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# Lattice Misfit and the Mechanism of High Temperature and Low Stress Creep of Ni-base Single Crystal Superalloys

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## ABSTRACT

The role of the misfit between the  $\gamma$ - and  $\gamma'$ -phase (lattice constants:  $\gamma' < \gamma$ ) during high temperature ( $>900^\circ\text{C}$ ) and low stress ( $<300\text{MPa}$ ) creep is re-considered. The presentation first provides an overview on how misfit was interpreted in the past. Then a new view is proposed, which is in line with mechanical and microstructural findings. The new results are based on a combination of creep testing of precisely oriented [001] miniature creep specimens and post mortem diffraction contrast scanning transmission electron microscopy (STEM) results for a single crystal superalloy (SX) of type ERBO-1 (CMSX4 type). It is proposed that the strong decrease of creep rate in the short early stages of creep is associated with the formation of regular dislocation networks. These shield the negative misfit between the  $\gamma$ - and  $\gamma'$ -phase. The networks form in the  $\gamma$ -phase, close to the  $\gamma/\gamma'$ -interfaces. It is shown that the spacing of dislocations in the networks remains constant during creep. Networks are however not static. They play an active role in forcing dislocations from both phases to undergo knitting reactions. Dynamic recovery is accomplished by pairs of  $\gamma$ -channel dislocations, which form superdislocations which cut the  $\gamma'$ -phase and annihilate by reacting with dislocations on the other side of the  $\gamma'$ -particle.  $\gamma'$ -cutting is initiated by dislocations which *knit out* from the networks. This process is coupled with the *knitting in* of new  $\gamma$ -channel dislocations to keep the misfit shielded. Knitting regular networks requires climb processes, which are suggested to be rate controlling. This new view of the high temperature and low stress creep mechanism of single crystal superalloys is discussed in the light of previous work published in the literature. Emphasis is also placed on the transition between rafting and topological inversion during tertiary high temperature and low stress creep.

**Keywords:** Single crystal superalloy, High-temperature deformation, Microstructure

## New Strengthening Mechanisms in Ni-Base Superalloys

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### ABSTRACT

Polycrystalline Ni-based superalloys are vital materials for disks in the hot section of aerospace and land-based turbine engines, and improving fuel efficiency and reducing greenhouse gas emissions compel increased performance at higher temperatures. In this presentation, two different approaches to improving high temperature strength and creep resistance are discussed. In the first approach, local ordered phases at stacking faults and microtwins can be accomplished by subtle changes to the alloy composition [1-2]. Multi-scale characterization has demonstrated that local phase transformation can lead to significant strengthening at elevated temperature, and a computational approach to aid in the design of alloys for promoting this new mechanism will be discussed. In the second approach, a novel additive-processing route for creating oxide dispersion strengthened (ODS) metallic alloys, recently developed by collaborators at NASA [3], has been utilized to design a an ODS solid solution alloy (GRX-810) which has exceptional high temperature properties [4]. This new additive ODS process enables the synthesis of ODS alloys in a single, additive processing step, thereby bypassing the conventional mechanical alloying process that is time-intensive and inconsistent with scale-up manufacturing. Detailed electron microscopy analysis has provided insights into the development of dislocation substructures during creep of GRX-810, and a model NiCoCr ODS alloy. The present status of this work and prospects for developing a wider range of ODS strengthened superalloys will be discussed.

### Acknowledgments

Support for this work at the Ohio State University provided by the National Science Foundation under Grant #2323717 GOALI: / DMREF: Multimodal design of revolutionary additive-enabled oxide dispersion strengthened superalloys.

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## Exploring Low-Temperature Creep in Titanium Alloys

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### ABSTRACT

Titanium alloys find application in the fan and compressor stages of aeroengines at temperatures to 500°C. We have explored creep in this temperature range in several alloys of titanium. A strong dynamic strain ageing effect originating from Si and C interactions with dislocations leads to a viscous drag-controlled regime around 450°C. Creep in this temperature domain is controlled by the glide of jogged screw dislocations and has been modelled through the interactions of solutes with the edge jogs. The temperature regime extending from ambient temperatures to about 200°C is particularly important in that dwell fatigue is known to exhibit significant life debits to about 120°C, and is influenced by static creep during hold at peak stresses approaching the yield stress in the fatigue cycle. We have therefore studied creep in this temperature domain in polycrystalline  $\alpha$  titanium, Ti-6wt%Al, and in bimodal Ti6242. Strong texture effects are present in the creep of the single-phase alloy, while the Ti6242 alloys shows a surprising exhaustion of creep as temperatures are increased to about 300°C.

### Acknowledgments

We are grateful to Pratt and Whitney, USA, in particular, Vasisht Venkatesh, Dheepa Srinivasan and Dave Furrer, for the support of this work. DB receives an INSA Senior Scientist fellowship. The presentation is based on the work of Priyanka Agrawal, Aparajita Pramanaiik, Atasi Ghosh, Shreya Mukherjee, Mainak Sen, and Girish Bojjawar and Prof. S. Karthikeyan.

## Creep Properties of Various Single Crystal Alloys and Conclusions for Beyond Nickel-Based Superalloys

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### ABSTRACT

Internal stress contributions in single and multi-phase alloys will be discussed and their influence on creep properties of single crystals quantified. Some stress contributions (solid solution, dislocation back stress, misfit, Orowan and cutting of strengthening phases) are well understood and can be theoretically estimated. Solid solution strengthening is way more critical to predict. This will be exemplified by creep observations of different alloys, mainly at 980°C, but also up to 1450°C in correlation with microstructures.

Conclusions of stress discussions and creep observations will be drawn for possible future materials beyond Ni-based superalloys. Further considerations like oxidation resistance and room temperature ductility are additionally of importance and considered.

**Keywords:** Single crystal superalloy, High-temperature deformation, Microstructure

### References:

[1] Uwe Glatzel, Felix Schleifer, Christian Gadelmeier, Fabian Krieg, Moritz Müller, Mike Mosbacher and Rainer Völkl

Quantification of Solid Solution Strengthening and Internal Stresses through Creep Testing of Ni-Containing Single Crystals at 980 °C

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## Precise computation of creep properties with basic models

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### ABSTRACT

Mechanical properties including creep have traditionally been described with empirical and semiempirical models involving a number of adjustable parameters. Such models are simple to use and a good fit to data is easily obtained but there are disadvantages. The models are not predictable and for identification of operating mechanisms, mistakes can easily be made. To avoid these problems, creep models derived from basic principles without involving adjustable parameters have been generated. In the presentation, it will be demonstrated that many properties can be computed with high precision. They include stress strain curves, creep rate at normal and high stresses covering power-law and power-law breakdown regimes, including primary, secondary and tertiary creep. Models for nucleation and growth of creep cavities have been formulated that allow for the prediction of creep rupture curves. Conditions at very low stresses normally have been considered where dislocation creep and diffusion creep are competing. Many of the creep models are valid over a very wide range of conditions verifying the validity of the basic models. Further details of the basic models can be found in a book that is freely downloadable at <https://link.springer.com/book/10.1007/978-3-031-49507-6>

**Keywords:** Creep, High-temperature deformation model

## Experimental Validation and Scientific Understanding of Ultrahigh Temperature Structural Materials

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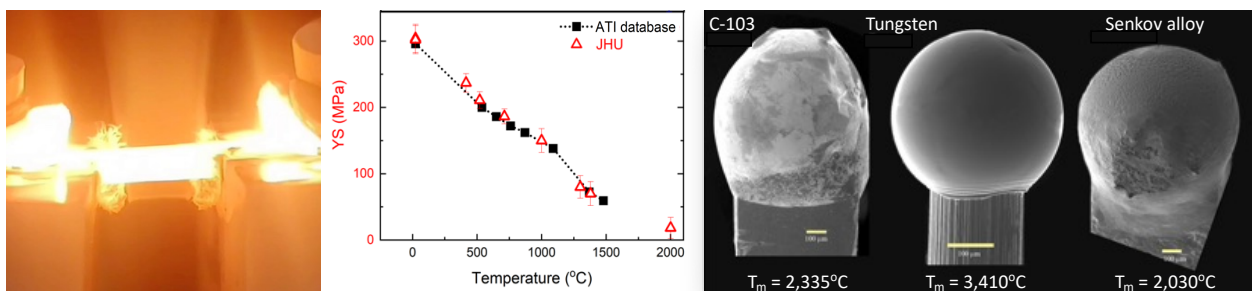
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### ABSTRACT

Modern transportation, power generation, space exploration, and high velocity flight all depend on the availability of materials that can be shaped and maintain their shape and strength at elevated temperatures. The pace of ultrahigh materials development and the need for mechanical property evaluation at ultrahigh temperatures (UHT), above 1500°C, are accelerating. However, current understanding of the high-temperature properties of prospective materials is inhibited by the limited availability of UHT facilities and the excessive cost of conducting standardized macro-scale experiments in extreme environments. The feasibility of performing a full battery of ultrahigh temperature macro-scale tests on emerging refractory or other exotic high temperature specimens remains a significant challenge, and alternative approaches for measuring and understanding mechanical behavior at these extremes are worth exploring. The challenges associated with ultrahigh temperature testing are multi-faceted and include the need to heat specimens to extreme temperatures without also heating the surrounding load frame and electronic instruments above their functional limits; the cost associated with obtaining sufficient quantities of developmental materials to produce macroscopic specimens; the need to avoid oxidation; precise temperature measurement and control; and implementing non-contact strain measurement at UHT.

With these challenges in mind, the current study was undertaken to develop a test methodology that could be used to accelerate materials discovery, data collection, and fundamental understanding of underlying deformation mechanisms. It is predicated on the use of Joule heating to provide rapid local heating of subscale specimens employing a grip-in-grip load frame that isolates the specimen, facilitates tensile loading, and fits within a vacuum chamber. Filtered ultraviolet illumination facilitates digital image correlation (DIC) and strain mapping. And multispectral pyrometry provides direct measurement of alloy emissivity and melting temperature and enables closed-loop temperature control. The tensile strengths of ATI C-103, WC3009, and various RMPEAs have been evaluated in high-vacuum at temperatures up to 2,000°C. The results from these experiments will be used to substantiate the new test methodology and to lay the foundation for a mechanistic interpretation of UHT plasticity and creep in RMPEAs. Single-crystalline UHT studies of ternary RMPEAs are also being used to determine whether dislocation glide is fundamentally different in these alloys than more traditional dilute refractory alloys.



UHT tensile specimen @ 1750°C | excellent agreement for ATI C-103 | T<sub>melt</sub> and emissivity measurements

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## Cantilever bending to study creep in inhomogeneous structures

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### ABSTRACT

It is known that cantilever bending, coupled to location-specific strain measurements using digital image correlation, provides a high throughput method of extracting creep laws by exploiting the variable stress state in a beam. The analyses rely on certain relationships that exist between stress, strain and position in homogeneous materials with tension-compression symmetry. Across the cross section, strain remains linear and the stress varies non-linearly depending on the creep stress exponent; along the length, stress remains proportional to the bending moment and there exist so-called invariant or skeletal points in both halves of the beam where the stress remains constant at a value predicted from linear elasticity. In the present work, we explore the extension of such analyses to more complex systems including composite beams, weldments and a material that is pronouncedly harder in compression than in tension. These examples include:

- (i) Creep rate estimation of miniature, roughly cuboid test pieces of steels extracted from used boiler tubes of thermal power plants, by additively extending them with a creep-resistant Inconel into cantilevers. It is shown that as long as the test coupon dimension along the beam length is  $\sim$  of the beam thickness, that useful data can be extracted.
- (ii) Extension of the analysis to the interfacial regions of dissimilar materials as found in weldments. The predominantly uniaxial stress state in a beam gives way to a multi-axial state at the interface owing to strain rate incompatibilities which enhance the hydrostatic component at the expense of the effective shear stress that drives creep. However, knowledge of the uniaxial power law coupled to finite element analysis allows the determination of the deviatoric stress from which the effective strain rate can be calculated and shown to be in agreement with the measured data.
- (iii) Room temperature, exponentially decaying creep in titanium alloys, which is responsible for dwell fatigue. Such creep takes place at high stresses that are comparable to or even larger than the yield stress. It is shown that despite the re-distribution of stress following yielding and the absence of steady state, that invariant points exist allowing creep laws to be extracted from a single test, provided tension-compression asymmetry is absent.
- (iv) Immediate direct evidence for the existence of tension-compression asymmetry. Highly textured Ti-6Al with the hard c-axis along the beam length show a shift in the neutral axis under creep that is consistent with the known asymmetry in the critical resolved shear stress for  $c+a$  slip.

### Acknowledgments

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# Localized phase transformation strengthening during creep of complex CoNiCr-based superalloys

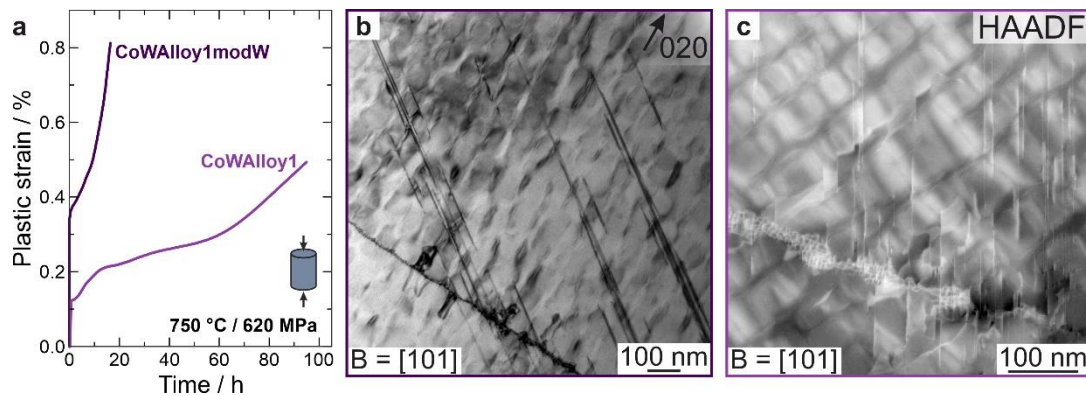
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## ABSTRACT

Segregation processes during high-temperature deformation can be either detrimental or beneficial in  $\gamma'$ -strengthened superalloys depending on which elements segregate. In intermediate CoNiCr-base superalloys only softening due to extensive microtwinning was observed so far. By analyzing the creep behavior of single- and polycrystalline CoNiCr-based superalloys at 750°C, we show that strengthening segregation behavior along various planar defects, including microtwinning, can be achieved simultaneously in this alloy class, which leads to an exceptionally high creep strength. While the alloys deformed under planar fault formation, a Ta-containing alloy outperformed the Ta-free alloy, where Ta was replaced by W. Atomic-scale energy dispersive spectroscopy investigations revealed strengthening local phase transformations along superlattice intrinsic and extrinsic stacking faults in both alloys. However, microtwinning is enhanced in the Ta-free alloy, while it is impeded in the Ta-containing alloy. Given the faster diffusivity of Ta compared to W, kinetic effects on localized phase transformation strengthening and their subsequent influence on creep behavior are discussed.



**Figure 1:** Creep behavior of CoNiCr-based superalloys CoWAlloy1 and CoWAlloy1modW. (a) Creep curves after compressive creep at 750 °C and 620 MPa. (b,c) Stacking fault-based defect structures after the interrupted creep tests close to dendrite boundaries in (b) CoWAlloy1modW, taken with  $g = 020$  in two-beam conditions close to the  $[101]$  zone axis, and (c) CoWAlloy1 using HAADF STEM in the  $[101]$  zone axis.

# High temperature compressive creep behavior of ZrB<sub>2</sub>-SiC-LaB<sub>6</sub> composites

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## ABSTRACT

A study was carried out to examine the effect of LaB<sub>6</sub> addition on compressive creep behavior of spark plasma sintered (SPSed) ZrB<sub>2</sub>-SiC composites at 1300-1400°C under 47 – 78 MPa stresses in laboratory air. The ZrB<sub>2</sub>-SiC composites with B<sub>4</sub>C and C as additives along with 10 or 14 vol% LaB<sub>6</sub>, referred to as ZSBCL-10 or ZSBCL-14, respectively was prepared by SPS at 1600°C and 1800°C. The steady-state creep rate at 1300°C has been found to be lower for 10 vol% LaB<sub>6</sub> composites sintered at 1800°C due to larger grain size, whereas the trend is reversed in the composites sintered at 1600°C due to cleaner interfaces as well as superior oxidation resistance of the ZSBCL-14. The stress exponents (n) obtained for creep of ZSBCL-10 (~1.3±0.1) and ZSBCL-14 (~2.6±0.2) sintered at 1800°C suggest respectively, diffusion and dislocation glide as the operating mechanisms. Grain boundary sliding promoted by finer grain-size and interfacial glassy phase, has been found to cause damage by intergranular cracking. The oxide scales formed during creep of composites sintered at 1800°C with higher crack density, have shown greater thickness than those by isothermal exposure at 1300°C for the similar duration. The value of n for 10 vol% LaB<sub>6</sub> composites sintered at 1600°C (~2±0.1), as well as the formation of intergranular microcracks at ZrB<sub>2</sub> matrix grain boundaries and ZrB<sub>2</sub>-SiC interfaces is suggestive of grain boundary sliding being the operative creep mechanism. On the other hand, the value of n ~ 1 obtained for the 14 vol% LaB<sub>6</sub> composites sintered at 1600°C is suggestive of diffusional creep being operative at 1300°C-1400°C. Due to cleaner interfaces and superior oxidation resistance, the ZSBCL-14 composite has exhibited lower steady-state creep rate at 1300°C than the ZSBCL-10. Further, the value of apparent activation energy as ~ 700 kJ/mol for 14 vol% LaB<sub>6</sub> composites in the temperature range of 1300°C-1400°C suggests that the ZrB<sub>2</sub> grain boundary diffusion is the rate-limiting mechanism of deformation. Thickness of the damaged outer layer containing cracks scales with temperature and applied stress, indicating their role in facilitating the ingress of oxygen causing oxide scale growth. However, a continuous reduction of the oxidation-induced defect density with depth to a limit of ~280 mm, indicates the predominance of creep-based deformation and damage at the inner core of samples.

# Creep behavior and deformation mechanisms of precipitation-strengthened refractory high entropy alloys

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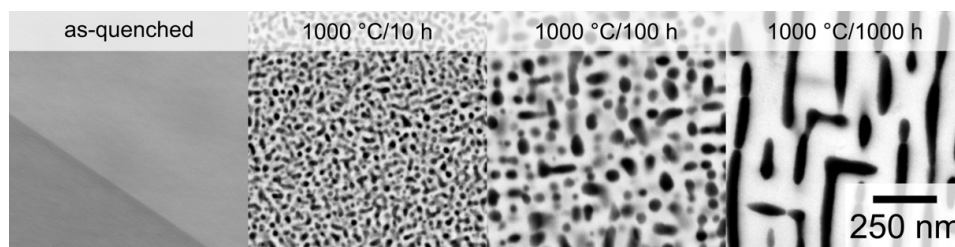
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## ABSTRACT

Refractory high entropy alloys (RHEA), composed of high melting, passivating, and lightweight elements, show great promise for high-temperature structural applications [1]. They have demonstrated exceptional properties surpassing those of Ni-based superalloys and other refractory alloys, such as remarkable high-temperature strength and superior corrosion resistance. However, the crucial aspect of creep resistance, essential for structural materials at elevated temperatures, has only been briefly addressed. A recent study by Gadelmeier et al. [2] investigated the tensile creep behavior of body-centered cubic (BCC) TiZrHfNbTa RHEA with a single-phase, polycrystalline microstructure at 980 °C and 1100 °C. The creep resistance at both temperatures was notably lower than that of a state-of-the-art, precipitation-strengthened, single-crystal Ni-based superalloy (CMSX-4). This discrepancy is attributed to phase decomposition during creep deformation and the BCC crystal structure of the alloy. Therefore, achieving a precipitation-strengthened condition is imperative for RHEA to exhibit competitive creep performance.



**Figure 1.** Scanning electron microscopy (SEM) micrographs (backscattered electron contrast) of 27.3Ta-27.3Mo-27.3Ti-8Cr-10Al (at.%) RHEA with primarily Z contrast taken within single grains after quenching and subsequent heat treatment at 1000°C for different durations [4].

Our alloy approach in the Ta-Mo-Ti-Cr-Al system has confirmed the existence of a two-phase microstructure with a disordered A2 matrix and ordered B2 precipitates in a certain compositional range, resembling that of Ni-based superalloys [3,4]. The formation of B2 in 82(Ta-Mo-Ti)-8Cr-10Al (in at.%) occurs through nucleation and growth-mediated precipitation, with a notably high solvus temperature of approximately 1050°C. Additionally, it maintains a stable ultrafine particle microstructure even after prolonged exposure to elevated temperatures, as illustrated in Figure 1 [4]. These findings suggest promising prospects for achieving high-temperature creep resistance in this alloy. Consequently, we present the current status of investigations into the creep behavior of this B2 precipitation-strengthened A2 alloy. Compression creep tests were conducted at elevated temperatures close to the solvus temperature and above, with varying constant true stresses to unveil the creep deformation behavior and underlying mechanisms. Subsequently, SEM/TEM were employed to examine the deformed microstructures at different creep strains. The discussion will encompass the impact of the coherent interface between the matrix and precipitates.

## References

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## **Some issues in creep of engineering materials and their resolution**

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### **ABSTRACT**

Some aspects of creep in superalloys and ferritic steels have not been fully explained hitherto. In the case of superalloys, for instance, the long-term creep behavior of superalloys shows almost no dependence on initial microstructure while there is significant dependence in the short-term. The role of gamma prime particles in defining this behavior has not been fully clarified. Similarly, in the case of ferritic steels, there is significant variance in the understanding of the nano/micro scale features controlling creep. The difficulty in experimentally observing nano-scale particles that pin dislocations has led to speculation that other observable features might be the controlling features. This talk will outline an approach which seeks to reconcile various observations in these materials into a coherent framework. The ability of this approach to predict long term creep behavior of these materials from initial microstructural observations will be demonstrated.

# Creep-fatigue assessment of EUROFER taking into account the impact of cyclic softening on its creep behavior

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## ABSTRACT

Recently, creep-fatigue (CF) rules in existing design criteria were modified for cyclic softening steels, e.g. EUROFER, taking into account the impact of cyclic softening on the evolution of creep damage and, hence, creep lifetime. Therefore, a cyclic softening stress factor was introduced whose dependence on cyclic softening is described by a power function and an additional material and temperature dependent parameter. For determining this parameter creep tests on material specimens pre-cycled/pre-softened to different levels need to be conducted which was successfully done for EUROFER at 550°C. In this work the creep tests on pre-softened EUROFER are extended to 450°C demonstrating similar remarkable effect of cyclic softening on creep properties as at 550°C. Moreover, the Monkman-Grant relationship between creep lifetime and minimum creep rate was also found being independent on cyclic softening and even independent on temperature indicating no change in creep damage mechanism within the temperature range investigated so far.

To verify the modified CF rules low cycle fatigue (LCF) tests with long relaxation as well as long creep holdtimes are conducted at 450°C and 550°C, respectively. Evaluating the results in comparison to the existing CF rules in the ASME-BPV and RCC-MRx design codes demonstrates once more the applicability of the modified CF rules to cyclic softening steels without being overly conservative.

## Design of Creep Resistant Aluminum Alloys

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### ABSTRACT

A variety of applications demand lightweight aluminum alloys that are creep resistant in the 250 – 450°C temperature range. Two recent developments in the design of creep resistant aluminum alloys will be described in this presentation. The first development is associated with design of creep resistant cast aluminum alloys. Cast AlCuMnZr (ACMZ) alloys with stabilized  $\theta'$ -Al<sub>2</sub>Cu phase microstructures have been gaining interest in recent years. ACMZ alloys have improved tensile response due to strengthening of grain bulks to 350°C by  $\theta'$  enriched microstructures but their creep response at low to intermediate stresses is demonstrated to be controlled by grain boundary microstructures. We will present an alloy design strategy wherein coarsening resistant Co, Ni and Fe rich phases in the grain boundary region impede grain boundary sliding at 300°C. The resulting Co-, Ni- and Fe- containing ACMZ alloys are among the most creep resistant cast aluminum alloys reported. The second development is associated with design of creep resistant additively manufactured aluminum alloys. A specific new composition in AlCeNiMnZr alloy with thermally stable microstructure and high-volume fraction of dispersoids with exceptional creep resistance to 400°C will be presented. In situ neutron diffraction experiments demonstrate that Orowan strengthening from dispersoids determines the creep resistance of the alloy. The two recent developments underline opportunities for modifying bulk and interfacial microstructures of aluminum alloys in order to enhance their creep resistance.

The total content should not exceed one page, including supporting figures.

### Acknowledgments

The research was co-sponsored by the Powertrain Materials Core Program (PMCP) under the Vehicle Technologies Office (VTO) and Advanced Materials & Manufacturing Technologies Office (AMMTO) under Office of Energy Efficiency and Renewable Energy (EERE), U.S. Department of Energy (DOE).

# Grain boundaries in the equiatomic CoCrFeMnNi high-entropy alloy and their impact on creep strength

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## ABSTRACT

Compression and tensile creep of the equiatomic CoCrFeMnNi high-entropy alloy was investigated at 1073 K and 1253 K. The high-entropy alloy was either in the polycrystalline (PX) or monocrystalline (SX) solid solution state. We find that the polycrystalline variant creeps significantly faster than the monocrystalline variant at low applied stresses. Additionally, the stress exponent is lower for the polycrystalline samples ( $n \sim 3$ ) than the monocrystalline samples ( $n \sim 5$ ), which, according to a standard interpretation of creep data, implies a curious transition from viscous glide to climb-controlled creep. Such a transition is difficult to rationalize given that our alloy composition and crystal structure remain the same and only the microstructure changes from polycrystalline to monocrystalline. We offer an alternative to the standard view where grain boundaries, which are only present in the polycrystalline material, serve as efficient sinks for dislocations and thus contribute to faster creep.

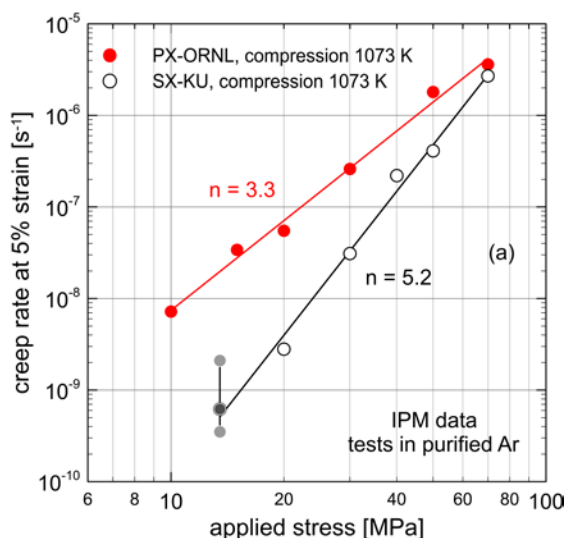


Figure 1: Double logarithmic plots compare secondary creep rates of the PX and SX variants of the CoCrFeMnNi solid solution and their dependences on the applied stress.

## Acknowledgments

Financial support was provided by Czech Science Foundation under the contract no. 14-22834S. CG and UG gratefully acknowledge financial support through DFG priority program SPP 2006 “Compositionally Complex Alloys - High Entropy Alloys”. Work at ORNL was sponsored by the U.S. Department of Energy, Office of Science, Basic Energy Sciences, Materials Sciences and Engineering Division.

# An Investigation of Low Temperature Creep Controlling Mechanisms in a Martensitic Spring Steel

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## ABSTRACT

The rising demand for electric vehicles urges automotive suppliers to push their limits on the sag resistance of suspension coil springs. Low temperature creep (LTC) investigations on high strength martensitic steels unveiled that the LTC strain follows a logarithmic creep law. However, the mechanism contributing to LTC as well as the LTC rate controlling mechanism in martensitic steels at ambient temperatures up to 423 K are not evident. The authors reviewed the existing LTC mechanism-based models, i.e. the strain hardening theory (SHT) and the exhaustion creep model (ECM), respectively, and benchmarked them against the experimental knowledge gained at the example of martensitic spring steels. None of these theories seamlessly reveal the nature of the LTC controlling mechanism.

Hence, in a first step, the authors provide revised versions of SHT and ECM, respectively. Furthermore, in a second step, the mechanical properties of inductive quenched and tempered (IQT) SAE 9254, and martempered SAE 9254 are determined under uni-axial tensile loading in the temperature  $T$  range of  $298 \text{ K} \leq T \leq 353 \text{ K}$  at a strain rate of  $2.5 \cdot 10^{-4} \text{ s}^{-1}$ . Subsequently, LTC deformation behavior of both IQT SAE 9254 and martempered SAE 9254 are studied in the above-mentioned temperature  $T$  range for a duration of 1 hr at each condition. The stress  $\sigma$  dependent LTC behavior of IQT SAE 9254 is studied in the stress  $\sigma$  range of  $1071 \text{ MPa} \leq \sigma \leq 1634 \text{ MPa}$ , and the stress  $\sigma$  range for martempered SAE 9254 is maintained as  $421 \text{ MPa} \leq \sigma \leq 632 \text{ MPa}$ .

A combined analysis by means of (i) a mechanism-based exhaustion creep model (ECM), and (ii) advanced microstructural characterization prior to and post LTC suggests that dislocation glide, mainly localized in the metastable retained austenite phase is one of the basic LTC contributing mechanism in SAE 9254. Furthermore, stress assisted martensitic transformation (SAMT), and, at elevated temperature  $T$ , strain induced martensitic transformation (SIMT) are considered as additional LTC contributing mechanisms. Furthermore, the considered approach suggests that the LTC rate controlling mechanisms are stress assisted recovery (SAR), SAMT and, at elevated temperatures  $T$ , SIMT.

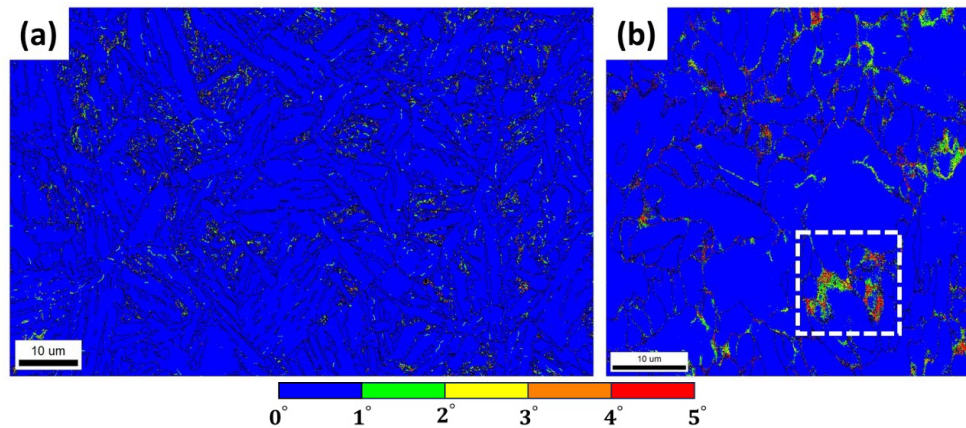


Figure 1: KAM mapping of martempered SAE 9254 (a) Prior to LTC, and (b) Post LTC

## Acknowledgments

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# Microstructure-sensitive modelling of intermediate temperature creep in polycrystalline Ni-based superalloys

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## ABSTRACT

Nickel-base superalloys are widely employed in hot parts of gas turbine engines due to their good mechanical properties at high temperatures. Specifically, improving the in-service performance of polycrystalline superalloys used in gas turbine discs to improve engine's efficiency requires increasing their creep resistance at intermediate temperatures between 650 °C and 800 °C. Although there is empirical understanding of creep at this temperature range, with stacking fault shear of the ( $\gamma$ ) matrix and ( $\gamma'$ )  $L1_2$  precipitates followed by microtwinning being the main deformation processes, there is virtually no predictive physics-based model able to capture the underlying mechanisms and their associated effects in creep strain. This work presents an integrated model for creep at intermediate temperatures in polycrystalline Ni-based superalloys [1]. The model is based on incorporating main rate-controlling mechanisms for dislocation partial and stacking fault propagation within the  $\gamma$  matrix and  $\gamma'$ ; these include solid solution strengthening, grain size and dislocation- $\gamma'$  shear effects [2]. The critical energy for stacking fault nucleation is obtained by minimising the energy to form a stacking fault from dislocation partials, which is promoted by local stress concentrations and the  $\gamma$ -stacking fault energy [3]. A force balance at the  $\gamma/\gamma'$  interface is performed -similar to the weak-pair coupling model- to estimate the ability of a newly formed stacking fault to cut a secondary  $\gamma'$ , defining the creep strain and defect density rates. It is shown that one of the main factors controlling the creep strain is the stacking fault energy in the  $\gamma$  as it dictates the stacking fault nucleation and shear rates. Similarly, it is demonstrated that the difference in the creep response between alloys having monomodal and multi-modal  $\gamma'$  particle size distributions is that the tertiary  $\gamma'$  athermally lowers the frequency of secondary  $\gamma'$  particle shear events. The model results are used to understand and predict creep deformation in experimental data of several polycrystalline superalloys, also highlighting the role of chemical composition in alloy's creep performance. Individual contributions to creep are studied from key microstructural features, i.e. grain size, multimodal  $\gamma'$  size distribution and  $\gamma'$  volume fraction, to showcase how the model can be used as tool for alloy and microstructure optimisation against creep. Lastly, it is discussed how the present work could be used as mathematical framework to incorporate other creep mechanisms defining a unified model for creep in Ni-based superalloys at wide different temperature and stress conditions.

## Acknowledgments

The author would like to acknowledge the Royal Academy of Engineering for his research fellowship funding.

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## **Creep of Dispersion Strengthened Materials – Emergence of Paradigms Challenging the Old Theories**

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### **ABSTRACT**

Creep theories have evolved from 1950s to accommodate for microstructural complexities in advanced materials. Nabarro-Herring diffusional creep model is a classic example of an elegant theory set in a simple microstructural discussion. Engineering materials are often microstructurally complex and derive their strength from a number of mechanisms. The creep deformation is analyzed in terms of parallel and sequential processes. In this overview, the emergence of mechanistic understanding will be presented by considering recent examples that challenge the conventional thinking. The approach of using single crystal blades in engines is built on eliminating grain boundaries to suppress creep deformation and failure mechanisms. Can nanocrystalline materials be creep resistant? Recent results show this possibility. The role of dispersion strengthening can be direct or indirect for diffusional and dislocation creep mechanisms. These examples will be highlighted to emphasize a few fundamental creep-resistant microstructural paradigms.

**Keywords:** Dispersion strengthening, High-temperature deformation

# The effects of secondary elements on the creep behavior of additively manufactured Ni-based superalloys

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## ABSTRACT

Metal 3D printing processes provide the potential for economic advantages and expanded manufacturing capabilities. However, the processing conditions of additive manufacturing (AM) result in unique material properties compared to similar materials produced by conventional synthesis methods. In particular, the mechanical behavior of the AM material can be affected by process related variations in material composition. To compare the distinctions between alloys produced by differing methods, high temperature creep experiments were performed on wrought and AM Inconel 718 (IN718). The elevated temperature creep ductility of the AM alloy was reduced compared to that of the wrought alloy. NanoSIMS and APT analyses revealed sulfur to be the possible cause of embrittlement. A compositionally modified AM IN718 alloy was synthesized with the goal of increasing the high temperature creep ductility by inhibiting the deleterious effects of sulfur in the material. The high temperature creep and fracture behavior for the modified alloy will be discussed.

## Acknowledgments

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## Multiscale modeling of high temperature deformation of Co-base superalloys

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### ABSTRACT

There has been a renewed interest in cobalt base superalloys for gas turbine applications for land-based electricity generation power plants owing to their excellent resistance to oxidation and sulfidization at elevated temperature (~800 °C), while retaining good mechanical properties such as strength and wear resistance. A new family of Co-base superalloys possessing ( $\gamma+\gamma'$ ) two-phase microstructure reminiscent of Ni-base superalloys have attracted significant attention owing to exceptional high mechanical properties. In this work, we focus on three Co-Ti-V based alloys developed in our lab with different Ti/V ratio. These alloys after appropriate heat treatments have a  $\gamma'$  (with cubic L1<sub>2</sub> structure) phase fraction in excess of 50%. High temperature tests revealed that the proof stress (at 1% plastic strain) and creep strength (based on Larson-Miller parameter for 1% creep strain) of the present class of alloys was superior to conventional solid solution strengthened Co-base superalloys, and many Ni-base superalloys. Proof stress variation with temperature revealed yield strength anomaly (YSA), i.e., the strength initially decreases with an increase in temperature, and then increases from 650 °C to 750 °C (peak temperature, T<sub>p</sub>), and at even higher temperatures, it decreases again, and this behavior is akin to many Ni-base superalloys. The proof stress at 750 °C decreased with an increase in vanadium concentration.

To explain the variation of mechanical properties with respect to temperature and composition, a multiscale modeling approach was used. At the lowest length scale, the effect of composition on elastic constants and lattice parameters was estimated in the two phases via *ab-initio* electronic structure calculations. These calculations were also used to determine the effect of composition on planar fault energies in the two phases—intrinsic stacking fault energy in  $\gamma$ , and APB(111), APB (001), CSF(111) and SISF(111) in  $\gamma'$ . Based on these calculations, it was concluded that at temperatures below T<sub>p</sub>, the  $\gamma'$  precipitates would be sheared (not bypassed) and that during ageing with growing precipitate size there is a transition from weak APB coupling of  $\frac{1}{2}\langle 110 \rangle$  dislocations to strong coupling of the same dislocations. Moreover, the YSA phenomenon could be explained in terms of anisotropy between APB energies on the {111} and {100} planes, and due to elastic anisotropy. Using these parameters, the thermodynamic and kinetic parameters relevant to YSA were also estimated to predict the peak temperature T<sub>p</sub> as a function of composition.

Since the ( $\gamma+\gamma'$ ) system is elastically heterogeneous with both phases being elastically anisotropic, and with a high phase fraction, traditional precipitate strengthening models from modulus and misfit strengthening cannot be used. To address this, lattice parameters and anisotropic elastic constants for both phases derived from *ab-initio* electronic structure calculations were used in the framework of a finite element model (FEM) to predict residual stresses in the  $\gamma$  channels due to lattice misfit. Experimental data on precipitate volume fraction, size and morphology were used to generate the appropriate pseudomicrostructure. Additionally, parameters relevant to solute strengthening of  $\gamma$  matrix, and order strengthening due to  $\gamma'$  also derived from *ab-initio* calculations, and residual stresses from FEM calculations were used in conjunction with discrete dislocation dynamics (DDD) to predict the effect composition on strength. In summary, this multiscale modeling effort has enabled a better understanding of the role of temperature and composition on high temperature mechanical properties in these alloys. Furthermore, this also underscores how such a modeling approach can enable rapid development of new alloys with the desired mechanical response.

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## Investigating the role of microsegregation on phase evolution, recrystallization and creep behaviour of LPBF IN939

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### ABSTRACT

Ni-base superalloys have been extensively used as the hot section components of gas turbine engines for several decades. To meet the ongoing advancements in design and a growing need for materials capable of withstanding elevated temperatures and loads, the metallurgical challenges have intensified, necessitating continuous improvements in processing routes to unlock the design challenges. Additive manufacturing (AM) plays a pivotal role by overcoming manufacturing constraints and producing near-net components efficiently in confined spaces. However, the major drawback of the AM process is its inherent microstructural features, such as columnar grain morphology, chemical and crystal heterogeneities, resulting in inferior mechanical properties, especially at elevated temperatures. Therefore, this study aims to mitigate these issues through optimized heat treatment to eliminate the heterogeneities through varied heat treatment, thereby achieving optimum creep properties.

For this study, IN939, a moderately weldable superalloy strengthened by  $\gamma'$  ( $\text{Ni}_3(\text{Al,Ti})$ ) precipitates, was selected. Specimens fabricated using laser powder bed fusion additive manufacturing were subjected to solution treatment in the temperature range of 1150–1250 °C, followed by a two-step aging treatment. Subsequently, a comprehensive understanding of the microstructural evolution was developed through microstructural characterization in the as-built and heat-treated conditions using SEM, TEM and STEM-EDS analysis. Further, high-temperature tensile and creep experiments were performed on the heat-treated specimens in the temperature range of 0.4 – 0.7  $T_m$  to elucidate the deformation behavior at elevated temperatures. Subsequently, the post-deformation microstructural characterization was carried out on deformed specimens to correlate the microstructure with the mechanical data which enabled a better understanding of the high temperature deformation mechanisms in this alloy.

Keywords: Additive manufacturing, Laser powder bed fusion, IN939, Creep, Recrystallization, Microsegregation

## Effect of Temperature and Stress on The Creep Behavior of 718 Plus Alloy Having a Uniform Bimodal Distribution of $\gamma'$ Precipitates

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### ABSTRACT

Allvac 718Plus alloy is a new generation  $\gamma'$ -strengthened nickel-based superalloy with excellent strength and corrosion resistance at high temperatures [1]. The alloy exhibits improved thermal properties up to 704°C. Though the creep properties of 718 plus with unimodal distribution of  $\gamma'$  precipitates have been thoroughly investigated [2], the effect of a uniform bimodal distribution of  $\gamma'$  on the creep properties of the alloy is lacking in the literature.

In the current study, 718 plus alloy was subjected to interrupted aging treatment to generate a bimodal distribution of  $\gamma'$  precipitates. Various bimodal  $\gamma'$  distributions were generated as a function of ageing duration, and the peak-aged distribution was identified through hardness tests. The volume fractions of the large and fine  $\gamma'$  precipitates evolved into a near 50:50% uniform distribution with average particle sizes of 72.3 nm and 12.5nm, respectively [3]. The creep tests at constant load (tensile loading; in elastic regime) were carried out at different temperatures (650 – 850°C) and stress values (0.6 – 0.8 times yield stress). The crept samples were characterized using SEM and TEM to investigate the deformation mechanism of the alloy under tensile load. The excellent stability of the microstructure in the lower temperature regime led to an extended steady-state creep. However, the higher deformation temperature led to a predominant accelerated creep due to the dissolution of fine  $\gamma'$  precipitates (decreasing volume fraction) and the growth of large  $\gamma'$  precipitates, thus effectively increasing the interparticle distance. Lower temperature and stress values show substantial evidence of grain boundary sliding. With increasing temperature and stress, the fracture mode in the crack growth region transitions from intergranular fracture to void formation and coalesces due to the dissolution of precipitates. Additionally, the effect of change in size and distribution of  $\gamma'$  precipitates on the deformation mechanism is thoroughly investigated. Furthermore, the growth of large  $\gamma'$  precipitates w.r.t the loading direction as a function of temperature and stress is also investigated.

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## Hot deformation of Zr alloys: microstructure and texture development

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### ABSTRACT

The high temperature compression behaviour of Zr, Zircaloy and Zr-2.5wt.%Nb is presented and compared. These were compressed at temperatures from 700 to 800 °C to intermediate strains and their microstructure and texture was determined using scanning electron microscopy and electron backscatter diffraction. At these temperatures Zr and Zircaloy remain in the  $\alpha$  phase whereas Zr-2.5Nb goes in to the  $\alpha+\beta$  phase field. The flow curves of Zr and Zircaloy showed a gradual work-hardening followed by gradual softening typical of dynamic recrystallization. However the flow curves of Zr-2.5Nb showed a sharp drop at  $\epsilon \approx 0.01$  that was related to plastic deformation induced  $\alpha$  to  $\beta$  phase transformation. Zr and Zircaloy developed a  $\langle 2\bar{1}\bar{1}3 \rangle$  component parallel to compression axis ( $\parallel$ CA) typical of  $\alpha$ -Zr alloys, whereas Zr-2.5Nb, that contained  $\alpha+\beta$  phase at 750 and 800 °C, developed a  $\langle 2\bar{1}\bar{1}0 \rangle$  component  $\parallel$ CA. It was shown that the formation of  $\langle 2\bar{1}\bar{1}0 \rangle_{\alpha-Zr} \parallel$ CA occurred by migration of  $\beta$ -Zr into  $\langle 2\bar{1}\bar{1}3 \rangle_{\alpha-Zr}$  followed by the reverse  $\beta \rightarrow \alpha$  transformation (through Burgers orientation relationship) driven by low flow stress in  $\langle 2\bar{1}\bar{1}0 \rangle_{\alpha-Zr}$  and low Nb content in the transforming  $\beta$ -Zr. It was concluded that the presence of  $\beta$ -Zr with  $\langle 001 \rangle_{\beta-Zr} \parallel$ CA and  $\langle 111 \rangle_{\beta-Zr} \parallel$ CA is a necessary condition for the formation of  $\langle 2\bar{1}\bar{1}0 \rangle \parallel$ CA in  $\alpha$ -Zr.

# Significance of nanostructuring on mechanical properties and structural relaxation of FeCoCrNi alloys examined by in-situ heating neutron diffraction

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## ABSTRACT

Structural evolution through nanostructuring and relaxation upon heating are examined by X-ray diffraction and in-situ heating neutron diffraction analyses, respectively, on additive-manufactured FeCoCrNi alloys, specifically 316L stainless steel [1] and equiatomic high-entropy alloys [2]. Significant structural changes occur in a very early stage of nanostructuring by high-pressure torsion, leading to severe lattice distortion by excess dislocations and defects. The sequential information on the structure relaxation during in-situ heating neutron diffraction analysis provides the texture development, linear thermal lattice expansion, and stress relaxation behaviors of the nanocrystalline steel with increasing temperature up to 1300K, as shown in Fig. 1 [1]. Together with the hardness measurements after heating, the results of structural evolution are interpreted to describe microstructural recovery, recrystallization and grain growth behaviors, and the thermal stability of the AM-treated alloys. There is considerable potential for utilizing nanostructuring for exploring post-manufacturing treatments to enhance physical and mechanical properties in the AM alloys.

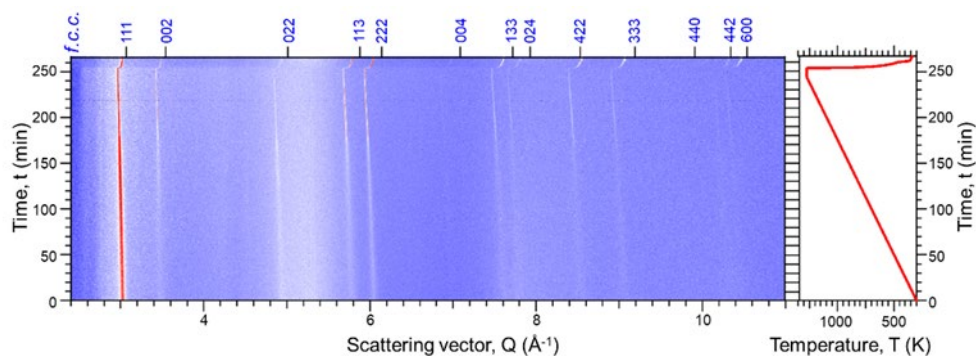


Fig.1 Contour plots showing variation in neutron diffraction patterns with time and temperature for the AM 316L SS after HPT for 15 turns [1].

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## **Elucidating the crack nucleation and propagation behaviour of a near alpha titanium alloy under thermomechanical fatigue**

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Rotating components, operating near the combustion chamber of gas turbine aeroengine, are exposed to thermal transients during their service life. The research of improved performances has increased the severity of loading conditions for these components such as high-pressure compressor discs and blades, where development of high temperature titanium alloys (IMI 685→IMI 829→IMI 834) and component design (compressor disk→blisk→bling) were improved together. While weight saving and high aerodynamic efficiency was the primary objective in the evolution of these designs, it has vectored towards the severe risk of crack nucleation and propagation due to thermomechanical fatigue. In the first part of the presentation, resistance of a high temperature titanium alloy, IMI 834, towards crack nucleation under thermomechanical fatigue will be presented. The influence of phase angle on the fatigue lives will be explored. In the second part of the presentation, crack propagation behaviour under thermomechanical fatigue of the same alloy, including the possible effects of crack closure, will be put forward. The experimental challenges in studying the thermomechanical fatigue behaviour, including both the crack nucleation as well as propagation, will be presented in detail.

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## Creep Rupture Behavior of Ferritic-Austenitic Dissimilar Joints of Grade 91 Steel Welded by Electron Beam and SMAW Processes

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### ABSTRACT

Austenitic alloys and Fe–Cr–Mo/W based Ferritic-Martensitic (FM) steels are being used in different sections of power generation systems and hence dissimilar weld joints (DWJs) between them are inevitable. In DWJs, creep failures at FM steel weld interface and at fine-grain/inter-critical heat affected zone (HAZ) of FM steels tend to cause premature failure of DWJs. Interface failure is a life limiting factor in DWJs and the research endeavors are underway worldwide to address interface failures in DWJs between above alloy systems, including modifications in filler wire composition and the development of novel fabrication techniques. In this context, the current study evaluates creep performance of DWJs, a) between Austenitic Alloy 800 and buttered Grade 91 (Gr.91) FM steel fabricated by Shielded Metal Arc Welding (SMAW) and b) between 316LN SS and Gr.91 made by Electron Beam (EB) welding without using any filler wire. The Alloy-800\_Gr.91 DWJ is a part of the trimetallic DWJ (316LN SS - Alloy 800 - Gr.91) being used in secondary sodium circuit of sodium cooled fast reactors. DWJ between Gr.91 (buttered with IN82) and Alloy-800, fabricated by conventional SMAW using IN182 electrode, revealed a stable interface (between buttered layer and Gr.91) free from deleterious coarse carbides and insignificant cavitation even after creep exposure of  $\approx 5800$  h at 873 K. The creep failures thus predominantly occurred at over-tempered region and at HAZ with only a 5–6% drop in % Reduction in area in reference to Gr.91 steel. In the case of 316LN SS\_Gr.91 EB-welded DWJ, compositional and microstructural gradients have been observed within the fusion zone. In spite of this, the narrow fusion zone and HAZ revealed remarkable creep rupture strength of the EB welded joints, surpassing 10,000 hours of exposure without significant interface degradation at 873 K. The above results indicated distinctive creep rupture behaviour of DWJs with no interface failures and with improvement in creep rupture strength compared to joints fabricated without buttering Gr.91 steel as shown in Fig.1 in the form of weld strength reduction factor. The study demonstrates that with adaption of advanced welding technique or careful selection of buttering process parameters, the reliability and performance of DWJs can be enhanced under creep loading conditions.

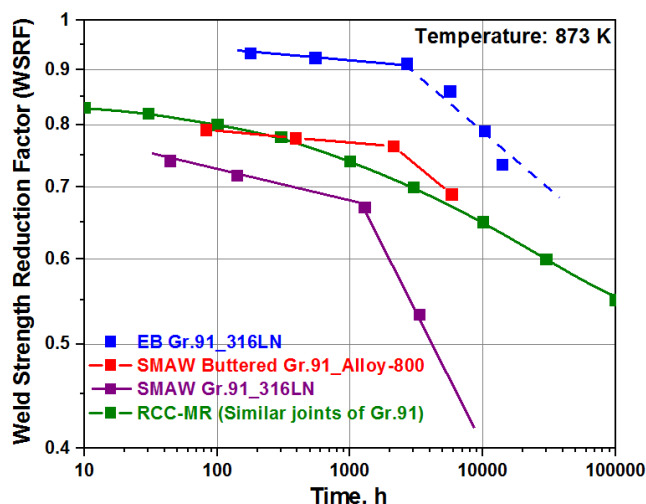


Fig.1: Creep weld strength reduction factor of DWJs fabricated by EB and SMAW welding techniques in comparison to that of Gr.91\_316LN DWJ (made without buttering Gr.91) by SMAW and to that specified in RCC-MR code; *Electrode material for SMAW weld joints is IN182. Reduction factor is the ratio of 'creep strength of weld joint' to 'creep strength of Gr.91 steel'.*

**Keywords:** Grade 91 steel, Alloy 800, 316LN SS, Dissimilar weld joint, Interface failure and Creep

## Understanding the High Temperature Creep Rupture Notch Sensitivity of Laser Powder Bed Fusion IN718 via Microstructural Characterization for Different Heat Treatments

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### Abstract

A characterization of Inconel 718 (IN718) microstructure manufactured by the laser powder bed fusion (LPBF) technique has been carried out as a function of three different heat treatments. Detailed analysis of the precipitation characteristics for each heat treatment was mapped using scanning electron microscope (SEM), transmission electron microscope (TEM)-high angle annular dark field (HAADF) imaging, and atom probe tomography (APT), revealing the occurrence of varying distributions and morphology of the strengthening  $\delta$ -Ni<sub>3</sub>Nb as well as the  $\gamma'$  and  $\gamma''$  phases. At stabilization temperatures below the  $\delta$  solvus, there was an excess of  $\gamma'$  and  $\delta$  precipitates and very little of the strengthening  $\gamma''$  phase. Adding a second homogenization cycle to enhance the  $\delta$  phase precipitation tended to further decrease the  $\gamma''$  precipitation. This occurrence of excessive  $\delta$ -phase (intra and inter-granular) and depletion of the strengthening  $\gamma''$  precipitates may have resulted in lower strength and ductility at elevated temperature (650°C). Comparisons made with the sample subjected to the standard AMS5663 (with a higher stabilization heat treatment) were able to reveal the differences in microstructure to correlate with improved creep behaviour.

**Keywords:** IN718, Additive manufacturing, powder bed fusion-laser beam,  $\gamma''$ -gamma double prime,  $\gamma'$ -gamma prime,  $\delta$ -delta, heat treatment, creep rupture, notch sensitivity

## **Grain boundary segregation behavior in Ni and Fe based alloys during diffusional creep**

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### **ABSTRACT**

Stress-driven diffusion atoms and point defects are considered basic driving mechanisms in various materials phenomena such as creep deformation, elemental segregation, self-assembly, and electromigration. In the Newtonian diffusional creep process, the Nabarro-Herring and Coble creep theories postulated that preferential flow of atoms toward the stress direction in exchange of vacancies governs the deformation rate in materials. These diffusional creep theories provide a compelling picture for materials scientists to design microstructures that improve the creep resistance. However, these theories remain vigorously debated due to a dearth of experimental evidence and poor prediction of creep rate. To help address these issues, the overarching goal of this study is to conduct hypothesis-driven research to understand the Newtonian diffusional creep mechanisms in Ni and Fe structural alloys such as Ni-Cr, Ni-Re, Fe-Cr-Ni and high entropy alloys. Specifically, this study tests the hypothesis of varied solute segregation to the grain boundaries depending on their orientation with respect to tensile loading axis. The grain boundaries in the undeformed or grip section of the specimen were compared to grain boundaries in the gauge section that are parallel or perpendicular to loading direction. The results reveal interesting insights into the competition between thermal equilibrium and diffusional creep induced segregation processes in the alloys. The experiments were further supported by calculations of vacancy energetics using self-consistent mean field theory combined with a novel chemo-mechanics coupled diffusion model for multicomponent alloys that predicts elemental segregation at grain boundaries. The mechanistic understanding of the diffusional creep mechanisms will enable new pathways for designing advanced creep-resistant materials for extreme environments.

### **Acknowledgments**

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# **CREEP-FATIGUE-ENVIRONMENT INTERACTIONS AND OTHER TIME AND TEMPERATURE DEPENDENT EFFECTS INFLUENCING PERFORMANCE OF SUPERALLOY 617**

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## **Abstract**

Alloy 617 is used extensively for heat exchanger components of very high temperature nuclear reactor (VHTR) and high temperature gas cooled reactors (HTGRs) and is a candidate material for superheater and turbines in advanced ultra supercritical (AUSC) thermal power plants due to its excellent high temperature mechanical properties. The design of heat exchanger components is primarily based upon the creep occurring at the operating temperatures. Many of these components are subjected to repeated thermal stresses during start-up and shut down or due to thermal transients occurring during service. Therefore, resistance to low cycle fatigue becomes a very important consideration in the design of the heat exchanging components. Further on-load periods at elevated temperatures introduce creep making creep-fatigue interaction (CFI) as an important aspect of the material's behaviour that limits the useful life of components. The occurrence of simultaneous Creep-fatigue interaction is often considered as the combination of fatigue and creep damage where fatigue damage is associated with the initiation and propagation of transgranular surface cracks and creep damage is associated with the intergranular creep cavitation. Creep-fatigue interaction testing at laboratory scale is carried out by imposing strain hold in continuous cycle fatigue either in tension or compression or both.

The Nickel-base Superalloy 617 and its variants are the major candidate materials for the heat exchanging components. Alloy 617 derives its high temperature solid solution strength by the addition of cobalt, molybdenum, and chromium. In addition, the intra and intergranular MC and  $M_{23}C_6$  provide the basic creep resistance. Authors have explored the creep-fatigue interaction behaviour of Alloy 617 for HTGR applications in simulated helium coolant environment at 950<sup>o</sup> C. The creep-fatigue interaction behaviour of a modified version of Alloy 617 having tighter chemical composition (Alloy 617M) has been studied at 700 °C for applications pertaining to AUSC plants. The deformation, damage and fracture behaviour of these alloys has been examined in detail by employing various dwell times in tension, compression and tension-compression at peak strains. In these studies, it has been noticed that creep damage gets enhanced due to synergistic interactions between fatigue, creep deformation, oxidation, deformation ratcheting, decarburisation, internal oxidation, phase changes etc. In this presentation, the complex damage behaviour of these alloys under hold time conditions will be dealt in detail. The formulation of CFI diagrams has been attempted based on the creep, LCF and CFI data.

# Role of molybdenum on high temperature deformation of Fe<sub>30</sub>Mn<sub>5</sub>Al<sub>1</sub>C(0-3) wt. % Mo Light Weight Austenitic Steels : Monotonic Tensile and Creep

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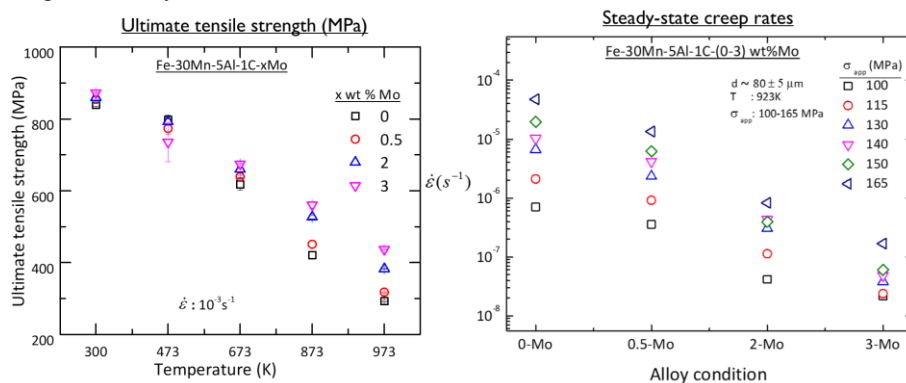
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## ABSTRACT

Lightweight austenitic steels based on the Fe-Mn-Al-C system can be candidate materials for high-temperature applications. The reports on understanding the behavior of these classes of alloys at high temperatures are limited. In the present work, four lightweight austenitic steels Fe<sub>30</sub>Mn<sub>5</sub>Al<sub>1</sub>C (0-3) wt % Mo were designed and processed through a vacuum induction melting route followed by hot-deformation and cold-working. The cold-rolled alloys were annealed to common grain size ( $\sim 80 \pm 5 \mu\text{m}$ ). Their deformation behavior is studied using uniaxial tensile tests in the temperature range of 300 - 973 K at an initial strain rate of  $10^{-3} \text{ s}^{-1}$  and tensile creep tests at 650°C in the stress range of 100-165 MPa respectively. The microstructural characterization is done at different stages using FESEM-EBSD and TEM. The flow strength of the alloys up to 673 K is dominated by solid solution strengthening by carbon than by molybdenum due to greater dislocation-solute interaction energy. However, beyond 673 K, the strength of the alloys increased with an increase in molybdenum by solid solution strengthening by molybdenum in 0.5 wt. % Mo and additionally by precipitation strengthening by molybdenum enriched carbides in 2 and 3 wt. % Mo alloys. On the other hand, a significant drop in fracture strain with an increase in molybdenum was observed at 973 K. This was seen in the form of a transition in fracture mechanism from transgranular ductile to quasi-ductile with intergranular cleavage fracture due to the formation of molybdenum-enriched carbides. Additionally, the serrated flow observed at specific temperatures is characterized and attributed to dynamic strain aging (DSA) phenomena occurring due to the interaction of dislocations with carbon at low temperatures and with molybdenum at high temperatures respectively. The creep studies revealed a decrease in steady-state creep rates with an increase in molybdenum at all stresses, with the stress exponents decreasing from 8.6 to 3.5 from 0 to 3 wt. % Mo. The calculated activation energies, the presence of primary creep, and stress exponents suggested that the dislocation-based creep mechanism could be the active creep mechanism among these alloys.



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# Fatigue and Creep-Fatigue Interaction Behaviour of Alloy 617M under High Cycle Fatigue

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## ABSTRACT

Present investigation deals with the high cycle fatigue (HCF) behaviour of alloy 617M (a chemistry-controlled variant of Inconel 617 with controlled composition and an important candidate material for the steam rotor forgings in the high temperature part of the Indian advanced ultra supercritical (AUSC) power plant) over a wide temperature range of 300-1023 K. The fatigue limit (corresponding to a cyclic life of  $10^7$  cycles) was observed to be significantly below the yield strength (YS) at ambient temperature. On the other hand, *run-out* ( $10^7$  cycles) at stresses significantly in excess of the YS was noticed in the temperature range of 853-973 K which was attributed to the occurrence of dynamic strain ageing (DSA) in the alloy. Constant life Haigh diagrams corresponding to different cyclic lives were constructed through tests performed using different *R*-ratios at a fixed temperature of 973 K. Furthermore, the influence of creep on the HCF performance was investigated by introducing periodic hold times at the mean stress ( $\sigma_m$ ) during the course of cycling at 973 K with a view to generating the design-relevant data for the material under combined action of high frequency cyclic loading and creep. Under such HCF-creep interaction tests, specimens were found to fail in certain stress amplitude ( $\sigma_a$ )- $\sigma_m$  combinations that otherwise resulted in run-outs in the absence of the creep holds, causing a deviation in the Haigh diagram. A few cyclic strain-controlled tests were also carried out on the alloy under isothermal and anisothermal cycling to assess the material performance in presence of high strain cyclic loading associated with start-stop operations. The observed life variations under different conditions are discussed in terms of the microstructural features and associated damage mechanisms.

## **Complexities of ferritic/martensitic (F/M) P91 steel weld joint under fatigue and creep-fatigue interaction loadings**

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### **ABSTRACT**

The Indian Prototype Fast Reactor uses modified 9Cr-1Mo ferritic/martensitic (P91) steel as a steam generator material, the weld joints (WJ) of which undergo phase transformations during fabrication by fusion welding. The change in mechanical properties within graded microstructure is a matter of concern as most failures occur in the weak links of the structure. Normal operations of the reactor (denote creep loading) and thermal transients during start-ups/shutdowns/temperature surge during reactor operations (denote fatigue loading) together resemble creep-fatigue interaction (CFI) loading waveforms. Thus, studies on the effect of various combinations of cyclic and creep loadings on the resultant microstructure and mechanical properties are necessary for safety and reactor operations reliability. To study the effects of waveforms, experiments were designed by applying dwells in either peak tension (T)/compression(C)/both T&C directions, with the periods of 10 min/30 min in consecutive cycles at elevated temperatures up to failure. The response of P91 WJ comprising of unaffected base metal (BM), heat affected zones (HAZ) (comprising of intercritical (IC), fine grains (FG), coarse grains (CG)) and weld metal (WM) under low cycle fatigue (LCF) (triangular waveform) and CFI (trapezoidal) loadings were different. Thus, their variations under LCF and CFI loadings shall be discussed with respect to the changes in the weld included angles, simulated microstructures (through heat treatments) and the different CFI waveforms employed (where symmetry, direction (tensile/compressive or both) and duration of the applied hold in the loading waveforms were altered). The need for a location specific microstructure-mechanical property evaluation, the dependence of cavitation propensities around second phase particles on the constituting microstructure and loading waveforms (under different T, C, TC CFI loadings), compressive dwell sensitivity and correlation between substructure and carbide stability in the WJ's constituents under various waveforms shall be discussed. Combination of X-ray diffraction (synchrotron) and EBSD techniques along with 2D hardness and metallography were utilized to reveal dislocations character, subgrains-carbide distribution to correlate with the CFI performance and failure locations. Need for miniature specimen testing shall be also highlighted.



## Utilizing heat treatment as a tool for creep life improvement and rejuvenation of modified 9 Cr steel post service exposure

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### ABSTRACT

Super-critical power plants have been set up for addressing the environmental concerns by reducing green house gas emissions. This is made possible by increasing operating steam temperatures, thereby, leading to improvement in efficiency of the power plant. High temperature components of super-critical power plant components use modified 9Cr steels which are capable of operating at relatively higher temperatures compared their sub-critical power plant counterparts using 1-2 Cr steels. In the present work, the modified 9Cr steel is subjected to a heat treatment containing an austenization treatment followed by a tempering treatment which enhances creep life by more than a factor of 2 in the temperature range of 550 to 650 °C. Characterization of the creep tested samples indicate numerous microstructural phenomena taking place such as precipitation of carbides, sub-grain coarsening, higher tetragonality of the martensitic phase and sluggish growth of  $M_{23}C_6$  carbides. The stress exponent values obtained between 4.5 to 5 indicating that the rate controlling creep deformation mechanism is dislocation climb controlled. Pinning of sub-grain boundaries by  $M_{23}C_6$  carbides was evident in the heat treated samples which caused lower creep rates and hence, higher creep resistance. In addition to this, a creep test progressing at 600 °C in the pristine condition was interrupted at life fraction ~0.5 and the same was subjected to the aforementioned heat treatment process. The sample obtained post the heat treatment was again subjected to a creep test at 600 °C using the same stress. It was observed that this sample showed an increase in creep life by a factor of ~4 compared to its corresponding as-received case. A detailed microstructural analysis revealed that the creep life of ferritic martensitic steels is predominantly governed by the coarsening kinetics of sub-grains. Faster sub-grain coarsening leads to higher creep damage accumulation. To mitigate sub-grain coarsening, synergistic effects are at play in the heat-treated condition such as the initial smaller sub-grain size and pinning pressure exerted by slow-coarsening  $M_{23}C_6$  carbides at their boundaries inhibiting their growth further. Moreover, the heat treated condition demonstrates a lower propensity for the formation of laves phases compared to the as-received condition as a result of shorter tempering durations. Overall, utilizing an apt heat treatment will not only enhance the creep life in modified 9Cr steel, but also helps in rejuvenating the creep life post service exposure.

**Keywords:** Creep; Modified 9 Cr steel; Sub-grain;  $M_{23}C_6$  precipitates; Pinning Pressure;

# **Irradiation creep in Zr-2.5%Nb alloy: a life limiting factor for pressure tube in pressurized heavy water reactor**

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## **ABSTRACT**

Pressure tubes made of Zr-2.5%Nb alloy are the most critical component in the pressurized heavy water reactors (PHWR). The extreme environment (e.g. high pressure, temperature and fast neutron flux) within the PHWR core induces dimensional changes (deformation) in pressure tubes which affect their useful service-life. It is widely believed that three mechanisms namely irradiation growth, thermal creep and irradiation creep are primarily responsible for dimensional changes of pressure tubes in PHWR. The deformation produced by aforementioned mechanisms manifests as elongation, diametral expansion, sag and wall thinning of pressure tube and the extent of this is dependent on exposure. Diametral creep may lead to the flow by-pass and the penalty to critical heat flux for fuel rods, longitudinal creep may lead to missing bearing support for end fitting provided in the lattice tube, and sagging may lead to interference with in-core components and potential contact between the pressure tube and calandria tube. In order to ensure integrity, safe and reliable operation and economic performance of the reactor, it is important to understand the factors that govern these mechanisms of deformation as well as to predict reliably the rate of deformation of pressure tubes. Several research and development efforts have been made in the past to understand the factors which influence the mechanisms of in-reactor deformation and its rate with the objectives to provide engineering solutions and to mitigate the mechanisms of degradation. As regards the rate of deformation, it is found that a host of variables such as neutron fluence, irradiation temperature, chemical composition and pre-irradiation material history (metallurgical texture, grain size/shape, extent of cold work, etc.) influence the in-reactor deformation. In order to reliably predict the deformation rate, all these variables must be simultaneously considered.

Diametral creep is the primary life limiting factor for the pressure tubes in PHWR. Irradiation creep plays critical role in governing the diametral creep. This study aims to explore the impact of alloy composition, microstructural parameters and texture on the irradiation creep of Zr-2.5%Nb pressure tubes. The mechanism of irradiation creep in Zr alloys will be reexamined. Drawing insights from the findings of this investigation, potential strategies for mitigating in-reactor dimensional changes in pressure tubes will be examined and discussed.

## Role of phase stability on strengthening in Multicomponent alloys

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### ABSTRACT

Multicomponent alloys commonly known as Medium and High entropy alloys have attracted lot of attention due to exceptional thermal stability and impressive mechanical properties. Although, early studies emphasized the high entropy effect capable of stabilizing single phase, several investigations in recent past have shown precipitate formation [1] and chemical ordering [2] to be a common phenomenon in these alloys. In this presentation, the role of defects on phase stability and its consequence on mechanical properties of two multicomponent alloys CoCrNi and AlCoCrNiFe will be discussed.

In CoCrNi alloys, it will be shown how deformation induced phase transition along with chemical ordering leads to strengthening during low temperature (@ 500°C) annealing [3]. Further, the importance of mobile dislocation on strength and ductility will also be discussed. In the eutectic AlCoCrNiFe alloy, observations from insitu synchrotron deformation will be used to evaluate the phase specific deformation characteristics and their role on strain hardening.

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## **An Overview of Thermal Creep Behaviour of Zr-2.5Nb Alloy – Effect of Manufacturing Route, Anisotropy, Cold work and Hydrogen**

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### **ABSTRACT**

Zr-2.5Nb alloy is extensively used for pressure tube applications in Indian Pressurized Heavy Water Reactors (IPHWR) in cold-worked and stress-relieved (CWSR) conditions. Under the operating conditions of internal pressure ~10 MPa (hoop stress ~ 130 MPa), temperature range ~253–293°C and flux ~  $3.5 \times 10^{17}$  n.m<sup>-2</sup>.s<sup>-1</sup>, thermal creep is one of the deformation modes prevalent in the regions of low neutron flux. To enhance the in-reactor performance, the manufacturing route for Zr-2.5Nb alloy has evolved with several modifications, which resulted in changes in the microstructure as well as chemical compositions. Depending on the manufacturing process, the Zr-2.5Nb alloy develops a strong texture due to its hexagonal close-packed structure (HCP), resulting in anisotropic mechanical properties including creep behaviour. In a reactor environment, hydrogen can be produced as a byproduct of the Zr/H<sub>2</sub>O corrosion reaction or through radiolysis of the water. Hydrogen, in solid solution or hydride form, is known to alter the mechanical properties of the Zr-2.5Nb alloy. The thermal creep behaviour of the Zr-2.5Nb alloy pressure tubes manufactured by different routes, and role of cold work, sample orientation and hydrogen content on the creep behavior of this material will be discussed in this talk. Thermal creep tests were carried out at 350°C, 400°C and 450°C at three normalised stress levels, which correspond to 0.7, 0.8 and 0.9 times the yield strength at the respective temperatures for all conditions. The rupture times, minimum creep rates, stress exponent and activation energies were determined to quantify the effect of different factors that influence the creep performance of the Zr-2.5Nb alloy. Further, scanning electron microscopy (SEM/EBSD)-based microstructural examinations were carried out to understand the effect of different factors and subsequent creep deformation on the Zr-2.5Nb alloy.

## Creep behavior of selectively laser melted Ti-6242

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### ABSTRACT

Ti-6Al-2Sn-4Zr-2Mo (Ti-6242) is a popular titanium alloy for compressor blade applications in aero-engines. Thermo-mechanical processing routes or investment casting routes are generally used for the fabrication of the compressor blades. The development of additive manufacturing techniques has opened a new route for manufacturing of near net shaped compressor blades. Along with a reduction in fabrication time, the higher fly to buy ratio makes development of compressor blades from additive manufacturing techniques an attractive proposition. However, there are very limited studies on additive manufactured Ti-6242 alloy. In this work we report the creep performance of Ti-6242 alloy produced using the selective laser melting approach. We evaluate the effect of laser power, scan rotation and layer height on the microstructure and mechanical properties of selective laser melted Ti-6242 alloy. We will report the creep behavior of the SLM alloy produced under different conditions and compare it against the creep behavior of conventional Ti-6242 bearing a fully lamellar structure. Creep tests conducted in the temperature range of 400 to 500 C are being analyzed to determine the stress exponent and activation energy of creep in the additive manufactured material and conventional Ti-6242 material.

### Acknowledgments (if any)

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# Creep and Stress Relaxation Response of Collagen Fibrils at the Nanoscale

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## ABSTRACT

Collagen fibrils, with diameters between a few tens to a few hundreds of nanometers, are the fundamental building blocks of connective mammalian tissues, such as bone, tendon, and skin. Although the tissue-level mechanical behavior of collagenous tissues is well studied, the time-dependent mechanical behavior of collagen at the nanometer length scales remains elusive. At the nanoscale, mechanical characterization presents a challenge, primarily due to limitations in achieving precise spatial and temporal control over stress or strain while maintaining molecular integrity. To bridge this knowledge gap, we devised an experimental approach [1-4] aimed at investigating the creep, stress relaxation, and mechanical behavior of individual collagen fibrils. Our methodology harnessed image-based edge detection and integrated closed-loop proportional–integral–derivative (PID) control to exert consistent force or stretch ratios on individual collagen fibrils via Microelectromechanical Systems (MEMS) devices [2]. This setup enabled real-time control of uniaxial tensile stress or strain with remarkable ~25 nm displacement accuracy. The experimental system was finely tuned to administer step inputs with rapid rise times ( $< 0.5$  s), minimal overshoot ( $< 5\%$ ), and negligible steady-state error ( $< 1\%$ ). We conducted creep and stress relaxation tests on collagen fibrils, both under partially hydrated conditions [2] and submerged inside phosphate buffered saline [1]. These fibrils exhibited strain-dependent stress relaxation and stress-dependent creep behavior across different deformation regimes observed in monotonic stress-strain curves, encompassing both the heel-toe and linear regimes. The observed behavior showcased non-linear viscoelastic properties well-captured by the adaptive quasi-linear viscoelastic model. Notably, this is the first time we have shown that hydrated mammalian collagen fibrils, the basic building blocks of connective tissues, exhibit nonlinear viscoelastic properties.

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# Creep and Creep Crack Growth in Unstable Microstructure

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## ABSTRACT

A stable microstructure is highly recommended to decelerate creep deformation and creep crack growth rate in elevated temperature applications. Hence, extensive care has been taken during the alloy design for components used in high-temperature applications such as advanced ultra-super-critical thermal power plants, aero engines, etc. The components are subjected to stringent working conditions during operation, which in turn influence the performance and life of the component by altering the microstructure. IN617 alloy has revealed 'non-classical creep behaviour' consisting of a minuscule primary and secondary regime followed by a prolonged tertiary regime during interrupted creep tests at two different test conditions, 700°C/275 MPa and 800°C/95 MPa. The post-creep analysis confirmed the dissolution and redeposition of carbides, generation of new twin boundaries and fine  $\gamma'$  precipitation as influencing factors for non-classical creep behaviour. The microstructural evolution during prolonged exposure is expected further to accelerate the creep crack growth rate in the component. Therefore, the alloy's creep crack growth behaviour has been investigated with different  $k_{\text{initial}}$  at 700°C correlating with a temperature-dependant fracture mechanics parameter ( $C^*$ ). The aforementioned microstructural alteration has also significantly impacted the creep crack growth rate.

**Keywords:** Creep, Creep Crack Growth Rate, Microstructure, Precipitation.

## Acknowledgements

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## Electric field assisted low temperature superplasticity in 3YSZ

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### ABSTRACT

Flash sintering is an electric field assisted sintering technique where a sample sinters in few seconds and at relatively much lower furnace temperatures. During flash sintering there is surge in electric power dissipation accompanied by non-linear increase in the electrical conductivity of the specimen. Not only the porous samples but dense polycrystals and single crystals also exhibit the flash phenomena, of course, they do not sinter.

In the present work we show that by application of modest dc electric field (100 V/Cm) superplasticity can be achieved in 3YSZ at furnace temperature as low as 800°C. Conventionally sintered dog-bone shaped samples of 3YSZ were subjected to constant load experiments in tension. The steady state strain rate was around  $4 \times 10^{-4} \text{ s}^{-1}$ . The electric field cumulatively caused Joule heating in the sample, reduction in flow stress required to cause plastic deformation and retarded grain growth. For the combination of field and temperature, there was a critical current density below which the samples did not show any elongation. The fractured surface of the deformed samples revealed minimal grain growth in the samples. The sample temperature in the state of flash was around 1400°C. The stress exponent was around 2 indicating grain boundary sliding as the dominant operating mechanism. It is postulated that point defects induced by electric field play an important role in the deformation. The method can be used for faster and low temperature forming of ceramic materials.



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# **A mean field dislocation density reliant physical model to predict the creep response and microstructure evolution of steel 304HCu**

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## **Abstract**

A novel physical approach to address the creep behavior of 304HCu austenitic stainless steel is presented. This approach combines a dislocation density reliant physical model with a continuum damage mechanics (CDM) model. Two different dislocation densities: mobile and forest, and dislocation mean free path are used to describe the substructure evolution in order to model the creep strain. The original Orowan's equation for estimating creep strain rate is modified employing CDM based softening parameters to take account of damage causing tertiary creep. The model is advantageous in the sense that with the ongoing creep, the evolution of different variables that are dislocation densities, dislocation mobility, dislocation velocity, internal stress, effective stress and damage evolution is tracked and discussed thoroughly. Furthermore, the model output is corroborated with experimental creep data of 304HCu steel published in the literature.

**Keywords:** Creep modelling; Dislocation density; Dislocation mobility; Creep damage; Internal stress;

## **Anisotropic creep behavior of CP-Ti plate at room temperature – insights into the deformation mechanisms**

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### **ABSTRACT**

Titanium (Ti) and its alloys are vulnerable to the peculiar phenomenon of room-temperature creep. They are reported to accumulate significant amounts of creep strains under ambient temperatures ( $T/T_m < 0.25$ ) at stresses much below the macroscopic yield strength. This limits their applicability in critical applications that require stringent dimensional tolerance. The present study investigates the room temperature creep behavior of a Grade 2 commercially pure Ti (CP-Ti) plate. The CP-Ti plate showed texture extremes in the two directions chosen for testing – the Rolling Direction (RD) and the Transverse Direction (TD). Monotonic tensile tests revealed appreciable differences in strength and fracture strains. Simultaneously, significant differences in creep strains were observed under equivalent loading conditions for the two directions. Utilizing electron backscattered diffraction (EBSD) and slip trace analysis provides insights into the operating deformation mechanisms. Additionally, observed differences in strength and accumulated strains are correlated with the distinct texture along the two directions.

### **Acknowledgments**

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# Effect of hydrogen on plasticity of $\alpha$ -Fe: a multi-scale assessment

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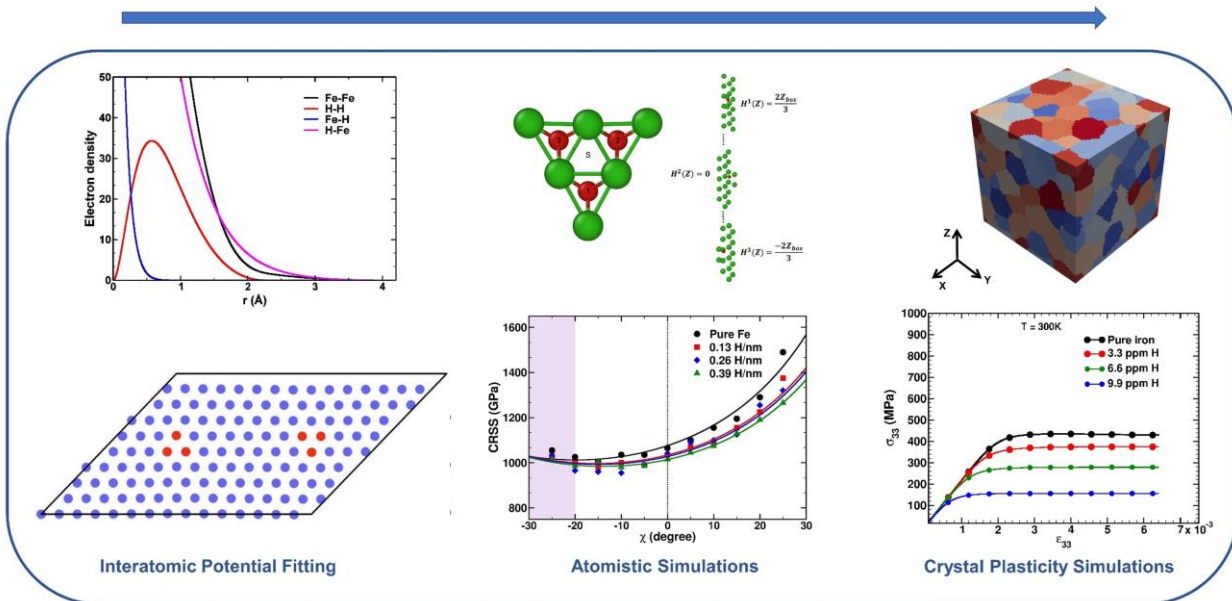
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## ABSTRACT

In this work, the effect of hydrogen on the dislocation-based plastic behavior of  $\alpha$ -Fe was examined by carrying out a multi-scale study. The influence of hydrogen concentration on the critical shear stress required for screw dislocation glide was quantified at the atomic length scale. The obtained variation in dislocation glide behavior was utilized to develop an accurate continuum description to examine the dislocation based plasticity in polycrystalline  $\alpha$ -Fe. To enable this study, a new Fe-H interatomic potential was developed that provide an accurate description of various hydrogen - defect configurations, which is essential to accurately study the effect of hydrogen on dislocation glide behavior. The screw dislocation core reconstruction observed due to the presence hydrogen was validated by performing large-scale DFT calculations based on the DFT-FE framework. To comprehensively quantify the effect of varying hydrogen concentration on the dislocation glide mechanics, a large number of atomistic simulations were carried out. Lastly, crystal plasticity simulations were performed to assess the ramifications of the atomistically observed variation in dislocation glide behavior introduced by hydrogen on the meso-scale deformation behavior of polycrystalline  $\alpha$ -Fe.



## Graphical Abstract

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# Creep behavior of Oxide Dispersion Strengthened Iron Aluminide (Fe<sub>3</sub>Al)

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## Abstract

Iron aluminide intermetallic (Fe<sub>3</sub>Al) can be a potential candidate for the development of new structural materials with improved performance due to their good oxidation-corrosion resistance in various environments, relatively low density and low cost. Even though room temperature mechanical strength is good, it suffers from low ductility (8%). Moreover, the transition temperature of Fe<sub>3</sub>Al to FeAl is 823K (550°C) limits the use of Fe<sub>3</sub>Al intermetallic as a structural material beyond 823K. Low ductility can essentially be improved by the selection of suitable alloy compositions, grain size control and dispersion strengthening, but achieving sufficient creep strength at the high temperatures remains an obstacle to significant engineering use of these materials and has been the subject of extensive recent effort. The main aim of the present work is to study the creep performance of Oxide Dispersion Strengthened (ODS) Fe<sub>3</sub>Al (Fe-12.6Al-5.3Cr-0.13Ti) over a range of temperatures of 823-923K and over a range of stresses of 150-250MPa. By analyzing the experimental creep data and fitting obtained Mukherjee-Bird Dorn creep equation, obtained strain hardening index of (n=16) and activation energy of (450 KJ/mol) the high value of strain hardening index and activation energy are due to the presence of fine oxide dispersoids. Micro structural characterization revealed the reduced size of dimples with increasing stress indicating the increased ductile nature of fracture. Energy dispersive X-ray spectroscopy (EDS) studies shows no oxide layer on fractured surface. Hardness measurement after creep test shows that hardness is decreased with increasing creep stress (from 425 to 394Hv1). From X-Ray diffraction (XRD) results show that there is no phase change at 823K with increasing stress and has shown signature peaks of Fe<sub>3</sub>Al as present in the initial bulk material. Dislocation density also calculated. As a function of test parameters, the overall creep behavior and associated mechanism of ODS-Fe<sub>3</sub>Al-Ti alloy will be presented.

**Key Words:** Oxide dispersion strengthened Iron Aluminides, Creep, Activation energy, Fe<sub>3</sub>Al, FeAl, Dislocation density

## **Solute defect interaction in superalloys during high-temperature deformation**

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Single crystal superalloys are multi-element ( $> \sim 10$  elements) alloys that are critical for the parts of high-temperature turbine engines. In operation, the alloy undergoes creep, i.e., plastic deformation under stress at high temperatures, producing defect structures. These structures are stabilized by the segregation of elements/solutes, influencing further deformation. Solute segregation effects to these defect structures produced during the creep of superalloys are believed to influence their high-temperature performance.

In this talk, I will briefly go through the solute-defect interactions of different superalloys plastically deformed at different temperatures and stress levels. Atomic-scale compositional and structural analysis of these defects are correlated with the segregation behavior on their local internal fault structure. These new observations highlight the possible creep rate limiting solutes whose content can be optimized for future alloys with higher temperature capabilities for more energy-efficient and cost-effective turbine engines with lower CO<sub>2</sub> emissions.

# Small Punch Creep Testing to Explore Deformation in PreFatigued and Welded Specimens

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## ABSTRACT

Evaluation of mechanical properties of materials using miniaturized specimen has become a necessity from the viewpoint of efficient utilization of available resources like raw material, time, manpower, infrastructure, and costs. Of the several such emerging techniques, Small Punch Creep (SPC) provides an excellent tool to study the time intensive creep behavior of a material by using specimens as small as 0.5 mm thick. As the testing standards are still evolving, it is essential to validate the results of SPC with the behavior so observed in conventional creep testing. Here, two such cases are discussed to showcase the utility of SPC for investigating the high temperature creep behavior. In the first case, as a part of possible remanent life investigation of a fatigued sample, a disc of 0.5 mm thickness of pre-fatigued Su-263 superalloy was tested for SPC. Effects of load and test temperature are shown for virgin and fatigued samples. An interesting feature of two-slope behavior in the creep curve is observed. The second case discusses extracting creep-related material constants for an individual heat affected zone (HAZ) of a dissimilar weld joint to help develop a realistic model to study stress analysis during conventional creep exposure. An individual HAZ was located, and miniature specimens were extracted for SPC testing to get the necessary material constants for further modeling and simulation.

# Can Creep Strength and Oxidation Resistance be Simultaneously Achieved for Co-based Single Crystal Superalloys?

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## ABSTRACT

The creep properties of Co-base superalloys consisting of FCC solid-solution based on Co and L<sub>12</sub> (Co<sub>3</sub>(Al,W) cuboidal precipitates have recently been reported to be improved beyond those of Ni-base superalloys of the 1<sup>st</sup> generation by simultaneous additions of Ta and Ti. But, the oxidation resistance of these Co-base superalloys containing Ta and Ti has been found not to be so good. A large amount of Cr additions (as well as a small amount of Si addition), on the other hand, has been known to improve the oxidation resistance of Co-based alloys above 900°C. However, nothing is known about how we can incorporate Cr and Si together with Ta and Ti in Co-based superalloys in a single-crystal form. Even this is possible, nothing is known about how these alloys behave in high-temperature creep strength and oxidation resistance. We have carried out experimental investigations on the phase equilibria, microstructures, oxidation resistance at 900°C and creep properties at 900 and 1000°C for a number of (Co<sub>0.8</sub>Ni<sub>0.2</sub>)-aAl-bW-cTa-dTi-8Cr-1Si alloys (*a*, *b*, *c* and *d*; variables in atomic percent) with, in most cases, their single crystals, to see if creep strength and oxidation resistance can simultaneously be achieved for Co-based single crystal superalloys.

Cr and Si alloyings decrease the  $\gamma'$  solvus temperature drastically, so that the  $\gamma'$  solvus temperature of the (Co<sub>0.8</sub>Ni<sub>0.2</sub>)-8Al-7W-8Cr-Si alloys never exceed 1200°C. However, substituting W with Ta is found to be effective in increasing the  $\gamma'$  solvus temperature without precipitation of any third phase (such as DO<sub>19</sub>). Similarly, substituting W and Al with Ti is also an effective way to increase the  $\gamma'$  solvus temperature. The  $\gamma'$  volume fraction at 1000°C for Ti-additions is higher than that for Ta additions, due probably to the higher solubility limit of Ti (8 at.% for Ti and 4 at.% Ta, respectively) and the higher thermal stability of the  $\gamma'$  phase at high temperature. Creep properties and oxidation resistance of (Co<sub>0.8</sub>Ni<sub>0.2</sub>)-8Al-2W-4Ta-8Cr-Si, (Co<sub>0.8</sub>Ni<sub>0.2</sub>)-5Al-4W-8Ti-8Cr-Si and (Co<sub>0.8</sub>Ni<sub>0.2</sub>)-6Al-2W-2Ta-6Ti-8Cr-Si alloys are investigated. The oxidation resistance of the Co-7Al-8W-2Ta-4Ti alloy is catastrophically bad, although this alloy is found to exhibit a reasonably high  $\gamma'$  volume fraction and  $\gamma'$  solvus temperature. When compared to the Co-7Al-8W-Ta-4Ti alloy, Cr, Si-alloyed Co-base superalloys exhibit superior oxidation resistance at 1000°C due to the formation of continuous oxide scales. The rupture time in tensile creep under the condition of 900°C/ 428 MPa for the (Co<sub>0.8</sub>Ni<sub>0.2</sub>)-8Al-2W-4Ta-8Cr-Si alloy is much shorter than that for the Co-7Al-8W-2Ta-4Ti alloy (approximately one thirteenth) due to the small  $\gamma'$  volume fraction at high temperatures. Thus, the creep strength and oxidation resistance is found not to be achieved simultaneously for Co-based superalloy single crystals.

## Acknowledgments (if any)

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# **Understanding the role of solute elements at atomic scale during creep deformation in CMSX-4 Ni base superalloys**

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The necessity for jet engines to operate at elevated temperatures for reducing carbon emissions, underscores the critical need for advancements in high-temperature materials. Nickel-base superalloys, which are prominently employed in the most thermally demanding sections of jet engines, such as turbine blades, have undergone continuous refinement. The introduction of 1% Rhenium (Re) into 2nd generation Nickel-based superalloys has resulted in a transformative two-fold extension in creep life—a pivotal advancement. Subsequent developments have seen a further escalation in Re content, reaching up to 2at%. Nevertheless, the challenges posed by the high density and cost of Rhenium hinder its seamless commercial utilization. This challenge compels researchers to explore alternative elements to Rhenium (Re) without compromising high-temperature properties. Despite dedicated and efforts in this pursuit, the identification of a suitable alternative has proven to be elusive.

Here, we have tried to address this critical gap by conducting a comprehensive creep deformation study, exploring two distinct conditions: an intermediate temperature, high-stress regime at (800°C, 800MPa) and a high-temperature, low-stress regime at (1000°C, 200MPa) in CMSX4, a representative 2nd generation Ni-base superalloys. Our focus centered on elucidating the evolution of structure and chemistry in defects that accommodate plastic deformation during the creep process by employing advanced correlative microscopy techniques, including cECCI, TEM, and APT.

Based on our results, we propose novel atomistic mechanism operating during intermediate and high temperature creep deformation. We have also highlighted the role of individual solute atoms in governing creep properties which may offer possible alternative of Re.



# Creep and Creep-Fatigue interaction in Ni-based superalloy having low volume fraction of $\gamma'$

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## ABSTRACT

Nickel-based superalloys are widely used in turbine blades, vanes and disk of aeroengines, where the components undergo very high temperatures. Interestingly, the current advancement of thermal powerplants, such as advanced ultra-super critical (A-USC) power plants, requires them to operate at a steam temperature of 760 °C and pressure of 35 MPa. Therefore, the currently used boiler tube steels are planned to be replaced by Ni-based superalloys in the A-USC power plants. Here, we will discuss the creep and creep-fatigue interaction of Ni-based superalloy, namely IN740H that is considered to be suited for A-USC power plants. This alloy showed high apparent creep stress exponents that increased monotonically at lower stresses and high creep activation energies. However, if compensated for the threshold stress, the stress exponent over the wide range of stresses became 4, and the true activation energy reduced to indicate solute atom diffusion in  $\gamma$ . The dependence of threshold stress on the temperature was established and compared with different high-temperature materials. Further, creep-fatigue experiments have been performed with change in strain amplitude as well as dwell time. The insights into creep mechanisms and creep-fatigue interactions, including the possible origin of threshold stress, highlights the importance of solute-drag that is obtained from transmission electron microscopy and atom probe tomography will be discussed.

## Acknowledgments

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# Comparison of Creep Characteristics of HEA Cantor Alloy and Binary Solid Solution Alloys

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## ABSTRACT

It is well recognized that the steady-state creep characteristics of solid solution alloys at  $0.6-0.7T_m$  ( $T_m$ : the melting point in Kelvin) is classified into two classes termed Alloy-type and Metal-type [1]. The characteristics are classified mainly by the stress dependence, temperature dependence and concentration dependence of the steady state creep rate, and their rate-controlling mechanisms in binary solid solutions are reasonably understood. Al-Mg solid solution alloys are the typical solid solutions which show the two types of behavior and transition from Alloy to Metal-type. The same transitions are reported in other binary solid solutions, such as  $\alpha$ Fe-Mo, Mg-Al, and so on. The typical difference between the two types of behavior is the stress exponent. It changes from about 3 to 5 with increasing the applied stress, which corresponds to the transition from the Alloy-type to the Metal-type. Similar behavior of the Alloy-type has been reported in Cantor alloy [2] and its change of the stress exponent from about 3 to 5 are reported, too [3]. In this presentation, creep behaviors of Cantor alloys are investigated in detail, and compared with that of binary solid solutions which show both two types of creep behavior. The existence of the effective stress, normalized creep rate, and other characteristic value such as the concentration exponent have been investigated. Creep and its transition behavior from Alloy to Metal-type in Cantor alloys are basically similar to that of binary solid solutions. Composition dependence on creep rate in Cantor alloy, however, seem slightly complex compared to that in binary solid solutions. Existence of the effective stress in Alloy-type behavior are consistent with viscous motion of dislocations in binary solid solutions and Cantor alloys. It is concluded that further investigations of the origin of the effective stress are required in solid solutions which consist of multi elements such as Cantor alloy and HEAs.

## Acknowledgments

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# Estimation of Residual Stresses in Aero-Engine Piping System

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## ABSTRACT

Thin welded pipes have extensive applications across various engineering sectors, including aero-engine piping systems, thermal and nuclear power plants, and the petroleum industry. This paper addresses the crucial task of estimating residual stresses in a fuel pipe assembly with characteristics similar to thin-walled pipes. Microcracks have been observed in the nipple-pipe weld section of this Nickel-based superalloy fuel pipe assembly, necessitating a comprehensive residual stress analysis. To achieve this, a robust computational procedure is introduced to assess temperature distribution and residual stress variations. Utilizing ABAQUS non-linear thermal analysis and the DFLUX subroutine, numerical simulations are employed to elucidate residual stress distribution within the fuel pipe assembly. Additionally, the study includes experimental validation using the blind hole drilling method to verify the accuracy of the numerical simulations. The results reveal a high degree of agreement between the simulated and experimental findings, affirming the effectiveness of the computational approach in accurately estimating residual stresses in complex thin-walled structures. Furthermore, the study explores practical strategies for mitigating residual stresses, including post-weld heat treatment and alterations to the fillet shape. These insights offer valuable contributions to enhancing the structural integrity of thin-walled pipes, with direct implications for safety and reliability in demanding engineering applications. This research enhances our understanding of residual stress phenomena in thin welded pipes and provides practical solutions to address associated challenges, contributing to the field of materials science and engineering.

**Keywords:** Fuel pipe assembly, Residual Stresses, ABAQUS simulation

## Acknowledgments

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# Precipitation-strengthened Ni-rich high entropy superalloy with excellent thermal stability and superior high temperature mechanical properties

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## ABSTRACT

Designing a novel class of alloy system by amalgamating the idea of high entropy alloy with superalloy has opened a new paradigm for the materials research community for developing advanced high temperature materials with excellent thermal stability and superior high temperature mechanical properties. In the present study, effect of Ti microalloying on phase evolution, microstructural development and mechanical properties of novel  $\text{Ni}_{46-x}\text{Co}_{20-x}\text{Al}_{12}\text{Cr}_8\text{Fe}_{12}\text{Mo}_2\text{Ti}_x$  ( $x = 1, 2, 3$  and  $4$ ) precipitation-strengthened high entropy superalloys (HESAs) were systematically investigated. The equilibrium phase formation by varying Ti content was predicted using Thermo-Calc software (*TCHEA-4.2* database). The proposed HESAs showed a broad  $\gamma + \gamma'$  equilibrium phase field over a long temperature range (from  $670^\circ\text{C}$  to a  $\gamma'$  solvus temperature  $1181^\circ\text{C}$ ) without presence of any brittle intermetallic phases. The microalloying of 8 at. % Ti, resulted in high volume fraction ( $\sim 62\%$ ) of continuous  $(\text{Ni})_3(\text{Al}, \text{Ti})$ -type ordered  $\gamma'$  precipitates within disordered face-centered cubic (FCC)  $\gamma$  matrix. The microstructure and the thermal properties of the HESAs showed excellent correlation to the thermodynamic predictions as shown in Fig. 1. Increasing in Ti content promotes the continuous partition of the of the  $\gamma'$  precipitates and reduces the content of Al and Ti in the FCC matrix, resulting in a large lattice mismatch between the  $\gamma$  matrix and  $\gamma'$  precipitates. The homogenized and aged  $\text{Ni}_{42}\text{Co}_{16}\text{Al}_{12}\text{Cr}_8\text{Fe}_{12}\text{Mo}_2\text{Ti}_8$  HESA exhibited superior mechanical properties at room-temperature and elevated temperature ( $800^\circ\text{C}$ ) owing to the higher volume fraction of  $\gamma'$  precipitates and increased anti-phase boundary energy (APBE). This pioneering work on developing Ni-rich HESA is anticipated to further widen the research on designing precipitation-strengthened Ni-rich HESAs with superior high temperature mechanical properties and to expand their scope to engineering applications.

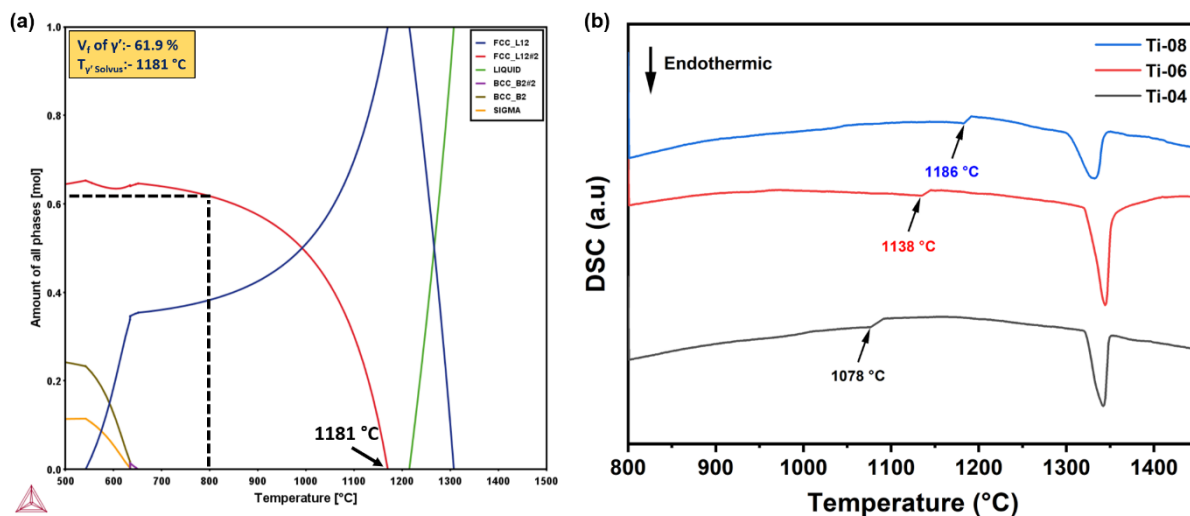


Fig. 1. (a) Equilibrium phase diagram of  $\text{Ni}_{42}\text{Co}_{16}\text{Al}_{12}\text{Cr}_8\text{Fe}_{12}\text{Mo}_2\text{Ti}_8$  HESA and (b) heat flow curves measured in DSC with a constant heating rate of 5 K/min

## Simulations of Creep in Crystalline Solids using Coarse-Grained Atomistics

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Overcoming the time scale limitations of atomistics can be achieved by switching from the state-space representation of Molecular Dynamics (MD) to a statistical-mechanics-based representation in phase space [1], where approximations such as maximum-entropy or Gaussian phase packets (GPP) evolve the atomistic ensemble in a time-coarsened fashion. In practice, this requires the computation of expensive high-dimensional integrals over all of the phase space of an atomistic ensemble which is commonly accomplished efficiently by low-order numerical quadrature. We show that numerical quadrature in this context comes with a set of inherent problems, which corrupt the accuracy of simulations, especially when dealing with crystal lattices with imperfections. As a remedy, we demonstrate that Graph Neural Networks, trained on Monte-Carlo data, can serve as a replacement for commonly used numerical quadrature rules, overcoming their deficiencies and significantly improving the accuracy [3]. Being a quasistatic minimization for equilibrium systems, GPP is computationally efficient and robust compared to MD or DFT with thermodynamic integration and can be used to find the energy barriers at a finite temperature using the nudged elastic band (NEB) calculations. Such NEB calculations can be used to simulate the diffusion of impurities over longer time scales than those accessible by MD simulations.

We showcase the computational efficiency of the GNN-assisted time-coarsened atomistic algorithm compared to MD simulations by benchmark studies on computing surface elastic parameters [2] and grain boundary energies of metals. Following this, we present long-timescale simulations of diffusion creep in pure metals and metallic alloys using on-the-fly NEB calculations for finding solute-hopping energy barriers to evolve the atomic concentrations staggered with a quasi-static minimization after every time step.

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# **Inverse Material Design using Generative Artificial Intelligence for High Mechanical Strength**

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## **ABSTRACT**

Autonomous materials discovery, tailored to specific properties through Generative Artificial Intelligence (AI) has been recently demonstrated to be a powerful tool for inverse materials design in the multi-dimensional composition space. It is time-consuming to design materials with functionality dependent properties using via the experimental route. AI can reduce the number of trial-and-error experiments required and hence guide experimentation accelerating the process. Leveraging high-throughput screening, Density Functional Theory, and deep learning, this study introduces a generative model to predict stable binary crystals with high Bulk Modulus. Applied to all possible binary systems across the periodic table, our model demonstrates efficacy of Generative Adversarial Networks (GAN), in obtaining new stable crystals with good mechanical properties: key for materials performance in multiple structural applications. The versatility of our approach, particularly in multicomponent systems, opens avenues for inverse materials design with optimal properties for varied applications.

# Multi-axial Cyclic Deformation Induced Phase Transitions in Aluminium using Large-scale molecular dynamic simulation

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## ABSTRACT

Aluminium has excessive importance for large scale production of automobile and aerospace spare parts due to its lightweight and non-corrosive nature. It is also suitable for numerous packaging applications such as edible things, electronic components, and medicines. I used multi-axial cyclic deformation approach to examine mechanical properties of aluminium using large scale molecular dynamics simulation. Multi-axial cyclic deformation referred to uni-, bi- and tri-axial direction used for cyclic deformations. I studied the formation of various kinds of hysteresis curve during multi-axial cyclic deformation. It is found that the hysteresis curve strictly dependent on the type of cyclic deformation. I also investigated the evolution of various crystalline phases and related dislocations during cyclic deformations. Body Centre Cubic (BCC) structure gradually formed during tri- and bi-axial cyclic deformations as shown in the following snapshots. Further, it is found that grain coarsening and twinning-detwining happened during fcc to bcc transition. For the characterization of cyclic deformation, various calculation methods such as adaptive common neighbour analysis, variation in total energy and dislocation extraction algorithms adapted. I believe that the results of present study will provide new understanding to the researchers for the design and characterization of aluminium based component.

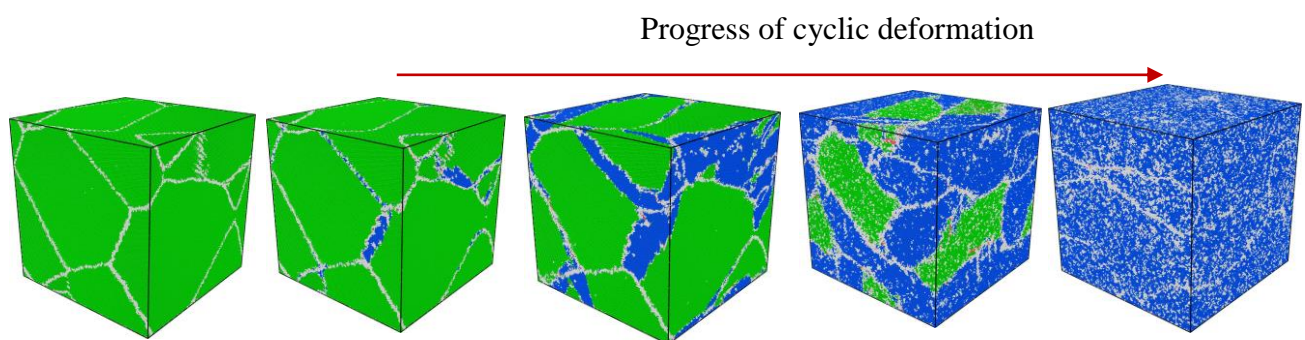


Figure: Snapshots at various instant during tri-axial tension-compression cyclic deformation of aluminium. Green, blue, red and gray colour atom depict fcc, bcc, hcp and other structures respectively. Red colour arrows shows the progress of cyclic deformations,

## **Creep deformation mechanisms in Inconel 617 explored through EBSD analysis at elevated temperatures**

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### **ABSTRACT**

The present study explored the creep behaviors of an innovative polycrystalline nickel-based superalloy across a broad range of applied stresses (120-300 MPa) and temperatures spanning from 700 °C to 800 °C. In addition to investigating the microstructure, the analysis investigated into the deformation mechanisms. The findings reveal that the creep curves for the Inconel 617 superalloy primarily comprise primary and accelerated stages, with the steady-state stage not prominently observed. As the applied stress increases at a constant temperature, the minimum creep rate rises, and the corresponding creep life decreases. The extended creep life at 750°C is attributed to the coupling of stacking faults within the  $\gamma'$  and deformation twinning.



## Creep behavior of solid solution strengthened Co-base superalloy at intermediate temperatures (650 – 900 °C)

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### ABSTRACT

Despite the existence of solid solution strengthened Co-base superalloys for several years, due to their microstructural complexity, their high-temperature deformation behaviour has yet to be understood thoroughly. However, interesting observations of the multi-principal element alloys in the recent past have rekindled interest towards the solid solution Co-base superalloys [1]. These alloys are considered to be candidate materials for alternatives to Ni-base superalloys due to their higher melting point, solid solution strengthening, and precipitation-forming ability. Notably, the continuous evolution of the carbides and their interaction with dislocations significantly affect the high-temperature mechanical properties of these alloys. Thus, understanding the creep behavior of these alloys at elevated temperatures is imperative. Intriguingly, earlier studies on these alloys have shown a distinct dependence of primary creep on applied stress and test temperature in the intermediate temperature range ( $\sim 0.55 - 0.7 T_m$ ) than at higher temperatures ( $> 0.7 T_m$ ) [2]. However, the underlying mechanism for such a behavior has not been elucidated, yet.

With this motivation, the present study was taken up to address the creep behavior of an FCC-stabilized Co-base superalloy Co-22Cr-22Ni-14W-2Fe-0.1C (wt. %). Constant load creep tests were conducted on solution-annealed specimens in the stress range of 80 – 400 MPa and at temperature range of 650 – 900 °C. All the creep tests were performed until achieving the steady state. Creep parameters, viz. activation energy and stress exponent, were evaluated. Further, substructure evolution with strain and temperature was characterized using transmission electron microscopy. The heterogeneity in the substructural features is rationalized using the internal stress measurements performed from the classical dislocation line tension analysis. Overall, an attempt has been made to identify the rate-controlling mechanisms during transient creep by correlating the substructure evolution with mechanical data and kinetic analysis.

Keywords: Cobalt-base superalloys, Creep, Transient creep, Substructure, Activation energy, Stress exponent, Transmission electron microscopy

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## **Investigation into Degradation Mechanism of Turbine Blades during Operation of Fighter Aircraft Engine**

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### **ABSTRACT**

Life of an aero-engine in particular for a fighter aircraft engine is dictated by the life of hot-end components which is directly influenced by the TET(turbine entry temperature) of the engine. Nickel base alloys for their excellent creep resistance are being used extensively for turbine module components of aero-engine. Manufacturing process of turbine blades has evolved from forging route to casting route as the forgeability of the material decreases with addition of refractory elements and increase in volume fraction of high temperature resistant phase such as Ni<sub>3</sub>Al ( $\gamma'$ ) in nickel base alloys. Further casting route has evolved from equiaxed to directional solidification with columnar grains and single crystal grain configuration to have higher creep life. Extensive cooling and protective coatings have been employed to reduce the metal temperature of turbine blade, but it increases the challenges for manufacturing as well as the additional damage mechanisms come into play.

This paper deals with the degradation in creep life, coating and residual stress of turbine blades of fighter aircraft engine. Three case studies namely a) Forged turbine blade of a straight flow turbojet engine, b) DS blade with columnar grains and c) SX turbine blade of a low by-pass turbofan engine is being investigated for the predominant degradation mechanisms limiting their life during operation.

Stress rupture life is the acceptance criteria for turbine blades during manufacturing & overhaul and it is affected by the increase in minimum creep rate during operation. Particularly, life is limited by creep growth for turbine blades of advanced engines operating at higher TET of about 1650K. Coating degradation indirectly limits the life of blade because of development of deleterious phases between coating substrate interface in the form of SRZ(secondary reaction zone). Further, base material is sacrificed during de-coating and recoating during overhaul. Therefore, selection of overlay and diffusion coating play an important role in life cycle management of turbine blade. Further, selection of coating get influenced by operating environment such as saline atmosphere and chemical. Provision of CMAS resistant coating may be made to resist such damage mechanism of coating. In addition to creep and oxidation, fatigue limits the life of turbine blades. In advanced aero-engines, favourable residual stress has been exploited to enhance fatigue life. Therefore, estimation of residual stress provides a qualitative measure of degradation in structural integrity of aero-engine components.

## **Elucidating Creep Deformation Mechanisms in Haynes 282 Subjected to Prolonged Ageing Treatment**

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### **ABSTRACT**

The creep behavior of a polycrystalline Nickel-based superalloy, Haynes 282, subjected to prolonged ageing treatment, was investigated. Ageing treatments were administered to hot-rolled and solutionized plates (as-received condition) at 750°C for durations of 46, 470, and 6000 hours. Creep rupture tests were subsequently conducted at temperatures of 750, 775, and 800°C, with applied stresses ranging from 300 to 420 MPa. Ageing resulted in the coarsening of  $\gamma'$  precipitates, a denser and more continuous network of grain boundary carbides, and the precipitation of a Mo-rich  $\mu$  phase, particularly notable at 6000 hours of ageing. Despite the prolonged ageing, grain growth was effectively limited due to the Zener pinning of grain boundary carbides. Creep rupture tests indicated that the creep rupture life increased with ageing duration up to 470 hours for each specific applied stress. However, at 6000 hours, the creep rupture life was observed to be lower than that of the as-received condition. Apparent stress exponents, derived from Norton's power law, ranged from 6 to 10. To determine the true creep deformation mechanism, threshold stresses were estimated, revealing a monotonic increase with ageing duration up to 6000 hours, attributed to the increased size of  $\gamma'$  precipitates and the associated resistance to  $\gamma'$  shearing. Consequently, the rate-controlling creep deformation mechanism transitioned from diffusion control in the as-received condition to dislocation-mediated mechanisms in the aged conditions. Thus, thermal ageing has a profound influence on the nature of the creep curve as well as the creep rupture duration. The findings provide critical insights into the microstructural evolution, stress response, and creep deformation mechanisms under prolonged ageing conditions, which are pivotal for optimizing the performance and reliability of Haynes 282 in high-temperature applications.

## **Effect of Ultrasonic shot peening on the fatigue behaviour of ‘Nickel’ free austenitic stainless steel**

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### **ABSTARCT**

Austenitic stainless steel stabilized by nitrogen, with very low content of undesired nickel (0.1 wt%), is a much better option for bioimplants in respect of the austenitic stainless steels stabilized with a high content of nickel ( $\geq 8$  wt%). For biomedical applications, in particular for orthopedic implants, resistance of the implant material against cyclic loading is an essential criterion. This investigation deals with the role of surface treatment by ultrasonic shot peening (USP) with 3 mm shots, for varying durations of 3-18 minutes, on low cycle fatigue behavior at total strain amplitudes of  $\pm 0.4\%$  to  $\pm 0.8\%$ . It was observed that while fatigue life was reduced due to USP at the highest strain amplitude of  $\pm 0.8\%$ , it was enhanced by  $\approx 18$  times at the lowest strain amplitude of  $\pm 0.4\%$ , following USP for 18 minutes.

**Keywords:** Ultrasonic shot peening; Low cycle fatigue; Compressive residual stress, Fatigue life

## Deformation of CoCrFeNi alloy thin films under thermal fatigue

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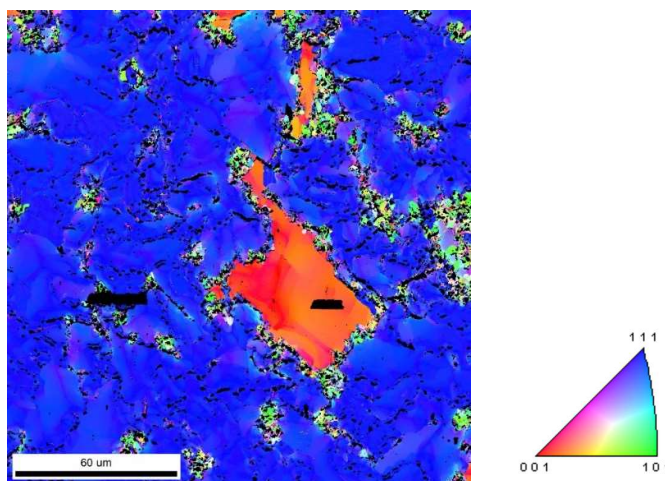
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Equiatomic CoCrFeNi alloy thin films were deposited on crystalline c-sapphire (basal plane  $\alpha - Al_2O_3$ ). As deposited films grew consistently homogenous, predominantly fcc and nanocrystalline. The heat treatment conditions were tuned to obtain fully dense thin films with tens of micrometers grain sizes. The texture showed a combination of close to  $\{001\}$  and close to  $\{111\}$  grains. Substrate curvature measurements indicated thermoelastic deformation with no significant plasticity until 673 K. Rapid thermal cycling to hundreds of thermal cycles between 473 – 973 K, showed the evidence of deformation through dislocation plasticity and twinning. Locations of heavy deformation exhibited the formation of recrystallized grains. The paper discusses possible implications to the theory and applications.



Electron Back-scattered diffraction (EBSD) - Inverse pole figure (IPF) map of CoCrFeNi alloy thin films after thermal cycling.

**Keywords:** Thin films, CoCrFeNi alloy, thermal fatigue

### Acknowledgments

- The DFG-ANR collaborative project “Analysis of the stability of High Entropy Alloys by Dewetting of thin films (AHEAD)” under the grant DE796/11-1 (MPIE), LU1175/22-1 (RU Bochum) and ANR-AHEAD-16-CE92-0015-01 (CINaM).
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## Low Cycle Fatigue Behaviour of a 316LN Austenitic Stainless Steel between 300 K-923 K

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### ABSTRACT

Low cycle fatigue (LCF) tests were conducted at a strain amplitude of  $\pm 0.6\%$  at three different temperatures, 300 K, 823 K and 923 K on a 316 LN austenitic stainless steel. At all three temperatures, the cyclic stress responses (CSR) are marked by an initial cyclic hardening, followed by softening. The initial hardening is found to increase with increase in temperature. The cyclic stress-strain hysteresis loops exhibited serrations at 823 K and 923 K, indicating the presence of strong dynamic strain ageing (DSA) at these temperatures. To rationalize the origin of initial hardening and softening at different temperatures, the friction stress and back stress values are computed from the measured stress-strain hysteresis loops. It is found that at 300 K, the reduction in friction stress is overcome by a marked increase in back stress, resulting in cyclic hardening in the initial cycles. On the other hand, the friction stress dominates over back stress at 923 K, leading to a stronger cyclic hardening response at 923 K. The softening is governed by both friction stress softening as well as back stress softening at all three temperatures. The back stress evolution with LCF cycles is subsequently modelled using a newly developed back stress deconvolution method [1] based on plasticity theory, which provides insight into the origins of back stress and dislocation substructure evolutions.

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# Mechanical Response and Fatigue Life Assessment of Additively Manufactured IN939 Superalloy Using Miniature Specimen Geometry

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## ABSTRACT

Additively manufactured (AM) alloys are being considered for light weight and improved design applications in aerospace and gas turbine sectors owing to the ability to manufacture near net shape light weight parts with improved designs. IN939 is a nickel-based superalloy used in several hot section components in a gas turbine in the cast form. There are few reports on additively manufactured IN939, especially related to small scale mechanical response. In order to accelerate development of new or modified heat treatments in such rapidly solidified structures, it is imperative to study their performance under monotonic and cyclic loading at service temperatures at small length scales.

In this study, room temperature and high temperature (700 °C) uniaxial tensile behaviour of laser powder bed fusion IN939 and its heat-treated counterparts have been evaluated via micro scale tensile testing. The same specimen geometry is further used to study the fatigue behaviour at 0.95% yield strength in the as-printed, direct aged and solution and aged conditions. At the alloy development stage, several other groups have shown the benefits of using small-scale test geometries in additively manufactured systems to carry out site-specific measurements. The tensile strength was found to increase at the expense of ductility, with the direct aged sample showing the highest strength but lowest elongation to fracture, whereas the solution and aged IN939 showed intermediate strength and ductility between as-printed and direct aged sample.

Fatigue life was found to inversely correlate with yield strength and directly with ductility between the two heat treatments. The as-printed counterparts exhibited the largest, yet highly variable room temperature fatigue life. Such a rapidly cooled, non-equilibrium microstructure will not be stable at elevated temperatures and hence their high temperature fatigue behavior was not evaluated in the as-printed condition. The direct aged sample showed a relatively poor fatigue life, despite having the highest strength, whereas the solution and aged showed a comparably intermediate fatigue life. In both the ageing condition, the high temperature fatigue life was found to be significantly lower than their room temperature fatigue life. These are discussed in terms of fracture features and their correlation to the microstructural features of the alloy.

# Creep life Prediction of Zr-2.5Nb alloy using the modified Larson-Miller Parameteric Technique

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## ABSTRACT

Zr-2.5Nb alloys are extensively used as Pressure Tube (PT) materials in Indian Pressurized Heavy Water Reactors. During a Loss of coolant accident, the PTs may fail due to creep in the longitudinal or circumferential direction. Larson-Miller (LM) parametric technique is widely accepted in industries to predict the creep life of metallic materials. In the present work, creep life of Zr-2.5Nb alloys has been studied using the Larson-Miller parametric technique. The technique is generally expressed as  $P = T [C + \log(t)]$ , where, P is the Larson-Miller parameter, T is the temperature in Kelvin, t is time and C is considered as a constant. C is taken as a constant because it is assumed that the constant stress lines in the graph of  $\log(t)$  vs.  $1/T$  when extrapolated meet the Y-axis at  $1/T=0$ . The present work is aimed at investigating the constant C for Zr-2.5Nb alloy and predicting creep life of the alloy. Accelerated creep tests were done on Zr-2.5Nb alloy in the stress and temperature range of 2-5MPa and 600-850°C for prediction of creep life using Larson-Miller technique. The results reveal that C is not a constant and is rather a function of stress. Moreover, the creep life predicted using the modified L-M technique gave a more conservative value compared to the traditional L-M technique.

## Acknowledgments

The authors are grateful to BARC, India, for providing the Zr-2.5Nb tubes necessary for the study.

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# Creep Mechanism at Low Stress and High Temperature in Single Crystals

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Harper-Dorn (H-D) creep, a mechanism at high temperatures ( $> 0.9T_m$ ) and at low stresses (i.e., at  $\sigma/G < 10^{-5}$ ), is observed in high purity metal or ceramic single crystals and polycrystals with large grain sizes. This mechanism has been debated ever since its proposition in 1957. The main issue of the debate is the observation of creep exponents ranging from 1 to 3 by different studies (see Fig. 1). A few authors reported a constant dislocation density ( $\rho$ ) in the entire stress range in the H-D creep, while some reported it to follow the Taylor relation with very low initial dislocation densities. However, none of the theories, so far, unified these observations or explain the mechanism of H-D Creep.

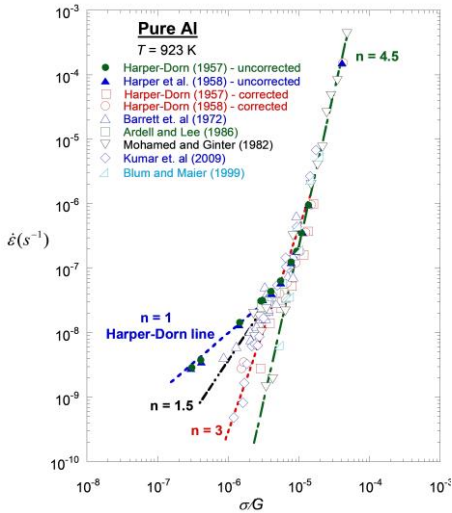


Fig. 1: Strain rate as a function of the normalized stress from several studies on Al. The dispute over the value of creep exponent in the low stress-high temperature regime is shown [1-6]. Given the large scatter in the steady-state strain rates at  $10^{-6}G$ , it is important to resolve the creep of materials in this regime.

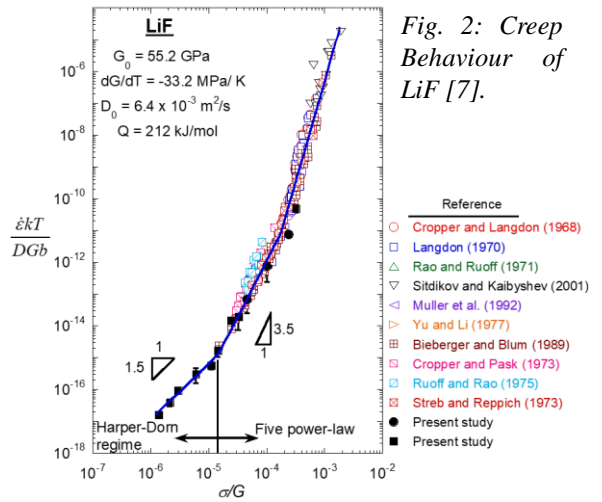


Fig. 2: Creep Behaviour of LiF [7].

This work examined the existing data on Al and subsequently tested high-purity LiF crystals in the H-D creep regime. LiF was selected as a model material because the dislocation quantification was well established, and sufficient studies existed at higher creep stresses. However, the creep of LiF has never been studied in the H-D regime. The LiF crystals were annealed up to 10,000 h at  $0.92 T_m$  to capture the reduction in  $\rho$ . The dislocation density showed reduction and then saturated at  $10^9 \text{ m}^{-2}$ . For the creep study, it was subjected to  $\sigma/G \sim 10^{-6}$  for 6 months until a steady state was attained. The  $\rho$  after creep was calculated and compared with the ‘frustration’ dislocation density after annealing. Long-term annealing was additionally conducted on NaCl and Al, which are simple cubic systems, to examine a similar phenomenon. It enabled us to comment on the validity of the observations in LiF can be extended to other material systems. The major findings from this work is that the creep exponent is 1.5 (see Fig. 2) and  $\rho$  was independent of the applied stress. This constant  $\rho$  observed in the H-D creep regime was similar to the ‘frustration’ dislocation density obtained in the LiF crystals after long-term annealing. The word ‘frustration’ here refers to the saturated  $\rho$  obtained after annealing. The  $\rho$  calculated from etch-pits was also verified from x-ray topography imaging of dislocations.

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# Elevated Temperature Deformation of Intermetallic Phases in Mg-Al-Ca Alloy at Small Length Scale

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## ABSTRACT

Intermetallic phases dispersed as network of micro- and nanoscale precipitates are desirable in engineering alloys due to their ability to improve strength and high temperature mechanical properties. Mg rich ternary Mg-Al-Ca alloy constitutes such a system having various types of intermetallics such as CaAl<sub>2</sub>, CaMg<sub>2</sub>, etc., dispersed as strengthening precipitates in Mg. These intermetallic phases are generally brittle at room temperature, but may undergo brittle to ductile transition (BDT) accompanied by loss in strength with temperature, or failure locally at the intermetallic-Mg interface. This study investigates the elevated temperature brittle to ductile transition behavior of the intermetallic phases using nano- and micromechanical testing. The nanoscale BDT and change in fracture behavior was studied using *in-situ* high temperature nanoindentation and micro-pillar splitting performed on single phase cast specimen of CaAl<sub>2</sub> intermetallic. A transition in deformation occurred ~450-500 °C with significant drop in nanoindentation hardness and increase in time-dependent deformation, increase in size of indents and absence of cracks around indent corners at high temperature. This was complemented by micro-pillar splitting experiments wherein cracking and brittle splitting of CaAl<sub>2</sub> pillars was observed up to 300 °C with increase in fracture toughness, followed by no pillar splitting from 400 °C due to transition into a more ductile behavior. Post-mortem transmission electron microscopy of the deformed region below nanoindents confirmed that this BDT of intermetallic was due to enhanced dislocation activity with temperature.

## Acknowledgments

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# Material Volume Reduction for Creep Testing Using Composite Cantilevers and Its Application for Residual Life Assessment

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## ABSTRACT

Digital image correlation (DIC) and finite element analysis (FEA) demonstrate that creep deformation in bending occurs primarily in  $\approx 30\%$  of the cantilever volume near the fixed end, especially when the creep stress exponent ranges from 5 to 7. As an alternative approach to minimize the material volume required for testing, the concept of fabricating composite cantilevers is proposed and validated in this study. A composite cantilever sample consists of an “active” creeping portion (e.g., T22 boiler steel) and an additively extended “passive” non-creeping portion (e.g., IN718). The volume reduction process involved varying the length of the “active” section,  $a$ , while keeping the total length of the cantilever,  $L$ , constant. The DIC measurements conducted at 600 °C to assess the creep behavior of T22 steel revealed that analytical expressions for monolithic cantilevers could aptly predict the constitutive steady-state creep laws from the composite cantilevers if measurements are made in a region at a critical distance away from the interface. FEA indicates that accurate stress estimation enables predicting monolithic creep behavior using composite samples with “ $a/L$ ” ratios of as small as 5%. Using the developed approach, the loss of creep resistance of T11 boiler steel that was in service for  $\sim 240,000$  h was ascertained in high throughput fashion using a composite cantilever having only 30 vol. % of the “active” material. Guidelines to minimize the volume fraction of the “active” portion in the composite cantilever and the implications of the observations for estimating the residual life of in-service high-temperature components are discussed.

## Acknowledgments

The authors thank Bharat Heavy Electricals Limited for providing the material to conduct this research work. The authors thank the IMPRINT program (including the Ministry of Human Resource Development and Ministry of Power, Government of India) of Government of India for the financial support. FH thanks the Prime Minister’s Research Fellowship (PMRF) for the contingency grant and financial support.

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# Assessment of mechanical properties of a Near $\alpha$ Titanium alloy disc with Dual microstructure

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## ABSTRACT

Titanium alloys are widely employed as compressor discs in aeroengines. The operating conditions in an aeroengine demands different and distinct, often contradicting mechanical property requirement from one location to another within a single compressor disc. The rim portion of the disc experiences high temperatures demanding better creep, fracture toughness and lower crack growth rate while the bore region is subjected to higher load fluctuations requiring higher strength and fatigue resistance. Even though the application demands location specific properties, currently these discs are subjected to a thermal process which provides uniform microstructure across the entire cross section resulting in achieving compromised optimum mechanical properties. Location specific properties can be achieved by tailoring the microstructure at different locations of an integral disc. To achieve different microstructure at different locations in an integral component, a selective heat treatment (SHT) process was devised and patented. Using this SHT process, the rim portion of the disc was heat treated to obtain a fully transformed  $\beta$  microstructure while maintaining an equiaxed globular  $\alpha$  + transformed  $\beta$  microstructure at the bore and thereby achieve dual microstructure in an integral disc of near  $\alpha$  titanium alloy. Producing such dual microstructure at the rim and bore results in a transition zone where the microstructure varies from one end to the other. Evaluating the performance of such dual microstructure discs with a transition zone is a challenging task as there are no established standards or procedures. To evaluate the mechanical properties (Tensile and Creep) of these dual microstructure discs, the proportion of microstructural constituents was varied along the gauge length of the sample in such a way to produce 0%, 25%, 50%, 75% and 100% fully transformed  $\beta$ . Suitable numerical approaches were used to gain insight on the tensile and creep behaviour of dual microstructure samples. Using the numerical procedure, the mechanical behaviour of the dual microstructure samples with varying microstructural constituents across the sample gauge length were predicted and compared with experimental results. The results obtained are presented and discussed here..

Key words: Titanium; Near alpha alloy; Dual Microstructure; Tensile; Creep.

## Acknowledgments

The authors thank Dr. R. Balamuralikrishnan, Outstanding scientist and Director, Defence Metallurgical Research Laboratory (DMRL) for his constant support and encouragement to publish this work. The funding provided by Defence Research and Development Organisation (DRDO) to carry out the work is acknowledged.

## Creep Behavior of Nitrogen Enhanced 316LN SS under Two-step Loading

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### ABSTRACT

Nitrogen enhanced 316LN SS with 0.12 wt.% N is being developed for primary side components of sodium cooled fast breeder reactor of India. The components in high temperature service experience both steady-state loading during operation as well as varying stresses during load ramps, shutdown/start-up, etc. It is therefore important to examine creep deformation under variable loading and in connection with this, the creep behavior of 316LN SS (solution heat treated at 1323 K/30-min) is examined under two-step loading (with upward ramp). Creep tests were conducted at 873 K, under varying stress in the range of 180-260 MPa, by loading the specimen at two stress levels repetitively with a definite time interval (1-3days), till failure. In all the tests, the accumulation of instantaneous strain and creep strain is particularly observed upon transition from low stress to high stress level, as shown in Fig.1a for the two-step loading between 180 and 200 MPa with an interval of 24 h. The rupture lives under two-step loading have been observed to be even lower than the lives corresponding to high stress level of the two-step loading, except in few conditions. An increase in time interval between the two stress levels accentuated creep strain accumulation and caused significant reduction in creep life. The rupture lives under two-step loading span up to ~7400 h. Creep life fraction that is estimated based on the linear damage summation varied mostly in the range of 0.28-1.0, wherein the lower fractions indicate the strong reduction in life. Further, long-term tests (up to ~10000 h) conducted with 7-28 days' time interval at two-step loading between 180-220 MPa also yielded life fractions within the above-mentioned range. In line with this, the increase in the observed creep rates (Fig.1b) are also significant which consequently led to exhaustion of creep ductility yielding low rupture-strain fractions in the range of 0.22-0.45, in reference to the rupture ductility at single stress levels. The above study though conducted at higher stress levels, the results emphasize that stress transients need to be taken into account in the design of components.

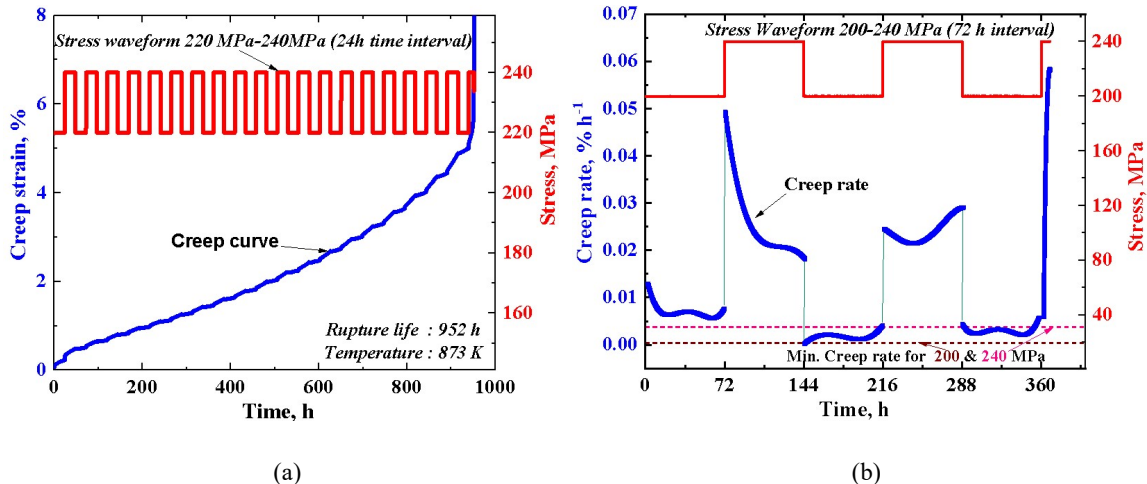


Fig.1: (a) Creep curve for two-step loading 220-240 MPa (24 h interval) and (b) Creep-rate variation for 200-240 MPa loading with 72 h time interval (minimum creep rate obtained from standard creep tests at 200 and 240 MPa are shown in dashed lines).

**Keywords:** 316LN stainless steel, Nitrogen content, Creep, Two-step loading, Life and strain fraction

# Comparison of creep and stress relaxation behaviour of additively manufactured AlSi10Mg

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## ABSTRACT

Al-Si-Mg series of alloys stand out as versatile Al-base alloys candidates for the emerging demands of automobile and electric vehicle industries due to their excellent strength-to-weight ratio. Notably, AlSi10Mg alloy is suitable for the laser powder bed fusion technique (LPBF) due to better weldability. Additively manufactured AlSi10Mg possess superior mechanical properties compared to their cast counterparts owing to fine and complex microstructure formation during LPBF processing. Nevertheless, understanding the deformation behaviour of AM-AlSi10Mg for prolonged exposure at high temperatures ( $\leq 250$  °C) is essential to ensure their durability in service. Comparison of time-dependent plastic deformation behaviour under constant strain and constant stress is required for the thermal activation analysis of AM AlSi10Mg.

The AM- AlSi10Mg specimens were prepared using the LPBF process, with the loading direction aligned parallel to the build direction and then subjected to different heat treatment conditions. Subsequently, uniaxial compression testing was done in the temperature range of 25–250 °C at strain rates ranging from  $10^{-4}$  to  $10^{-1}$  s<sup>-1</sup>. Further, high-temperature constant strain (stress relaxation) experiments under compression loading were also performed under the above testing conditions. Constant stress compression creep experiments were conducted in the temperature range of 0.4–0.8  $T_m$ , at the stress range of 100–200 MPa. Post-deformation microstructural analyses were carried out to correlate with the mechanical data and to elucidate the high-temperature deformation behaviour of AM-AlSi10Mg. Comparing the creep and stress relaxation parameters obtained from creep and stress relaxation experiments together with dominant rate-controlling deformation mechanisms, a constitutive high-temperature deformation model was developed.

Acknowledgements: The authors acknowledge the research funding via the Institute of Eminence (IoE) initiative of IIT Madras for establishing the IoE Research Centre in Additive Manufacturing.

# Additive manufacturing of orthorhombic titanium aluminide Ti<sub>2</sub>AlNb

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## ABSTRACT

Titanium alloys based on orthorhombic titanium aluminide Ti<sub>2</sub>AlNb are promising refractory materials for aircraft engine parts in the operating temperature up to 750 °C. Parts made of Ti<sub>2</sub>AlNb-based alloys by traditional technologies, such as casting and metal forming, have not yet found wide application due to the sensitivity of processability and mechanical properties in chemical composition and microstructure compared with commercial solid-solution-based titanium alloys. In the last three decades, metal additive manufacturing (MAM) has attracted the attention of scientists and engineers for the production of intermetallic alloys based on Ti<sub>2</sub>AlNb. This report summarizes the recent achievements in the production of O-phase-based Ti alloys using MAM, including the analysis of the feedstock materials, technological processes, machines, microstructure, phase composition and mechanical properties. Powder bed fusion (PBF) and direct energy deposition (DED) are the most widely employed MAM processes to produce O-phase alloys. MAM provides fully dense, fine-grained material with a superior combination of mechanical properties at room temperature.

More detailed characterization of the Ti<sub>2</sub>AlNb alloys manufactured using laser powder bed fusion (L-PBF) with preheating in the range 200–700 °C is made. The effect of process parameters on porosity, microstructure, phase and chemical compositions, texture, physical and mechanical properties were studied using the methods of hydrostatic weighing, scanning and transmission electron microscopy, X-ray diffraction and energy-dispersive X-ray spectroscopy, microindentation and compression testing. An increase of volumetric energy density during L-PBF in the range from 28 J/mm<sup>3</sup> to 139 J/mm<sup>3</sup> contributes to: 1) the increase in the relative density of the resulting O-alloy coupons from 97 to 99.9%, 2) the intensification of the solidification <001>-fiber texture of β/B2-solid solution, 3) decrease in Al, increase in Nb content and lesser enrichment with oxygen in as-built coupons. The separation the O-alloy samples from the platform and along the building direction results in the imbalance of residual stresses accompanied by the crack formation in samples fused on a substrate preheated at 200°C. The typical defects (pores, lack of fusion, chemical inhomogeneity) after SLM are discussed. The relationship between the resulting level of properties and a microstructure-texture in an O-alloy state formed of as-built SLM samples are considered. Preheating at 600 and 700 °C resulted in termination of cracks and precipitation of refined O-phase. A combination of physical and mechanical properties of the synthesized O-alloys coupons were determined.

Further research on MAM for the production of critical parts made of Ti<sub>2</sub>AlNb-based alloys can be focused on a detailed study of the influence of post-processing and chemical composition on the formation of the structure and mechanical properties, including cyclic loading, fracture toughness, and creep resistance.

## Acknowledgments

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## Insights of orientation-dependent spheroidization during secondary thermo-mechanically processing of Ti-6Al-4V alloys

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### ABSTRACT

Ti-6Al-4V is a two-phase ( $\alpha+\beta$ ) titanium alloy composed of 96% by volume of  $\alpha$  (hcp,  $P6_3/mmc$ ) and ~4% by volume of  $\beta$  (bcc,  $Im3m$ ) phases. The two phases withhold Burger orientation relationship (BOR) i.e.  $\{0001\}_\alpha \parallel \{110\}_\beta$  and  $\langle 11\bar{2}0 \rangle_\alpha \parallel \langle 111 \rangle_\beta$  [1]. A major microstructural evolution occurs in secondary TMP of Ti-6Al-4V alloy via spheroidization, in which lamellar  $\alpha$  phase transforms to equiaxed morphology [2]. Despite knowing that the spheroidization response of lamellar ( $\alpha+\beta$ ) colonies can significantly vary due to orientation of microstructural features [3], the exact nature and controlling factors of this heterogeneous spheroidization require further exploration. Also indispensable is a thorough knowledge of the site-specific  $\beta$ -phase penetration through the  $\alpha$  lamellae during spheroidization. The impact of modification in  $\alpha/\beta$  interface coherency during deformation and its role on the ensuing spheroidization response via the diffusion of solute atoms during TMP is not been thoroughly investigated

To this end, as-cast Ti-6Al-4V alloy having a fully-transformed lamellar microstructure is deformed by uniaxial hot compression up to a true strain of ~0.69 over an intermediate and high deformation temperature range i.e. at 800°C and 1000°C respectively at a constant true strain rate of  $1s^{-1}$ . The CD||ND  $\alpha$ -inverse pole figure (IPF) maps reveals diverse deformation response within similar  $\alpha$ -colonies that is supported by the Taylor and Schmid Factor maps plotted considering Basal  $\langle a \rangle$ :  $\{0002\} \langle 11\bar{2}0 \rangle$  and Prismatic  $\langle a \rangle$ :  $\{1\bar{1}00\} \langle 11\bar{2}0 \rangle$  slips. Moreover, the intra-lamellar substructure formation was investigated through point-to-point misorientation distribution maps. It demonstrated the formation of low and high angle boundaries (LABs and HABs) in adjacent lamellas confirming the heterogeneous  $\beta$  penetration. Furthermore, the analysis of BOR and the expected deviation as a function of colony orientation were carried to scrutinize the  $\beta$  diffusion at the  $\alpha/\beta$  interfaces. Finally, an understanding on orientation driven site-specific spheroidization was developed, which will be advantageous for manufacturing optimized structural components.

### Acknowledgments (if any)

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# Primary Creep Behaviour of Ti-6Al and Ti-G4 at Low Temperatures

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## Abstract

Titanium and its alloys show surprising dwell-induced debits in life in stress-controlled low cycle fatigue at ambient temperatures to about 120°C. These effects are attributed to unusual creep at stresses close to the yield stress over this temperature regime. Hence, Ti-6Al (1300 wppm of oxygen) and Ti-G4 (5000 wppm of oxygen) were chosen to study the role of Al and oxygen on the creep behaviour in single phase, polycrystalline Ti. Both alloys had a grain size of  $70 \pm 25 \mu\text{m}$  after heat treatment. Tensile tests, strain rate jump tests, and creep tests at 0.85, 0.9, and 0.95 of the yield strength were carried out at RT, 120°C, and 200°C. Slip trace analysis, along with high-resolution digital image correlation on the crept samples, revealed the activation of various slip systems and grain level strain localization at different temperatures.

## Microstructure and creep behaviour of SiC nanoparticles added Mg-Al-Ca-Mn alloy

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### ABSTRACT

The influence of SiC nanoparticles (SiC<sub>np</sub>) additions on the microstructure and creep behaviour of squeeze-cast Mg-5.0Al-2.0Ca-0.3Mn (AXM520) alloy is investigated. The concentrations of the SiC<sub>np</sub> are varied from 0.5 to 3.0 (wt.%), and the nanocomposites (NCs) are abbreviated as NC0.5SiC, NC1.0SiC, NC2.0SiC and NC3.0SiC. The microstructures of the AXM520 alloy and NCs consist of  $\alpha$ -Mg, a eutectic of  $\alpha$ -Mg and (Mg,Al)<sub>2</sub>Ca (C36), and an Al<sub>8</sub>Mn<sub>5</sub> phase. Additionally, the SiC phase is also present in the NCs. The continuous network of the C36 phase in the NCs is fragmented and becomes discontinuous with the increase in the fraction of the SiC<sub>np</sub>. All the NCs revealed improved creep performance compared to the AXM520 alloy. The creep rate of the NCs decreases with the increase in the SiC<sub>np</sub> content. The NC2.0SiC exhibited an improvement in creep resistance by 73.2% compared to the alloy. However, the creep resistance deteriorated since the amount of the nanoparticles was further increased in the NC3.0SiC, leading to agglomeration. The stress exponents range from 5.0 to 6.7, and activation energies range from 89.8 to 101.8 kJ/mol, implying that the creep in the materials is controlled by the climb of dislocation assisted by the pipe diffusion. The pile-ups of dislocations took place around the C36 phase and near the SiC<sub>np</sub>. The additional strengthening owing to the presence of the SiC<sub>np</sub> in the NCs was responsible for their improved creep performance compared to the AXM520 alloy.

Keywords: Magnesium alloy; Nanocomposite; Squeeze-casting; Microstructure; Creep; Dislocation climb

## Creep Properties of Alloy D9I Stainless Steel subjected to Prior Thermal Aging

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### ABSTRACT

Alloy D9I austenitic stainless steel (Titanium added 14Cr-15Ni SS) in 20% cold work (CW) condition is a candidate material for sodium cooled fast breeder reactor (SFR) core components viz., clad and hexcan which experience intense neutron flux and elevated temperature mechanical loading during service. Nucleation of fine secondary Ti(C,N) precipitates on dislocations contribute to an enhanced resistance to radiation induced damage and creep strength of the Alloy D9I. Transient conditions such as failure of coolant pumps, power ramps during operation, etc. result in inadvertent thermal exposure that can reach the clad hot spot temperature of 973 K. Though such exposures are only for a brief duration, substantial microstructural changes can occur during this short-term aging thereby influencing the creep performance. In this investigation, creep tests were carried out at 973 K and 150-250 MPa on 20% CW Alloy D9I SS aged at 973 K for 40 hours (CW + aged condition) to simulate the cumulative effect of thermal transients. Comparisons were drawn with respect to three other conditions of this alloy: 20 % prior cold worked (CW), as-received variant (ASR) and as-received plus aged (Aged).

The variation of rupture life of all the four variants is depicted in Fig. 1(a). The rupture lives for the ASR variant were marginally higher than that of the Aged variant at all stress levels. However, the rupture lives of these two variants were considerably lower when compared to CW and CW+Aged variants. The rupture lives were nearly the same for the CW and CW+Aged variants at all stress levels except 150 MPa. At this stress level, the rupture life of the CW+Aged variant was almost twice that of the CW variant. The distinct variation in creep properties between the CW and CW+Aged variants tested at 150 MPa appeared to be strongly dependent on the stability of the cold work induced dislocation substructure. While extensive recovery was evident in case of the CW variant (Fig. 1(b)), retention of the cold work induced dislocation tangles (Fig. 1(c)) was observed in CW+Aged variant. The stability of the dislocation substructure influenced the precipitation of secondary Ti(C,N). The relative prevalence of  $M_{23}C_6$  and secondary Ti(C,N) precipitates in turn dictated the resistance of the respective microstructure to creep cavitation. To elucidate this finding, an account of the precipitate size distribution and its effect on the cavitation propensity and fracture behaviour is presented.

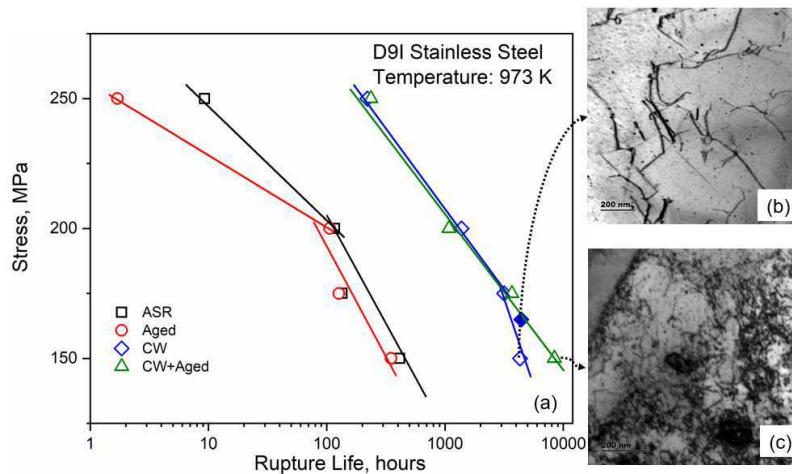


Fig. 1: a) Variation of rupture life with applied stress for the four variants at 973 K, b) TEM micrograph showing recovered substructure for CW variant tested at 150 MPa and c) TEM micrograph showing dislocation tangles for CW+aged variant tested at 150 MPa.

## **Type IV Cracking in Grade 91 Steel Weld Joint Under Creep Exposure**

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### **ABSTRACT**

Grade 91 steel is extensively being used in the nuclear and fossil fuel fired power plants. The steel derives its high temperature creep strength from the tempered martensitic lath structure, transformation induced dislocation density,  $M_{23}C_6$  and MX precipitates, boundaries and solid solution strengthening. In the present study, activated-tungsten inert gas welding (A-TIG) technique is employed to fabricate the grade 91 steel weld joints. The weld thermal cycle leads to variation in microstructure and mechanical properties across the weld joint. The soft zone formation occurs in the outer edge of heat affected zone (inter-critical region). The weld joints exposed at 923 K and lower stress conditions led to premature failure of the joints than the base metal. The difference in creep rupture strength between the weld joint and base metal has increased with decrease in applied stress. Further, the creep rupture strength of the grade 91 steel weld joints was enhanced through (i) thermo-mechanical treatment which enhanced the MX precipitation and (ii) addition of boron in grade 91 steel which reduced the coarsening of  $M_{23}C_6$  precipitates. The microstructure evolution during welding, post weld heat treatment and creep exposure have been examined in order to elucidate the creep behaviour of the weld joints.

# Effect of Re-Solutionizing Treatment on Creep Rupture Behavior of Nuclear Grade Nitrogen Enhanced 316LN Stainless Steel

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## ABSTRACT

316LN stainless steel (SS) with ~0.07 wt.% N is being used for fabricating sodium-cooled fast reactor (SFR) components such as reactor vessel, intermediate heat exchanger, primary sodium pump, etc. which experience temperatures below 873 K. Currently, nitrogen enhanced variant with 0.12% N is being developed to improve mechanical properties of 316LN SS. The material is usually given solution treatment at 1323-1423 K on the final wrought product (i.e., plate product in this study), as per the RCC-MR code. However, it must be mentioned that, at times it is required to give re-solutionizing treatment to material due to intermediate fabrication works involved in manufacturing a component. In this context, it is proposed to examine the effect of re-solutionizing treatment on creep properties of 316LN SS with 0.12% N, for which re-solutionizing treatment was carried out on specimen blanks at 1373 K for one hour. Re-solutionizing enhanced the grain size of the as-received material from ~60  $\mu\text{m}$  to ~120  $\mu\text{m}$  which is below that specified (viz., 180  $\mu\text{m}$ ) in RCC-MR code. Creep tests were performed at 873 K in the stress range of 180-260 MPa in both as-received and re-solutionized conditions. Creep curves revealed an early onset of prolonged tertiary regime in re-solutionized variant with relatively shorter primary and secondary regimes compared to the as-received 316LN SS (Fig.1). As a result, the re-solutionized variant exhibited lower creep life and rupture ductility than as-received 316LN SS, with about 50% reduction in life at the lowest stress level. It may however be mentioned that, at all the stress levels, the minimum creep rate was comparable in both the variants indicating similar governing deformation mechanism. In spite of this, the deformation and damage accumulated in secondary and tertiary creep regimes is observed to induce a predominantly low ductile intergranular fracture in re-solutionized variant at all stress levels (a typical fractograph shown in Fig.1). In contrast, the fracture mode in as-received variant is found to depend on the stress level, with intergranular fractures at low (< 220 MPa) and high stress levels (> 230 MPa) and transgranular fractures at 220-230 MPa. This is observed to be in par with the anomalous trend in rupture ductility for the as-received variant.

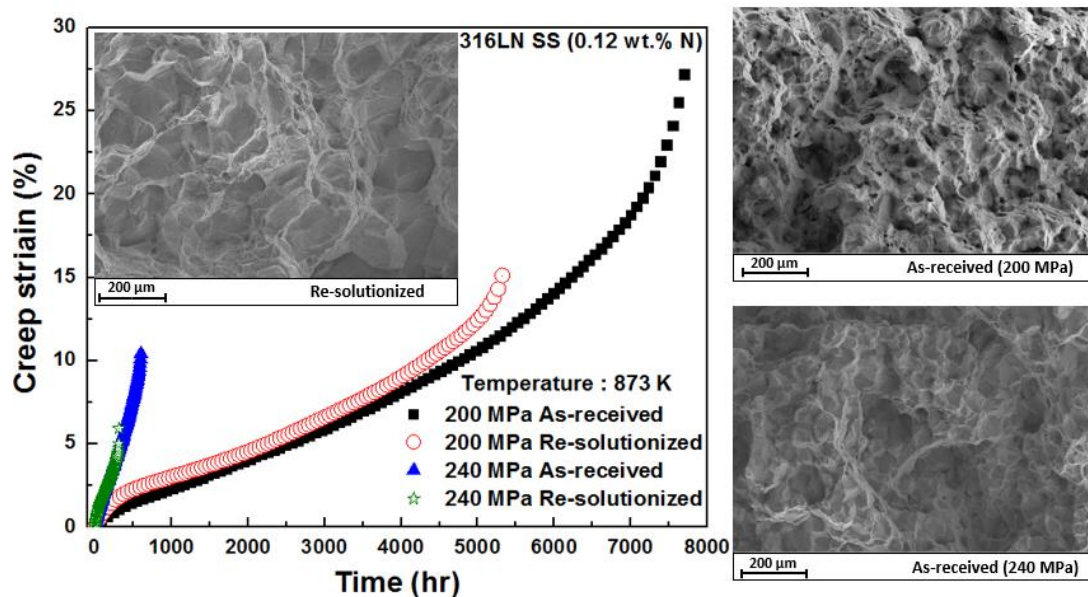


Fig.1: Comparison of creep deformation and fracture characteristics of nitrogen enhanced 316LN SS in as-received and re-solutionized conditions.

## Elevated temperature mechanical properties of cold-formed steel

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### ABSTRACT

Mechanical properties play a significant role in the fire safety design of cold-formed steel (CFS) structures due to the rapid reduction in mechanical properties such as yield strength (0.2% proof stress) and elastic modulus under fire conditions and related decline in the ultimate strength resulting in early failure. Hence, there is a need to fully understand the deterioration characteristics of yield strength and elastic modulus of CFS at elevated temperatures. Limited studies have been carried out to investigate the deterioration of mechanical properties of CFS at elevated temperatures and values of these properties at different temperatures are not well reported [Rokilan and Mahendran (2020), Li and Young (2017)]. The most commonly used method to assess the mechanical properties of CFS is to perform tensile coupon tests based on either the steady state or the transient state test method. Although the transient state test method is considered to be more realistic in simulating the behaviour of a real fire including the creep effect, the steady state test method is commonly used as it is easier to conduct than the transient state test method and provides the stress-strain curves directly [Kankanamge and Mahendran (2011)]. Hence, in this research the steady state test method was used. Therefore, tensile coupon tests were conducted at different temperatures ranging approximately from 21°C to 850°C for obtaining the mechanical properties of CFS structural material. In steady state tests, specimens were heated up to target temperatures that were held constant, and then loaded until fracture. Tensile coupon specimens extracted from CFS lipped channels were loaded under different nominal temperatures mainly, 21°C, 100°C, 200°C, 300°C, 400°C, 500°C, 550°C, 600°C, 650°C, 700°C, 750°C, 800°C and 850°C. The heating rate was set as 50 °C/min for all the steady state tests. After the target temperature was reached, an additional 20 min of heating, as required by the ASTM E 21 (2009), was maintained to ensure that the specimen obtained uniform temperature distribution. A 10 ton MTS Universal Testing Machine (UTM) was used to carry out the tensile coupon tests with a high temperature furnace which can heat specimens up to 1000 °C. The shapes and dimensions of coupon specimens were prepared in accordance with the ASTM E 8 (2016) for tensile testing of metallic materials. This study included CFS of grades G550. Yield strength (0.2% proof stress), elastic modulus, ultimate strength and ultimate strain of CFS at elevated temperatures are investigated in detail.

### Acknowledgments

The research was funded by CSIR-CBRI.

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# Finite Element Modeling for better understanding Martensite Decomposition within an Additively Manufactured Ti64 during Small Punch Creep Tests

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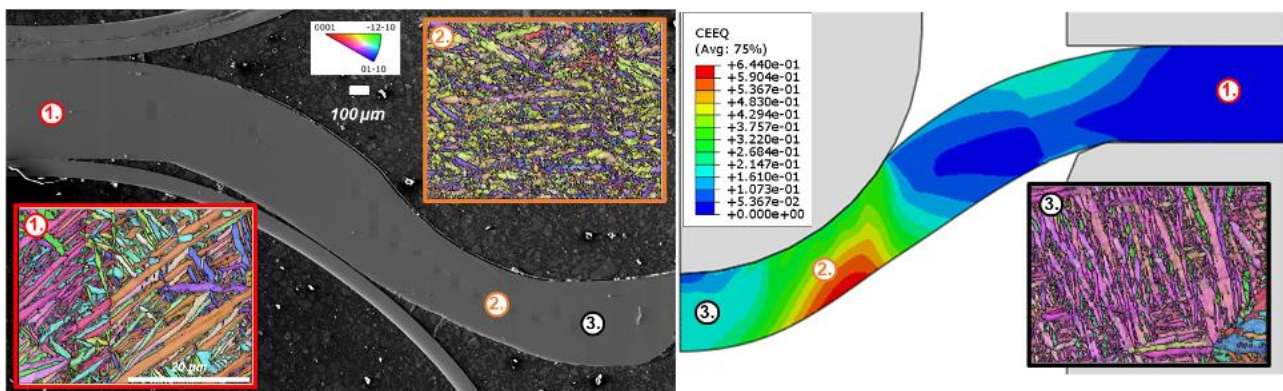
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## ABSTRACT

Miniaturization of mechanical tests have gained high interest over the last few decades. In this frame, Small Punch Testing (SPT) methods were developed in the early 1980's. The technique uses disc-shaped specimens of 10 mm (or 8 mm) diameter and 0.5 mm thick. The specimen is mechanically loaded via a punch either through constant displacement rate control mode (SPT) or load control mode (SPCT). Plastic and creep properties are then deduced [1].

Titanium alloys are widely used in the aerospace industry thanks to their excellent mechanical properties, good corrosion resistance and low density. The Ti64 (Ti-6Al-4V) is the most used titanium alloy, classically exhibiting an  $\alpha+\beta$  microstructure. Recent progress of Additive Manufacturing (AM) of Ti64 alloys relies on the use of the Laser-Powder Bed Fusion (L-PBF) technique consisting of selectively melting a powdered material thanks to a laser beam. Such manufacturing processes lead to high cooling rates and thus non-equilibrium microstructure and may generate high residual stresses. The L-PBF Ti64 exhibits an  $\alpha'$  martensitic microstructure in large prior  $\beta$  grains elongated along the manufacturing direction. The martensitic microstructure exhibits higher strength than the cast and wrought  $\alpha+\beta$  structure. However, the creep behavior of the AM Ti64 is less known and the stability of the martensite during creep tests also deserves to be studied. It is the goal of the present study to examine those properties and also to take advantage of the variety of local mechanical state within SPCT samples as a way of screening its effects on the martensite decomposition [2].

In the present study, we examine the creep behavior of L-PBF Ti64 through SPCT for temperatures ranging from 400°C to 500°C and stresses ranging from 250 MPa to 500 MPa. Comparisons with uniaxial creep data are carried out to validate the use of SPCT for Titanium alloys. Then, creep parameters such as Norton exponent are deduced, and the creep mechanisms are discussed. Interrupted SPCT experiments are performed in the steady creep strain rate state. Great attention is paid to the evolution of the microstructure and especially the martensite decomposition. The local mechanical state obtained thanks to Finite Element Modeling (FEM) provides a better understanding of the microstructure evolution [2,3].



**Figure 1:** Investigation of Martensite decomposition occurring at 450 °C - 400 MPa within SPCT sample via EBSD and its link to the local equivalent creep strain obtained by FEM

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## **Creep Behavior of Stainless Steel 316L Manufactured by Laser Powder Bed Fusion**

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Metal additive manufacturing (Metal AM) continues to gain momentum. Many companies explore the manufacturing of innovative products, including components for safety-critical applications. Despite the intensive research of recent years, a fundamental understanding of the process-structure-property relationships remains challenging due to, i.a., the inherently complex and highly hierarchical microstructures arising from the wide range of build process parameter variability. This contribution presents the results of an experimental study on the creep behavior of an austenitic 316L stainless steel produced by laser powder bed fusion (PBF-LB/M/316L) with an emphasis on understanding the effects of microstructure on creep mechanisms. Hot tensile tests and constant force creep tests at 600 °C and 650 °C, X-ray computed tomography, as well as optical and electron microscopy were performed. The produced PBF-LB/M/316L exhibits a low void population (< 0.01 %) resulting from the manufacturing parameters used and which allowed us to understand the effects of other microstructural aspects on creep behavior, e.g., grain morphology and dislocation substructure. A hot-rolled variant of 316L was also tested as a reference. The produced PBF-LB/M/316L possesses shorter primary and secondary creep stages and times to rupture and smaller creep stress exponents than the hot-rolled variant. Overall, the creep damage is more extensive in the PBF-LB/M/316L and is characterized as predominantly intergranular. It is considered that the damage behavior is mainly impacted by the formation of precipitates at the grain boundaries combined with their unfavorable orientation. The dislocation substructure and local elemental segregation appear to have a decisive impact on the overall creep behavior.

### **Acknowledgments**

This work was supported by the BAM focus area materials project AGIL “Microstructure development in additively manufactured metallic components: from powder to mechanical failure”.



## Microstructural evolution and mechanical behavior of as-built IN939 at room and elevated temperature fabricated by PBF - LB

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### ABSTRACT

Ni-based superalloy, Inconel 939 (commonly referred as IN939), has been widely applied in manufacture many aircraft engine components exposed to higher temperatures, such as fuel nozzles, vanes, and gas turbine blades. These components contain complicated geometries that cannot be produced by using traditional manufacturing methods (castings and forgings). The alloy is also considered as difficult-to-machine due to their high mechanical properties, which are also maintained upto 850 °C. Hence, new manufacturing techniques, such as additive manufacturing (AM), come into the focus of research in the academia and industries. However, the complicated thermal schedule and processing parameters of this technique influence the microstructure of additively manufactured superalloys significantly, with respect to the conventional one. Thus, further research is crucial for understanding the microstructure-processing relationship and its impact on the resulting mechanical properties, particularly at the service temperature, prior to the successful utilization of the AM-superalloy. To advance the knowledge-base, the present study aims to systematically investigate the evolution in microstructure, residual stress and mechanical behavior at the different regions of IN939 superalloy processed by powder bed fusion – laser beam (PBF – LB). Microstructures of the PBF-LB IN939 superalloy are noted to consist of  $\gamma$ ,  $\gamma'$ , and  $\eta$  phases at top and bottom parts. Interestingly, Kernel average misorientation (KAM) map shows higher plastic strain at the bottom compared to top parts of the alloy. This is related to the occurrence of higher residual stress at the bottom parts of the additively manufactured part, in comparison to the top. Hardness tests (macro-, micro-, and nano-) are also conducted at top and bottom parts of the samples. It is found that bottom parts of the sample have higher hardness values as compared to the top parts of the alloy. Tensile tests are conducted at various temperatures (room temperature, 750 °C, and 850 °C) on the PBF-LB IN 939 alloy. While digital image correlation assisted in understanding the strain non-uniformity generated during tensile deformation at room temperature, properties at elevated temperature are evaluated to assess the performance of the alloy at service condition. Increase in yield strength with temperature indicates that the alloy exhibits yield strength anomaly (YSA). Interestingly, YSA behavior is noted to attain a peak at temperatures where these materials are most commonly used, making this study significant. Hot compression behavior and deformation mechanisms are also investigated using compression tests at these temperatures. The results show that the dominant deformation mechanisms are closely related to the temperature.

# **Creep behaviour of Hastelloy-X processed by selective laser melting**

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## **Abstract**

In this work, a detailed investigation of creep behaviour was carried out for Hastelloy X processed by selective laser melting (SLM). Tensile creep tests were performed in two directions, namely the vertical and horizontal directions. Tests were performed at 800°C at four different initial stresses (150, 125, 100, 75 MPa) to determine the stress exponent ( $n$ ) values. The resultant  $n$  values indicate the dislocation climb as the underlying mechanism of creep deformation in both direction specimens. Creep properties and stress rupture properties were found to be better for the vertical-direction samples than those for horizontal-direction samples. Epitaxial grain morphology of the vertical-direction samples was identified as the primary cause of better creep performance due to the slow cavitation and subsequent crack propagation process attributed to the less availability of the transverse grain boundaries as opposed to the more available transverse grain boundaries in the case of checker-board morphology of horizontal-build samples.

Keywords: Creep; Selective laser melting; Hastelloy X; Grain morphology

# High Temperature Deformation Studies using 3D, Digital Image Correlation.

S.Raviprakash<sup>1</sup> and Abhishek.G<sup>1</sup>

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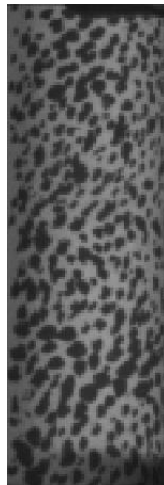
## ABSTRACT

In this paper, the authors have successfully carried out experiments at High Temperatures for the following tests using 3D Digital Image Correlation Technique:-

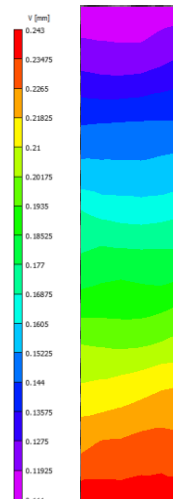
- 1) High Temperature Low Cycle Fatigue (LCF) at 600 °C, 800 °C and 900 °C.
- 2) In-Phase Thermo Mechanical Fatigue (TMF) Cycle:- 200 °C to 600 °C.
- 3) High Temperature Creep Studies at 600 °C.



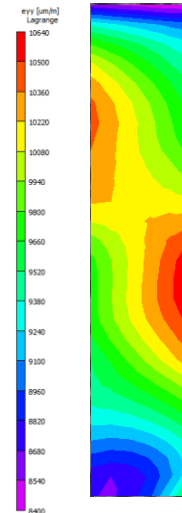
Speckle image  
at Room Temp.



Deformed image  
at 600 °C.



“V” Displacement  
Loading Direction (Y) at 600 °C



$\epsilon_{yy}$  at 600 °C

## References :-

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S. Abotula, P. Naik Parrikar, Anil Rajesh, and A. Shukla -  
*Department of Mechanical, Industrial & Systems Engineering, University of Rhode Island.*

## Mechanical Properties of Grain Boundaries in Forsterite Bicrystals

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Understanding the rheological properties of olivine, the most abundant mineral in Earth's mantle, is crucial for unravelling the dynamics of upper-mantle convection. While extensive research has focused on olivine rheology, little attention has been given to the specific behavior of olivine grain boundaries, which play a key role in mantle deformation [1,2]. Current models describing grain-boundary deformation [3] are primarily phenomenological, making it challenging to extrapolate laboratory findings to natural conditions.

To address this gap, we conducted a series of deformation experiments on olivine (forsterite) bicrystals, where a single grain boundary was oriented for direct shear at atmospheric pressure and 1300 or 1400°C. Experiments were conducted in a uniaxial creep apparatus with resolved shear stress ranging from 1 to 30 MPa. The stress-strain relationship revealed a linear dependence (creep exponent,  $n$ , of  $\sim 1$ ) on applied stress, indicating that dislocation creep, commonly observed in single crystals and coarse-grained polycrystalline aggregates, was not the dominant deformation mechanism under these conditions. Direct observations of grain-boundary sliding in a geologic material were made for the first time (Fig. 1), however, the linear viscous behaviour measured was likely controlled by a documented chemical reaction between the olivine and the alumina pistons. EBSD mapping confirmed that both olivine grains in the bicrystal remained internally undeformed. Our results demonstrated that the bicrystal boundary is weaker than the forsterite grain interior, but the viscosity was dominated by the olivine-alumina reaction. The direct evidence of grain-boundary sliding was observed through fiducial scratch marker displacement in SEM (see Fig. 1). However, the strain geometry remains complex, necessitating further experiments to determine the overall strain distribution in the samples.

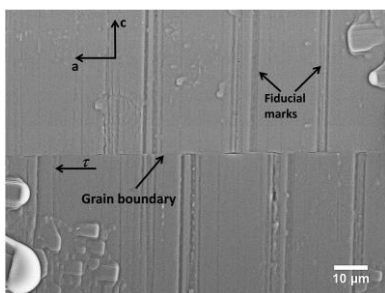


Fig. 1: Microstructure showing the displacement of fiducial marks across the grain boundary after creep conducted at 1400°C. The fiducial marks are indicated. The grain boundary is the horizontal line in the centre as indicated by an arrow. The crystal structure of forsterite is orthorhombic with different  $a$ ,  $b$  and  $c$  axes. Here  $a$  and  $c$ -axis are indicated. The direction of the shear was along  $a$ -direction indicated with  $\tau$ .

In summary, our research provides unprecedented direct observations of grain-boundary sliding in olivine [4], shedding light on the intricate mechanisms governing mantle deformation. The observed differences in creep behavior between bicrystals and traditional forsterite samples underscore the importance of considering grain-boundary effects in comprehensive rheological models of the mantle.

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# Low-cycle fatigue response of CrFeNi multi-principal element alloy

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## Abstract

In the present work, the low-cycle fatigue (LCF) behavior of single-phase, face-centered cubic (FCC) equiatomic CrFeNi Multi-principal element alloy (MPEAs) [1,2] is investigated at room temperature. The fully reversed strain-controlled fatigue tests were conducted in air under different strain amplitudes (0.3%, 0.5%, and 0.7%). The measured cyclic stress response of CrFeNi reveals two distinct stages: a rapid increase (i.e., cyclic hardening stage), followed by a relatively gradual decrease (i.e., cyclic softening stage) until failure. The rate and amount of cyclic hardening increased with the increase in strain amplitude. Upon comparing with similar grain sized CoCrFeMnNi [3] and 316L steel [4], CrFeNi exhibits a similar tensile peak stress response. Differently, CrFeNi shows highest tensile peak stress at first cycle followed by CoCrFeMnNi and 316L steel. Furthermore, the cyclic hardening trend is as following: CrFeNi < 316L steel < CoCrFeMnNi. Electron-microscopy investigations reveal no noticeable change in grain size, texture, and annealing twin density after specimen failure. TEM investigations are under progress to reveal dislocation structure evolution.

Furthermore, the evolution of effective stress ( $\sigma_e$ ) and back stress ( $\sigma_b$ ) of CrFeNi are analysed by partitioning its cyclic hysteresis loops via Cottrell's method (see Figure 1). Here the effective stress signifies the stress required to overcome obstacles such as lattice friction, solutes, and individual dislocations, whereas the back stress reflects long-range resistance stress from obstacles such as sub-grain structures, grain boundaries and strain incompatibilities. It was found that the cyclic stress response of CrFeNi predominantly originates from the back stress evolution. Back stress also increases significantly with increasing strain amplitude. However, the change of effective stress is relatively insignificant with altering cycle number and strain amplitude. Comparison with 316L austenitic steel and CoCrFeMnNi brings out the peculiarity of CrFeNi cyclic stress response.

**Keywords:** Low-cycle fatigue; multi-principal element alloys (MPEAs); Effective stress; Back stress

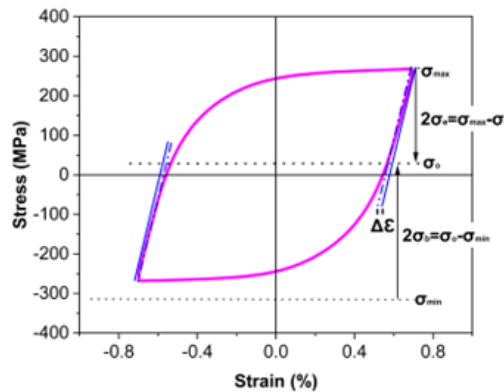


Figure 1: Schematic figure showing Cottrell's method of calculating effective stress ( $\sigma_e$ ) and back stress ( $\sigma_b$ ). Here,  $\sigma_0$  is measured as the offset yield strength corresponding to an offset plastic strain ( $\Delta\epsilon$ ) of 0.1%.

# On the influence of stacking fault energy on the creep behavior of Ni based solid solution alloys

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## ABSTRACT

A Power law relation ( $\dot{\epsilon} \propto \gamma^q$ ) between the creep rate ( $\dot{\epsilon}$ ) and stacking fault energy ( $\gamma$ ) of a material in a dislocation climb controlled creep region has been well accepted since 1965 and the noted stacking fault energy exponent ( $q$ ) is  $\sim 3$  [1]. However, certain solid solution alloys were deviated from this observation. The current study was aimed to explore the relationship between creep rate and stacking fault energy by considering f.c.c. based single phase solid solution alloys such as binary Ni- (x) Co alloys, where x=10,33, and 66 at% and a concentrated solid solution alloy NiCoCrFe. All Ni-Co binary solid solution alloys and NiCoCrFe alloy followed the dislocation climb controlled creep mechanism at 1015 K and 990 K, respectively. Creep deformation did not result in notable second phase formation in these alloys. The observed stacking fault energy exponent of binary Ni-Co alloys is  $q \sim 2$ . Interestingly, despite the similar range of experimentally evaluated stacking fault energies of Ni-60Co (10 – 36 mJ m<sup>-2</sup>) and NiCoCrFe (11 – 26 mJ m<sup>-2</sup>) alloys, the creep rates of the former alloy were observed to be significantly higher. A distinct difference in their crept microstructures also can be noted, where Ni-60Co alloy showed cell structure and the planar band structure was prevalent in NiCoCrFe alloy. The possible reasons behind the difference in creep rates and crept microstructures of Ni-60Co and NiCoCrFe alloys were discussed from the perspective of stacking fault energy at high temperature and internal stresses.

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# Effect of Stacking Fault Energy on Creep Behavior of FCC Medium Entropy Alloys

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## ABSTRACT

In this study, we focus on face-centered cubic (fcc) single-phase alloys within the Ni-Co-Cr-Fe system to investigate the effect of stacking fault energy (SFE) on creep deformation. To achieve alloys with a single fcc crystal structure, we have employed a thermodynamic-based CALPHAD approach to determine the compositions. These compositions are further optimized for their Ni, Co, and Cr ratios, aiming to modify their SFE. The SFEs of these alloys are calculated using first-principles calculations of the generalized stacking fault energy (GSFE), inducing inelastic shear on the (111) plane in the  $[11\bar{2}]$  slip direction, incorporating all aspects of density functional theory (DFT). Additionally, observations of partial dislocations are conducted through transmission electron microscopy (TEM) studies to experimentally evaluate the SFE. After establishing the microstructures through thermomechanical processing, the alloys are subjected to constant stress creep tests at temperatures ranging from 0.5 to 0.7  $T_m$ . The collected creep data is then analyzed alongside microstructural characterization to comprehend the influence of SFE on creep deformation behavior.

Keywords: Complex Concentrated Alloys, Stacking Fault Energy, Density Functional Theory

# Insight into the determination of threshold stress using constant-load uniaxial creep test in a Zr-2.5Nb alloy

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## ABSTRACT

In this study, the thermal and threshold creep behaviour of cold-worked and stress-relieved (CWSR) Zr-2.5Nb material is investigated at 350°C, 400°C and 450°C. Using flat creep specimens with their axis in the axial direction of the pressure tube, constant-load uniaxial creep tests were carried out in accordance with ASTM 139-20. The specimens were tested at three normalised stress levels, which corresponded to 0.7, 0.8 and 0.9 times the yield strength. The stress exponents and activation energy were determined in the temperature range of 350°C-450°C. Li and Langdon [1] proposed a simple method to estimate the threshold stress, defined as the limiting stress corresponding to a strain rate of  $10^{-10} \text{ s}^{-1}$ . The proposed method recommends extrapolation of the minimum creep rate vs. stress plot to the strain rate of  $10^{-10} \text{ s}^{-1}$ . The x-intercept on the  $\sigma$ -axis determines the threshold stress. Towards evaluating the threshold creep behavior, stress increment creep tests were conducted at the estimated threshold stress as the initial stress level. It was found that the extrapolation method overestimated the threshold stress. The minimum creep rates, obtained at the estimated threshold values, were found to be atleast one order higher than the proposed threshold creep rate value of  $10^{-10} \text{ s}^{-1}$ . The discrepancy in the obtained minimum creep rates at the threshold stress value could be attributed to the invalidity of the extrapolation method from creep data of the limited stress range to determine the threshold stress. Nevertheless, for a given material, the effect of cold work or annealed microstructure on the threshold stress needs to be investigated.

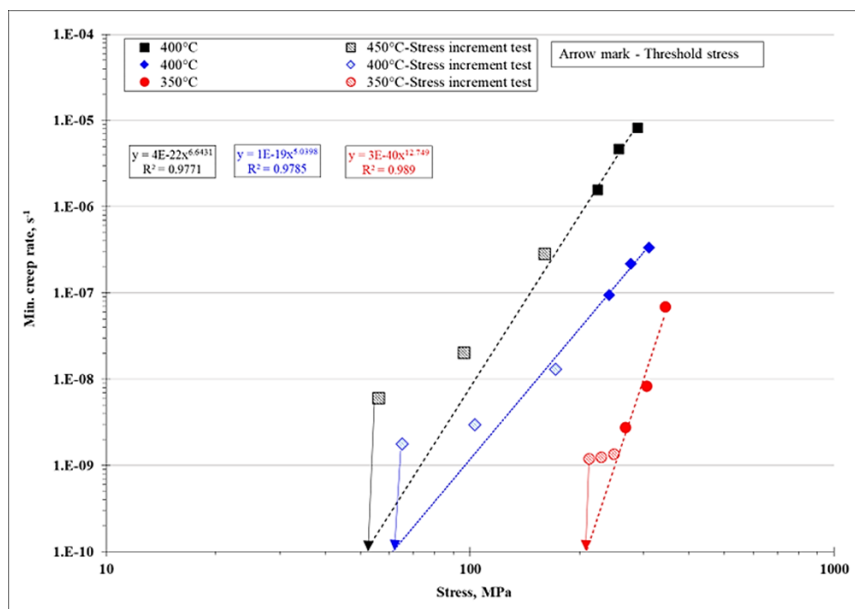


Fig. 1: Estimation of threshold stress

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# A multiscale material modelling methodology to consider effect of irradiation damage on mechanical properties of zirconium alloys

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## ABSTRACT

Zirconium alloys are widely used as structural materials in the nuclear industry due to their low neutron absorption cross section, excellent mechanical properties and corrosion resistance [1],[2]. During the operation of a nuclear reactor, the structural components like fuel cladding, which are made of zircaloy-4, undergo neutron irradiation which causes degradation of their mechanical properties due to irradiation-induced damage. The notable features of degradation are the loss of ductility, increase in yield strength and decrease in fracture toughness. The degradation can be mainly attributed to the formation of microstructural defects like irradiation-induced cascades, dislocation loops, voids etc., produced by neutron irradiation. In this work, molecular dynamics and kinetic Monte-Carlo simulations have been carried out to evaluate defect density and defect size evolution in irradiated Zirconium alloys (Fig. 1 and Fig. 2). Simulations have been carried out for room temperature and operating temperature of the reactor and for different dpa levels of irradiation damage. The results of kinetic Monte-Carlo simulations have been coupled to crystal plasticity based models in order to evaluate the plastic flow response and corresponding stress-strain data at different irradiation levels. The radiation damage in terms of defect density is used as input to the simulations. The obtained stress strain data is then modelled with Ramberg-Osgood expression in order to parametrize the model in terms of material parameters. The parameters of Ramberg-Osgood are evaluated as a function of dpa levels of irradiation. Coupling Ramberg-Osgood parameters with Gurson-Trevergaard-Needleman damage model, the fracture toughness of values of Zirconium alloys at different irradiation levels have been predicted. Some of the results of multiscale simulations have also been compared with data from literature to verify the methodology (Fig. 3). This approach shall be helpful in predicting the properties of irradiated materials which are crucial for design and safety analysis of nuclear reactors.

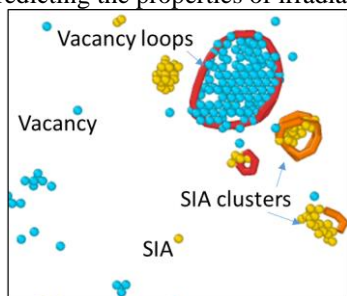


Fig 1. Primary radiation damage in Zr from MD simulations

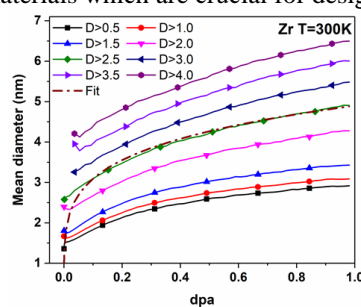


Fig. 2 Defect size evolution as function of dose (dpa) from KMC simulations

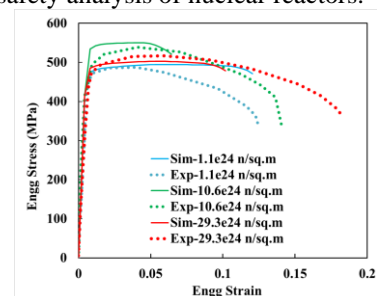


Fig. 3 Crystal plasticity finite element simulations of the stress strain data of Zr-4 at different neutron fluences (Experiments taken from Cockeram et.al [3])

**Key Words:** Irradiation Damage; Multiscale Material Modelling; Crystal Plasticity Simulation; Mechanical Property.

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## Monotonic Fracture Studies on Dissimilar Metal Pipe Weld Joints

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### ABSTRACT

Dissimilar metal (DM) weld joints are used for joining pressure vessel nozzles made of low alloy steel with piping made of austenitic steel in the typical light water reactors. The highly heterogeneous nature of these joints in terms of their microstructure, mechanical, thermal, and fracture properties, has limited the ability of current fracture assessment methods in accurate analysis. This paper presents details of monotonic fracture studies carried out on four DM pipe weld joints made of SA 508 Gr. 3 Cl-1 low alloy steel and SA 312 Type 304LN austenitic stainless steel. The pipe weld joints were of 324 mm outer diameter and 24.5 mm thickness having through-thickness crack in the circumferential direction of the pipe. The weld joints had a buttering layer of Nickel-rich Inconel alloy (ENiCrFe-3) material on the low alloy steel portion. The initial part-through notch was located in the base metal of low alloy steel portion in two cases (DW-F-3 and DW-F-5) and in the heat affected zone of low alloy steel portion in the remaining two cases (DW-HZF-4 and DW-HZF-7). Before carrying out fracture tests, fatigue crack growth (FCG) tests were carried out on the DM pipe weld joints under constant amplitude sinusoidal cyclic loading till crack growth became through-thickness and further grew till 1/8<sup>th</sup> of the circumference of the pipe weld joint. FCG tests on two pipe weld specimens (DW-F-3 and DW-HZF-4) were carried out in air environment and on the remaining two specimens (DW-F-5 and DW-HZF-7) under reactor water environment. Subsequently, fracture tests were carried out on the pipe weld specimens with through-thickness crack under static and four point bend monotonic loading; intermittent unloading was carried out at regular intervals to evaluate the stiffness of the pipe weld specimen with respect to crack growth. During the fracture tests, load, load-line displacement (LLD), crack mouth opening displacement (CMOD) and crack length in circumferential direction were monitored. The surface crack length was measured using image processing technique and crack mouth opening displacement was measured using clip gauges. The maximum measured load (MML) for the pipe weld specimens varied from 1008.16 kN to 1100.90 kN and the corresponding LLD varied from 182.35 mm to 194.47 mm. The MML was lowest for the specimen DW-F-3. Figure 1 shows comparison of load vs. crack length and Fig. 2 shows comparison of load vs. CMOD. Crack length at the end of fracture tests varied from 138.50 mm to 171.23 mm. The CMOD observed was highest for the specimen DW-HZF-7.

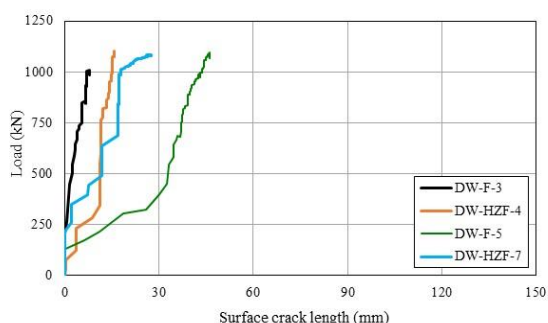


Fig. 1 Load vs. crack length

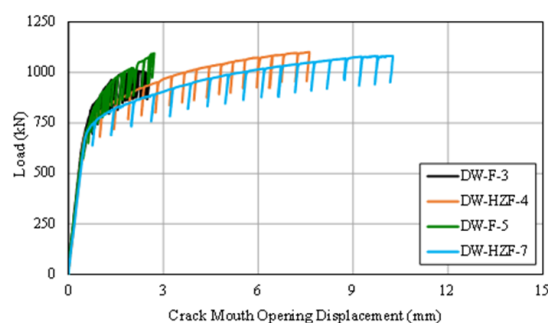


Fig. 2 Load vs. CMOD

### Acknowledgements

The abstract is based on the work carried out as part of Sponsored Research Project - SSP 22541 titled "Fatigue Crack Growth Rate (FCGR) Studies on Bi-metallic Pipe Welds". The paper has been assigned the registration number CSIR-SERC-1089/2023.

# Significance of creep behavior in advanced-grade clad materials for high-temperature applications: A critical review

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## ABSTRACT

In recent years, mechanical component failures, particularly in turbine blades, valves, pressure vessels, tubes, discs, and thermal shields, have increased due to combined effect of creep, vibration, wear, corrosion, and foreign particle damage. To enhance corrosion and wear resistance researcher suggested to employ advanced-grade material like P91, P92, IN738, IN939, GTD111, Nimonic 105 as substrate and Co-based or Ni- based or Fe-based super alloy as cladding material with aid of advanced welding processes. Furthermore, to address issues with hardness variation in the Heat-Affected Zone (HAZ), a buffer layer between the substrate and cladding material is suggested. While these functionally-graded materials improve corrosion and wear resistance, it's crucial to test their durability under high temperatures and pressures condition. Creep damage is a critical concern for long-term performance of a component requires precise deformation and rupture life prediction for cost-effective maintenance. First, the significance and importance of creep investigation of advanced-grade clad components used for high temperature applications are explained. Creep phenomena in metallic material is explained followed by effect of various parameter such as stress and temperature, microstructure, chemical composition, heat treatments, etc. on the creep behavior of the advanced grade materials are discussed in detail. This manuscript provides the detail insight about various creep deformation mechanism such as diffusion, dislocation creep and grain boundary sliding and their dominance during the high stress and temperature level. Further, recently developed creep model without multiple adjustable parameters such as Kachanov-Rabotnov, Theta projection and Sine hyperbolic models are explored with the help of case studies and their applications, merits and demerits are presented in details. Finally, future developments in the creep analysis of clad components, as well as the creep prediction model, are highlighted. This review paper is intended to provide a solid basis for the investigation of creep behaviour of clad components for high temperature applicability in order to acquire a detailed understanding of all the essential factors associated with component sustainability.

**Keywords:** Creep, Cladding, Advanced-grade material, High temperature, Super alloy

## High temperature deformation behaviour of Al-Cu-Mn-Zr (ACMZ) alloy

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### ABSTRACT

Age hardening Al-Cu alloy is a potential candidate for replacing non-critical part in automotive engine and aerospace component. However, high temperature application of these alloys remains a challenge due to their degradation in mechanical property at high temperature. Al-Cu alloy is primarily strengthened by metastable  $\theta'$  precipitates which undergo transformation and form stable  $\theta$  precipitates and subsequent coarsening occurs when they are exposed at an elevated temperature ( $>200$  °C) for prolonged amount of time [1]. The coarse  $\theta$  precipitates are detrimental for the mechanical properties of these alloys making it unsuitable for high temperature application. A simultaneous addition of Mn and Zr in the Al-Cu alloy helps the metastable  $\theta'$  precipitates stabilize at elevated temperature [2]. This segregation of these trace elements results in an overall reduction of the interfacial energy of the  $\theta'$  precipitates making it more stable at elevated temperature (300-350 °C).

Industrial application of a cast alloy usually requires a suitable thermo-mechanical processing. Mn and Zr modification to Al-Cu alloy when coupled with pre-aging deformation exhibit further alteration in precipitation kinetics. Apart from the influence on precipitation, deformation can also heavily influence the texture formation in these alloys. In this study an Al-6wt% Cu alloy with minor addition of Mn (0.3 wt%) and Zr (0.15 wt%) was prepared by convention melting-casting route. The alloy has been uniaxially compressed over a wide range of strain rate ( $0.01\text{s}^{-1}$ - $1\text{s}^{-1}$ ) and temperature (200-400°C). A comprehensive processing map has been developed which help to detect different deformation mechanism/s at different strain rate-temperature region. Further studies have been carried out on the texture evolution on these different regions in order to correlate the texture formation with the associated deformation mechanism. This study will provide us with a comprehensive wrought processing route of this high temperature resistant aluminium alloy with industrial application potential.

*Keywords: Aluminium alloy, processing map, high temperature deformation*

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# Hot deformation behavior and Processing map of Al-12Ce-0.7Sc alloy

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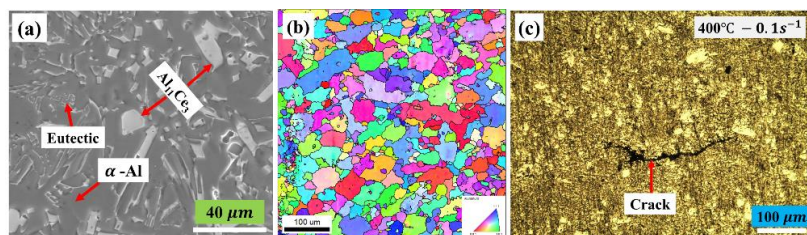
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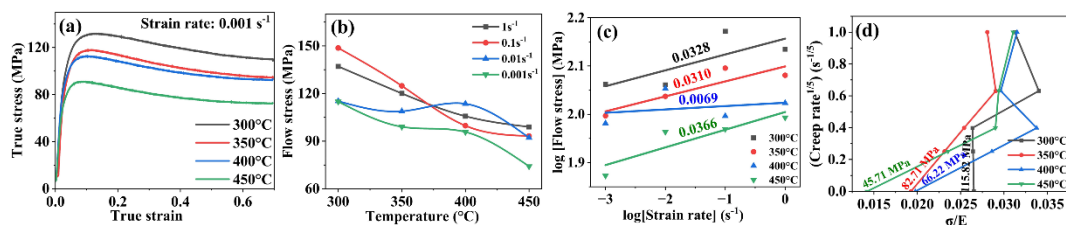
## ABSTRACT

This study investigates the hot deformation behavior of precipitation-hardened stir-cast Al-12Ce-0.7Sc (wt.%) alloy using an isothermal hot compression test using a wide range of temperature and strain rates: 300 – 450°C / 0.001 – 1 s<sup>-1</sup>. The true stress-strain curves show a typical dynamic recovery behavior. The dynamic strain aging (DSA) characteristic was observed in the temperature and strain rates range of 350 – 450°C / 0.01 – 0.001 s<sup>-1</sup>. The sine-hyperbolic Arrhenius constitutive equation and strain-compensated constitutive model were used to predict the flow stress of the materials under different deformation conditions. Further, hyperbolic stress exponent (*n*) was found to be 18 – 51, depending on the strain rate and test temperature. The apparent stress exponent (*n*) is related to the presence of threshold stress, mainly attributed to the presence of micron-sized Al<sub>11</sub>Ce<sub>3</sub> and nano-sized Al<sub>3</sub>Sc precipitates.

Similarly, the sub-grain formation also enhanced the stress exponent. The activation energy of the deformation (*Q*<sub>app</sub>) was found to be 67.52 KJ/mol, which reveals the presence of pipe diffusion as the rate-controlling mechanism for the DSA. The strain rate sensitivity (*m*) was found to be in the range of 0.00695 – 0.03662 from 300 – 400°C. At 400°C, the workability was reduced significantly due to the presence of DSA. The creep threshold stresses (*σ*<sub>th</sub>) were linearly extrapolated to the zero-strain rate using the Langenburg-Bergman plot, considering the stress exponent as (*n* = 5). The processing map was also developed at a strain of 0.5, based on the dynamic materials model (DMM), to obtain the optimum processing window for the hot deformation. The safe hot processing window for this alloy is at 350°C / 0.01 s<sup>-1</sup>. Further, the microstructural evaluations of the hot compression specimens were investigated using an optical and scanning electron microscope. The grain size and misorientation angle of the hot compressed specimens for the safe processing windows were investigated using the Electron backscattered diffraction (EBSD) technique.



**Fig.1.** Initial micrograph images of Al-12Ce-0.7Sc alloy (a) SEM micrograph; (b) EBSD; (c) optical micrograph of the hot compressed specimen.



**Fig.2.** (a) True stress-strain curve; (b) flow stress variation with temperature plot; (c) strain rate sensitivity plot; (d) Langenburg-Bergman pot for creep rate extrapolation.

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# Understanding the role of microsegregation on creep rupture behaviour of LPBF processed IN718

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## ABSTRACT

The microsegregation resulting from the rapid solidification inherent during the laser powder bed fusion additive manufacturing (LPBF-AM) process plays a pivotal role in influencing the precipitation behaviour of strengthening microstructural constituents in superalloys. Consequently, the microstructure developed during the LPBF process coupled with the subsequent post-heat treatment and service conditions exhibit distinct deformation behaviour. Therefore, the current study focuses on understanding the role of microsegregation on the precipitation behaviour of LPBF-AM IN718 alloy and its consequent influence on creep behaviour.

LPBF processed as-built IN718 samples were subjected to solution treatment at 1080 °C for 1 hour, followed by the conventional double ageing treatment employed for wrought IN718 alloys. Subsequently, samples were subjected to 2000 h of long-term exposure at 650 °C, wherein a detailed analysis of their microstructural evolution revealed a progressive precipitation of  $\delta$ -phase ( $\text{Ni}_3\text{Nb}$ ) throughout the duration of exposure. Further, tensile creep experiments were conducted to quantitatively assess and comprehend the creep rupture behaviour. The premature precipitation of  $\delta$ -phase, attributed to microsegregation, has demonstrated substantial variations in creep rupture life, emphasizing its notable impact on its creep behaviour.

## Acknowledgment

We express our sincere gratitude to the Gas Turbine Research Establishment (GTRE), Bengaluru for their invaluable support and funding, which has been instrumental in the successful execution of this project.

# Precipitation and Segregation Behavior in 602 CA Nickel-Based Alloy during High-Temperature Deformation

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## ABSTRACT

Alloy 602 CA is a well-known nickel-chromium-iron alloy recognized for its exceptional resistance to creep deformation and its remarkable ability to resist corrosion, even when subjected to extremely high temperatures, up to 1423 K. This alloy holds significant relevance in various applications within the thermal, chemical, and petrochemical industries. These remarkable properties can be attributed to its microstructure, which contains carbide precipitates. The formation of primary Cr<sub>23</sub>C<sub>6</sub> carbides has been extensively studied, and they are uniformly distributed within the microstructure as well as along the grain boundaries. In recent years, researchers have delved into the evolution of the microstructure, segregation at grain boundaries, and phase transitions in Alloy 602 CA, with a particular focus on grain boundary diffusion. It has been observed that distinct M<sub>23</sub>C<sub>6</sub>-type carbide precipitates tend to form predominantly on high-angle grain boundary sites. However, this precipitation behavior requires further investigation to fully understand the morphology of these precipitates, which is influenced by the nature of the grain boundaries.

Therefore, this study explores the formation of carbides at grain boundaries and the underlying mechanisms, especially in the context of creep deformation. A comprehensive understanding of precipitation behavior is addressed by carrying compression and tensile creep tests under conditions ranging from 723 K to 1073 K and stress levels between 20 MPa and 400 MPa. A thorough microstructural analysis of the crept samples has been carried to address the formation of carbides in relation to the nature of grain boundaries and the stress state at the grain boundaries.

# In-situ characterization of the creep behavior in single-crystalline Co-base superalloys using high-energy X-ray diffraction

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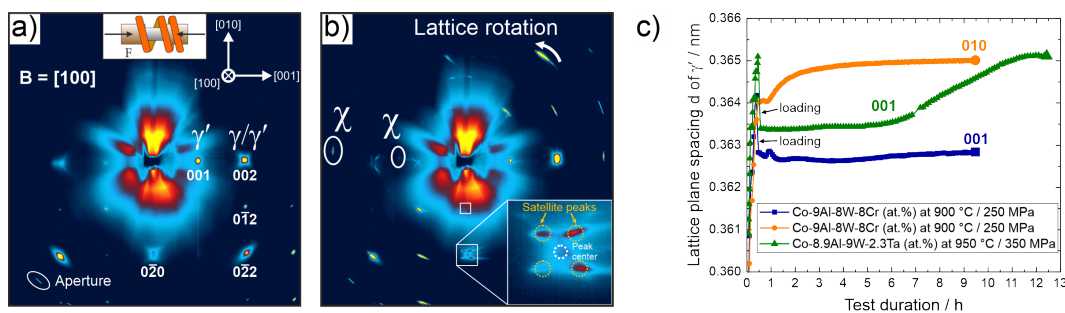
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## ABSTRACT

Gaining direct insights in the deformation processes that occur during creep testing of superalloys at high temperatures is challenging and hence most of the studies e.g. regarding the analysis of the active deformation mechanisms are only conducted post-mortem at room temperature. In this study, an advanced method will be presented which allows for the in-situ characterization of the creep behavior at high temperatures using high-energy synchrotron X-ray diffraction. For this, a compressive load was applied to cylindrical samples along the [001]-crystallographic direction and a photon energy of 100 keV was used to study the creep deformation in two single-crystalline Co-base superalloys. Due to the high photon energy and the corresponding short wavelength the Ewald's sphere is relatively flat which leads to the intersection of several reflections with the Ewald's sphere in reciprocal space. Therefore, after orienting the samples accordingly, the transmitted X-ray beam forms a diffraction pattern with a 4-fold symmetry on a 2D area detector showing both  $\gamma'$  superlattice and  $\gamma/\gamma'$  fundamental reflections in two independent crystallographic directions on just one detector image which is similar to the one in a TEM along the [100] zone axis, see Figure 1 a). With this, the evolution of the reflections during creep can directly be observed in reciprocal space. For a Cr-containing alloy with a relatively low  $\gamma/\gamma'$  lattice misfit at the test temperature, the significantly different evolution of the  $\gamma'$  lattice plane spacings along the [001] and [010] direction (see Figure 1 c)) can be attributed to the loss of coherency between the  $\gamma$  and the  $\gamma'$  phase during the early stages of creep deformation. For a Ta-containing superalloy with a high  $\gamma/\gamma'$  lattice misfit, the significant increase of the  $\gamma'$  lattice plane spacing along the [001] direction (see Figure 1 c)) can be attributed to the pronounced directional coarsening (rafting) of the  $\gamma'$  phase and the resulting reduction of the coherency stresses along the [001] direction. Furthermore, a lattice rotation as well as the deformation-induced formation of the  $\chi$ -phase towards the end of the creep test can be observed in this alloy, see Figure 1 b). Another important aspect is the observation of satellite peaks around the actual center of a respective reflection due to an off-centered intersection of the Ewald's sphere with the 3D extended reciprocal lattice rods (relrods), e.g. see Figure 1 b). For the Ta-containing superalloy, the formation of these relrods is associated with the deformation of the  $\gamma'$  precipitates by the formation of stacking faults on the  $\gamma'$  {111} planes which corresponds well to the observed stacking faults after ex-situ creep tests [1]. By applying this new and advanced testing setup, it is now possible for the first time to simultaneously study in-situ the evolution of lattice plane spacings in different crystallographic directions, phase formation as well as deformation mechanisms in Co-base superalloys during creep at high temperatures.



**Figure 1:** 2D high-energy X-ray diffraction pattern during in-situ creep testing of a Co-8.9Al-9.0W-2.3Ta superalloy at the (a) beginning and (b) end of the in-situ creep test and c) Evolution of the lattice plane spacing of the  $\gamma'$  phase in different crystallographic directions during in-situ creep testing of a Co-9Al-8W-8Cr and a Co-8.9Al-9.0W-2.3Ta superalloy.

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# Determination of high temperature creep properties of Zircaloy-4 clad tube of Indian PHWR and its application in prediction of clad burst behavior

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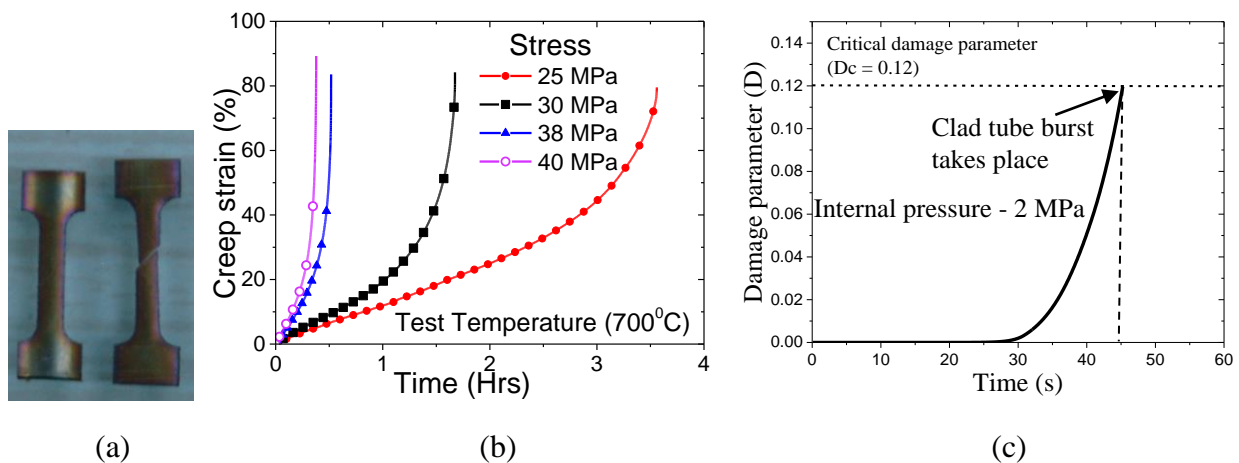
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## ABSTRACT

Zircaloy-4 is used in fabrication of Indian clad tubes of pressurized heavy water reactor (PHWR). Operating temperature of PHWR is around 300°C, however, during postulated events like loss of coolant accident along with station blackout, the heat removal from the fuel bundles will get severely impacted [1]. This shall cause the clad tube to deform progressively due to creep. The temperature of the clad tube may reach upto 1000°C or above causing the clad tube to undergo creep deformation under internal pressure of fission gas. The amount of creep deformation and subsequent burst of clad tube is important from the safety point of view of fuel bundles [2]. The time taken by the clad tube to reach critical damage for its burst is necessary to understand the progression of severe accident and plan the operators response to mitigate consequences of severe accident. In this work, samples were prepared from the longitudinal orientation of the clad tube as shown in Fig. 1(a). Since outer diameter of clad tube is 15.2 mm [3], the design and preparation of the samples to carry out the creep test has been a challenge. Creep tests were conducted in the temperature range of 600-1000°C at different stress levels. The creep curve obtained at 700°C at different stresses ranging from 25 to 40MPa is shown in Fig. 1(b). The activation energy and hardening exponent of the Norton's creep rate equation have been determined at different temperatures. Using these creep rate equations, the deformation of fuel clad is modelled under different high temperature loading conditions in order to predict the deformation behavior of clad tubes. The time taken by the clad tube to burst is determined using critical material damage parameter obtained using an unified damage model as shown in Fig. 1(c). The results of this work is used to study sequence of core damage in nuclear reactors during severe accident scenario.



**Fig.1:** (a) Specimen before and after test (b) Creep curve obtained at 700°C at different stress levels (c) Time taken for the clad tube to burst using critical damage parameter.

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## Examination of Creep of Dissimilar Metallic Joints using DIC-augmented-Bending Creep

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### ABSTRACT

Dissimilar metallic joints are widely used in various high-temperature engineering components, such as nuclear reactors, heat exchangers, and, more recently, in repair and restoration of turbine blades. At elevated temperatures, dissimilar metallic joints are susceptible to premature failure due to creep. In the present study, an additively manufactured hybrid sample of two aluminum alloys (AlSiMg and AlMgSc) showing widely different creep response has been selected as a model system to study the creep properties of dissimilar metallic joints. At first, the creep response of monolithic materials was evaluated using bending creep wherein the strain field on one of the flat surfaces was measured using digital image correlation (DIC). Hybrid samples were machined in two different configurations to study the creep of joints, such that in the first configuration the interface was perpendicular to the length of the cantilever sample, and in second configuration the interface was along the length of the sample. In the first configuration, the experimental results showed that the triaxial region spans up to a small distance from the interface and beyond that, the monolithic properties of the base materials can be extracted. In the second configuration, a shift in the neutral axis was observed towards the creep-resistant side due to load redistribution. It shows that a creep-resistant material can be used to reinforce a softer material to improve its creep behavior significantly.

**Keywords:** Dissimilar metallic joints, creep, High-throughput testing, DIC

# Effect of stress triaxiality on high temperature deformation behavior of SS316LN austenitic stainless steel and calibration of Johnson-Cook material damage model

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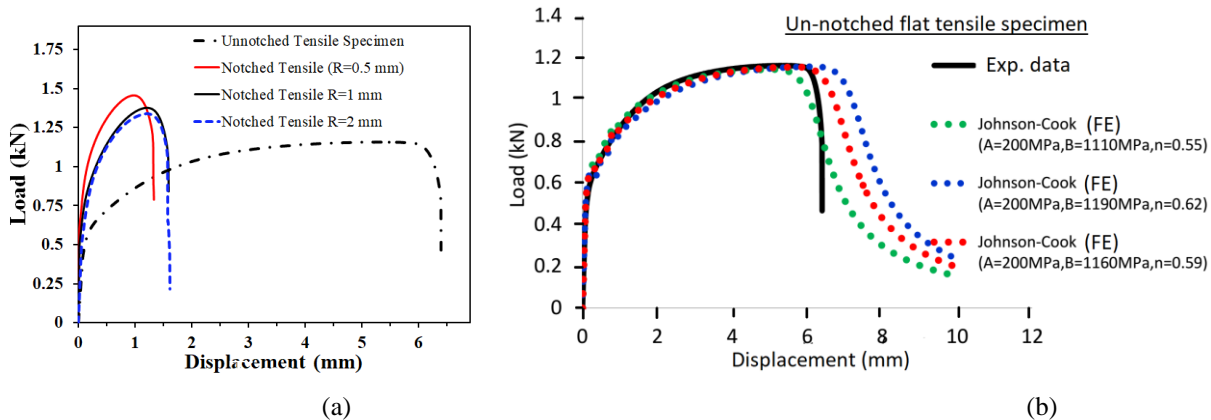
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## ABSTRACT

Austenitic grade stainless steel SS316LN is used for fabrication of vessels and various other critical core components of fast breeder type nuclear reactors. For ensuring structural integrity of reactor components for various type of postulated loading conditions, finite element (FE) analysis of components are carried out. For FE analysis, the data regarding high temperature deformation behavior of the material is required in the form of true stress-strain curve. In addition, Johnson-Cook [JC] [1] type deformation and damage model are required and these have been used widely in literature [2]. The crack initiation in ductile materials follows the mechanism of void nucleation, growth and coalescence and the fracture strain is a function of stress triaxiality. However, evaluation of fracture strain in laboratory specimens for various values of stress triaxiality requires introduction of stress discontinuities, such as notches. On the other hand, introduction of notches creates gradient in strain and stress fields across the cross-section of specimen in the notched region, which is usually the failure zone. In order to address this issue, FE analysis of the specimens are required so as to evaluate the evolution of strain and stress field as a function of loading till the instance of fracture. In this work, both notched and un-notched specimens have been tested at different temperatures and a typical load-displacement data is shown in Fig. 1(a). It may be noted that the load carrying capacity of the notched specimens are higher compared to the un-notched specimens and the maximum load increases with decrease in notch radius. The displacement at fracture also reduces with decrease in notch radius. This is due to increase in stress triaxiality and corresponding suppression of plastic deformation in the notched region of the specimen. The load-deformation behavior of these specimens have been simulated with JC material model and a typical comparison between experimental data and FE analysis is shown in Fig. 1(b). The parameters of JC model has been calibrated with one type of specimens and their applicability for other type of specimens has been verified by comparing the results of simulation with experiment. The variation of parameters of JC model with temperature has also been studied and evaluated for SS316LN stainless steel. The results of this work is very important for integrity analysis of nuclear reactor components.



**Fig.1:** (a) Load-displacement data for SS316LN tensile specimens (notched vs un-notched) and its dependence on notch radius; (b) Numerical simulation of un-notched tensile specimen with Johnson-Cook material model with various parameters and its comparison with experimental data.

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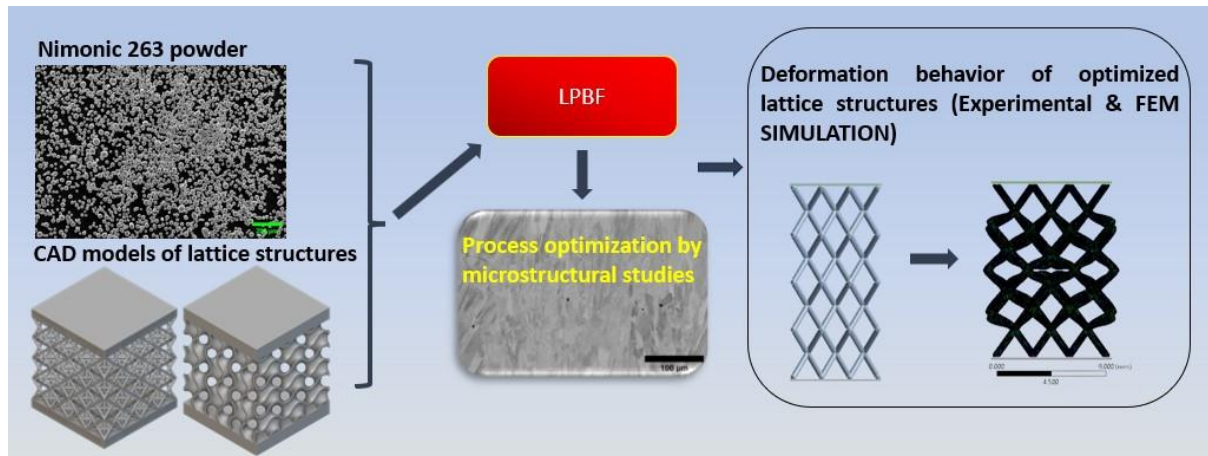
# High-temperature deformation behaviour of Nimonic 263 lattice structures

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## ABSTRACT



Superalloy foams possessing a high strength-to-weight ratio find the niche for high-temperature applications such as aerospace, powerplants, nuclear reactors, etc. Among the Ni-based superalloys, Nimonic 263 has a high service temperature of up to 800 °C due to its, high-temperature strength. However, superalloy foams are challenging to be processed using conventional methods. Hence, additive manufacturing using the laser powder bed fusion (LPBF) process can be explored to produce superalloy foams, which are also termed as lattice structures or non-stochastic foams. Materials processed by LPBF have microstructural deficiencies such as lack of fusion, keyhole, and balling defects. These defects can result in performance degradation at high temperatures due to subpar mechanical properties. To understand the high-temperature deformation behaviour of Nimonic 263 lattice structures, CAD models of BCC, FCC, BCCXYZ, FCCXYZ, octet, hexagonal-honeycomb, Gyroid, Schwarz, and Primitive structures were generated using nTopology and subsequently fabricated employing LPBF process. The fabricated lattice structures have two different microstructures (as-built, without gamma prime precipitation, and heat-treated with gamma prime precipitates). These structures were deformed in compression at room temperature and at elevated temperatures from 200 to 800 °C. Microstructural characterization was performed to investigate the role of defects, carbides, and precipitates on the high-temperature compressive deformation behaviour. The FEA modelling was also implemented using the ANSYS mechanical solver module to simulate the deformation and strain localization during the compression of these modelled structures. Finally, the experimental and theoretical deformation behaviour is correlated with the established models for the deformation of foams.

## Acknowledgements

The authors acknowledge the funding from the Centre of Excellence for Additive Manufacturing (CEAM), Indian Institute of Technology Madras.

# Deformation behaviour of gradient nanostructured nickel at high temperature

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## ABSTRACT

The potential failure modes of RF microelectromechanical systems (RF-MEMS) include dielectric charging, fatigue, contact degradation, and creep [1]. High-temperature tensile properties and creep behaviour for RF-MEMS device applications are investigated in the present work. The nanocrystalline metals used for MEMS devices, including gold, nickel, copper, aluminium, and silicon, have shown many extraordinary properties compared to their coarse-grain (CG) counterparts. Nickel and nickel-based alloys are known to possess favourable properties that make them promising candidates to realize movable structures for MEMS applications. Nanocrystalline (NC) nickel exhibits ultimate strength five times higher than CG nickel [2]. However, there is a reduction in % elongation from 7.5 to 2.9 for NC compared to CG Ni [3].

Gradient nanostructures (GNS) exhibit superior strength and ductility compared to their homogeneous counterparts, as well as better corrosion resistance and wear properties. They are defined by a gradient of grain sizes throughout the thickness from the surface to the core. The % elongation of GNS Ni was around 54% more compared to the homogeneous NC nickel [3]. Zeng et al. comprehended the plastic deformation partition in a two-dimensional (2D) gradient sample by using a finite element-based crystal plasticity constitutive model. Their simulations effectively demonstrate that the gradient samples have a strain gradient. Due to the structural inhomogeneity in the material, the coarse grains plastically yield first and sustain higher plastic strain during deformation than the fine grains, giving rise to the layer-by-layer elastic-plastic transition as well as strain partitioning. As a result, strain gradients are developed inside the material, and geometrically necessary dislocations (GNDs) are generated [4]. The GNDs undergoes pile up and produces back-stress which consequently help with preventing necking during tensile testing, thus improving ductility [5]. The superior material properties may make gradient nickel better candidates for RF-MEMS devices with higher performance and reliability for high-temperature applications.

In the present work, pure Ni samples possessing a gradient structure with a change in the grain size ranging from a few nm to hundreds of nanometers are prepared by electrodeposition technique, changing current density and saccharine amount in the bath. The Vicker's hardness value of the homogeneous NC nickel electrodeposited at 100 mA/cm<sup>2</sup> is 630 MPa. However, when the current density was changed linearly from 25 to 100 mA/cm<sup>2</sup> the hardness value increased to 694 MPa, which is 10% higher than that of the homogeneous NC counterpart. Tensile tests from ambient conditions to 400°C will be carried out to determine the effect of temperature on the prevalent modes of deformation. Creep tests will be performed at temperature 250°C and 400°C. The samples will be characterized using XRD to measure crystallite size, lattice strain, and preferred orientation. Microstructural characterization will be carried out using a scanning electron microscope (SEM) to study the failure mechanism during deformation.

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# Temperature-Driven Failure in Fluorapatites: An Atomistic-Scale Study

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Fluorapatite (FAP) is a type of calcium phosphate mineral with chemical composition  $\text{Ca}_{10}(\text{PO}_4)_6\text{F}_2$ <sup>[1]</sup>. It possesses hexagonal symmetry, which endows it with five independent (direction-dependent) elastic constants. Though commonly employed in medical applications, such as dental restoration and protective coating on metallic implants<sup>[2]</sup>, FAP's ability to undergo diverse isomorphic substitutions<sup>[3]</sup>, makes it adaptable for various uses. Notably, FAP shows promise for emerging high-temperature applications, such as carbon capture from direct sources<sup>[4]</sup>, and nuclear waste sequestration<sup>[5]</sup>. Therefore, it is crucial to understand how the deformation mechanism varies with temperature and loading direction. In this work, molecular dynamics (MD) simulations are conducted using an 8×8×8 supercell containing 34,816 atoms. Though several potential models for apatites have been proposed in the literature<sup>[6]</sup>, but based on our earlier work<sup>[7]</sup>, the FAP is modelled through the core-shell-based Buckingham potential. The system is subjected to tensile loading in different directions: along the x-axis (crystallographic axis a), y-axis (forming a 30-degree angle with crystallographic axis b), and z-axis (along crystallographic axis c). Our simulations reveal that the strength and deformation behavior along the different directions are distinct. We attribute this to the lack of isotropy in the single crystal FAP. We also investigate the effect of increasing the temperature for each loading case. At lower temperatures, FAP exhibits the highest elastic constants along the z-axis; however, for temperatures exceeding 500K, the elastic constant along the x-axis takes precedence. We show that at relatively lower temperatures of up to 500K, loading along the x-direction causes peak stress within the system. However, from 800K to 1200K, the peak stress is observed when the loading occurs along the y-direction. In contrast, FAP fails at much lower peak stress when loaded along the z-direction, regardless of the temperature considered. This indicates that the maximum energy absorption per unit volume without inducing permanent distortion (modulus of resilience) is the smallest for the z-axis direction.

## Acknowledgments

AR thanks CSIR, India, for providing the fellowship towards her PhD.

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# **UNDERSTANDING THE DWELL FATIGUE AND CREEP PROPERTIES OF IMI834 INSIGHTS FROM EXPERIMENTS AND SIMULATIONS**

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## **ABSTRACT**

Cold dwell fatigue is a well-known phenomenon in titanium alloys where stress holds at ambient temperatures, reducing fatigue life. This study investigates the anisotropy of dwell fatigue behavior of the IMI834 in forged disc along axial and radial directions experimentally and correlates it with 2D microstructure using crystal plasticity Fast Fourier transform simulations. Cyclic and dwell fatigue tests with peak stress of 95% of yield stress show anisotropy of dwell fatigue properties along axial and transverse directions. 2-Dimensional EBSD microstructures are obtained to study the effect of localized grain orientation combinations and texture evolution during dwell fatigue loading using DAMASK software. Fractography confirms that the fracture-initiated region is faceted in nature in all microstructures. EBSD analysis is done on the microcracks formed under the fractured surface to understand any relationship between the orientation of grains and crack initiation. Most of the microcracks formed have been observed in the primary alpha grains, which are basal oriented in nature. Creep tests are done at 95% of yield stress at room temperature. ECCI technique is used to understand the slip traces in the grain containing microcrack and its neighbors.

## Creep response of yttria containing hot powder forged ODS Steels

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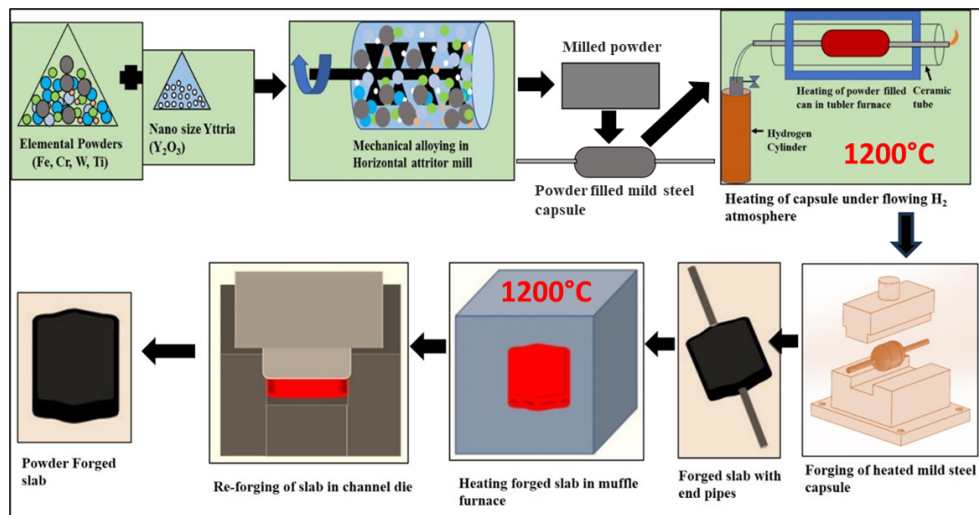
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### ABSTRACT

The aim of this study is to investigate the creep behavior of two types of ferritic ODS steel: Fe-18Cr-2W (ODS-0) and Fe-18Cr-2W-0.285Ti-0.5Y<sub>2</sub>O<sub>3</sub> (ODS-1), produced by hot powder forging. The fabrication route of these steels are shown in Fig.1. Creep tests were conducted in a temperature range of 600°C to 700°C and a stress range of 120MPa to 230MPa. The as-forged ODS steels exhibit an equiaxed, strain-free grain structure. After creep, the microstructure of Ti and yttria-containing ODS steel remains stable, while steel without Ti and yttria shows elongated grain structures. Significant improvement in creep life is observed in Ti and yttria-containing ODS steel compared to the one without Ti and yttria. The creep mechanism in these test regimes is discussed. Sections of samples near creep fracture surfaces revealed void formation at triple junctions, followed by crack propagation normal to the loading direction, leading to final failure. The fracture surface of the creep-tested samples shows mixed mode (cleavage + ductile dimple) failure.



**Fig.1:** Schematic diagram of the hot powder forging route.

### Acknowledgments

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## Creep of carbide-strengthened superalloys

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### ABSTRACT

Carbide-strengthened superalloys have been used for various high-temperature applications in turbine engines. This demands the development of a fundamental understanding of their high-temperature deformation behaviour specifically creep deformation mechanisms over a wide range of temperatures and stress. However, the widely known dislocation creep theories have not been successful in explaining the primary creep regime and underlying overall creep deformation mechanisms at larger strains. This is due to the continuous evolution of microstructural constituents and dislocation structure. Thus, the present work aims to understand the role of stacking fault energy and microconstituents on the creep behaviour of carbide-strengthened superalloys using two model superalloys (Co-22Cr-22Ni-14W-3Fe-1Mn-0.1C, Ni-22Cr-14W-5Co-3Fe-2Mo-0.1C (wt.%)). Constant load tensile creep tests were conducted on the solution-annealed specimens in the temperature range of 650 – 900 °C and the stress range of 50 – 300 MPa till steady-state was reached. Further, internal stress and its evolution with strain were estimated using the load drop experiments during creep testing. From the detailed transmission electron microscopy studies, the substructure evolution was analyzed. Together, the distinct role of various microstructural features and stacking fault energy of the respective alloys is correlated with their creep behaviour.

Keywords: Superalloys, Creep, Internal stress, Substructure

# Investigation of high temperature creep deformation behavior of alloy 690 used in nuclear reprocessing plants

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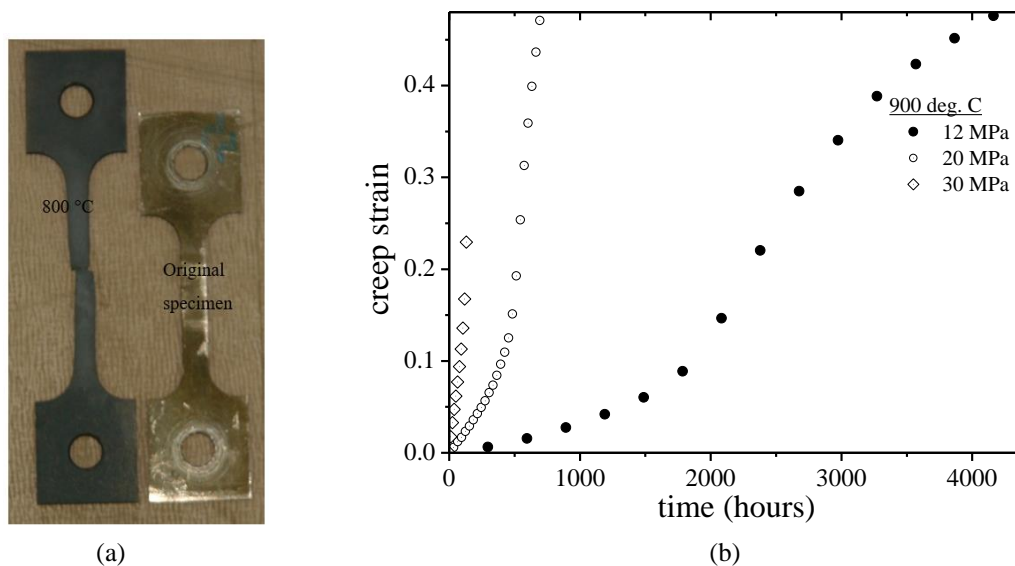
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## ABSTRACT

Alloy 690 is a nickel based alloy with high chromium content of 28% approximately. It has been used in fabrication of steam generator tubes and processing vessels of nuclear reprocessing plants[1]. Since the temperature of reprocessing vessels may reach upto 1100°C during its operation, evaluation of high temperature creep deformation is of utmost importance. The amount of creep deformation is necessary for performing the design life and ageing studies of various components made up of alloy 690 material. In this work, a systematic investigation of creep deformation of alloy 690 material in the temperature range of 600 to 1000°C under different stress levels has been carried out. The 10 mm thick alloy 690 plate used for fabrication of reprocessing vessels is used in manufacturing of different specimens. The specimen used for evaluation of creep properties is shown in Fig. 1(a). The specimen was designed according to ASTM E8 standard [2]. The creep curve has been obtained at different temperature and stress conditions. The comparison of creep data at 700°C at different stress levels is shown in Fig. 1(b). Similar results were obtained at other temperatures. Using these data, the steady state creep rate were evaluated which is used to evaluate the activation energy and hardening exponent of alloy 690 material. These parameters has been used for determining the Norton's creep rate equation which can be used for modelling the creep deformation of alloy 690 material under different operating conditions. Larson Miller parameters have been determined at various temperature and stress levels. These data shall be useful in design and safety analysis of various reprocessing components.



**Fig.1:** (a) Specimen before and after test (b) Creep curve obtained at 900°C at different stress levels.

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# **Rapid estimation of dwell fatigue life in titanium alloys by statistically analyzing the characteristics of deformation events at the nanometer scale**

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## **ABSTRACT**

This study illustrates the connection between the characteristics of deformation events at the nanometer scale, which develop during early cycling, and the long-term performance of a titanium Ti64 alloy. Automated imaging within a scanning electron microscope is utilized to conduct high-resolution digital image correlation measurements during deformation over large fields of view. A computer vision algorithm then extracts the characteristics of the observed deformation events. The characteristics of the most intense deformation events during the first loading cycle are found to be indicative of long-term performance. The reduction (dwell debit) in the lifetime of the titanium alloy subjected to different load hold periods is analyzed by examining the change in the intensity of deformation events during the first dwell-fatigue cycle. A direct correlation is observed between the increase in the intensity of the most intense deformation events during the hold and the dwell debit.

# Unveiling the Impact of Strain Gradients and Surface Proximity Effects on Creep Behavior

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## ABSTRACT

In this study, we examine the relative dominance of strain gradient and surface proximity effects in cantilevers with reference to uniaxially loaded specimens using aluminum as a case study. Under uniaxial loading, near-surface regions developed creep-compliant dislocation substructures with lower dislocation density and larger subgrain size. This surface-affected region varied inversely with applied stress and was limited by grain size. When the surface-affected region matched the sample's characteristic dimension, overall creep resistance decreased, leading to load-shedding between the weaker surface and the stronger interior.

Conversely, meso-scale cantilevers less than 1 mm thick and thinner exhibited diverse behaviors—hardening, softening, and bulk-like—depending on the strain gradient's magnitude. Regions with significant strain gradients showed refined dislocation structures, enhancing creep resistance, while areas with minimal strain gradients experienced increased creep rates due to surface effects. These findings highlight the importance of strain gradients, which can overshadow surface proximity-induced softening after a certain magnitude ( $\sim 0.05 \text{ mm}^{-1}$ ). This work advances our understanding of material behavior under creep conditions, with potential implications for designing structures with strain gradients.

## Transitional Creep Mechanisms in Zircalloys

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### ABSTRACT

Zircalloys have been used as nuclear fuel cladding in light water reactors due to their low absorption cross-section for thermal neutrons, good mechanical properties, and good corrosion resistance. Creep is one of the primary performance degrading mechanisms in Zircalloys during reactor service owing to the presence of high temperature and stresses at the reactor core. As a result, prediction of creep life of Zircalloys under relevant reactor service conditions becomes critical for structural integrity and safety of nuclear reactors. To this end, knowledge of transitions in creep mechanisms and descriptive models of creep rates as a function of stress, temperature and microstructure are essential components in creep life prediction. In this paper, we present the results of uniaxial and biaxial creep studies of Zircaloy-4 and Nb-added Zircaloy under a range of temperature and stress conditions. Creep mechanisms have been detected by a combinatorial analysis of creep parameters such as stress exponent and activation energy, deformation microstructure by transmission electron microscopy and model-experimental data comparison. Details of the creep mechanisms such as diffusional creep, dislocation climb, dislocation cross-slip and Orowan bypassing active in the Zircalloys and their implications on creep-life prediction during dry storage will be discussed in this presentation. Furthermore, the influence of hydriding – an inevitable microstructural change occurring in Zircalloys during light water reactor operation – on the creep behavior will be presented.

### Acknowledgments (if any)

This research was supported by DOE-NEUP under the Integrated Research Project (IRP) entitled, 'Fuel Aging in Storage and Transportation (FAST)', and US National Science Foundation [grant number DMR0968825].

## **Microstructural evolution and creep rupture behaviour of magnetically impelled arc butt welded dissimilar joints of P91 and Super304H steel pipes**

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### **ABSTRACT**

Dissimilar joints of ferritic-martensitic steels and austenitic stainless steels are used in critical sections (superheater and reheater) of advanced ultra-supercritical boilers. The primary limitation hindering the operational conditions of the dissimilar joint of ferritic-martensitic steels and austenitic steels efficiency is the microstructural instability in the wide heat-affected zone (HAZ) of ferritic-martensitic steels resulting from the shallow temperature gradient due to the conventional welding processes. The formation of such unstable microstructure in the weldment further impacts their reliability in demanding long-term microstructural stability, and creep strength during service. Among various processes, magnetically impelled arc butt (MIAB) welding is a unique thermo-mechanical welding technique for joining closed tubular structures with localized heating and melting, resulting in fine/narrow HAZ.

A systematic investigation was undertaken to analyse the microstructural evolution during the joining of dissimilar joints of P91 and Super304H steels using MIAB welding. The weldment demonstrated a fine/narrow HAZ adjacent to the faying surface of both P91 and Super304H. Intriguingly, there was no significant change in hardness across the weld interface towards the Super304H, whereas there was an increase in hardness across the P91 weldment. A detailed microstructural evolution was characterised using optical and scanning electron microscopy (SEM). The creep strength of the weldment was evaluated at temperatures 550 – 650 °C at different stress levels of 150 – 250 MPa. The creep rupture of the weldment was analyzed by correlating the mechanical data with microstructural characteristics.

## Creep Damage Mechanisms and Prediction of Creep Properties of DS CM-247 Alloy

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### ABSTRACT

A study was conducted to investigate creep damage mechanisms and predict the creep behavior of a directionally solidified nickel-based superalloy (DSCM247) at three different temperatures: 850°C, 950°C, and 1050°C, while subjecting the material to a stress range spanning from 150MPa to 500MPa using the concepts of  $\theta$ -Projection technique and continuum damage mechanics (CDM) approach. In this current investigation, an effort has been undertaken to fully uncover the capabilities of the alternate current potential drop (ACPD) technique for assessing creep strain and damage accumulation. And demonstrated the ACPD technique's capability to quantify creep strain and damage both in smooth and notch specimens. The data depicting the evolution of damage acquired from creep tests performed under identical stress and temperature conditions, utilizing the ACPD technique for both smooth and notched specimens were coupled with creep constitution equation to generate/predict creep curves following the concepts of strain equivalence hypothesis of continuum damage mechanics (CDM) and validated with experimental creep curves. Also, a methodology based on CDM concept has been developed to predict important creep parameters such as threshold creep strain ( $\epsilon_{th}$ ), critical creep strain ( $\epsilon_{cr}$ ) and time to rupture ( $t_r$ ). Further, the CDM predicted  $\epsilon_{th}$ ,  $\epsilon_{cr}$  and  $t_r$  values as well as the values derived from the creep curves predicted by  $\theta$ - projection technique were successfully validated with the experimental values. Detailed microstructure and damage evolution during creep of specimens with and without notch were characterized using Alternate Current Potential Drop (ACPD), scanning electron microscopy (SEM), electron back scattered diffraction (EBSD) techniques.

# Deformation response and microstructure and micro-texture evolution during hot compression of Ti-6Al-2V-1Fe-1Cr alloy

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## ABSTRACT

Titanium alloys are widely used as structural materials in the field of automotive, aerospace, and medical industries due to their excellent high-temperature specific strength and corrosion resistance. In the present study, we investigated a new two-phase ( $\alpha+\beta$ ) Ti alloy Ti-6Al-2V-1Fe-1Cr (Ti-6211), which has finer prior  $\beta$  grains. Presence of strong  $\beta$  stabilizer (Fe, Cr) leads to increase the amount of  $\beta$  phase fraction in this alloy. Higher amount of  $\beta$  phase fraction, finer prior  $\beta$  grains leads to increase the strength and ductility as well as decrease the  $\beta$ -transus of this alloy than Ti-6Al-4V alloy. The alloy was cast in a vacuum arc melting furnace and studied for the microstructure and chemical composition. Metallography, thermal analysis, CALPHAD approach were used to estimate the  $\beta$ -transus temperature. Microstructure studies reveal the presence of basket weave structure with finer  $\beta$  grain size ( $\sim 572 \pm 240 \mu\text{m}$ ) and higher  $\beta$  volume fraction ( $\sim 16\%$ ) than Ti-6Al-4V alloy in as-cast condition. The  $\beta$ -transus temperature of this newly developed alloy estimated to be  $970 \pm 5^\circ\text{C}$ .

The as-cast alloy underwent isothermal hot compression across a range of temperatures ( $725^\circ\text{C}$  to  $925^\circ\text{C}$  with an interval of  $50^\circ\text{C}$ ) while maintaining a constant strain rate of  $1\text{s}^{-1}$ . The compressed specimens were examined for the microstructural changes through SEM. Microstructure reveals the bending and kinking of  $\alpha$  lamellae for near  $\alpha$  test temperatures whereas at higher temperatures, fragmentation of  $\alpha$  lamellae takes place. Microstructural observations confirm the initiation of spheroidization at  $825^\circ\text{C}$ . In order to investigate the microstructural details in terms of orientation, EBSD scans were conducted on the specimens deformed at  $825^\circ\text{C}$ ,  $875^\circ\text{C}$  and  $925^\circ\text{C}$ .



# Degradation of mechanical properties of Nano-twinned Copper with temperature

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## ABSTRACT

The recently proposed “Moore’s law of Packaging” states that the number of interconnects in a package will continue to double every few years providing the necessary performance boost from one generation to the next. This has led to a significant push towards advanced packaging techniques like 2.5D and 3D heterogeneous integration. These architectures drive fine pitch first level interconnects requiring direct Cu-Cu bonding while eliminating solders. Nano-twinned (NT) Cu is emerging as a key material for interconnects in advanced packaging, especially for direct Cu-Cu bonding. In NT-Cu, dislocation motions are restricted by the twin boundaries (TBs) increasing the strength of the material. However, these dislocations can still penetrate through the TBs, exhibiting an excellent combination of strength and ductility. The electrical resistivity offered by the TBs is extremely low compared to the high-angle grain boundaries (GBs), contributing negligibly to the overall electrical resistance of the material, unlike other strengthening mechanisms. It also possesses high resistance to electromigration due to the low diffusivity path of the TB and thus improving the reliability and performance of the microelectronic devices. NT-Cu restricts the formation of Kirkendall voids as the TB act as the sink for the vacancies.

Here, we report temperature (25°C to 200°C) and strain rate ( $1.6 \times 10^{-5}$  to  $5.4 \times 10^{-4} \text{ s}^{-1}$ ) dependent mechanical properties of electrodeposited NT-Cu having grain size of  $242 \pm 116 \text{ nm}$  and  $74 \text{ nm}$  twin spacing in as deposited condition. Our high temperature tensile tests results showed softening of NT-Cu from 25°C to 200°C and increase in strength when strain rate was increased from  $1.6 \times 10^{-5}$  to  $5.4 \times 10^{-4} \text{ s}^{-1}$ . Tensile strength decreased by 90 % from 25°C to 200°C at constant strain rate of  $5.4 \times 10^{-4} \text{ s}^{-1}$  while it decreased by 23% by decreasing strain rate from  $5.4 \times 10^{-4} \text{ s}^{-1}$  to  $1.6 \times 10^{-5} \text{ s}^{-1}$  at 25°C. A large increase (3.5X) was observed in the ductility at high temperatures whereas modulus decreased by  $\sim 86\%$  at highest temperature (200°C) and lowest strain rate ( $1.6 \times 10^{-5} \text{ s}^{-1}$ ). A significant drop of 67% in the strain hardening exponent may be due to detwinning and twin coarsening causing softening at high temperatures. Further microstructural analysis using FIB and EBSD can confirm the actual failure mechanisms. These results are relevant for thermomechanical modelling of the interconnects as well as for the process development for direct Cu-Cu bonding.

**Keywords:** Nano-twinned Cu; High temperature tensile test; Mechanical properties; Interconnects; Semiconductor Packaging;

## Research Data Management for Creep Reference Data of Ni-Based Superalloys

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In times of digital data processing and sharing, a central challenge for the Materials Science and Engineering (MSE) community is the complete digital representation of materials, including their relevant processing and microstructure details and the resulting properties. This digital representation, supported by digital infrastructures, enables entirely new qualities of collaborative work and is central to how we will conduct MSE research and develop products in the future. The main challenges are the composition, shaping, and description of the data so that it can be easily interpreted, trusted, and re-used. This presentation introduces our current effort to develop a best practice framework for generating and distributing creep reference datasets for Ni-based superalloys, which builds on previous approaches [1,2]. Researchers can use such datasets to compare their own experimental or simulation results, used, for instance, to perform simulation-based alloy design and optimization or in the verification of the own testing equipment. The concept includes building an easily accessible digital infrastructure to annotate and discover the datasets and the integration of an ontology that aligns with the necessary metadata schema. With the help of an ontology, the reference dataset for creep can be semantically described, thus enabling the digital representation and reuse of shared concepts from domain experts and the community. To achieve this, it is necessary to establish a defined vocabulary with corresponding relations and annotations. The interplay between a digital infrastructure, the agreement on the necessary metadata and related metadata schema, and an underlying ontology will ensure that data and results can be easily shared, taken up, and re-used, thus fulfilling the FAIR principles (Findable, Accessible, Interoperable, Reusable). Furthermore, we aim to develop a widely accepted definition of reference data and data quality levels and to reach a self-supported community process. We intend to motivate the discussion about reference material datasets, necessary metadata, and future collaborations within the creep community.

### Acknowledgments

This project is funded by the scientific consortium NFDI-MatWerk of the national research data infrastructure (NFDI) of the Federal Republic of Germany (funding reference no.: NFDI 38/1, DFG project no.: 460247524).

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# Inference of grain boundary sliding and co-rotation of $\alpha$ and $\beta$ phases during superplastic deformation of Zr-2.5%Nb at 700 °C through EBSD measurements

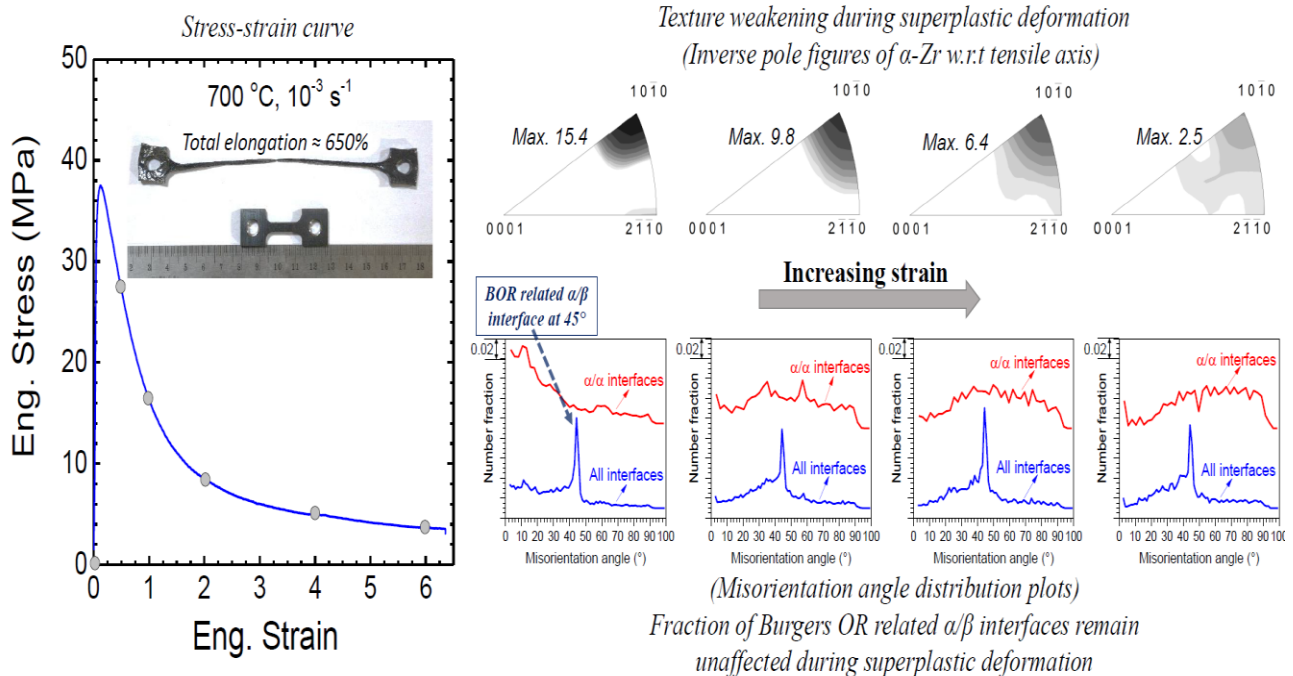
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## ABSTRACT

Present study explains the micromechanics of superplastic deformation of Zr-2.5 wt.%Nb alloy using EBSD analysis. The alloy when deformed in tension at 700 °C and  $10^{-3} \text{ s}^{-1}$  exhibited superplastic flow with total elongation of 650%. Different samples were deformed to intermediate strain levels for characterizing microstructure evolution and subsequently, establishing the underlying micro-mechanisms. EBSD measurements showed that local misorientations within grains did not develop during the superplastic flow. Texture weakening in  $\alpha$  and  $\beta$ -Zr phases was observed and it has been shown that the resultant texture is not due to slip-based deformation. Misorientation angle distribution plots revealed a peak at 45° that corresponded to the interface angle between the Burgers OR related  $\alpha$  and  $\beta$  phases. The fraction of these interfaces remained unaffected during the superplastic flow. It is concluded that the superplastic flow in Zr-2.5Nb at 700 °C is accommodated by Rachinger grain boundary sliding and co-rotation of  $\alpha$  and  $\beta$  grains.



## Acknowledgments

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G. Bharat Reddy et al., Scripta Materialia **234** (2023) 115567

## **Physically based model to study the creep deformation behavior in the nickel based disk superalloys**

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### **ABSTRACT**

This research work focuses on the development of a dislocation creep model to simulate the creep curves, along with the prediction of microstructure evolution in the nickel based superalloy, that is primarily used for the manufacturing of the turbine disk of aeroengine. The mechanisms that are responsible for microstructure evolution during creep includes dislocation generation, glide recovery and climb recovery. The total creep deformation is evaluated through the combination of strain fractions obtained from Orowan's equation and grain boundary sliding mechanism. The simulated creep curves are validated with the experimental creep curves and depict a reasonable agreement. The evolution of different variables such as dislocation densities (mobile and immobile), mean free path, dislocation glide velocity, climb velocity, internal stress, effective stress and climb stress are also discussed meticulously.

## **Limit load and ovalization studies on carbon steel elbows under in-plane bending**

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### **ABSTRACT**

Elbows form an integral part of a piping system because they are used for changing the direction of flow in the nuclear power plant industry. They are relatively more flexible than the straight portion of the piping system. Hence, they tend to deform easily and absorb more energy due to displacements of the system under various loading conditions. They tend to undergo plastic deformation under these conditions, leading to unserviceable situations. The most common type of loading is bending which deforms the elbows and leads to failure. Along with the external loadings, these elbows experience internal pressure from the liquid or gas they are conveying. Hence, estimating the load carrying capacity and the extent of deformation of the elbows is pertinent. Limit load and ovalization of the elbows are dependent on various parameters such as thickness, mean radius, bent radius, internal pressure, yield stress, notch size and deformed cross-section diameter of the elbow.

In the present study, numerical simulations were carried out on two types of carbon steel elbows to determine the limit load and the ovalization. For this study, healthy elbows and elbows having circumferential through-wall notch at intrados were considered. These elbows are studied with and without internal pressure and subjected to in-plane bending moment. Since the notch is at intrados, opening moment is applied for elbows having circumferential through-wall notch as well as healthy elbows.

From the results of experimental studies, load-displacement curves were obtained. From these curves, limit load values were calculated using the twice elastic slope (TES) method as per ASME code. From the finite element analysis, the limit load values were determined. In the literature, different limit load evaluation expressions were proposed by various researchers by using the results of numerical studies. Such limit load evaluation expressions proposed by Chattopadhyay and Tomar (2006), Chang-Sik and Kim (2006) and Hong et. al. (2010) are considered in the present study for comparison of the limit loads.

Ovalization, in general, indicates the deformation of the cross-section of an elbow under loading. The ovalization of these elbows was experimentally measured and compared with the deformation values obtained from the numerical studies.

**KEYWORDS:** Carbon Steel Elbows, Internal Pressure, Circumferential Through-Wall Crack, Limit load, Ovalization.

## **Thermo-mechanical analysis of notched piping components using phase field approach**

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### **ABSTRACT**

With the increase in industrialization, demand for materials that can sustain different loading conditions are increasing. In this regard, fracture assessment of material dealing with crack initiation and crack growth behaviour becomes of paramount importance to ensure adequate safety under different loading conditions. Pipelines play an irreplaceable role in long-distance oil and gas transportation. Leaks in oil and gas pipelines can cause significant damage to people, property, and the environment. The Main Heat Transport (MHT) system of nuclear power plants employs stainless steel weldments at many critical joints in piping components made of SS304LN austenitic steel. The maximum temperature inside these piping components may reach up to 300 °C. The loading condition at such an environmental state may reduce the fracture resistance and load-carrying capacity of components, and hence assessing the fracture behaviour of pipelines under operating conditions is vital for its structural integrity. Here, an attempt is made to numerically simulate the fracture behaviour of notched piping components under thermo-mechanical analysis using a phase field (PF) approach.

The PF approach has gained considerable attention in solving complex crack propagation problems because of its mesh independence capabilities. PF approach is based on the energy minimization principle, which will deal with strain energy, thermal energy and fracture energy in conjunction for thermo–mechanical analysis. This approach represents crack initiation and propagation phenomena within a unified framework by a scalar PF variable discriminating the uncracked and cracked surface by 0 and 1, respectively. A degradation function accounting for the loss of stiffness in the material is also introduced in the mathematical modeling. The coupled equilibrium equations thus formed are numerically solved by three-dimensional (3D) finite element discretization interfaced in CAE software using different user-defined subroutines. Numerical results thus obtained for load-loadline displacement (LLD), crack propagation path, and temperature variation profile from PF approach are validated with experimental results of notched piping components subjected to 4-point bending under monotonic loading scenario at elevated temperature. The proposed phase-field approach seems promising to deal with the fracture behaviour of structural components with multi-physics phenomena.

**KEYWORDS** : Piping components, Phase Field, Thermo-Mechanical, Finite Element Analysis, User-Defined Subroutines.

# Estimation of $\beta$ -transus temperature of Ti-6Al-2V-1Fe-1Cr alloy

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## ABSTRACT

Titanium alloys are widely used as structural materials in the field of automotive, aerospace, and medical industries due to their excellent high-temperature specific strength and corrosion resistance. Ti-6Al-4V alloy is most popular for structural applications due to its better combination of mechanical properties and biocompatibility. However, the presence of large prior  $\beta$  grains and thick  $\alpha$  lamellae in Ti-6Al-4V alloy restricts its use in as-cast condition. In the present study, we investigated a new two-phase ( $\alpha+\beta$ ) Ti alloy Ti-6Al-2V-1Fe-1Cr (Ti-6211), which has finer prior  $\beta$  grains. Presence of strong  $\beta$  stabilizer (Fe, Cr) leads to increase the amount of  $\beta$  phase fraction in this alloy. Higher amount of  $\beta$  phase fraction, finer prior  $\beta$  grains leads to increase the strength and ductility as well as decrease the  $\beta$ -transus of this alloy than Ti-6Al-4V alloy. Apart from this, lesser toxicity can be expected from this alloy due to presence of lower amount of V.

The alloy was cast in a vacuum arc melting furnace and studied for the microstructure and chemical composition. Metallography, thermal analysis, in-situ high-temperature X-ray diffraction, and CALPHAD approach were used to estimate the  $\beta$ -transus temperature. The estimation of  $\beta$ -transus is important for developing the TMP processing parameters. Microstructure studies reveal the presence of basket weave structure with finer  $\beta$  grain size ( $\sim 572 \pm 240 \mu\text{m}$ ) and higher  $\beta$  volume fraction ( $\sim 16\%$ ) than Ti-6Al-4V alloy in as-cast condition. CALPHAD calculation estimates the  $\beta$ -transus to be  $\sim 976^\circ\text{C}$ . Thermal analysis by differential scanning calorimetry (DSC) and differential thermal analysis (DTA) provides the temperature range of  $750^\circ\text{C}$  to  $975^\circ\text{C}$  for  $\alpha$  to  $\beta$  phase transformation.

Based on the thermal analysis, metallographic studies were carried out on heat-treated samples to estimate the exact  $\beta$ -transus. The samples were heat treated with a heating rate of  $5^\circ\text{C}$  per minute in the range of  $750^\circ\text{C}$  to  $975^\circ\text{C}$  and soaked for 30 minutes at target temperature before rapid quenching in ice water. The micrograph of the sample heat treated at  $\sim 970^\circ\text{C}$  shows the presence of acicular or martensitic  $\alpha$ , which confirms the  $\beta$ -transus of  $970 \pm 5^\circ\text{C}$ . The error of  $5^\circ\text{C}$  is due to the inaccuracy in temperature measurement and delay in quenching. In-situ high-temperature X-ray diffraction show intense  $\beta$  peaks at  $970^\circ\text{C}$ , which confirms the  $\beta$ -transus to be  $\sim 970^\circ\text{C}$ . Finally, the  $\beta$ -approach curve ( $\beta$  volume fraction of as a function of temperature) was determined within the temperature range  $750^\circ\text{C}$  to  $970^\circ\text{C}$ .

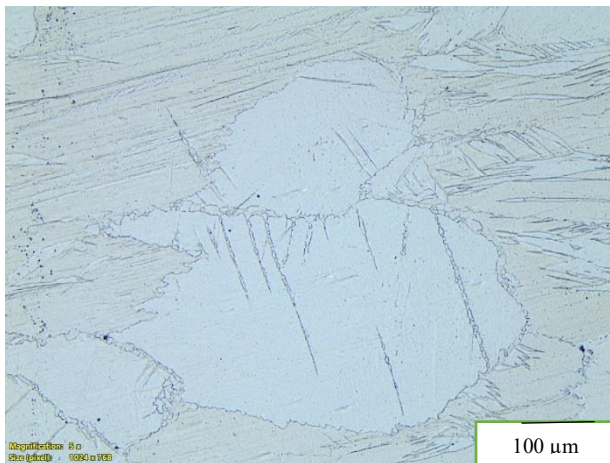
## Mechanical and microstructural modification of Mg AZ31 alloy using friction stir process & heat treatment

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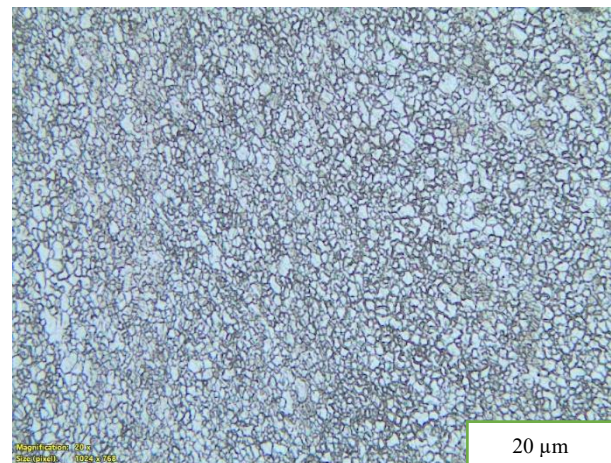
### ABSTRACT

Magnesium base alloys are one of the lightest metallic structural materials and hence are considered as the potential replacements for Al-base components in the automobile and aerospace sectors. It has the potential to increase fuel efficiency and significantly control global environmental pollution. Most of the structural magnesium alloys exhibit insufficient room temperature ductility and anisotropy, constituting a bottleneck for their widespread application. Multiple research projects showed that microstructure and texture modification could enhance Mechanical properties at RT. Severe plastic deformation (SPD) and heat treatment (HT) have a high probability of reducing the problem in Mg alloy.

The friction Stir process (FSP) and different HT modifications in the microstructure and texture help in reducing twin and hence Mechanical properties. It can also control flow stress and reduce defects by controlling parameters. Comprehensive characterization of AZ31 at various processing stages (as received, after Friction Stir Processing) establishment of correlations between texture and microstructure evolution and the RT compression test, Fracture toughness test in RD, TD, RD & TD 45°. After FSP grain refinement, dynamic recrystallization, improved material flow, and reduced internal stress



AS Received sample AZ31 Optical  
Microscope



After FSP AZ31 OM

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Friction stir welding and processing “Materials Science and Engineering” Volume 50, Issues 1–2, 31 August 2005, Pages 1-78 (<https://doi.org/10.1016/j.mser.2005.07.001>)



# **CREEP BEHAVIOUR OF ADDITIVE MANUFACTURING – A REVIEW**

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## **ABSTRACT**

Creep is a slow process which is independent of time. It is defined as a permanent deformation caused by stress like mechanical, electrical, etc. It can be prevented by the alloys and microstructure of the corresponding metals, in addition to its melting point. It is reduced by depressing the temperature, stress level and increasing the stiffness. The study explores the influence of process parameters such as layer thickness and infill density. Additive manufacturing (AM) plays an important role in industrial productions. AM or Additive Layer Manufacturing (ALM) is a technical process in which the 3D objects are constructed by using computer based process. In this paper, we review the creep behaviour of Additive Manufacturing which is in trend nowadays. The process of preventing this behaviour is widely discussed here. Finally this paper addresses the creep behaviour of metals using additive manufacturing. This review serves as a valuable resource for engineers and researchers aiming to optimize AM processes and design components with enhanced creep resistance.

Key words : creep, additive manufacturing, additive layer manufacturing, micro structure

# **Development of a computational framework to predict the ductile to brittle transition of ferritic steel**

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## **ABSTRACT**

Irradiation damage can induce significant microstructural changes in nuclear materials, leading to the nucleation of nano-sized precipitates and an enhancement of dislocation activity. This, in turn, results in phenomena such as swelling, creep, hardening, and embrittlement. The Charpy impact test serves as an efficient method to assess the shift in the ductile-brittle transition temperature (DBTT), quantifying the extent of irradiation damage. A cohesive finite element method (CFEM)-based framework is developed to capture different fracture micro-mechanisms in the DBTT regime. To account for the stochastic nature of fracture, we digitally generate statistically equivalent microstructure sample sets (SEMSS), in which cohesive elements are inserted throughout the microstructure (inside the grain and along the grain boundary). Further, this microstructure is tied to the notch of the Charpy specimen. Cohesive elements follow a temperature-dependent bilinear traction-separation law derived from the unit cell method, while experimental stress-strain curve is used for bulk elements. Our framework provides a reasonable prediction of Charpy impact energy at different temperatures and successfully captures the ductile-to-brittle transition of ferritic steel. The numerical results were found to be in good agreement with the experimental data reported in the literature.

## **Acknowledgments**

This work was funded through Start-up Research Grant (SRG) scheme by DST.

## Addressing functional fatigue challenges in shape memory alloys

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### ABSTRACT

Shape memory alloys (SMAs) are progressively finding broader applications in biomedical, robotic and aerospace industries. The properties like superelasticity and shape memory effect make SMAs a promising material for applications where greater flexibility and shape morphing capabilities are desired. Most of the applications require SMAs to function reversibly and repeatedly for a large number of cycles. Unfortunately, traditional SMAs are prone to functional fatigue, i.e. after a few cycles, SMAs lose their superelastic and shape recovery abilities. Consequently, the quest for synthesizing highly reversible SMAs has been at the forefront of SMA research in the recent times.

SMAs owe their properties to the material undergoing a martensitic phase transformation between two crystalline phases which is accompanied by the formation of a rich microstructure in the martensitic phase. The emerging microstructural features are successfully explained by the geometrically non-linear theory of martensite, which views the microstructure as a configuration of compatible strains that minimize the elastic energy. The reversibility of the transformation is governed by the mechanical compatibility between the transforming phases. The strongest conditions of mechanical compatibility between the two phases known till date are the cofactor conditions, which is a set of relations between the lattice parameters of the two phases. These conditions allow the formation of elastically stress free interfaces between the two phases, which can move back and forth through the crystal to facilitate the transformation. Alloys that have been tuned to satisfy the cofactor conditions have shown extremely low transformation hysteresis and high reversibility. Motivated by the cofactor conditions, we propose a new set of conditions which expand the possibilities of formation of stress-free interfaces between the two phases and surpass the limitations of the cofactor conditions. Additionally, we showcase some theoretically predicted microstructures achievable under the new conditions and provide insights into how they may result in enhancing the shape recoverability of the material. The suggested conditions can serve as guidelines for alloy development and lead to the discovery of novel SMAs with unprecedented functionality.

# Fracture Behaviour of Homogeneous Rocks in Compression: Insights from Discrete Element Method Analysis

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## ABSTRACT

Understanding the strength and deformability of rocks is crucial for the design, construction, and stability assessment of slopes, foundations, and underground excavations in civil and mining engineering. However, it is impossible to simulate all the natural conditions in the laboratory due to operational and instrumental constraints. Therefore, numerical modelling of rock fracturing is essential to acquire the required detailed insights. Considering the crystalline nature of the rock grains, one suitable numerical modelling approach is utilizing discrete element methods (DEM). DEM has the capability to explicitly represent both fracture systems and grain geometry. In DEM, deformable blocks are discretized into finite-difference tetrahedral elements, referred to as finite-difference zones, to incorporate internal deformation within each block. In view of the above, the current study aims to investigate the fracturing and damage evolution of rock (i.e. quasi-brittle material) at the grain scale through the discrete element method. A three-dimensional numerical model, based on Voronoi tessellation, is established to evaluate the compressive failure of rocks under uniaxial stress condition. The model is developed incorporating realistic geometrical and mechanical data obtained from literature. Furthermore, a tensile softening contact model is employed, utilizing randomly generated polyhedral geometries to investigate the post-peak behaviours and failure pattern. The model was validated through a series of micro-parameter adjustment and comparing the resultant stress-strain behaviour with the literature data. Further parametric studies revealed insights related to the effect of cohesion, internal angle of friction on the fracture behaviour of sandstone under various ambient conditions.

**Keywords:** Sandstone, DEM, Voronoi Tessellations, Fracture, Stress-Strain curve

# Unraveling the Complexities of Concrete Creep: Microstructural Insights and Analytical Modeling

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## ABSTRACT

Creep, a significant contributor to the gradual deterioration of concrete structures over time, remains challenging to predict accurately despite extensive research. Its unpredictability stems from the complex interplay of concrete properties, loading conditions, and environmental factors. Furthermore, the fundamental mechanism of creep remains incompletely understood, with various theories proposed by eminent researchers lacking unanimous acceptance. Nonetheless, the scientific community generally agrees that creep originates at the micro-level of cement paste. Recent advancements in characterization techniques have improved our understanding of cement paste microstructures, paving the way for promising modeling techniques to estimate macro-level creep. Analytical methods based on physics, capable of simulating micro-level phenomena accurately, have gained popularity. These models consider interactions among various shapes of cement hydrates and their pore structures at micro and nano scales. This study investigates the impact of cement-hydration product shapes at the micro and nano levels on macroscopic creep properties using micromechanical analytical modeling in MATLAB. The model, utilizing Eshelby theory and continuum micromechanics homogenization approaches, predicts that increasing the aspect ratios of hydrate shapes leads to a reduction in macroscopic creep in cement paste.

**KEYWORDS:** *Cement, creep, multiscale modeling, continuum micromechanics*

# Effect of substrate surface roughness and charging conditions on hydrogen embrittlement susceptibility of dual phase steels using electrochemical permeation technique

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Electrochemical hydrogen (H) permeation technique using the Devanathan-Stachurski method is widely used to calculate effective diffusion coefficient of hydrogen as well as reversible trap density in steel using successive rise and decay transients thereby providing important data to evaluate hydrogen embrittlement susceptibility. Critical to such measurements is the establishment of a steady-state hydrogen permeation flux on the hydrogen detection side for a given charging current density on the hydrogen entry side. In this work, it is shown that hydrogen entry side substrate surface roughness, H-charging current density value as well as electrodeposited nickel coating thickness on the H-detection side are critical to achieve this constant steady-state hydrogen permeation flux on the detection side. Hydrogen entry side steel substrate roughness severely affects the critical diameter of the molecular hydrogen gas bubble formed by recombination of atomic hydrogen, before desorption from the steel surface that determines the constant hydrogen permeation flux on the detection side. Corresponding chronopotentiometry measurements on the hydrogen charging side show visible serrations in the entry side potential vs time due to hydrogen bubbles causing Ohmic potential drops that dynamically influence the steady-state hydrogen permeation flux on the detection side. Further, it is shown that a given H-charging current density value that although provides a steady-state hydrogen permeation flux can still cause embrittlement by generation of dislocations which decreases effective hydrogen diffusion coefficient as observed from increasing time lag for successive permeation transients. Hydrogen permeation results using vastly different microstructures such as dual phase ferrite/martensite, ferritic (interstitial free) and martensite steels with corresponding contrasting surface defect densities are shown to highlight the role of H-charging parameters and H-entry surface roughness needed to achieve steady-state permeation flux measurements. These results are relevant to establish a framework for improving the current ASTM G148 [1] standard for reliable measurement and interpretation of electrochemical H-permeation data.

## Acknowledgment

This work was funded through Startup Research Grant (SRG), No. SRG/2021/000001, Science and Engineering Research Board (SERB), India. The authors thank Mr. Sanjay Manda and Prof. Indradev Samajdar, IIT Bombay, for their assistance in using the EBSD facility. The authors also thank PD Dr. Michael Rohwerder, Max-Planck-Institut für Eisenforschung GmbH, Düsseldorf for fruitful discussions.

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# A comprehensive Review of Numerical Models to study Volumetric Creep of Soft Clays

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## ABSTRACT

Soft clays are considered as problematic soils because of their low shear strength, high compressibility, high water content and high plasticity. Time-dependent deformation known as secondary consolidation or creep is significant in case of geomaterials like soft clays. Ignoring the contribution of secondary consolidation towards total settlement of the geomaterial would result in significant errors in the design. In case of embankments, which is an inevitable part of the transportation infrastructure, time-delayed settlement is a major concern when constructed over soft clays. The post-construction settlement due to creep under the embankment load must be quantified to understand the load transfer mechanism and overall stability, and neglecting the creep component will result in an underestimation of the settlement characteristics. Several constitutive models have been developed to study this time-dependent behaviour of soil including empirical models, rheological models, and general stress-strain models based on visco-plasticity theory. Because of the adaptability to be implemented in a finite element framework, visco-plastic models are more commonly adopted. This article reviews different constitutive models that have been developed to study the volumetric creep behaviour of soils including isotropic models, anisotropic models, isotache concept, etc. As experimental determination or field measurements may not be feasible in many cases, these numerical models will be practical. The article discusses different model parameters, determination of these parameters, also the capabilities and limitations of these models in capturing the time-dependent behaviour.

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# Three-Point Bending Analysis of Mixed-Mode Crack Growth in Functionally Graded Materials: An Adaptive Phase Field Approach

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## ABSTRACT

Functionally graded materials (FGMs) are advanced materials designed with a gradual transition in composition, microstructure, or characteristics, enabling seamless shifts from one material to another. As FGMs gain prominence across various industries, understanding and predicting fracture behavior become crucial for design considerations [1]. Depending on their composition and microstructure, these materials have different critical energy release rates under different modes. The material's fracture behavior may be affected by this variation [2]. Phase-field fracture modeling (PFM) is a popular method for predicting fracture, as it helps to track the morphology, branching, and propagation of arbitrary cracks. The practical implementation of the PF technique is often hindered by the requirement of highly refined mesh to capture small-length scale values accurately. By adaptively refining the mesh when required, this problem can be addressed. In this paper, the adaptive refinement is carried out based on a threshold value of the phase-field variable. The simulation begins with a fairly coarse element discretization, and a fine discretization develops near the crack path, while the area far from the crack retains coarser discretization. The mesh refinement is carried out using quadtree decomposition, and the elements with hanging nodes are handled as  $n$ -sided polygons. The adaptive approach improves computing efficiency by significantly lowering the number of elements while maintaining solution accuracy. This study provides insight into how the critical energy release rate ratio affects the fracture behavior of Poly-Methyl Meth-Acrylate (PMMA) specimen subjected to three-point bending using an adaptive PFM. The results show that the peak load on the load-displacement graph changes noticeably, and the fracture path remains unaltered despite variations in the energy release rate. Similarly, the difference in crack propagation path and the stiffness change in load-displacement behavior is observed by varying the location of the applied load and the crack with respect to the distance between supports. This comprehensive examination enhances our understanding of mixed-mode crack propagation in FGMs, offering valuable insights to improve analysis and design methodologies for such materials.

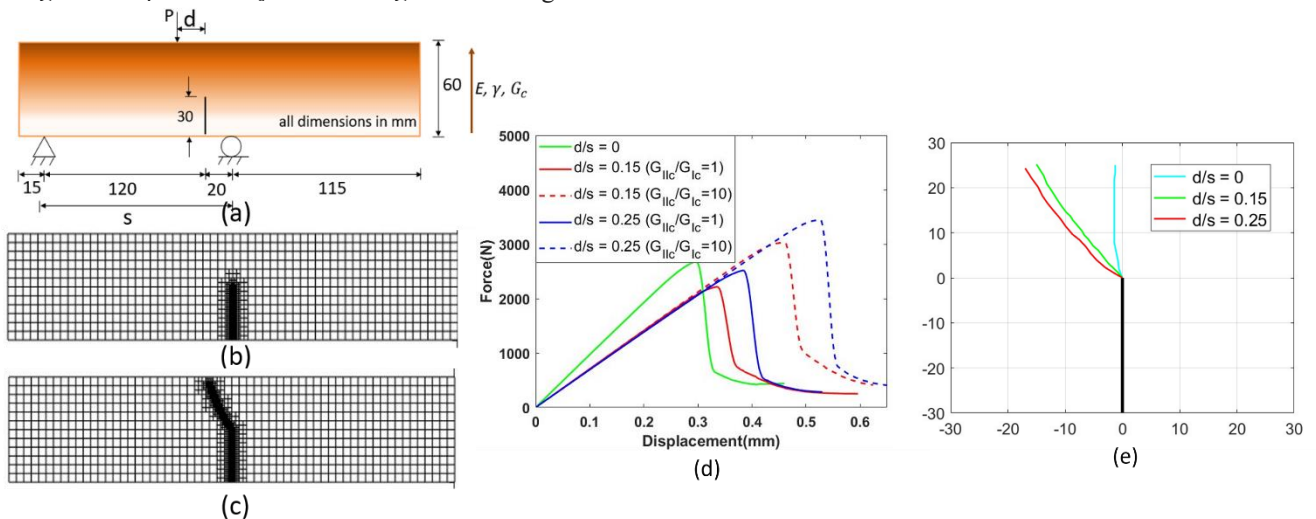


Figure: (a) Geometry and boundary conditions of PMMA specimen, (b) adaptive mesh at initial and (c) final stage, (d) load-displacement graph, and (e) comparison of crack path.

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## Study of effect of high temperature and specimen dimensions on fracture toughness of silicate glass

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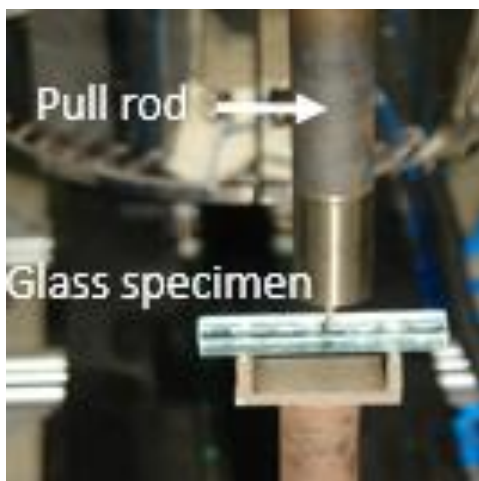
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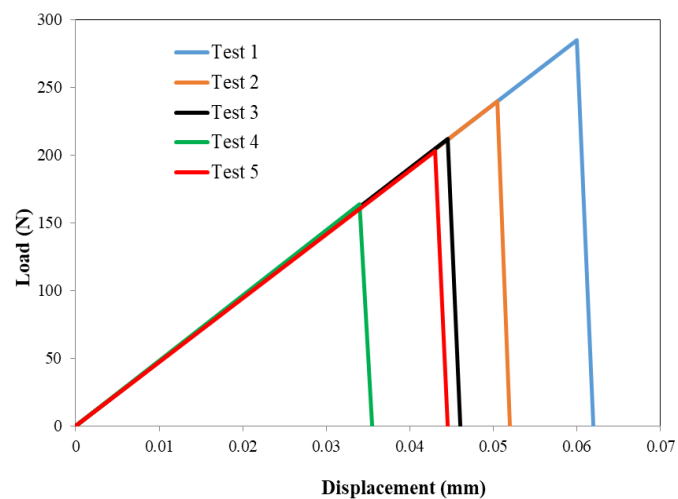
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### ABSTRACT

Silicate glass possess excellent chemical resistance and chemical stability. Silica glass can be used at high temperatures around 300°C due to its high deformation temperature. These materials are brittle at room temperature, however, their high temperature fracture properties need to be developed. The fracture strength of glass follows a statistical distribution. Generally, the fracture strength follows Weibull model due to its extreme event of fracture of glass [1-2]. In this work, different specimens have been prepared using suitable techniques to evaluate the fracture toughness of glass at different temperature. Three point bend tests have been carried out on different thickness of glass in order to understand the effect of thickness, span and width of specimen on fracture toughness of glass material. Annealing of the glass samples procured through market has been carried out in order to determine the effect on annealing on fracture toughness. Issue related to the annealing is discussed in [3]. Effects of annealing and changes in stress state on fracture toughness of bulk metallic glass is obtained in [4]. Test data shows a large scatter in fracture properties of the glass material. Statistical analysis of fracture toughness has been carried out to evaluate the parameters of Weibull model. It was seen that as the annealing of glass specimens were carried out, there is a corresponding reduction in the fracture strength of glass. This is due to the inherent stresses present inside the glass during its fabrication. Effect of annealing is predominant at room temperature as compared to 300°C. It has been observed that with the increase in temperature, the fracture toughness of the glass material increases. The temperature dependence of fracture toughness has been obtained. These data shall be useful in design studies related to glass material.



(a)



(b)

**Fig.1:** (a) Specimen loaded on the machine (b) Load-displacement data obtained from testing of 4 mm thick glass samples.

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# A deep learning approach to model coarsening with Cahn-Hilliard equation

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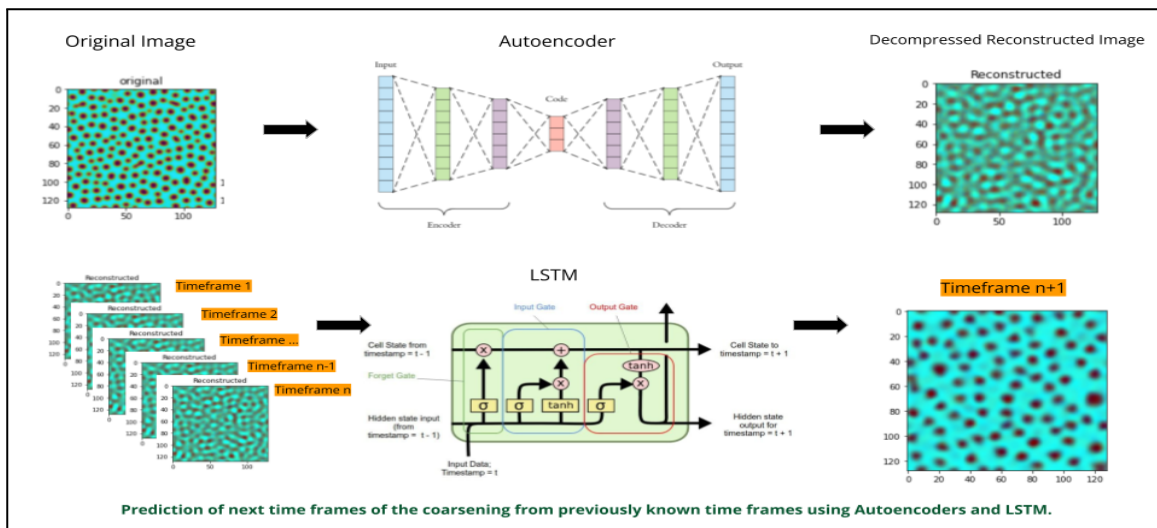
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Modeling microstructure evolution and coarsening is essential for optimizing material properties and designing advanced materials. The Cahn-Hilliard equation is the widely accepted derivative method to simulate these processes [1, 2, 3]. The equation takes into account parameters like the mobility of interfaces, diffusion coefficients, and local composition to predict the morphological changes that occur during coarsening. One of the shortcomings of phase field modeling is a high computational complexity and a large number of boundary conditions that need assumptions.

In this work, a deep learning approach is applied to the Cahn-Hilliard equation to model coarsening. Initially the phase separation and coarsening are modeled using Fourier transform and Cahn-Hilliard equations. The deep learning model is applied to predict the microstructure evolution at larger timescales that are computationally expensive in conventional modeling. The deep learning architecture is designed to effectively capture the spatio-temporal correlations and features in the phase field images. A combination of Autoencoders and LSTM (Long Short Term Memory) can be employed, where the Autoencoder extracts spatial features from individual phase field images, and the LSTM captures temporal dependencies by modeling the sequence of images. The model is trained to predict the next time frame of the microstructure evolution given the current state.

The deep learning model developed that is trained and validated, can be further utilized to predict the coarsening behavior of unseen microstructures. Given an initial microstructure, the model can generate a sequence of predicted microstructure states that can be compared with experimental observations or simulations from well established models to validate the accuracy and effectiveness of the deep learning approach, extending its applicability to a wide range of materials problems.



## Acknowledgements

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# Microstructure and high temperature tensile deformation of reduced activation ferritic steel (Fe-15Cr-2W) processed by mechanical alloying and powder forging

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## ABSTRACT

Reduced activation ferritic steels are considered as potential candidates for nuclear reactors cladding tube. In the present study reduced activation ferritic steel of composition Fe-15Cr-2W was prepared by powder metallurgy route. Mechanically alloyed powder was produced by using high energy attritor mill (Simoloyer) followed by hot powder forging at 1200°C in H<sub>2</sub> atmosphere. X-ray diffraction and TEM analysis confirms complete dissolution of alloying elements after 10h of mechanical alloying. The forged alloy was tested for mechanical properties (yield strength, ultimate tensile strength, and elongation) at room temperature and elevated temperatures (600°C and 700°C). The yield strength (YS) and ultimate tensile strength (UTS) at room temperature (RT) were found to be 532 MPa and 944 MPa respectively and the total elongation (TE) was 10%. Testing at elevated temperatures manifested a reduction in YS and UTS along with an increase in ductility. At 600°C, the YS and UTS were found to be 495 MPa and 920 MPa respectively and the TE was 12% while at 700°C, YS and UTS were reduced to 258 MPa and 354 MPa respectively and TE increased to 25%. This change in strength and ductility at elevated temperatures is attributed to the change in the deformation mechanisms from athermal dislocation glide to thermally activated viscous creep. Fractography of tensile tested samples at room temperature reveals mixed type of fracture, changing to that of ductile nature at elevated temperature, indicated by fine and coarse dimples present on the fractured surface.

## Acknowledgments

The authors are grateful to Science and Engineering Research Board (SERB), Dept. of Science and Technology, Govt. of India for providing financial assistance to carry out research under the grant No. SER-1618-MMD (CRG/2020/004572).

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# **Predicting Mechanical Properties of Hemp Fiber Reinforced Composites Across Ambient Temperatures: Integrating Experimental and Numerical Approaches**

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## **ABSTRACT**

The global shift towards sustainability is fueling increased interest in the utilization of bio-based natural fiber-reinforced composites. This study specifically concentrates on investigating the properties of natural fiber-reinforced composites across various ambient temperatures, ranging from normal to the glass transition temperature of the hosting medium. Hemp fiber is selected as the reinforcement material, and a polyester matrix is chosen as the matrix material, given their preferences in automotive applications. Additionally, a nano carbon filler is incorporated into the composite. In this research, experiments are conducted to predict the tensile strength, Young's modulus, and elongation of the hybrid composite at room temperature. Simulation studies and a machine learning regression approach are employed to further forecast the behavior of the prepared composite, aiming to generate a Master curve for the tested composites. Leveraging time-temperature superposition methods allows estimating the material behavior at desired temperatures based on data from tensile strength and time of failure at different temperature exposures. For comparative analysis, composites are prepared both with and without the infusion of nano-reinforcement. The present work plays a crucial role in the effective design of natural fiber-reinforced composites under different ambient temperatures. The goal is to further expand the application of these sustainable materials.

Keywords: Natural fiber reinforced composites, tensile strength, tensile modulus, elongation, ambient temperature.

## Thermal stability of $\beta$ -NiAl bond coats with Reactive Elements addition

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### ABSTRACT

High melting intermetallic such as  $\beta$ -NiAl has gained significant attention in the last six decades.  $\beta$ -NiAl has found its application in Gas turbine blade as an interlayer in between the Ni-Superalloy and Ceramic top coat. This interlayer is known as bond coat material, mainly used to improve the oxidation resistance of the base Superalloy. It has been experimentally observed that  $\beta$ -NiAl lowers the rate of oxidation. However, a minor addition of certain reactive elements (such as Hf, Zr, Y, Ce etc.) to  $\beta$ -NiAl has shown massive improvement in oxidation resistance thereby increasing the operating temperature of turbine blades. This results in a so-called “Reactive Element Effect”. The available kinetics data for oxide growth justify these observations. However, the mechanisms proposed to explain this effect is still not much clear and it needs further investigation.

From practical aspects, observing the thermal stability of  $\beta$ -NiAl with increase in temperature is our prior objective in this study. To examine the thermal stability,  $\beta$ -NiAl samples along-with reactive elements are synthesized using Vacuum arc melting under Argon atmosphere. Heat treatments are performed in vacuum furnace and the samples are characterized using X-ray diffraction (XRD), Scanning electron microscopy (SEM) and Energy dispersive spectroscopy (EDS) to observe the Reactive Element Effect on the grain size, morphology and phase stability of  $\beta$ -NiAl.

Keywords: Thermal stability, Turbine blade,  $\beta$ -NiAl, Reactive Element Effect, Heat treatment

# Prediction of Fatigue Crack Growth Behavior at Elevated Temperatures in Nickel based Super-alloys using Machine Learning

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## ABSTRACT

Materials exposed to elevated temperatures in applications such as heat exchangers of nuclear reactors, gas-turbines, jet engines, oil refineries and so on, are expected to retain their structural integrity while performing their intended task, that is, designed for 'damage tolerance'. The essential requirement of a damage tolerant design in such applications is to establish the fatigue crack growth rates (FCGR) at elevated temperatures. Performing FCGR tests at elevated temperatures is a tedious and expensive task adding to the cost of the product itself. Efforts are in place amongst the scientific community to minimize repetitive tests by using numerical techniques of predicting the FCGR, without compromising safety. This implies that the accuracy of prediction needs to be extremely good and consistent. The present work explores the use of machine learning (ML) technique for the prediction of fatigue crack growth behaviour in Nickel based super-alloys at varying elevated temperatures. The algorithm used for the proposed study is an artificial neural network (ANN) that takes in various physical and material properties as 'input' to generate the entire FCGR curve at a given temperature and load ratio. The correlation is developed by, training the algorithm with experimental data of similar materials from literature. The hyper-parameters of the algorithm are optimized during the training process to achieve the best possible generalization and prediction accuracy. The algorithm is validated by performing multi-fold cross validation and subsequently testing it on a completely new set of data. The predicted FCGR curve in the present work is in close correlation with that of the experimental results available in the literature. Incorporating the machine learning and deep learning techniques improves the prediction accuracy while providing easy and quick solutions to otherwise expensive and time-consuming repetitive tasks. However, these techniques are rapidly evolving and improving, and hence are not an alternate to the experimental assessment of structural integrity.

## Acknowledgments

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## **Room Temperature and High Temperature Fatigue Behaviour of Additively Manufactured Hybrid XH67\_IN718 via Micro-scale Fatigue Testing**

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### **ABSTRACT**

Additively Manufactured alloys are known to possess sufficient tensile strength and ductility combination, matching their conventionally processed counterparts, and yet fail to perform under cyclic loading conditions. Assessment of fatigue behavior of these alloys, especially during alloy development stage, necessitates small volume fatigue testing, to replicate component geometry. The study includes a thorough microstructural evaluation of a new Hybrid IN718-XH67, manufactured by laser powder bed fusion (LPBF) for both as-printed and heat-treated conditions. The standard AMS 5662 heat treatment comprises solution annealing, involving heating at 980 °C for 1 hour followed by air cooling. Subsequently, aging takes place at 720 °C for 8 hours. The cooling process in a furnace follows, descending to 620 °C at a rate of 50 °C/h, where the alloy is held for 8 hours. Finally, the alloy undergoes air cooling to reach ambient temperature. Tensile property analysis is conducted to determine the hybrid alloy's mechanical strength in its as-printed state, with subsequent evaluation of heat treatment condition, both at room temperature and elevated temperature. Furthermore, the fatigue behaviour of the hybrid alloy is systematically investigated at 0.95% yield strength. Results will be discussed in the context of observed failure mechanism, tensile properties and the hierarchical microstructure.

# Fracture Behavior of Additively Manufactured SS316L under Fatigue Loading with Miniature Specimen: Effect of Microstructural Hierarchy

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## ABSTRACT

Additive manufacturing (AM) has opened up new possibilities for manufacturing metallic components involving complex geometry or tailored microstructure. However, AM results in anisotropy of properties due to the directional and cellular nature of microstructure, along with defects such as micro-porosities, quenched-in-dislocations or solidification cracks, offering unique opportunities and challenges. The effect of molten pool boundaries, grain boundaries and cell boundaries as obstacles to dislocation motion at different hierarchically shrinking length scales leads to a complex fracture behavior, upon cyclic loading. Low cycle Fatigue life of Laser Powder Bed Fusion (LPBF) processed Stainless Steel 316L (SS 316L) were investigated in the plastic strain regime, to discern these effects in an accelerated manner. Miniature tensile Specimens were machined along the build direction using electro-discharge machining (EDM). Microhardness, tensile tests and fatigue tests were conducted using digital image correlation (DIC) for full field strain mapping and high-throughput testing under an optical or scanning electron microscope. Specimens were tested at room temperature in their as-built condition as well as after vacuum annealing at 950° C and 1200° C for 3 hrs., which led to changes in grain morphology and different extents of recrystallisation and grain growth. The microstructure is investigated post fatigue loading to determine the origin of damage nucleation and evolution. The cyclic stress-strain response along with the associated hysteresis is discussed with respect to the microstructure and damage evolution, leading to final fracture.



## **Constitutive modeling of materials' cyclic hardening and softening behavior for steel under low cycle fatigue**

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### **ABSTRACT**

The austenitic steels, like 304L SS, 321 SS, 316L SS, etc., when undergoing strain-controlled low cycle fatigue (LCF), exhibit some or all of the cyclic features like initial hardening, softening, saturation, and secondary hardening. The conventional Chaboche's non-linear isotropic and kinematic hardening laws are modified to predict the changes in isotropic and kinematic hardening laws evident with cycling (throughout life) and strain amplitude. Thus, a universal constitutive model is proposed according to the analysis of cyclic deformation to capture the changes in loop shape and peak stress. The results are shown here, with the low cycle fatigue test data of 304L SS and 321 SS for strain amplitudes performed at room temperature at a constant strain rate of  $1 \times 10^{-3} \text{ s}^{-1}$ . The agreement between experimental data and simulated results suggests that the proposed model works well in predicting cyclic hardening-softening followed by secondary hardening or softening-hardening behaviors shown by materials. Thus, it is believed that the methodology can be used in modeling the complex material behavior irrespective of the sequence of the cyclic features exhibited. Moreover, the fatigue life predicted based on the cyclic plastic strain energy density, estimated from the simulated hysteresis loops, lies within a scatter band of  $\pm 1.5$ .

## **Microstructural Evolution and Creep behavior of Additively Manufactured near $\alpha$ Ti6242 Titanium alloy**

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### **ABSTRACT**

The recent advancements in additive manufacturing (AM) process to produce a complex/near-net-shape components, especially for aerospace structural applications gained a lot of attention. Among various Ti-alloys developed for high-temperature applications, near- $\alpha$  Ti-6Al-2Sn-4Zr-2Mo (Ti6242) alloy has been an important one for aeroengine applications due to its higher creep resistance. In this study, we investigate the microstructural evolution and its effect on the room temperature and elevated temperature (550 °C) tensile and creep properties of Ti6242 alloy fabricated via laser powder bed fusion. The post-process of Hot Isostatic Pressing (HIP) treatment (above  $\beta$  transus temperature  $T_{\beta} \sim 1020$  °C) results in the  $\beta$  grain refinement and the formation of  $\alpha$  lath in prior  $\beta$  grains. In addition, baseline heat treatment leads to the formation of very fine  $\alpha$  lath in prior  $\beta$  grains ( $V_f$  of  $\beta$  phase  $\sim 4$  to  $8$  %), which gives a high yield of  $986 \pm 3$  MPa and  $578$  MPa with elongation to fracture of  $22 \pm 2$  % and  $29$  % at room temperature and  $550$  °C, values are par to those wrought counterpart as well as reported LPBF Ti6242 alloy. The alloy exhibits an excellent creep-resistant behavior when tested at  $550$  °C at a stress range of  $300$ - $400$  MPa. In addition, the correspondence of uniaxial creep equivalence with bending creep was also established. Detailed electron microscopy results will be presented for the tensile and crept samples to correlate deformation and microstructural features that elucidate the mechanisms.

### **Acknowledgements:**

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# Effect of Static and Cyclic Loading on the Stability of Retained austenite in Martempered SAE9254 Steel

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## ABSTRACT

The surge in demand for electric motor vehicles, driven by stringent emission reduction mandates for automotive suppliers, necessitates advancements in the sag resistance of suspension coil springs. Despite this imperative, the mechanisms underlying sag loss in spring steel coil springs remain elusive. Previous studies on spring steels suggest the involvement of phase transformation and load partitioning between martensite and austenite during deformation. This study delves into the impact of loading on the stability of retained austenite ( $\gamma_R$ ) in martempered spring steel, particularly in advanced high-strength spring steels. Microstructural and mechanical analyses, encompassing X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM) with electron backscatter diffraction (EBSD), as well as tensile and fatigue testing, were employed. Martempering heat treatments are applied to the spring steel wire to discern the effects of loading on  $\gamma_R$  stability. Tensile tests are conducted with interruptions at various strains, employing an exceedingly slow strain rate proximal to that of creep. Our findings from interrupted slow strain rate (ISSR) tests reveal that  $\gamma_R$  transforms well below the material's yield strength (YS), providing a reference for subsequent fatigue testing. Fatigue tests are conducted at 35% of the YS and interrupted at different cycles (ranging from 10 to 10,000 cycles) to scrutinise the seldom-explored impact of cyclic loading on  $\gamma_R$ . The results offer valuable insights into the relationship between loading conditions and the stability of  $\gamma_R$ , thereby contributing to a deeper understanding of sag resistance in coil springs. This research not only addresses the current gap in knowledge regarding the mechanical behaviour of advanced high-strength spring steels but also underscores the significance of considering loading effects on  $\gamma_R$  stability in the pursuit of developing resilient suspension systems for the evolving landscape of electric vehicles in the automotive industry.

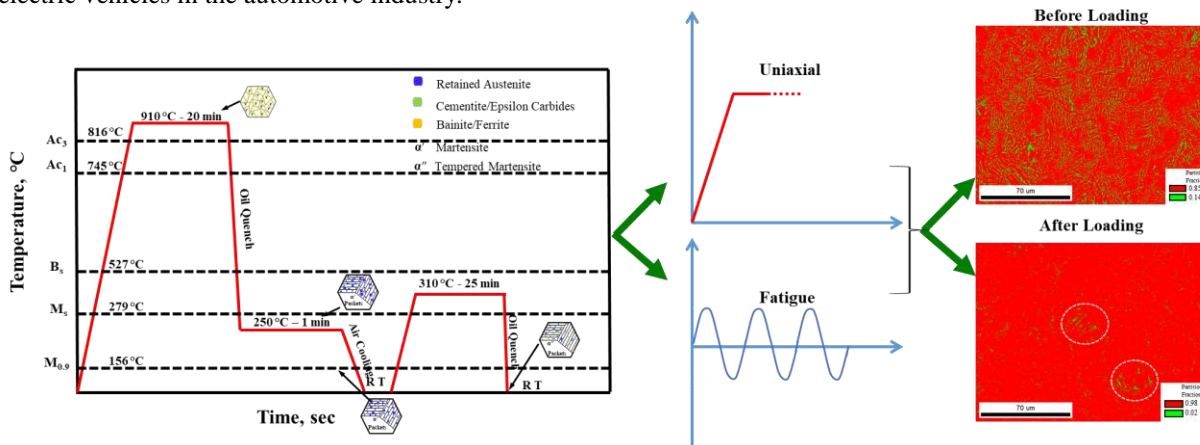


Figure 1: Heat treatment cycle utilised for martempering heat treatment and effect of loading on the microstructure.

## Acknowledgements

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# A high-throughput approach to decipher the dynamic deformation behaviour of FCC-Copper

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## Abstract

Dynamic deformation of materials comprising deformation at a high strain rate ( $\dot{\epsilon} > 1 \text{ s}^{-1}$ ) is a comparatively less explored field in mechanical behaviour of materials compared to that of quasistatic deformation. In addition to providing fundamental insights into dislocation processes as well as offering different deformation mechanisms, dynamic deformation of different classes of materials is important for technological applications. The response of materials for experiments in the dynamic regime is completely different from the response in the quasistatic regime due to strain rate hardening and activation of additional deformation mechanisms like twinning, phase transformation, shear banding, and amorphous phase formation. Hence, it is very important to perform controlled experiments under dynamic and quasistatic regimes to obtain the constitutive behaviour of materials over a wide strain rate regime. This includes performing experiments in different modes of loading with constant strain rate, strain, temperature, and microstructure. Unlike quasistatic testing using a universal testing machine, dynamic deformation is studied using the split Hopkinson pressure bar apparatus for loading in compression, tension, and torsion. The SHPB technique developed for testing in the strain rate regime of  $10^2 - 10^4 \text{ s}^{-1}$  has inherent complexity as it is based on the one-dimensional propagation of elastic waves and it is very difficult to control the strain rate, strain, and even temperature (due to adiabatic heating). Nevertheless, multiple studies have been performed over a wide range of strain rates, strain, and temperatures to obtain constitutive responses of different classes of materials. Most high-throughput mechanical testing facilities like nanoindentation, small punch tests, micro-pillar compression tests, and in situ testing focus on the quasistatic regime while the similar high-throughput testing facilities are missing in dynamics regime.

The current work focuses upon studying the dynamic deformation behaviour of FCC-Copper in a high-throughput manner by using a split Hopkinson pressure bar, digital image correlation setup with a high-speed camera, nanoindentation and electron back scatter diffraction facility. Efforts will be directed to design specimens with suitable geometry to obtain different levels of strain rate and strain in the same sample. This will be followed by extracting the stress-strain response of the material from different regions to obtain the constitutive response of the material and this will be coupled with a detailed microstructural characterization to establish the microstructure-property linkages. The results obtained will then be matched with the constitutive response obtained from conventional testing to validate the fidelity of our high-throughput approach.

# Understanding deformation mechanisms in the Hall-Petch breakdown regime of Ni-W alloys

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## ABSTRACT

The substantially higher strengths of nanocrystalline metals has been attributed to the well-known Hall-Petch relationship  $\sigma = \sigma_o + k_{HP}d^{-1/2}$  where  $\sigma$  is the yield stress,  $\sigma_o$  is the lattice friction stress,  $d$  is the grain size and  $k_{HP}$  is the Hall-Petch constant. The Hall-Petch scaling breaks down at grain sizes below  $\approx 10$ nm. The dominant deformation mechanism in the Hall-Petch breakdown or grain boundary weakening regime are not well-understood, with weakening being attributed variously to diffusion creep, grain rotation, and grain boundary sliding. Typically, specimens with the finest grain sizes are annealed to obtain the coarser grain sizes, and the influence of such annealing on strength is not clear. Several studies have attributed the grain boundary weakening regime to enhanced grain boundary segregation or reduced internal stresses caused by annealing.

This study focuses on characterizing the dominant deformation mechanisms in the Hall-Petch breakdown regime. The Ni-W system was chosen as the model alloy, where W is known to segregate at the grain boundary. The Hall-Petch behaviour was studied using nanoindentation hardness measurements, and the hardness observed in Ni-W alloys (3-15 %W) ranged from 6 to 15 GPa for grain sizes from 29 nm down to 3 nm. Solute segregation was studied extensively to understand the role of solute chemistry at the GB, using Atom Probe Tomography (APT) experiments.

Three alloys with 3, 8 and 15 at% W were studied. Different grain sizes were obtained at a constant W content by annealing at various temperatures. A clear Hall-Petch breakdown was observed in the three compositions. Measurements revealed that solute segregation did not increase upon annealing, so that the GB weakening regime cannot be attributed to variations in segregation.

Annealing introduces multiple microstructural changes simultaneously and it is difficult to separate the contribution from each process. Four alloys with W content  $19 \pm 1$  at% and grain sizes ranging from 8 to 3 nm were synthesized by slightly modifying the deposition conditions, without annealing. A clear grain boundary weakening regime was observed, suggesting that the grain boundary weakening regime is not related solely to annealing effects.

## **Tailoring the high temperature micromechanical behavior of Inconel 718 alloy using significant microstructural modification**

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### **ABSTRACT**

Inconel 718 is one of the widely accepted precipitate-hardened polycrystalline nickel based superalloys used in turbine engine materials where it withstands its properties at higher temperatures (~650 °C) at the exhaust regions exhibiting superior creep resistance, fatigue strength etc. due to the presence of interesting intermetallic precipitates such as  $\gamma'$  (gamma prime, Ni<sub>3</sub>Al/Ti),  $\gamma''$  (gamma double prime, Ni<sub>3</sub>Nb) providing strength to the material where  $\gamma$  (gamma, solid solution strengthening) matrix provides ductility to the material. But other brittle precipitates like  $\delta$  (delta, Ni<sub>3</sub>Nb), carbides, laves phase etc. shows unpredictable behavior at various temperatures effecting the mechanical strength and its performance.

This project focuses on understanding evolution of various precipitates at elevated temperatures and investigate new heat treatment strategies to get the balance between ductility and strength at higher temperatures increasing the upper limit of its service temperature (~650 °C). The broader outcome of this project results in reduction of carbon emissions from aviation sector as the superalloy's service temperature gives way to choose for higher turbine outlet temperature thereby increasing combustion efficiency effecting the content of exhaust gases.

The methodology includes performing various heat treatments on wrought Inconel 718 to tailor the high temperature micromechanical behavior of the alloy by optimizing the size, volume fraction, concentration and distribution of the metallic precipitates, conduct mechanical tests like fatigue creep, high cycle fatigue, uniaxial tension test with different strain rates at both room temperature and at elevated temperatures and observe the microstructural features of the material to compare and analyze the mechanical properties.

Finally, concluding the modified heat treatment techniques succeeded in obtaining a substantial integrity in microstructural features of the material by which certain percentage of the mechanical properties are enhanced. In the later stages, we include physics based experimentally informed micromechanical modeling of the material including temperature and orientation dependencies through UMAT and Abaqus platforms to simulate for complex real time boundary conditions and this model can be extended for other superalloys in future research works.

# Life Prediction of Annealed Titanium Extra Low Interstitial Alloy for Biomedical and Aerospace Application

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## ABSTRACT

The aerospace and biomedical industries both frequently use titanium alloys because of their exceptional combination of mechanical properties, corrosion resistance, and biocompatibility. One of these alloys, Extra Low Interstitial (ELI) titanium, is highly desired for critical applications because of its enhanced mechanical and chemical properties following a specialized annealing process.

This study examines the life prediction of annealed titanium ELI with a focus on its durability and long-term performance. The study uses a thorough strategy that includes extended finite element method, mechanical testing, and material characterization. This study aims to predict the service life in room condition, and simulated body fluid of annealed Titanium ELI components by analyzing the microstructural features, mechanical characteristics, and environmental factors.

The use of titanium ELI in the biomedical industry includes surgical instruments, dental prosthetics, and orthopedic implants. It is essential for aerospace structures, engine parts, and light, strong parts. It is crucial for both industries to guarantee the long-term reliability of these key elements.

A thorough life prediction technique that considers elements like corrosion resistance, fatigue test, fracture toughness and cyclic loading is used for the investigation. This model advances knowledge of the behavior of the material and aids in performance and safety prediction for biomedical and aerospace applications.

In conclusion, by giving a deeper understanding of material behavior in the room condition, and simulated body fluid environments, the study on the life prediction of annealed Titanium ELI for biomedical and aerospace applications is crucial for the advancement of these industries. The body fluid environment sample's having fracture toughness and life are less than the room condition sample. The experimental life of the body fluid environment specimen's shows significant degradation of component life.

## Acknowledgments

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## **Development of High-temperature DIC (up to 1800 °C) for strain measurements**

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### **ABSTRACT**

The demand for high-speed vehicles (above Mach 5) and energy efficiency is motivating the development and testing of new high-temperature materials. One of the major challenges associated with high-temperature mechanical testing is strain measurement. Here we have established a digital image correlation technique that can measure strain up to 1800 °C. Isothermal experiments are conducted on three high-temperature materials, Nb-C103, C<sub>f</sub>/SiC, and β-NiAl at temperatures up to 1800 °C. The testing is done in the air as well as in an inert environment. The standard deviation in the strain at isothermal conditions is within  $4 \times 10^{-4}$  which is well within the acceptable range. The coefficient of thermal expansion of C<sub>f</sub>/SiC is measured and validated with literature. The speckle paint is stable enough to run long experiments and the DIC system is flexible enough to be integrated with any mechanical testing system if the sample is accessible through a viewport.

Keywords : DIC, Strain measurement, High-temperature



# High-temperature deformation of CoCrNi multicomponent alloy consisting of hierarchical microstructure

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## ABSTRACT

Multicomponent alloys have given us the freedom to explore the unsearched phase field of alloy systems for better physical and mechanical properties. Moreover, tailoring the microstructure by introducing heterogeneity in microstructural features has led the materials community to build high-performance systems that can push working stress limits when in application. The subset of CoCrFeMnNi multicomponent alloy and its alloy composite have been investigated by researchers for their mechanical properties at various temperature ranges. In this context, the equiatomic CoCrNi alloy has demonstrated remarkable mechanical performance at elevated temperatures. Thus, the present work focuses on the creep deformation of the alloy with a hierarchical-phase microstructure based on the Co-Cr-Ni system. The alloy was processed via the melting route and thermomechanical processing techniques. The constant load creep tests were performed in the temperature range of 600–750 °C and at compressive stresses of 100–200 MPa. The insights into the creep deformation are brought up by evaluating the activation energy and the stress exponents. A comprehensive microstructural assessment, utilizing electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM), was carried out on crept samples to explore the micromechanisms and elucidate the role of interfaces present at various hierarchical levels.

Keywords: Multicomponent alloy; Hierarchical microstructure; Creep Deformation

## Understanding Phase transformation during creep deformation of extruded Magnesium-Rare Earth alloys

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### ABSTRACT

Light-weight Magnesium (Mg) alloys attract great attention for automotive applications, especially in power train components. It acknowledges the potential of rare earth elements, specifically cerium (Ce) and dysprosium (Dy), as alloying additions to enhance the mechanical properties of Mg alloys at both room and high temperatures. However, the precise role of these elements is not yet well understood, particularly regarding local phase transformations, solute segregation, defects-solute interaction, and site occupancy.

To address this knowledge gap, the study focuses on investigating the creep deformation behavior of extruded Mg alloys, namely pure Mg, Mg-0.5Ce, and Mg-8Dy-0.5Zr-0.5Ce (by wt.%), under compressive loading. The microstructural analysis of the alloys before and after creep is conducted using various techniques such as Optical Microscope (OM), Scanning Electron Microscopy (SEM), Electron Back-Scattered Diffraction (EBSD), Transmission Electron Microscopy (TEM), and Atom Probe Tomography (APT).

The results reveal the formation of metastable phases after the extrusion process, which remain stable during creep. Understanding these phase transformations is crucial for accurately predicting the performance of the alloys at high temperatures. The study also sheds light on the role of Ce and Dy and their influence on the deformation behavior of the alloy. TEM and APT observations indicate the segregation of Ce and Dy at interfaces, including grain boundaries and micro-twins induced by creep.

Overall, the obtained results provide valuable insights into the creep deformation behavior and microstructural changes of Mg alloys with Ce and Dy additions. This understanding is essential for optimizing the mechanical properties and predicting the performance of lightweight Mg alloys in automotive applications, particularly in powertrain components subjected to high-temperature conditions. The findings are discussed in relation to the measured creep properties of the alloy.

**Keywords:** Creep Deformation, Ce-segregation, Dy-segregation, Twins, HAADF-STEM, APT

## Bending Creep of Ti6242 at Room Temperature

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### ABSTRACT

Room temperature (RT) creep serves as the underlying mechanism responsible for Dwell Life Debit observed in Titanium alloys [1]. This phenomenon is characterized by a decrease in fatigue life resulting from the introduction of a dwell time at maximum load within an otherwise conventional cyclic loading. The present work delves into comprehending RT creep in Titanium alloy, Ti6242, under tension and compression at various stress levels. For studying power law creep, cantilever bending has been proven to be an instrumental high-throughput technique [2]. When a beam is loaded transversely at one end, a stress gradient emerges along its length and depth. Through bending, coupled with digital image correlation (DIC), it is possible to extract creep responses at various stress levels from a single sample. In a conventional uniaxial creep test, the stress (or load) remains constant. To account for this, in bending, creep strains are extracted from stress-invariant points. However, the creep strain accumulated at room temperature in titanium alloys is significantly smaller than the elastic strain and hence the stress redistribution can be neglected. In the light of this assumption, the creep strains can be extracted from any points. In Titanium, creep strains are significant only at stresses near the yield stress. Therefore, the beam must be bent in a manner that subjects the outermost fibres to a stress value close to the yield strength. Although, the strain values given by DIC at the edges of region of interest is not valid, they can be back calculated from the fact that a plane remains a plane even after bending. The deformation mechanisms responsible for RT creep are examined by slip trace analysis.

**Keywords:** RT Creep, Bending, Digital Image Correlation, Genetic Algorithm

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# Creep and stress relaxation in thin films: Experiments, modelling, and materials science-based insights for reliability improvement

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## ABSTRACT

Materials used in microelectronics and integrated circuits can be subjected to temperatures as high as 400 °C during their operation. Since these thin films are primarily deposited on silicon substrates and due to the significant difference in the thermal expansion coefficients of Si and the metallic films, they develop substantial stresses ranging from several hundreds of Mega-pascals to even Giga-pascals [1]. Under these conditions, the films tend to relax their stresses through creep, formation of hillocks, interfacial sliding, void formation, and fracture, which deteriorates the life of the thin film devices [2]. Hence, comprehending these stress relaxation mechanisms and trying to constrain them becomes essential. In this study, we performed stress relaxation studies on Aluminium thin films of 200 nm thickness from temperatures ranging from 200 to 400 °C. As the primary goal of this study is to correlate creep and stress relaxation, the effects of interfacial sliding were avoided using an interlayer of TiN of 20 nm thickness, and the hillock formations were minimised using a passivating layer of TiN of 20 nm thickness. This ensures that most of the stress relaxation occurs due to creep. These stress relaxation measurements were done using a custom-built laser curvature setup. These results are then utilised to predict the dominant creep mechanisms. These experimental results have also been backed by FEM simulations which show agreement with each other.

**Keywords:** Thin films; Stress relaxation; hillocks; passivating layer.

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## Small Punch Creep – correlation with uniaxial creep testing?

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### ABSTRACT

The Small Punch Creep (SPC) testing has been found to be quite useful testing technique since its inception. The requirement of lesser volume of material has been its unique selling point. However, in terms of interpretation of the results it has been quite challenging in comparison to its uniaxial counterpart, majorly due to its multiaxial stress state. To get meaningful information from this technique various attempts have been made in the past to establish a correlation between the SPC and uniaxial creep testing. These efforts have yielded some empirical relations correlating entities in from both the techniques [1], [2]. In the present study a comparison of SPC and Uniaxial test on Aluminum has been done at 300°C. It has been observed that the empirical relations do not accurately correlate the stresses and strains obtained in these two types of testing for Aluminum. FEM modelling was also done for the SPC testing using Norton power law as creep constitutive equation. The parameters for the Norton power law were obtained from the uniaxial creep tests. It was observed that the FEM results were an underestimate of the experimental results. Based upon these observations it was inferred that the correlation between these two techniques, if at all it exists, may not be quite straightforward and further different ways to approach this problem are discussed in this study.

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# High Throughput Bending Creep of Directionally Solidified AlCoCrFeNiTi Alloy

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This study introduces an innovative method for efficiently determining the creep properties of high-temperature materials through high-throughput testing, employing digital image correlation [1,2]. The focus is on the AlCoCrFeNiTi alloy [3-5], synthesized by directional solidification, known for its exceptional strength and unique properties. Experimental investigations, including compression, and bending creep tests, were conducted at 750°C.

Capitalizing on the inhomogeneous stress and strain distribution within a cantilever subjected to bending, we leverage this characteristic to extract multiple creep curves from a single test. Stresses at key points were determined using existing analytical solutions [5,6]. Uniaxial tests spanning 300 to 500 MPa initial stress were complemented by bending tests designed to induce similar stress levels. A detailed comparison between bending and uniaxial creep is presented, including the results of verification studies on additional alloys.

This methodology not only expedites testing but also minimizes material usage, energy consumption, and manual labour. This research showcases a reliable and time-efficient approach to exploring the creep behavior of high-temperature materials. The technique is particularly advantageous for characterizing precious alloys with limited dimensions. Microstructural heterogeneity may exist in specimens tested under bending load, however, it can still be correlated to the mechanical properties with modern high-resolution characterization methods. Stress and resulting strain can be directly compared in a single specimen, ensuring uniform manufacturing, and heating history. This method eliminates the possible errors due to testing with different rigs, which could impair the accuracy of studies based on individual tests.

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## Mechanical Properties of Grain Boundaries in Forsterite Bicrystals

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Understanding the rheological properties of olivine, the most abundant mineral in Earth's mantle, is crucial for unravelling the dynamics of upper-mantle convection. While extensive research has focused on olivine rheology, little attention has been given to the specific behavior of olivine grain boundaries, which play a key role in mantle deformation [1,2]. Current models describing grain-boundary deformation [3] are primarily phenomenological, making it challenging to extrapolate laboratory findings to natural conditions.

To address this gap, we conducted a series of deformation experiments on olivine (forsterite) bicrystals, where a single grain boundary was oriented for direct shear at atmospheric pressure and 1300 or 1400°C. Experiments were conducted in a uniaxial creep apparatus with resolved shear stress ranging from 1 to 30 MPa. The stress-strain relationship revealed a linear dependence (creep exponent,  $n$ , of  $\sim 1$ ) on applied stress, indicating that dislocation creep, commonly observed in single crystals and coarse-grained polycrystalline aggregates, was not the dominant deformation mechanism under these conditions. Direct observations of grain-boundary sliding in a geologic material were made for the first time (Fig. 1), however, the linear viscous behaviour measured was likely controlled by a documented chemical reaction between the olivine and the alumina pistons. EBSD mapping confirmed that both olivine grains in the bicrystal remained internally undeformed. Our results demonstrated that the bicrystal boundary is weaker than the forsterite grain interior, but the viscosity was dominated by the olivine-alumina reaction. The direct evidence of grain-boundary sliding was observed through fiducial scratch marker displacement in SEM (see Fig. 1). However, the strain geometry remains complex, necessitating further experiments to determine the overall strain distribution in the samples.

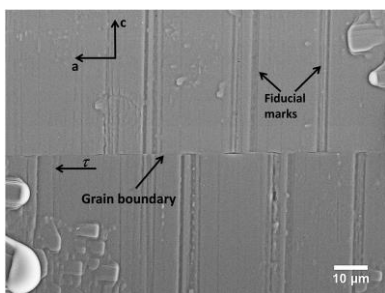


Fig. 1: Microstructure showing the displacement of fiducial marks across the grain boundary after creep conducted at 1400°C. The fiducial marks are indicated. The grain boundary is the horizontal line in the centre as indicated by an arrow. The crystal structure of forsterite is orthorhombic with different  $a$ ,  $b$  and  $c$  axes. Here  $a$  and  $c$ -axis are indicated. The direction of the shear was along  $a$ -direction indicated with  $\tau$ .

In summary, our research provides unprecedented direct observations of grain-boundary sliding in olivine [4], shedding light on the intricate mechanisms governing mantle deformation. The observed differences in creep behavior between bicrystals and traditional forsterite samples underscore the importance of considering grain-boundary effects in comprehensive rheological models of the mantle.

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## **Effect of Joint Thickness and Electric Current on Creep Behaviour of Cu-Sn Joints**

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### **ABSTRACT**

Tin (Sn) is widely used in microelectronics and electrical devices as a primary solder material due to its excellent solderability, good corrosion resistance and low melting point. The miniaturization of microelectronic chips has led to the use of miniature Pb-free Sn-rich solder joints in interconnects for 3D stacking of dies. However, downsizing the solder joint size has resulted in a reduction in the volume of compliant Sn and an increase in brittle intermetallic phases. The small size and constrained geometry of these microscale joints and the stiffness mismatch between the compliant Sn and rigid substrates limit their ability to deform. Furthermore, the distribution and orientation of anisotropic Sn grains and the size and distribution of second-phase precipitates vary as the joint thickness decreases. Therefore, the mechanical behaviour of bulk Sn cannot be directly applied to predict the response of miniature joints accurately.

This study investigates the effect of joint thickness on the creep behaviour of thin Cu-Sn joints. By using the Cu-Sn system, the variability due to microstructures can be reduced, and the effect of joint thickness on mechanical behaviour can be captured accurately. Results revealed that the creep resistance increased with the decrease in joint thickness, although there was no change in the creep mechanism from pure tin. The creep test on pure Sn in the presence of electric current was also performed to make the study more relevant for the microelectronics industry. But there was no significant change in creep behaviour in the test regime of electric current, and only joule heating due to electric current was responsible for the creep of Sn. Furthermore, the fractured surfaces were studied to get some scientific perspective of the creep behaviour of joints and Sn in different conditions.

The findings from this study provide valuable insights into the design and reliability considerations for Cu-Sn and SAC joints in electronic devices. Engineers can enhance the long-term performance and durability of electronic components by optimising joint thickness. Future research may explore additional factors that influence the creep behaviour of Cu-Sn joints, such as electric current and grain size, their number and orientations, to further advance our understanding of their mechanical properties and improve their structural reliability.



# Hydrogen embrittlement in non ferrous metals: A review

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## ABSTRACT

Metal damage caused by hydrogen has been referred to as hydrogen embrittlement (HE). HE is a well-known phenomenon in metals. However, the inherent effect of hydrogen in non ferrous metals on mechanical and corrosion behaviour has not revealed in detail. This review provides a thorough examination of the effects of hydrogen on the mechanical and corrosion behaviours of non-ferrous alloys, focusing on materials such as aluminum, titanium, copper, and magnesium. Special attention is given to the influence of microstructural alterations and internal crystal structures during HE. Furthermore, it addresses the occurrence of HE in advanced manufacturing processes for non-ferrous metals, highlighting its implications for industrial applications. Numerous mechanisms that causes to formation of HE in non ferrous alloys are discussed in detail. In addition to detailing the mechanisms, this review explores the preventive techniques designed to mitigate the onset of HE.

Keywords: hydrogen embrittlement, non-ferrous metals, mechanical behaviour, corrosion behaviour.

# NUMERICAL ANALYSIS OF J INTEGRAL FACTOR AND STRESS INTENSITY FACTOR FOR Al–SiC COMPOSITES.

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## Abstract

Man-made material that comprises more than an element with wide inconsistency in their properties such as physical, chemical and mechanical properties. Composite is a material which is lighter in weight, toughest and stronger than ordinary material. Materials used in current case are lagging with its mechanical properties, to enhance the properties of material they are reinforced with other elements to form a sustainable material known as MMC. The reinforcing material may be in the form of Vibrissae, discrete solid and fiber. Fatigue fracture is a major problem in complex structure and it is very significant for the components made of metals. Metal matrix composite (MMC) material has a crucial role in reducing the fracture rate. Though it is an effective method for assessment of damages on the various metal matrix composite specimens is a complex technique. The cost for the preparation of multiple specimens are high. Finite element analysis (FEA) method had become an effective method to test the fracture toughness of metal matrix composite. This article represents computational analysis of Fracture toughness, Stress intensity factor and J integral factor in the distinguished metal matrix composites using 2D compact tension (2D-CT) specimen. In this numerical method the mechanical properties, compositions of different Aluminum with silicon carbide are varied to evaluate and identify the composite with greater fracture toughness and fatigue strength. The numerical results thus obtained have been compared with the experiments reported in earlier study and highest Young's modulus of 121 GPa is attained in A5083 + 30 vol% SiC-oxid composites that contain an (Al + Mg + Si)–MgAl<sub>2</sub>O<sub>4</sub>–SiC [1]. FEM analysis on samples is carried out using ABAQUS CAE.

**Keywords:** Fracture toughness, Metal matrix composite, Al-SiC, ABAQUS CAE.

# Finite element simulation of high temperature fracture toughness of SS316LN stainless steel using damage model and its comparison with experimental data

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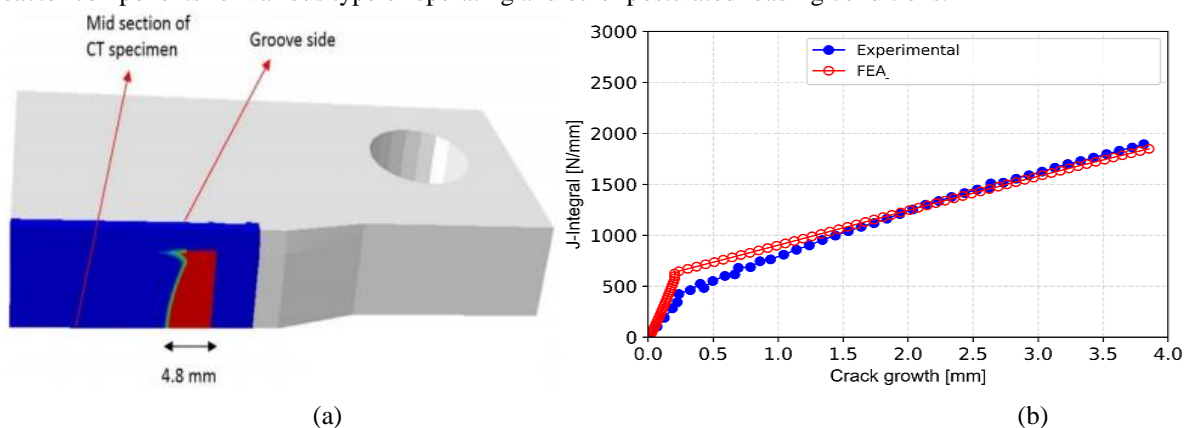
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## ABSTRACT

Crack initiation and subsequent growth in ductile materials takes place through the mechanism of void nucleation, growth and coalescence. Ductile materials like austenitic stainless steel SS316LN are used in fabrication of vessels and core components of fast breeder reactors and these reactors operate at high temperature, which can be of the order of 500-550°C. Evaluation of component level data, e.g., crack initiation toughness and fracture resistance curve may not be possible as the size of the components are large and hence, specialized test setups are required. In order to address this issue, finite element (FE) simulation of the component with a postulated crack is usually carried out and the corresponding load-deformation and crack growth behavior is evaluated. However, this analysis requires use of validated material plastic deformation and damage models, which need to be calibrated using laboratory scale experiment of fracture specimens. There are various types of material damage models, which are used by various researchers [1] and one of the popular model is due to Gurson [2], Tvergaard and Needleman [3]. It has been shown [1] that the parameters of these models are independent of specimen or component geometry and these are only dependent on the type of material used in the fracture test. Hence, it is possible to evaluate the parameters from specimen level tests such as those of smooth and notched tensile specimens as these are easy to fabricate and test. In this work, different types of tensile specimens manufactured from SS316LN stainless steel have been tested at different temperatures upto 600°C and the results have been used to calibrate the material damage model through FE simulation. In order to validate the applicability and ensure the accuracy of the damage model to simulate high temperature crack growth behavior in SS316LN steel, compact tension (CT) specimens have been tested at different temperatures upto 600°C and these have been analyzed through material damage models. The one-half of the CT specimen as modeled in this work is shown in Fig. 1(a). The typical damage contour showing crack growth for an applied load-line displacement of 10.5 mm is also shown. The corresponding J-R curve as evaluated from FE analysis is compared with experimental data in Fig. 1(b). It can be observed that the damage model is able to predict the fracture resistance data of SS316LN stainless steel very accurately and hence, this can be used to predict crack initiation and growth in actual reactor components for various type of operating and other postulated loading conditions.



**Fig.1:** (a) FE simulation of crack front propagation (shown in red) in CT specimen for applied load-line displacement of 10.5 mm; (b) FE simulation of fracture resistance (J-R curve) of CT specimen and its comparison with experiment.

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# Machine Learning-CALPHAD Assisted Material Design of L12-Strengthened Ni-Al-Co-Cr-Fe-Ti Complex Concentrated Superalloy for High Strength and Multi-Property Optimization

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## ABSTRACT

The demand for high-performance superalloys that offer superior temperature capability and thermal stability is paramount in various high-temperature applications, such as the hot sections of aircraft gas turbine engines. This study highlights the potential of statistical inference from machine learning (ML) in designing Ni-based complex concentrated superalloys (CCSAs) by leveraging the AutoSciKit Learn library and CALPHAD data from Thermocalc. By training ML algorithms on a comprehensive database of experimental and theoretical data, we efficiently designed a new Ni-based CCSA with unique targeted properties. A reduction in Co and the complete elimination of Fe result in precipitates transforming into more rounded cuboidal shapes and elevating the  $\gamma'$  solvus temperature, alongside a reduced lattice misfit of 0.26%. Notably, the Co<sub>10</sub>Fe<sub>0</sub> variant stands out for its high  $\gamma'$  solvus temperature (1245°C), substantial  $\gamma'$  volume fraction, cost-effectiveness, and low density. APT studies indicate an increase in the partitioning of the  $\gamma'$  stabilizer (Al) and a decrease in the partitioning of the  $\gamma'$  destabilizer (Cr), elucidating the rise in the  $\gamma'$  solvus temperature in the Co<sub>10</sub>Fe<sub>0</sub> alloy. High magnification HAADF-STEM images from the [001] oriented grain of the Co<sub>10</sub>Fe<sub>0</sub> alloy aged at 900°C for 50 hours clearly show rounded cuboidal precipitates. Fast Fourier transform (FFT) analysis reveals superlattice reflections characteristic of L1<sub>2</sub> ordering from the precipitates and face-centered cubic (FCC) reflections from the matrix, confirming the presence of a purely  $\gamma$ - $\gamma'$  structure at 900°C. High-resolution STEM-HAADF imaging indicates significant coherency between these precipitates and the matrix, characterized by a cube-on-cube orientation relationship. Long-term isothermal annealing studies up to 1000 hours at 900°C on lab-scale samples revealed that the alloy displays exceptional high-temperature thermal stability (coarsening rate of 8.01 nm<sup>3</sup>/s), consistent with TC-Prisma predictions, and comparable to commercial Ni-based superalloys such as CMSX-4, demonstrating the effectiveness of our ML-based approach. Significantly, the alloy design circumvents the emergence of any deleterious phases, achieving a high  $\gamma'$  solvus temperature, cost-efficiency, and low-density. The optimized Co<sub>10</sub>Fe<sub>0</sub> variant exhibits exceptional high-temperature strength of 1.06 GPa at 770°C, demonstrating its potential as a promising material. This study offers a promising pathway for the rapid and efficient design of low-cost advanced superalloys with unique targeted properties, suitable for high-temperature applications.

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