CREEP 2021

15th International Conference on Creep and Fracture of Engineering Materials and Structures

2021
14–16 JUNE
DIGITAL

FRIEDRICH-ALEXANDER UNIVERSITÄT ERLANGEN-NÜRNBERG

SFB/Transregio 103
SUPERALLOY SINGLE CRYSTALS

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Programme structure • Creep 2021

Monday 14 June 2021		Tuesday 15 June 2021		Wednesday 16 June 2021	
Meeting 1	Meeting 2	Meeting 1	Meeting 2	Meeting 1	Meeting 2
08:50–10:00		09:00–10:00		09:00–10:00	
Conference opening and Invited talks I		Invited talks V		Invited talks VII	
Coffee break		*Coffee break*		*Coffee break*	
10:30–11:15	10:30–11:30	10:30–11:30	10:30–11:30	10:30–11:30	
Abstract talks I	Abstract talks II	Invited talks VI	Abstract talks VI	Abstract talks X	
Lunch		*Lunch*		*Lunch*	
12:30–13:30	12:30–13:30	12:30–13:30		12:30–13:15	12:30–13:30
Invited talks II	Abstract talks III	Abstract talks VII		Invited talks VIII	Abstract talks XI
Coffee break		*Coffee break*		*Coffee break*	
14:00–15:00	14:00–15:00	14:00–15:00		14:00–15:00	14:00–15:00
Invited talks III	Abstract talks IV	Abstract talks VIII		Abstract talks XII	Abstract talks XIII
Coffee break		*Coffee break*		*Coffee break*	
15:30–16:30	15:30–16:30	15:30–16:30		15:30–16:40	
Invited talks IV	Abstract talks V	Abstract talks IX		Invited talks IX and conference closing	

Scientific Programme – 14 June 2021

08:50–10:00 Conference opening and invited talks I

Creep of single-crystals of nickel-base g-alloy at high temperatures

Viguier, Bernard, CIRIMAT - University of Toulouse, Toulouse
Epishin, Alexander, Technical University of Berlin, Berlin
Bernard, Fedelich, Federal Institute for Materials Research and Testing (BAM), Berlin

Porosity in single-crystal nickel-base superalloys is removed by hot isostatic pressing (HIP) at temperatures above g-solvus where the material is very soft and ductile. In order to investigate the mechanisms operating at such high temperatures a g-analogue of superalloy CMSX-4 has been designed. Compared to CMSX-4 this alloy contains reduced amount of g-forming elements and therefore has no g-phase which change the creep mechanisms as well as distorts the high temperature dislocation configurations during cooling. The alloy was solidified as [001], [011], [123] and [111] single-crystals and tested under creep conditions at temperatures 1150 °C, 1225 °C and 1288 °C. The last temperature, 1288 °C, is of our special interest because it is a temperature of commercial HIP of CMSX-4 being slightly higher the g-solvus temperature of this alloy equal to about 1280 °C. The creep tests performed at 1288 °C showed that the g-alloy has creep strength close to that of CMSX-4 as well as CMSX-4 exhibits very high creep anisotropy. The creep rate increases by an order of magnitude with changing the orientation from [001] to [111]. The creep rates of [011] and [123] orientations are in between. Such a character of creep anisotropy fits with the orientation change of Schmid factor for octahedral glide system. This deformation mode was also identified by the shape of cross-section of a deformed [011] single-crystal. From TEM investigations and accompanying modelling follows that at 1288 °C dislocations stochastically cross subgrains almost without interaction and therefore this dislocation movement does not results in significant hardening and coarsening of the material. Accordingly the creep curves exhibit nearly constant strain rates, see Fig. 1. However, a little interaction of gliding dislocations still exists which follows from the formation of new low angle boundaries inside pre-existing subgrains as identified by EBSD. Creep tests at 1150 °C and 1225 °C were performed in order to check whether the strong creep anisotropy remains at lower temperatures. Creep tests showed that in temperature range between 1150 °C and 1288 °C the creep anisotropy does not change which could indicate the same deformation mode, namely octahedral glide. It is different for nickel-base superalloys which creep anisotropy decreases with decreasing testing temperatures below g-solvus due to precipitation of the strengthening g¢-phase. From the value of estimated creep activated energy equal to about 400 kJ/mole one could conclude that the creep mechanism is octahedral glide of dislocations retarded by trailed atoms of rhenium and tungsten.

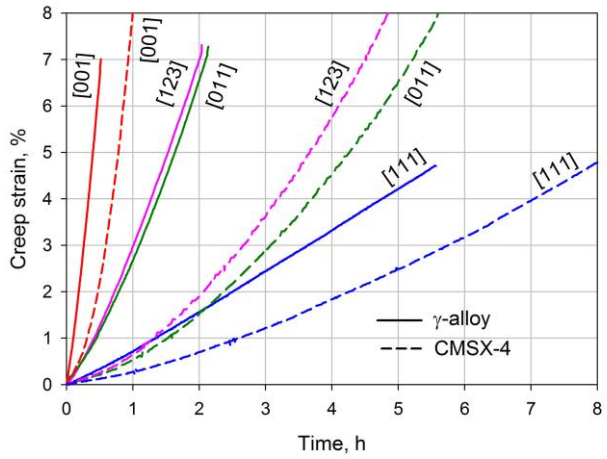

Figure 1: Creep curves of g-alloy (solid lines) compared with those of CMSX-4 (dashed lines) [1] at 1288 °C. Creep tests of g-alloy were interrupted.

References:

[1] A. Epishin, B. Fedelich, G. Nolze et al., MMT A, 49, pp. 3973–3987 (2018).

Novel creep mechanisms in a Co-base superalloy based on planar fault formation by segregation-assisted glide and climb of partial dislocations

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In this work we present a dedicated high-resolution TEM study of planar faults and associated partial dislocations which formed in a multicomponent single crystal Co-base superalloy (ERBOCo-1: Co32Ni8Al6Cr5W2.5Ti0.5Ta [1]) during uniaxial creep along [001] at 850 °C. In our earlier study we have shown that creep deformation in this alloy involves extensive formation of planar faults including superintrinsic and superextrinsic stacking faults (SISF, SESF) by glide of partial dislocations [2]. HR-STEM imaging combined with geometric phase analysis and STEM-EDXS revealed structural and chemical complexity of the planar faults and associated partial dislocations.
Here we report on a novel route of creating SISF by climb of Frank partial dislocations via vacancy condensation. One example of such partial dislocation is depicted in Figure 1(a). The inset shows the inserted {111} half-plane and the leading Frank partial dislocation which is formed by splitting of a Lomer-type dislocation according to a/2 <1-10> → a/3 <111> SISF a/6 <11-2>. Interestingly, the Frank partial dislocation is driven forward by an osmotic force that overcomes the elastic climb force acting in opposite direction. This mechanism is only sustainable because the Frank partial serves as an internal vacancy sink absorbing vacancies emitted by climbing channel dislocations. Furthermore, the movement of said dislocations is highly dependent on segregation of solutes to the fault and dislocation core, lowering the energy associated with the creation of the SISF.

Figure 1(b) shows another example of a planar fault, which changes its nature from intrinsic at the bottom to extrinsic at the top. While this structure has been reported before by Vorontsov et al [3], it was never experimentally observed in a Co-based system. One key difference here is, the attachment of an anti-phase boundary (APB) segment changing the local chemistry and dislocation structure. We believe that the APB serves as a diffusion channel funneling γ-forming elements like Co and Cr towards the defect. Figure 1(c) presents the EDXS element distribution maps, showing the segregation at the SISF, SESF and APB, respectively.

All defect structures created during creep involve a diffusion of solutes towards and away from dislocation cores and planar fault planes [4, 5]. So far, it is not clear, if the segregation effects are a necessity for the creation process of faults or if they also occur at low temperatures and fast deformation rates. Potential microscopic studies which may allow answering this question will be discussed.

[1] N. Volz et al., Metallurgical and Materials Transactions A 49(9) (2018) 4099-4109.
[2] M. Lenz et al. , Acta Materialia 166 (2019) 597-610.
[3] V.A. Vorontsov et al. , Acta Materialia 60(12) (2012) 4866-4878.
[4] S.K. Makineni et al. , Scripta Materialia 157 (2018) 62-66.
[5] S.K. Makineni et al., Acta Materialia 155 (2018) 362-371.

On the effect of stress state and crystallography on microstructural evolution in single crystal Ni-base superalloys

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Thome, Pascal, Ruhr-University Bochum, Institute for materials science, Bochum
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Neuking, Klaus, Ruhr-University Bochum, Institute for materials science, Bochum
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Rafting, the directional coarsening of the ordered γ'-phase, is a well-known microstructural instability in single crystal Ni-base superalloys, where the atoms of two phases occupy a common fcc base lattice. The lattice constants of the two phases are not the same and this gives rise to coherency stresses and strains. During [001] tensile creep testing, for negative misfit alloys, one observes the gradual transformations ofthe γ'-cubes to γ'-rafts, which form perpendicular to the direction of the applied tensile stress. In the present work, we study rafting processes which occur in circular notched cylindrical specimens during [001] tensile testing. It is well known that stress redistribution occurs in these specimens, such that high notch root stresses in loading direction decrease while they increase in the center of the specimens. We combine interrupted creep testing with quantitative analysis of SEM micrographs to show that there are [001] tensile loading creep conditions, where the kinetic of rafting follows the kinetic of stress redistribution. Moreover, we show how crystallographic loading directions ([001], [110] and [111]) affect the creep behavior and the microstructural damage evolution. We show that large scale lattice rotations in the notch root cross section only manifest themselves during the final rupture events.

10:30–11:15 Abstract talks I

Influence of the cast skin on the microstructure and creep behavior of single-crystal thin-walled specimens of the nickel-based superalloy M247LC

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Introduction

Inside the turbine blades there are fine structures due to the complex cooling channels to ensure the use at high temperatures. These thin partition walls are uncoated and can"t be processed anymore afterwards. Previous investigations already showed that the creep properties deteriorate with decreasing wall thickness. In this work we investigate the creep behavior depending on the cast wall thickness by casting single-crystal thin-walled specimens with a minimum wall thickness down to 0.4 mm. The influence of the cast skin on the microstructure and creep behavior of these thin cast structures is analyzed.

Methods

After the adaptation of the conventional investment casting process by using 3D-printer for the production of positive models single-crystal thin-walled specimens with different wall thicknesses of the nickel-based superalloy M247 LC are cast via Bridgman process in a vacuum induction furnace. The cast specimens are heat treated in a vacuum cold wall furnace under vacuum and argon. By that we investigate the influence of the heat treatment on the microstructure especially in the near-surface region. The microstructure is characterized by SEM and EDS. The creep behavior is investigated depending on the cast wall thickness. To investigate the influence of the cast skin on the creep behavior, creep tests are carried out on thin-cast specimens with and without cast skin.

Results

After heat treatment under vacuum a single-phase surface layer is formed. EDS-measurement shows that this layer is rich in nickel and aluminum, whereas the chromium and cobalt content is depleted. This suggests that a single-phase γ'-layer was formed. After creep deformation the microstructural characterization shows the formation of oxides and a γ'-depletion zone in the near-surface region. The creep properties deteriorate with decreasing wall thickness. Creep tests for the investigation of the influence of the cast skin on specimens with a wall thickness of 2 mm show that there is no significant change in creep behavior. The results regarding microstructure and creep testing are shown in the figure. Further creep tests are carried out on thinner samples, since the influence of the cast skin increases with decreasing wall thickness due to the high surface to volume ratio.

Conclusions

The formation of the single-phase γ'-layer is caused by the sublimation of chromium during heat treatment under vacuum at 10-4 Pa due to its low vapor pressure. The deterioration of creep properties in thin-cast single-crystal structures of nickel-based superalloys is related to surface degradation mechanisms such as oxidation and γ'-depletion. Because of the high surface to volume ratio, these degradations and also the cast skin have a bigger influence on creep properties than in bulk specimens.

How concentrations of individual alloy elements affect high temperature and low stress creep on Ni-base single crystal superalloys

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Variations of chemical compositions of multicomponent Ni-base superalloy single crystals (SXs) affect microstructures, local alloy chemistries and mechanical properties. The present study compares two alloys. ERBO/1 (CMSX-4 type) a well characterized reference alloy with Re is compared to the new Re-free SX ERBO/15, where Re, which has a beneficial effect on high temperature and low stress creep strength, is replaced by increased levels of Ti, W and Mo. It is shown that when subjected to appropriate heat treatments, ERBO/15 can show the same or even better resistance against tensile creep as ERBO/1. An effort is made to vary the Mo and W concentrations in ERBO/15 considering two variants, ERBO/15-Mo (Mo reduced from 2.6 to 1.3 at.% balanced by an increase of Ni) and ERBO/15-W (W reduced from 2.5 to 1.3% balanced by an increase of Ni). It is shown that under the same multiple step heat treatment, these two ERBO/15 variants have a coarser microstructure (same channel widths but larger γ' particles sizes) and higher γ' volume fraction and deform faster than the ERBO/15 base alloy. The results are discussed on the basis of a microstructural analysis in the TEM and in the light of thermodynamic calculations. Re, Mo and W all partition to the γ-channels and it seems reasonable to assume that they slow down dislocation climb. Interrupted creep tests are combined with transmission electron microscopy to study the effect of the small variations in alloy composition on microstructure and creep deformation mechanisms in the high temperature and low stress creep regime.

Influence of solution temperature on microstructure and creep properties of A 4.5%Re/3%Ru single crystal nickel-based superalloy

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By means of selecting crystal technique, a4.5%Re/3%Ru single crystal nickel-base superalloy with [001] orientation was prepared under high temperature gradient condition in the directional solidificationfurnace. Aftersolution treated athighest temperature of 1325 °C, 1328 °Cand aging treated, themicrostructure observation and creep properties of the superalloy treated by various regimes were carriedout. The results show that a few eutectic microstructure existsin the interdendritic regionof the alloy at lower temperature solution treatment, while no eutectic microstructure is detected in the alloy at high temperature solution treatment.The creep life of the former at 1100 °C/137MPais measured to be 321 h, while the creep life of the latter is meartured to be 476h. After elevated solution temperature, the the increasing extent of the alloy creep lives obtains about 48.3%. The measuring results of atom probe show that the atoms Re, W and Moareenriched in the γ phase near γ/γ ' interfaceto form the peak concentration of refractory elements, which may hinder the dislocation movement and delay the dislocation shearinginto the γ ' phase. This is considered to be one of the reasons for the alloy displaying a better creep resistance at high temperature. In the laterstage of creep, the deformation mechanism of alloy is dislocation shearingintotherafted γ ' phase, whereinthe dislocations of shearing in γ ' phase may cross-slip from {111} to {100}to form the K-Wlock with the non-planar core structure, which can inhibit the slippingand cross-slippingof dislocationsto improve the creep resistance of alloy at hightemperature.While the interaction of atomsRu with Re and W may reserve some Re and W atoms in γ ' phase, for delayingthe diffusion of elements, to keep the K-Wlocksin the γ ' phase of the alloy at high temperature, which isconsidered to be another reason of the alloy possessing the good creep performance at hightemperature.

10:30–11:30 Abstract talks II

Grain Boundary Precipitation Strengthening Mechanism in Creep - Modeling and Experiments -
Takeyama, Masao, Tokyo Institute of Technology, Materials Science and Engineering, Tokyo

Objective of this paper is to create a stir in the conventional creep strengthening mechanism, specifically focusing on the role of grain-boundary precipitates in creep deformation. Take a look at Fig. 1. Which microstructure is more creep resistant? Many of you undoubtedly say (b). Contrary to expectation, however, the answer is (a), where most of the grain boundaries are covered by TCP Fe2Nb Laves phase and the precipitation density in grain interiors is obviously lower, in comparison with those in the other. The higher the fraction of grain-boundary area covered by the Laves phase (ρ), the lower the creep rate, along with the equation shown in Fig 2. This strengthening method is called "Grain-boundary precipitation strengthening (GBPS)". Based on this strengthening mechanism, we designed novel austenitic heat resistant steels strengthened by the GB Laves phase, in conjunction with thermodynamics and kinetics in Fe-Cr-Ni-Nb system, and proved that the steels exhibit superior creep rupture strengths at 1073 K, comparable to those of Ni based alloys (A263, IN617). These results clearly demonstrate that creep deformation preferentially occurs along grain boundaries (mantle region) and how important to suppress the localized deformation by decorating the grain boundaries with thermodynamically stable phases. By extending this GBPS knowledge, grain-size dependence of creep resistance was modeled based on "core-mantle" concept, by taking grain interiors for core. The model suggests that, in case of ρ=0 (no GB precipitates, that is,γ single phase), the creep resistance decreases with decreasing grain size and the slope of the grain-size exponent is -1 in logarithmic plot of the minimum creep rate against the grain size, even under creep conditions where dislocation climb is the rate-controlling process. With increasingρ to 1 (full coverage of GBs), the slope becomes shallower with no grain-size dependency in the creep rate. We confirmed the validity of the change in grain-size dependency withρ by experiments using the Fe-Cr-Ni-Nb model steels. These findings are apparently different from what textbooks are telling where the grain-size dependency occurs in diffusional creep regime and no dependency in dislocation creep regime. In this presentation, detailed GBPS with the core-mantle concept together with the experimental evidences to support the above new findings will be shown in conjunction with the microstructure analyses. This study was partially supported by the Advanced Low Carbon Technology Research and Development Program (ALCA) of the Japan Science and Technology Agency (JST) (No. JPMJAL1005).

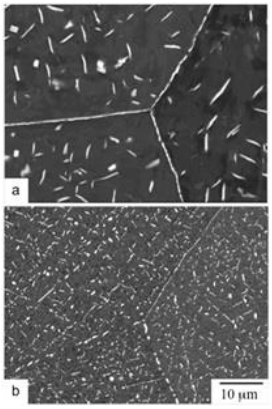

Fig. 1 Microstructures of a novel austenitic model steel with Fe2Nb Laves Phase: (a) ρ=90%, (b) ρ=50%

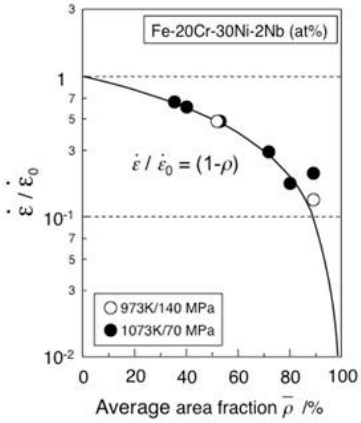

Fig. 2 Change in the normalized creep rate withρ in a novel austenitic model steel.

Effect of grain-boundary precipitates on the grain size dependence of creep in austenitic steels

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Takeyama, Masao, Tokyo Institute of Technology, School of Materials and Chemical Technology, Dept. of Mat. Sci. & Eng., Tokyo

In creep theory, it is widely believed that minimum creep rate is insensitive to grain size, d, in power low creep region of the stress exponent, n, is about 4 to 6 where dislocation climb is the rate controlling process. In contrast, minimum creep rate increases with decreasing d under diffusional creep region of n=1 where the grain size exponentρ is -2 and -3 for volume diffusion and grain boundary diffusion, respectively, known as Nabarro-Herring creep and Coble creep. Although there have been a number of reports grain-size dependence of minimum creep rate withρ in the range of -1 to -2, the dependence was in most cases interpreted in terms of diffusional creep or grain-boundary sliding. However, very limited studies have been systematically conducted how grain-boundary precipitates affect the grain-size dependence of minimum creep rate in the dislocation creep region. Takeyama et al. have proposed a novel creep strengthening mechanism, called "grain-boundary precipitation strengthening (GBPS)", that is, the higher the grain-boundary covering ratio, ρ, with thermodynamically stable precipitates such as a-W (bcc) phase or Fe2Nb (C14) Laves phase, the lower the minimum creep rate in proportional to (1-ρ). This suggests that the grain-size dependence of minimum creep rate varies with ρ. Therefore, in this study, grain size dependence of the minimum creep rate in the power law creep region has been investigated using model steels with various values of ρ. Two steels were prepared: one is γ single phase of Fe-20Cr-30Ni and the other Fe-20Cr-30Ni-2Nb (at.%) with γ Fe2Nb two phases at 1273 K. These steels were homogenized to adjust the grain sizes from 50 mm to 700 mm. Then, 2Nb steel was aged in the two-phase region to havep of 0.8 to nearly 1.0. Creep tests were conducted at 1073 K under a constant stress of 20 MPa to 70 MPa in air. We first confirmed that the rate controlling process of all creep tested under the conditions is in dislocation climb region, since the n is about 5 to 6 (Fig. 1). In case of the steel with ρ=0, the p is nearly equal to -1, whereas it is nearly 0 in the steel with ρ=0.98 (Fig. 2). Thus, it is obvious that the existence of grain-boundary Laves phase reduces grain size dependence of the minimum creep rate. These results are fairly in good agreement with the calculated grain size dependences of the minimum creep rate based on a core-mantle model by taking the GBPS mechanism into account, as shown in Fig. 2. Therefore, unlike the theory written in textbooks, grain-size dependence of the creep rate should vary, depending on the amount of grain-boundary precipitates. Detailed mechanism of the dependency will be discussed in conjunction with GBPS and microstructure analysis along the grain boundaries. This study was supported by the Advanced Low Carbon Technology Research and Development Program (ALCA) of the Japan Science and Technology Agency (JST) (No. JPMJAL1005).

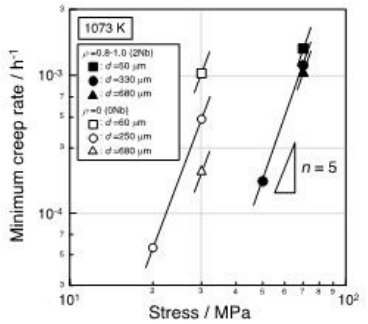

Fig. 1 Stress/minimum creep rate curves of the steels studied with variousρ at 1073 K, showing the n nearly equal to 5 in the region of dislocation creep.

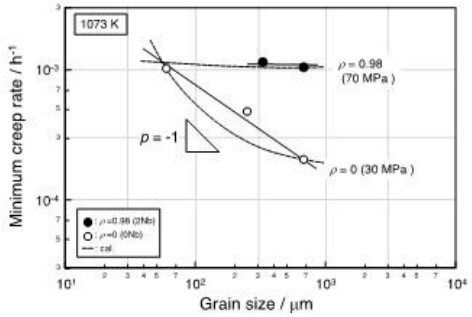

Fig. 2 Grain size dependence of minimum creep rate in the steels with no grain-boundary precipitates (ρ=0) and with the precipitates (ρ=0.8 and 0.98), showing a distinct difference in the dependency.

High temperature and low stress creep behavior of the refractory chemically complex alloy $AlMo_{0.5}NbTa_{0.5}TiZr$

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Glatzel, Uwe, University of Bayreuth, Lehrstuhl Metallische Werkstoffe, Bayreuth
Agudo Jácome, Leonardo, Federal Institute for Materials Research and Testing (BAM), Materials Engineering, Berlin

The refractory chemically complex alloy (rCCA) AlMo0.5NbTa0.5TiZr, with a density of 7.4 g/cm3, shows a compressive ultimate strength of 772 MPa at 1000 °C, comparatively surpassing Ni-base and other rCCAs[1]. Its dual-phase microstructure, with a high volume fraction (≈ 62%) of cuboidal and plate-like particles coherently embedded in a continuous matrix, resembles the well-known pattern of the γ/γ'' in Ni-base superalloys. Its developers have thus implied that it could stand as structural alloy for high temperature (HT) applications. However, this rCCA presents fundamental differences when compared to Ni-base superalloys. Its microstructure results from a spinodal decomposition, where a bcc phase forms the precipitates while a B2 phase builds the continuous matrix. Here, we report the HT creep properties and the underlying microstructural changes of the rCCA AlMo0.5NbTa0.5TiZr to propose deformation and degradation micromecanisms for this regime.

The material was produced by arc-melting[2] and subsequently heat treated in argon: at 1400 °C for 24 h[2] plus a hot isostatic pressure treatment at 1370 °C and 170 MPa for 4 h, with a cooling rate of 10 K/min[3]. Miniaturized tensile specimens (≈ 28 x 7 x 2 mm) were cut by wire electrical discharge machining, and polished to a quality of 1 µm. Creep tests were conducted in vacuum in the respective temperature and stress range 800-1200 °C and 30-120 MPa. For observation, thin slices were extracted from the gauge length, away from the fracture surface, grinded to a thickness of 100 µm, and electropolished to electron transparency. The microstructure was observed on the electropolished specimens using scanning (S) as well as transmission (T) electron microscopy (EM).

The Norton plot shown in Fig. 1 gives Norton exponents of about 3.1 and 3.2 for temperatures of 1000 and 1100 °C, respectively. Curiously, creep rate minima are very close for a stress level of 30. The starting microstructure shown in Fig. 2 reflects a macroscopically lean coarse grain structure (a) and a microscopically fine-meshed basketweave (b) structure with coherency dislocations only around coarsened particles usually close to subgrain boundaries (b and c). Results are discussed on the base of variations of this starting microstructure after interrupted and ruptured creep tests.

Figure captions:

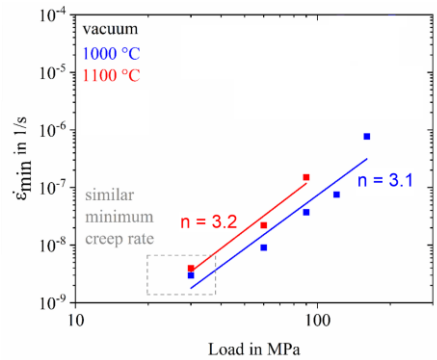

Fig. 1: Norton plot for creep tests at 1000 and 1100 °C under vacuum.

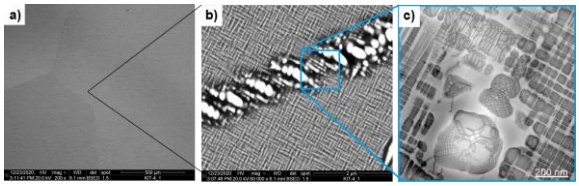

Fig. 2: heat-treated microstructure before creep. SEM images of (a) coarse grain structure and (b) sub-grain boundaries, and (c) BF STEM of coarse bcc particles decorated by dislocation networks.

References:

Senkov ON, Isheim D, Seidman DN, Pilchak AL. Development of a refractory high entropy superalloy Entropy 18 (2016) 102
By Alexander Kaufmann at Karlsruher Institut für Technologie.
By Inmaculada Lopez Galilea and Benjamin Ruttert at Ruhr-Universität Bochum.
Funding of DFG projects GL 181/56-1 of the priority program SPP2006 CCA-HEA and AG 191/3-1 is acknowledged.

Creep life assessment method of welded joint of in-service piping considering data scatter
Yaguchi, Masatsugu, Central Research Institute of Electric Power Industry, Materials Science Laboratory, Yokosuka

There is a large amount of scatter in the creep properties of welded joints of the steels; however, the scatter is not considered in conventional remaining life assessments of welded joint of in-service piping. Thus, the author has developed a new method of assessing the individual creep properties of welded portions of actual pipes. This paper explains the concept of the assessment method and also describes the actual implementation of the method for Grade 91 steel as an example. In the method, the creep property of the welded joint is related to that of each base metal because the creep properties of welded joints strongly depend on the creep life properties of the corresponding base metals, as previously shown by the author's work. Microstructure analyses and small punch creep tests on samples cut from the base metals at the outer surface of pipes in service were conducted, and the results were compared with a material data base to estimate the creep property of each base metal of the target pipe. The precision of the creep remaining life assessment of pipes is significantly improved using the developed method because it can consider variations of the creep properties of their materials, which are not considered in existing life assessment methods. Then, the method was applied to the welded joints of the pipes in ultra-super critical power stations during periodical inspection, and creep remaining lives of the components were examined.

12:30–13:30 Invited talks II

Creep behaviour in aerospace alloys under varying stress and temperature
Whittaker, Mark, Swansea University, Institute of Structural Materials, Swansea
Harrison, Will, Swansea University, Institute of Structural Materials, Swansea
Gray, Veronica, Swansea University, Institute of Structural Materials, Swansea
Williams, Steve, Rolls-Royce plc, Derby

In the development of more accurate lifing models for safety critical aerospace components, knowledge of the development of evolving stress conditions is required in order to make appropriate predictions. At the high temperatures experienced in components such as turbine discs, creep behaviour is therefore a particularly important input into such models. Traditional approaches to modelling creep deformation under such circumstances are based around data conducted under isostress and isothermal conditions, enabling the development of constitutive relationships. However, in many applications both stress and temperature may vary, particularly during a full cycle, and the results of these changes are not well understood, either in terms of microstructural behaviour or predictive capability.

The current paper considers the effect of repeated variation of stress and temperature in high performance alloys for aerospace applications. In particular, variations between Low Temperature High Stress (LTHS) to High Temperature Low Stress (HTLS) conditions are considered in terms of dislocation behaviour and overall deformation characteristics and linked to previous efforts to understand material behaviour in terms of the recently developed Wilshire Equations.

Constitutive modelling of creep curves in Ni-based superalloys
Mohles, Volker, RUB, ICAMS, Bochum
Zomorodpoosh, Setareh, RUB, ICAMS, Bochum
Jiang, Yuxun, RUB, ICAMS, Bochum
Steinbach, Ingo, RUB, ICAMS, Bochum

Recently, a data-driven creep model has been developed that successfully describes creep experiments for single crystal superalloys depending on composition, temperature and stress. However, the approach offers no physical links to the incremental nature of microstructure evolution. Therefore, as a new constitutive approach has been developed based on the Kocks-Mecking plasticity model. It is able to describe creep curves with a single dislocation density as a state variable. The evolution of the dislocation density is described by a dislocation generation rate, a dynamic annihilation rate, and static recovery. The generation rate depends on the present dislocations as well as an external limitation of the free dislocation path. The idea of this limitation is the width of γ-channels in-between the γ'-particles in Ni-based superalloys. It is known that the channel width increases with time, so that the creep model can account for the structural change of the γ'-particles (rafting). With this as a first ansatz for softening, the model is able to cover the creep stages I, II, and III. The strain rate is described by thermally activated dislocation slip and climbing. Static recovery is described as a thermally activated process as well. The corresponding activation parameters define the strain rate dependence on temperature and the mechanical load, and they offer the option to account for dependences on alloy composition. Using a pre-calculated channel width as a function of strain from phase-field simulations, the creep model is tested by fitting it to measured creep curves.

Data-based creep modeling strategy: application to Co-based superalloys

Zomorodpoosh, Setareh, Ruhr-University Bochum, ICAMS, Bochum
Roslyakova, Irina, Ruhr-University Bochum, ICAMS, Bochum
Steinbach, Ingo, Ruhr-University Bochum, ICAMS, Bochum

Understanding the creep behavior at high temperatures is an important area of many technologies such as nuclear power plants, turbine blades, jet engines, and other engineering applications [1]. Therefore, modeling and prediction of creep behavior have been a topic of much interest. This study presents an overview of the recent developments of data-driven creep models depend on the composition, temperature and stress focused on Co-based single-crystal superalloys. The modeling strategy is based on a combination of statistical and machine learning modeling approaches. To guarantee an accurate description and robust results, known physical laws and relationships between materials properties have been considered as a basis for regression functions. The model with the best performance has been identified based on statistical criteria and evaluated with the available creep experimental data of Co-base superalloys, which have been provided by the collaborative research center SFB/TR 103, project B3 [2]. Furthermore, a current creep database with built-in data mining methods for storage and automated process of raw data [3] has been extended for different types of experiments such as double shear and compression tests.

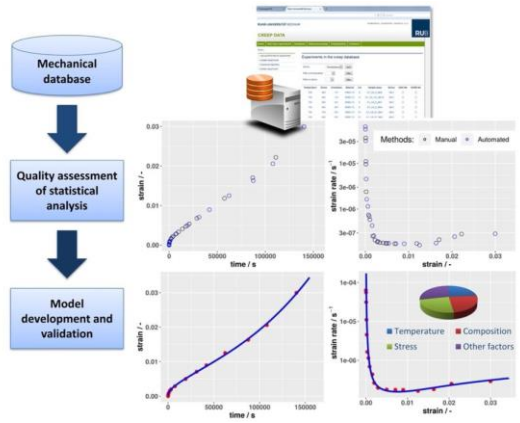

Fig. 1: Development of creep model for Co-base superalloy; experimental data from paper by N. Volz, et al. [4].

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12:30–13:30 Abstract talks III

Evaluation of harper-dorn creep in LiF single crystals

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Kassner, Michael E., University of Southern California, Aerospace and Mechanical Engineering, Los Angeles
Kumar, Praveen, Indian Institute of Sciene Bangalore, Materials Engineering, Bangalore

Ever since the first observation of Harper-Dorn (H-D) creep in 1957, it has been constantly under debate and criticism. H-D creep is observed at stresses generally below 10-5G and at temperatures which are above 0.9Tm. It is usually characterized by a stress exponent of 1, an activation energy equal to that for self-diffusion and the dislocation density has been suggested to be constant and independent of the applied stress in most of the cases. In this study, compression creep of high purity [100] oriented LiF single crystals was conducted at 0.95Tm at stresses ranging from 10-6G to 4 × 10-4G. The dislocation structure and the dislocation density were evaluated using dislocation etch pit analysis. It was observed that there is a transition of creep exponent from 4 to 1.6 ± 0.1 at a normalized stress of 10-5G. In the low-stress regime (i.e., below 10-5 G) the dislocation density in these crept crystals was constant. At higher stresses, the dislocation density showed a square dependence on the applied stress. Along with this, LiF single crystal were annealed up to 10,000 hours to study the effect of long-term annealing on the change in dislocation density in these crystals. Some of the crystals were also pre-strained in order to study the effect of pre-straining on evolution of dislocation density with annealing. A reduction of about 1.5 orders of magnitude in the dislocation density was observed up to 150 hours beyond which no reduction in the dislocation density in these crystals was observed even up to 10,000 hours. The dislocation density in LiF single crystals showed a minimum saturation value, for both as-received and pre-strained samples, below which no further reduction in dislocation density is possible in these crystals. This minimum saturated value of dislocation density is suggested to be the reason for the constant dislocation density which is observed in the H-D creep regime in the present study. On the other hand, if the initial dislocation densities are much lower than this saturation limit, a higher creep exponent is expected. Based on the Orowan equation, it is suggested that the climb velocity of dislocation can be related to a stress exponent which is greater than 1.

The role of grain boundary mobility in diffusional creep

Delannay, Francis, UCLouvain, IMMC, Louvain-la-Neuve

We revisit the model of diffusional creep by accounting for the dependence of the diffusion potential on grain boundary curvature. The study is carried out through two case-studies: the deformation of a lattice of columnar grains in conditions of Coble creep, and the rotation of a grain embedded in a polycrystal in conditions of Nabarro-Herring creep or Coble creep.
The analysis reveals that, unless grain boundary mobility is infinite, grain boundary curvature is dynamically induced by strain rate. A link is established between the curvature distribution and the transfer of diffusion fluxes across grain boundaries.
For the two case-studies, the equation expressing the balance of grain boundary motions at steady-state is solved for calculating, within a range of grain boundary mobilities, the grain boundary profiles, the diffusion fluxes, and the contributions to power dissipation arising from curvature. The latter contributions are found to scale closely as the square of grain size. It follows that the dissipation contribution due to curvature is larger in conditions of Nabarro-Herring creep.
In conditions of Coble creep, the dissipation contribution due to curvature translates into a lower bound for the apparent boundary viscosity parameter to be used in numerical simulations. This lower bound is consistent with previous identifications of the parameter in literature.
The classical model assuming flat grain boundaries with transfer of fluxes via triple junctions emerges as a particular case involving the implicit assumption of an infinite grain boundary mobility.

Creep behaviour of pure, solution and precipitation hardening alloys under tensile and compressive conditions

Zhu, Qiang, Southern University of Science and Technology, Mechanical and Energy Engineering, Shenzhen
Li, Xin, Southern University of Science and Technology, Mechanical and Energy Engineering, Shenzhen
Xu, Zhen, Southern University of Science and Technology, Mechanical and Energy Engineering, Shenzhen

Creep behaviour of engine blades is normally characterised by creep testing under tensile stressing conditions. Creep testing is a time-consuming process and it is desirable to accelerate the creep testing process. A new technique for speeding up the creep testing process in terms of multi-tests under compressive stressing conditions has been developed. In order to understand quantitative and qualitative relationship between creep behaviour under tensile and compressive stressing conditions, systematic creep tests of pure, solution and precipitation hardening alloys were carried out. This presentation will show the testing results of the creep tests. Microstructural evolution during the compressive creep tests will be also presented.

Inter- and extrapolation using analytical methods: evaluations regarding time-based and strain-based creep design properties

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Kontermann, Christian, University of Technology Darmstadt, Chair and Institute for Materials Technology, Darmstadt
Schwienheer, Michael, University of Technology Darmstadt, Chair and Institute for Materials Technology, Darmstadt
Speicher, Magdalena, University Stuttgart, Materials Testing Institute, Stuttgart,
Oechsner, Matthias, University of Technology Darmstadt, Chair and Institute for Materials Technology, Darmstadt

In the past as well as in recent years, several analytical methods for the interpolation and extrapolation of various creep design properties have been developed or evolved. Various methods are already implemented in numeric tools and therefore accessible for practical application in material development and product design.
Whilst standards and guidelines for creep testing presently suggest a limitation of the extrapolation factor to 3 in time direction for creep rupture data, neither specific suggestions regarding extrapolation in temperature direction nor with respect to interpolation and extrapolation of further time-based or strain-based creep properties are given. Research work providing appropriate recommendations and guidelines is strongly demanded. Within that given framework this publication focuses on the examination of established as well as novel methods for the analytical interpolation and extrapolation of creep design properties required for high temperature applications.
Within a recently finished research project, a variety of analytical and phenomenological methods were subjected to systematic in-depth analyses comprising the interpolation and extrapolation of time-based creep data (Rupture Time, Time to Specific Strain) as well as the interpolation and extrapolation of strain-based creep data (Minimum Creep Rate). Individual influencing factors and sensitivities as well as the individual application possibilities and application restrictions regarding different types of heat resistant steels were determined. Based on these findings present recommendations regarding the interpolation and extrapolation of creep rupture data are examined and recommendations regarding the interpolation and extrapolation of Creep Strain Limits as well as Minimum Creep Rates are proposed in this contribution.

14:00–15:00 Invited talks III

Stress exponents in basic modelling of creep

Sandström, Rolf, KTH Royal Institute of Technology, Materials Science and Engineering, Stockholm
Zhang, Jing, KTH Royal Institute of Technology, Materials Science and Engineering, Stockholm

To avoid limitations with empirical approaches one of the authors has developed creep models without the use of adjustable parameters. We refer to such models as basic. For example dislocation models for the creep rate of aluminium and copper have successfully described the variation of the stress exponent over a wide range of temperatures and stresses [1,2].
Practically all basic models for the creep rate gave a stress exponent of 3 to 5. The continuous deformation at a constant stress is a characteristic feature of creep and is controlled by recovery of dislocations. In the present paper the recovery model is reanalysed. The established model of recovery in creep is based on the interaction of individual dislocations. However, if dislocation loops and dipoles are taken into account, the resulting stress exponent is increased.
At very low stresses, the apparent stress exponent often takes values close to unity which can be taken as the controlling mechanism being diffusion creep. However, this is not necessarily the case. At sufficiently low stresses it is practically impossible to perform the creep tests long enough to ensure that the secondary stage is reached. Thus, the creep rate is taken from the primary stage where the creep rate is higher.
The behaviour at normal creep stresses as well as at very low stresses is illustrated in Figure 1 for the austenitic 17Cr12Ni2Mo stainless steel 316H. The experimental data are from [3]. In the normal creep range with stresses from 80 to 300 MPa, the creep exponent is 7. The model values for secondary creep rate at low stresses give a stress exponent of 5 where the pipe-diffusion is no longer active. The experimental values at low stresses (<10 MPa) in [3] are taken from primary creep curves after 1000 h. Using the primary creep curve model in [4], the creep rate has been assessed. The apparent stress exponent in both model and experiments is close to unity, much higher than for the modelled secondary creep rate at the same stresses. It can be concluded that the creep model can at least approximately describe the experimental values at very low stresses. It should be recognised that an extrapolation of up to eight orders of magnitude in creep rate is involved from the normal creep range. Similar results have previously been obtained for copper [4].

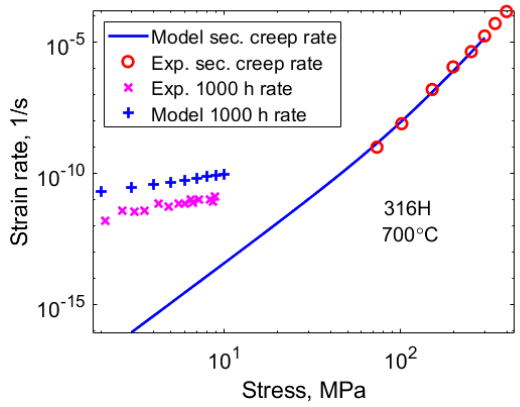

Figure 1. Creep rate versus stress for 316H at 700ºC. The creep rate at low stresses is evaluated from primary creep curves after 1000 h creep.

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Maximal strain rate sensitivity of quasi-stationary deformation strength when subgrain size matches grain size

Eisenlohr, Philip, Michigan State University, Chemical Engineering and Material Science, East Lansing
Blum, Wolfgang, Friedrich-Alexander University of Erlangen-Nuremberg, Erlangen

When the mean grain size d is comparable to the mean subgrain size w, for instance after grain refinement or deformation at high temperature and low stress, a significant fraction of grains is free from subgrains.Easy recovery of dislocations at high-angle boundaries makes subgrain-free grains softer than the grains that contain subgrains and low-angle boundaries.The observed inverse stress dependence of the quasi-stationary subgrain size $w_{qs} \propto 1/\sigma$leads to a significant variation of the fraction of subgrain-free grains with stress σ.A model is presented to demonstrate that this variation significantly enhances the rate-dependence of deformation strength, thus stabilizing against fracture.

This is not a pipe – issues of representation and reality in creep modelling

Brear, John, John Brear - Plant Integrity, Llanelli

Models in general

A scientific model is a formal representation of an idea – that is, of a hypothesis or of a theory. The methodology of modelling is therefore a particularisation of general scientific method and thus subject to the same discipline and rigour. In the present context, a materials model is a mathematical representation of some physical process, or combination of processes, whereby a material evolves from one state to another. Any proposed model should therefore represent both the state and the manner of its evolution. Any materials state or process can be modelled to the extent to which it can be described, since a mathematical representation is merely a description in a particularly formal language. A creep model is a materials model that includes a certain class of mechanical effects. No model is "right" in absolute terms, since a model is only a description of reality. In practice, the "right" model is the one that yields the best useful compromise between its descriptive and predictive capability and its simplicity of formulation and application.

Issues with creep modelling

Primarily addressing mechanistically informed creep models appropriate to component life prediction, this paper considers issues that can complicate their formulation and their interpretation. These issues include:

The process of deriving a model – the relation between empiricism and logical inference or deduction

The difference between state and coordinate variables

What is a true materials property – such as creep rate – as opposed to the result of a process – such as proof strength, UTS, rupture life?

The representation of discrete processes by continuum approximations

The need to separate materials, geometry and loading factors

The treatment of initial, final and boundary conditions

Allowing for extraneous, interfering mechanisms

The problem of accidental similarities between mathematical formulae, and the dangers of making false inferences therefrom

Examples illustrating these are given, taken from the author"s own work or from the literature.

Intention

The intention of this paper is to stimulate discussion.

14:00–14:45 Abstract talks IV

On the role of stacking fault shearing in CoNi-base superalloys during high temperature creep

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Volz, Nicklas, Friedrich-Alexander University of Erlangen-Nuremberg, Department of Materials Science & Engineering, Institute I: General Materials Properties, Erlangen
Amon, L., Friedrich-Alexander University of Erlangen-Nuremberg, Department of Materials Science & Engineering, Institute I: General Materials Properties, Erlangen
Göken, Mathias, Friedrich-Alexander University of Erlangen-Nuremberg, Department of Materials Science & Engineering, Institute I: General Materials Properties, Erlangen
Neumeier, Steffen, Friedrich-Alexander University of Erlangen-Nuremberg, Department of Materials Science & Engineering, Institute I: General Materials Properties, Erlangen

In contrast to Ni-base superalloys, the γ precipitates in Co-base and CoNi-base superalloys are already sheared in the beginning creep stages under the formation of superlattice intrinsic or extrinsic stacking faults (SISFs/SESFs) in the intermediate temperature and stress regime.[1–3] Despite these shearing events, the creep strength of quaternary single-crystalline (SX) Co-base superalloys is similar to 1st generation Ni-base superalloys.[4]
The present work aims to clarify the significance of stacking fault shearing and to evaluate the strengthening and/or softening effect of high stacking fault densities during creep in CoNi-base superalloys. Using transmission electron microscopy, the occurring deformation mechanisms are investigated in different creep regimes. By quantifying the defect and planar fault densities, the contribution of different deformation mechanisms to the overall plastic strain and their impact on the creep curves are calculated. Special emphasis is laid on the strengthening effect of stacking fault interactions. In order to further investigate this strengthening effect, extreme stacking fault densities, i.e. up to 40 stacking faults per precipitate, are introduced into the specimens prior to the creep experiments via constant strain-rate compression experiments. Subsequently, the creep strength is evaluated as a function of the initial stacking fault density for the different creep regimes. Finally, the thermal stability of the stacking fault structures are addressed and implications for the creep behavior are discussed.

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In situ real time study of stresses of a single crystal superalloyusing far-field X-ray diffraction

Schenk, Thomas, Institut Jean Lamour, SI2M, Nancy
Jaques, Alain, Institut Jean Lamour, SI2M, Nancy
Trehorel, Roxane, Institut Jean Lamour, SI2M, Nancy

During high temperature creep Ni-based single crystal superalloys develop a rafted microstructure with alternate semi-coherent layers of a ductile fcc γ matrix (channels) and hard γ' phase (rafts) with a L12 Ni3Al structure, perpendicular to the [001] tensile axis.
Using a simple multilayer-model, stresses and strains within both phases can be calculated: provided we know the applied load, the free lattice mismatch δ(T) and the $\delta^{\perp}$ mismatch (in the [100] and [010] directions, due to dislocations in the γ/ γ interface) between both phases.
The evolution of $\delta^{\perp}$ has been previously recorded by in situ triple-crystal synchrotron X-ray diffractometry (TCD) at 300 s intervals using high resolution diffraction measurements of the (200) diffraction peak during in situ high temperature (950°C-1100°C) creep tests, but the interval between successive measurements is too large to record the transient behaviour of the material following load or temperature jumps, especially the onset of plasticity within the γ rafts.
We present here observations with a new experimental setup: far field 3D imaging (FFXRD) of a single crystal diffraction peak in transmission.
This technique allows to follow the evolution of $\delta^{\perp}$ during transients in creep tests retaining a high (10-5) absolute precision.

Advantages and drawbacks of FFXRD compared to TCD will be discussed, and a short analysis of the experimental results for one specimen will be given.

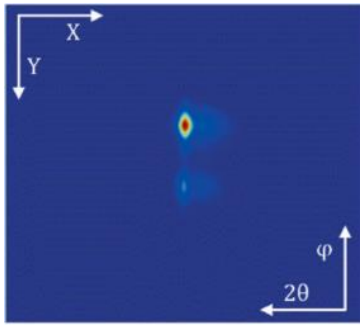

Figure 1: Recorded (200) far field spot

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Introduction of first-principles methods into fundamental creep models for austenitic steels

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Sandström, Rolf, KTH Royal Institute of Technology, Materials Science and Engineering, Stockholm
Zhang, Jing, KTH Royal Institute of Technology, Materials Science and Engineering, Stockholm
Korzhavyi, Pavel, KTH Royal Institute of Technology, Materials Science and Engineering, Stockholm

Austenitic stainless steels are candidate materials for future power plants, including fossil fuel-fired and nuclear power plants, where the materials are subjected to high temperatures and stresses. Creep is an important factor influencing the service security of the materials. Unlike the short-term tensile or hardness tests, creep tests often take years and consequently, predictive modeling of creep is essential. To facilitate the research and development, it is necessary to understand the long-term creep rupture life-controlling mechanism. Instead of using empirical models, fundamental models have been proposed, where the evolution of microstructure and defects are involved. Brittle creep rupture models are proposed based on the creep cavitation models. Dislocation generation, precipitation hardening contribution, solid solution hardening and splitting of dislocations are considered in models for both ductile and brittle creep rupture. The parameters involved in the models are well defined. Some physical parameters could only be obtained from the first-principles calculations like the elastic constants and lattice misfit parameters. In the first-principles calculations, the open-source code EMTO (Exact Muffin Tin Orbitals) was used to compute the high-temperature total free energy, including the electronic, magnetic and vibration contribution, with CPA-DLM (Coherent Potential Approximation-Disordered Local Moment) approach, where the paramagnetic was considered. The precipitation kinetics was calculated with the thermodynamic software MatCalc. Thus, multiscale models could be established across the atomic-scale, micro-scale and macro-scale. Figure 1 shows the creep rupture prediction of HR3C (25Cr20NiNbN) austenitic stainless steels based on the proposed multiscale fundamental models at temperatures of 650-750 °C.

15:30–16:15 Invited talks IV

Deformation mechanisms in *Immm*-Ni_2 (Cr,Mo,W)-containing Haynes® 244® superalloy

Titus, Michael, Purdue University, Materials Engineering, West Lafayette
Mann, Thomas, Purdue University, Materials Engineering, West Lafayette
Fahrmann, Michael, Haynes International, Kokomo

The Ni-based superalloy Haynes® 244® is strengthened by a Immm-Ni2(Cr,Mo,W) body-centered orthorhombic (BCO) intermetallic phase. Due to the low symmetry of this phase, the deformation mechanisms are expected to be complex, and a myriad of planar defects are predicted to be observed on the {013} planes, which are nearly co-planar to the face-centered cubic (FCC) matrix {111} planes. These defects include those analogous to the γ'-Ni3Al phase: superlattice intrinsic/extrinsic stacking faults, antiphase boundaries, and complex stacking faults, however other planar defects also exist. To assist in the identification of deformation pathways, we utilized ab-initio density functional theory calculations to determine the generalized stacking fault energy surface or γ-surface. Favorable, low-energy deformation pathways and unfavorable barriers have been identified. These pathways along with preliminary compression creep results will be presented, and comparisons will be made to similar Ni-based alloy systems containing γ' and γ'' precipitates.

Characterisation of the creep behaviour of novel pesting-resistant Mo-Si-Ti alloys

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Kauffmann, Alexander, Karlsruhe Institute of Technology, Institute for Applied Materials, Karlsruhe
Heilmaier, Martin, Karlsruhe Institute of Technology, Institute for Applied Materials, Karlsruhe

Mo-Si based alloys are attractive candidates for potential high temperature applications, especially as alternatives for Ni-based superalloys, due to their high solidus temperatures resulting in promising creep behaviour. However, the main challenge for industrial application is the insufficient oxidation behavior at temperatures below 1000 °C (pesting) [1-5]. Several alloying strategies have been developed, among which macro-alloying with Ti was found to be the most effective approach for both, improving the oxidation behaviour [2, 6, 7] and achieving a significant reduction in density down to 6-7 g/cm3 [8]. The ternary Mo-Si-Ti system profits from eutectic (L → MoSS Ti5Si3) and eutectoid phase reactions (Mo3Si → MoSS Mo5Si3) leading to fine-scaled microstructures. The eutectic alloy Mo-20Si-52.8Ti (at%) possesses an outstanding oxidation behaviour even at the critical temperature of 800 °C [8]. However, this first pesting-resistant alloy exhibits minor creep resistance compared to the Ti-lean eutectoid alloy Mo-21Si-34Ti, which undergoes severe pesting. Further alloy design revealed that a minimum nominal Ti content of 43 at% is mandatory to ensure pesting-resistance [9]. With the aim of improving the creep resistance of Mo-Si-Ti alloys by guaranteeing the pesting-resistance and still a reasonable reduction in density, in this study, the Mo content is adjusted within this chemical composition range in order to increase the solidus temperature. These requirements are met by the intermediate eutectic-eutectoid alloy Mo-21Si-47Ti with a density of 6.3 g/cm3. Thereby, focus is placed on the characterisation of the creep behaviour by assessing the role of the solidus temperature of the investigated alloys and the

individual phases MoSS, Ti5Si3 and Mo5Si3 being present in the microstructures. The first is found to determine the creep resistance of the these alloys as shown by investigations at the same homologous temperature. Furthermore, the influence of the microstructural scale is found to be negligible, as an artificially coarsened eutectic alloy does not show any improvement in creep resistance. However, for further improvement in creep resistance, primary solidified Mo5Si3 is considered as strengthening phase. It is revealed that eutectic-eutectoid alloys with primary Mo5Si3-crystals possess higher minimum creep rates. Therefore, the impact of varying volume fractions of primary Mo5Si3 on the creep behaviour is analysed while the oxidation behaviour in the pesting regime is studied.

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15:30–16:30 Abstract talks V

Recovery strain in AlTiVNbZr0.25 at 1073K

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Sklenička, Václav, Institute of Physics of Materials, Czech Academy of Sciences, Advanced High-temperature Materials, Brno,

Dislocation motion causes generation and storage of dislocations as well as local recovery of dislocation line length. The strain associated with recovery (recovery strain) was investigated during compressive creep at 1073 K of the high entropy alloyAlTiVNbZr0.25by performing sudden stress reductions. Large reductions suppress work hardening so that the strain rate is solely given by the recovery strain rate. As the dislocation density and its recovery rate decline after the stress reduction, the recovery strain rate also declines. Finally a quasi-stationary state with balance of generation and recovery of dislocations is established at the reduced level of stress. The transient response to the stress reduction is modelled on the basis of class A (alloy class) creep behaviour, assuming a wide distribution of localeffective stresses driving viscous dislocation motion during recovery.

Creep properties of high entropy alloy CrMnFeCoNi in single crystalline state from 700 °C to 1050 °C

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Glatzel, Uwe, University of Bayreuth, Metals and Alloy, Bayreuth

The equiatomic and single-phase high entropy alloy CrMnFeCoNi also known as Cantor alloy, is an ideal candidate for the study of solid solution hardening in a statistically distributed crystal lattice. The Cantor alloy shows extraordinary mechanical properties, for example a strong temperature-dependent yield strength, accompanied by just a small strain rate dependence. This effect is not expected for face-centered cubic (fcc) metals.
To investigate the fcc CrMnFeCoNi alloy in more detail, the focus lies on the entropy affecting mechanical, especially creep properties. Using an arc-melting furnace, master alloys are produced under argon in the first step. Then the pre-alloyed buttons are cast as a single-crystal (SX) by induction casting under argon for further characterization. Technical pure nickel (fcc) is also cast in single crystalline state in the same way and is used as a reference material. The produced single crystals have a crystal orientation close to the [001]-direction and show a homogenous element distribution. For the mechanical testing of the alloys, miniature specimens are prepared by electrical discharge machining (EDM). Creep tests are carried out under vacuum (4·10-4Pa) and in air at temperatures of 700 °C, 980 °C and 1050 °C at different loads.
The results of the creep experiments are used to determine the Norton-exponent as well as the Larson-Miller parameter at a strain level of 2 % for both CrMnFeCoNi and pure Ni. Finally, this interesting findings are compared and discussed with respect to creep results of nickel-based superalloys using the Norton plot as well as the Larson Miller plot.

Creep-corrosion of Al0.1CoCrFeNi high entropy alloy in a molteneutectic salt

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Molten salt corrosion of Al0.1CoCrFeNi HEA samples under biaxial tension was investigated. The surfaces of samples were subjected to as much as 70 MPa biaxial tension and molten Na2SO4-NaCl simultaneously using a novel disc-bend configuration for up to 12 hours. The resulting structure of the exposed surfaces and cross-sections were examined using scanning electron microscopy (SEM) and elemental composition maps and profiles were determined using energy dispersive spectroscopy (EDS). While the present HEA is fairly corrosion resistant when unloaded, formation of relatively large pores, deep penetration of a Cr-depleted reaction layer, and dissolution of external oxide films occurred for the samples under biaxial tension. Based on these findings, it is clear that the influence of even modest amounts of creep deformation on corrosion in molten salt is severely detrimental for the present HEA. This effect can be attributed to dislocation facilitated diffusion within the reaction layer.

A novel displacement cascade driven irradiation creep mechanism in α-zirconium and copper

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Metals and alloys, such as stainless steels and zirconium alloys, used as structural materials in the nuclear core of pressurized water undergo irradiation creep deformation. The mechanical behavior is well characterized at the macroscopic level [1]. Yet, the underlying microscopic mechanisms are still unclear [2]. Indeed, many theoretical mechanisms have been proposed in the literature, but only few experimental results were conclusive.
Recent in situ TEM straining experiments under ion irradiation conducted on a zirconium alloy (Zircaloy-4) have demonstrated that, at high stress levels, dislocations pinned on irradiation induced point defects clusters start to glide once under irradiation [3]. Same results were then observed for pure copper, which is considered as a model material for stainless steels and in metallurgy in general. One of the proposed hypotheses for this phenomenon was that the observed dislocation glide assisted by irradiation was due to a direct interaction between the displacement cascade and the pinned dislocation. Indeed, we suggest that, if the stress is high enough, a displacement cascade occurring very near the pinning point of the dislocation would induce the release and glide of the dislocation.
A molecular dynamics study in α-zirconium was conducted to test this hypothesis. The interaction of a screw dislocation with an interstitial loop was first studied. The objective was to pin the dislocation on the irradiation defect. The possible unpinning of the dislocation by a PKA (Primary Knock-on Atom) for stresses slightly lower than the unpinning stress was then studied. Based on these numerical simulations, a simple analytical probabilistic model was proposed to explain the irradiation creep deformation under high applied stress.

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Scientific Programme – 15 June 2021

09:00–10:00 Invited talks V

Creep behavior of Mg-Y-Zn ternary dilute solid solution alloys
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Yttrium is one of the most attractive alloying elements for magnesium to improve high temperature creep strength. The strengthening effect of yttrium addition was observed even in small addition, which is explained as the dislocation locking effect by the segregation of yttrium to stacking faults between partial dislocation pairs on the basal planes. Moreover, this effect is enhanced by the simultaneous microalloying of zinc. On the other hand, the concentration dependence of solute yttrium for creep strength of Mg-Y-Zn ternary alloys is smaller than that of Mg-Y binary alloys. Transmission electron microscopic observation has revealed that many planar type (intrinsic type II) stacking faults are formed spread out elliptically on the (0001) magnesium matrix planes in crept Mg-Y-Zn alloys. The net solution concentration of yttrium diminishes in Mg-0.87Y-0.02Zn alloy as creep progresses since the segregation of yttrium and zinc occurs in these planar type stacking faults.
The addition of yttrium also changes the temperature dependence of activation energy for creep of magnesium. In many hcp metals and alloys, two kinds of dislocation creep regions with different activation energy are observed. Transition temperature between two dislocation creep mechanisms of pure magnesium is around 700K. However, since the suppression of the recovery process by the dislocation locking effect is higher in dislocation climb than dislocation cross slip, high temperature dislocation creep region is observed below 500K in Mg-Y-Zn solid solution alloys. Furthermore, many step-like c-dislocations are found on the basal and the non-basal planes after creep at 450 K. The dislocation segment on the non-basal planes of these step-like c-dislocations are expected to act as obstacles to the dislocation slip on the primary slip system (the basal slip). Effects of the "forest dislocation hardening" by step-like c-dislocations of Mg-Y-Zn dilute solid solution alloys were also examined in this investigation. The deformation mechanisms and rate-controlling factors of creep in these alloys will be discussed based on creep parameters and microstructural observations.

Interface Strengthening in α-Mg/C14–Mg_2Ca Eutectic Alloy
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In tensile tests, α-Mg/C14–Mg2Ca eutectic alloy with a lamellar structure is plastically deformed above 473 K but ruptures before yielding at temperatures below 448 K. This study investigates the effect of the α/C14 interface on the creep strength of α-Mg/C14–Mg2Ca eutectic alloy at 473 K under 40 MPa stress. The creep curves of the alloy exhibited three stages: a normal transient creep stage, minimum creep-rate stage, and accelerating stage. The minimum creep rate was proportional to the lamellar spacing, indicating that the α/C14 lamellar interface plays a creep-strengthening role. In high-resolution transmission electron microscope observations of the specimens after the creep test, a dislocations appeared within the α-Mg lamellae and were randomly distributed on the α/C14 interface. It was deduced that the α/C14 interface presents a barrier to dislocation motion and does not recover its dislocation caused by the creep test.

Understanding the high creep resistance of MRI 230D magnesium alloy through nanoindentation and atom probe tomography

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Due to their low density, magnesium alloys are very appealing for light weight construction. However, the use of the most commonly used magnesium alloy AZ91 in structural applications is limited to low temperature applications as creep becomes an important issue for this alloy above temperatures of about 150°C. Several alloys with an improved creep resistance have been developed in the past, for example the MRI230D alloy or Ca alloyed AZ91 variants. However, there is an ongoing discussion in literature regarding the mechanisms of the improved creep resistance. One factor claimed responsible for the improved creep resistance are the intermetallic phases which form during casting process. Another possible factor is an increased creep resistance of the Mg crystal itself, caused by precipitation.

To gain more insight in the improved creep resistance of the alloy MRI230D, nanoindentation measurements have been performed on the different microstructural phases of as cast, creep deformed and heat treated samples of MRI230D. For comparison Ca alloyed AZ91 variants have also been investigated. These nanoindentation measurements clearly show that the intermetallic phase (IP) of the alloy MRI230D has a higher strength compared to the Ca alloyed AZ91 variants. Even more important, the IP of the alloy MRI230D does not loose strength during creep deformation. High temperature nanoindentation measurements at a temperature of 200°C clearly show that the IP of the MRI230D alloy maintains its strength when compared to room temperature measurements. This is in clear contrast to the Ca alloyed AZ91 variants, where the IP is significantly softer at 200°C than at room temperature. Atom probe measurements have been used to gain insight in the differences in term of chemical composition between the IPs of MRI230D and the Ca alloyed AZ91 variants in order to understand the dissimilar behaviour in terms of strength loss with increasing temperature.

10:30–11:30 Invited talk VI

Creep response of additively manufactured Mo-Si-B alloys

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Schmelzer, Janett, Otto-von-Guericke University Magdeburg, Mechanical Engineering, IWF, Magdeburg
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Refractory metal alloys, e.g. from the Mo-Si-B system, are potential candidates for applications in gas turbines. Additive manufacturing (AM) of such alloys is quite challenging due to their ultra-high melting point above 2000°C and the strong tendency to oxidize at intermediate temperatures. This presentation will show the successful processing of three-phase Mo-Si-B alloys from pre-alloyed powders by two different laser-based techniques. The characteristics of AM materials will be compared to similar alloy compositions processed by powder metallurgy, ingot metallurgy and directional solidification.

It will be demonstrated that the AM materials are crack-free and the constituents are homogeneously distributed. The microstructure is very fine as can be compared to powder metallurgically processed Mo-Si-B. Interestingly, the brittle-ductile-transition temperature of AM Mo-Si-B materials is lower in comparison to similar alloy compositions produced by other processing methods. Creep response is investigated by stress-controlled creep tests at temperatures > 1000°C; these temperatures are considered as possible application temperatures for turbine blades. In general, the creep resistance of Mo-Si-B alloys depends not only on the microstructure size scale but also on the morphology and volume fraction of i) the comparatively weak Mo solid solution phase and ii) the creep resistant silicide phases. The strong sensitivity of creep response on these microstructural features will be shown using examples of alloys from the Mo solid solution primary solidification area as compared to eutectic Mo-Si-B alloys with a high volume fraction of silicide phases. Furthermore, the experimental data were used as a basis for thermal and mechanical analyses by the finite element method using a simple geometry of a turbine blade.

How particle populations change during long term creep of a 12% chromium tempered martensite ferritic steel

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Tempered martensite ferritic steels (TMFSs) contain 9 to 12% Cr. They are often used for steam headers and other thick section pipes which operate at temperatures up to 600°C in fossil-fired power plants. Thus they have to withstand mechanical loads at high temperatures, where creep limits there exploitable service life. It is well known that microstructure strongly influences creep. Most importantly, carbides, nitrides and Laves phase particles play a key role as obstacles to dislocation motion. It is also well known, that particle populations, which form during the heat treatment of the materials, evolve during creep. Particles grow and change their chemical compositions. New particles (like Laves phase and Z-phase) nucleate and grow during creep. In the present work, we take a new look at material states, which had been previously investigated as thin foils in the TEM by Aghajani et al. [Acta Mater. 57(2009)]. This time, we make replicas, which allow characterizing the morphology, crystallography, and chemistry of particles in the absence of the magnetic matrix. This gives special emphasis to phases like Z-phase, which in thin foils can be easily overlooked. The material states investigated in the present work were subjected to creep at 550°C at 120 MPa. The rupture time of the steel which was investigated, a German grade X20 CrMoV 12 1, was 139 000 hours. In addition to this final state, a number of intermediately interrupted specimens were available (interruptions: 12, 51, 81 k-hours). This unique set of specimens in combination with the replica technique and advanced analytical transmission electron microscopy allowed us to obtain new insight into how microstructure and especially particle populations evolve under conditions of long term creep. Most importantly it is shown that the Laves phase particles nucleate after 12 k-hours and have an almost constant chemical composition throughout creep. The Z-phase is also detected after 12 k-hours and its composition evolves as creep proceeds. The results are discussed with respect to creep life assessments and considering previous results which were reported in the literature.

Creep deformation behaviour of cold worked Ti-modified 14Cr-15Ni austenitic stainless steel

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Ti-modified 14Cr-15Ni austenitic stainless steel has been developed for nuclear fuel cladding and wrapper material for its superior creep strength and better void swelling resistance during service. Creep behavior of indigenously developed Ti-modified 14Cr-15Ni austenitic stainless steel in solution annealed, 10, 20, 30, and 40 percent cold worked conditions was studied at 973 K and at various stress levels. Increase in cold work level resulted in denser dislocation network resulting in prominent low angle grain boundaries. Low angle boundary fraction increased as the amount of cold working increased from 10 to 40 percent prior to creep loading. The variation in the fraction of low angle impacted the creep rupture strength of the steel. During creep exposure of the cold worked steels, softening occurred due to both recovery and recrystallization. The extent to which recrystallization and recovery occurred during creep was dependent on the level of cold work. Subgrain formation and recrystallization resulted in enhanced recovery and variation in the creep strength cold worked steel. Precipitation of fine titanium carbides during creep exposure effectively pinned the dislocations, which also contributed to enhanced creep strength of steel.

10:30–11:15 Abstract talks VI

Microstructural degradation and residual creep life of 9Cr longitudinal elbow weldment after 81,544 hours service

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Shimoda, Junji, Mitsubishi Power, Ltd., Nagasaki
Fujita, Masaaki, Mitsubishi Power, Ltd., Nagasaki

Mod. 9 Cr -1 Mo steel (Gr. 91) is superior to low alloy steel in creep rupture strength and corrosion resistance, and has been used in great numbers in main piping and header of USC power boilers in all over the world. However, the creep strength of the welded joint of this steel is essentially lower than that of the base metal. The long-term creep rupture strength of the base metal and the welded joint is weaker than the strength extrapolated from the short-term creep rupture test, and the estimated value of the long-term creep rupture strength is greatly affected by the quantity of the long-term test data under low stress region. In fact, in Japan, U.S.A. and Europe, the review of long-time creep strength is carried out multiple times, and the extrapolated creep rupture strength of base metal and joint is reexamined low in each time.

The number of USC power plants operated for more than 100,000 hours is increasing. It is important to grasp the accurate ultra-long-term creep rupture strength from the viewpoint of those maintenance and management.
In this study, the total creep rupture life was obtained by adding the remaining life estimated by the creep rupture tests and the plant operation time. And then the validity of creep rupture life by proposed formula was evaluated by comparing the total estimated life by this experiment.

A seam-welded elbow serviced at 610 °C for 81,544 h as a high-temperature reheat piping was investigated for test material. Remaining creep rupture life at serviced condition was expected by creep rupture tests. Two methods, the Larson-Miller method and the Iso-Stress test, were applied to estimate the remaining life. The longest creep rupture time is 29,450 h.

Although there were some differences in the remaining life estimated by the Larson-Miller and Iso-Stress methods, the total creep rupture life estimated in this study was almost equal to the estimated value of the latest creep life evaluation equation. Therefore, it is concluded that the validity of the creep life evaluation equation was confirmed. However, it is necessary to pay attention, because different results may be obtained in other heats.
Extremely softened coarse grain microstructure with 140 HV in Vickers hardness was confirmed in weld metal and a part of HAZ. This was considered to be ferrite grains produced by recrystallization during long operation. And, in the HAZ, the creep cavity was generated, and it seemed due to creep damage with the long-term operation.

Field Dislocation Mechanics and its extension to the Mesoscale: application to Poly-Crystal viscoplastic behavior of Uranium Dioxide

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Uranium dioxide ceramics (UO2) are widely used as nuclear fuels in current Nuclear Pressurized Water Reactors (PWRs). Under irradiation, it is well known that the dislocation flow governs its viscoplastic behavior that determines the material performances.

In the present work, our purpose was to take into account the three dislocations slip systems (⟨110⟩{100}, ⟨110⟩{110} and ⟨110⟩{111}) in the viscoplastic model, which was developed by considering the dislocation glide. This model is based on the continuous theory Field Dislocation Mechanics (FDM) suggested by A. Acharya [1], the Geometrically Necessary Dislocation densities (GNDs) are described through the Nye dislocation density tensor.

In fact, the theory allows to consider the GNDs motion by using the dislocation transport equation, which is numerically solved with Fast Fourier Transform approach. Then, the extension of FDM is used to include crystal plasticity for Statistically-Stored Dislocations (SSDs) that is defined as Phenomenological Mesoscopic Field Dislocations Mechanics [2] (PMFDM). PMFDM allowed us to investigate collective evolution of dislocation density at mesoscopic scale. In contrast with classic plastic formulation, the model allowed us to distinguish both populations of dislocations: SSDs and GNDs and the resulting phenomena arising from each one, which are produced at different scales. The model is used to investigate i) the plastic flow due to the GNDs in addition to SSDs, ii) the forest hardening by the mean of the generalized Taylors equation, and iii) the collective evolution of dislocations as well as their pile-up on the grain boundaries, the so-called Hall-Petch effect.

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Effect of strain gradients on creep response of cantilevers in five-power-law regime

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Cantilevers register a gradient stress state across their thickness and length as a result of applied load. The resultant strain distribution across the cantilever varies according to the stress distribution and leads to a strain gradient. The effect of strain gradient on general plasticity in beams has been reported in many previous studies; however, their effect on the creep response has not been investigated thoroughly.

The strain gradients across a cantilever is associated with geometrically necessary dislocations that result in hardening the material"s flow properties during plastic bending, thus resulting in a sample size effect. During creep, the effect of additional dislocation density can either be attributed to an increase in the overall dislocation density or to the interaction of geometrically necessary dislocations with statistical dislocations causing entanglement of dislocation networks. Additionally, the uniaxial sample size effect in power-law creep leads to a softening in creep response. In this context the interplay between the strain gradient hardening and uniaxial-sample size effect on creep is of much interest. On one hand, the strain gradient effect may lead to a hardening in creep response of cantilevers in the dislocation creep regime. On the other hand, the dislocation network as rate determining parameters leads to softening in creep resistance as a function of decrease in sample size. The softening in creep response occurs because of the free surface effect and contribution from grain boundary diffusion in a limiting case when the sample size is comparable to the crystallite size.

In this study creep tests were performed by bending cantilevers of commercially pure polycrystals, high purity single and polycrystal of aluminum and commercially pure lead in power law regime. Digital image correlation was used in conjunction with bending tests to obtain 2D strain fields. The resultant creep response was compared with bulk uniaxial creep response in the size range of 5 – 0.5 mm. It was observed that the cantilevers of thickness ~ 0.5 mm are more creep resistant in steady state than the bulk 5 mm samples . The hardening appears to vary linearly with the inverse of sample size. The activation energy for creep in samples subject to strain gradient effect was found to be same as that of the bulk samples. The creep substructures obtained in the presence and absence of strain gradient hardening were compared in terms of dislocation substructure sizes recorded from EBSD micrographs. At equivalent stresses the dislocation substructures in samples subject to strain gradient hardening were observed to be finer as compared to the bulk structures. The sessile nature of GND"s is attributed to possible interaction between the GND"s and statistical dislocations which leads to the formation of finer substructures and therefore hinders mobile dislocation motion (glide component of the sequential glide-climb process).

12:30–13:30 Abstract talks VII

Creep properties studies on yttrium-added Hastelloy-X superalloy built by selective laser melting

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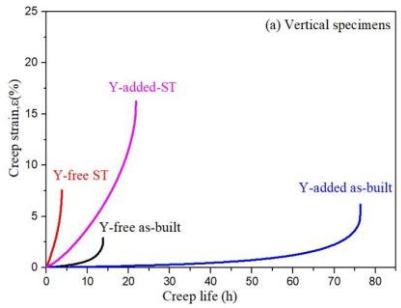
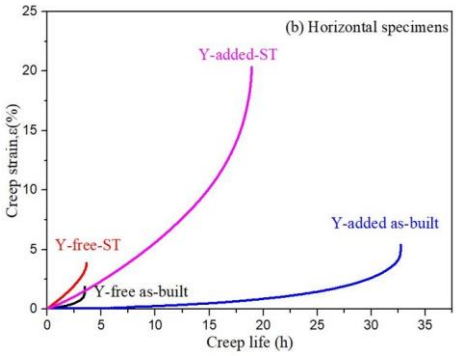
Hastelloy X is a solid solution strengthening Ni-base superalloy widely used in critical sections of the aero-engine such as transition ducts, combustor cans, etc. This study's primary purpose is to understand the effect of yttrium on creep properties in Hastelloy-X. In this present study, two kinds of Hastelloy-X specimens with 0.046 wt.% yttrium and yttrium-free were built from pre-alloyed powders (Table 1) using EOSM 290 with the same processing parameters. The solution heat treatment (ST, 1177°C for 2 h) was conducted. A creep test was conducted at 900°C and 80 MPa. The creep stress was applied along with the vertical and horizontal directions.

	Ni	Cr	Mo	W	Co	Fe	C	Si	Mn	Y
Y-free	Bal.	21.98	8.94	0.54	1.48	18.04	0.082	0.09	<0.01	-
Y-added	Bal.	22.25	8.86	0.60	1.54	18.26	0.073	0.01	-	0.046

Table 1. Chemical compositions of two kinds of the alloy (mass %)





Investigation of primary creep regeneration (PCR) for 316H and 10%Cr steels

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Primary creep regeneration (PCR) is an important phenomenon observed under stress-varying creep conditions. For some materials and loading conditions, a load reversal might clear the previous strain hardening memory and lead to a high creep rate period upon reloading, and therefore re-occurrence of the primary creep stage (i.e. PCR). This study investigates the PCR behaviour of two high-temperature steels with very different metallurgical-microstructural characteristics at their respective application temperatures; namely 316H austenitic stainless steel at 650 °C and a 10%Cr martensitic steel at 600 °C. A series of high-temperature stress-varying creep tests for each steel were conducted to investigate the sensitivity of PCR to the different loading parameters (e.g. reverse-loading magnitude and duration) [1-3]. The observations were employed to develop a phenomenological model for representation of the PCR phenomenon and its sensitivity to the different loading parameters under stress-varying creep loading conditions.

Furthermore, interrupted stress-varying creep tests were performed to generate representative samples for the microstructural state of the steels before, during and after stress transients (ex-situ experiments). Different advanced microstructural investigation techniques, such as transmission electron microscopy (TEM), electron backscatter diffraction (EBSD) and neutron diffraction (ND) have been employed to characterize the microstructural parameters (such as dislocation density and lattice strain) of the samples from the ex-situ experiments. In addition, observations from a set of in-situ synchrotron X-ray diffraction experiments have been exploited to continuously monitor the dislocation density and lattice strain evolution during stress-varying creep loading conditions. The observations from mechanical, ex-situ, and in-situ experiments are being explored for development and effectiveness verification of a physically-based model.

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Effect of welding on stress relaxation cracking of 316L(N) austenitic stainless steel

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The high temperature gradients in stainless steels resulting from welding induce residual stresses, strain, as well as the precipitation of undesirable phases at grain boundaries in the heat-affected zone (HAZ) [1]. When exposed to high temperatures (higher than 500°C), the relaxation of these residual stresses can further damage the grain boundaries near the growing precipitates leading to intergranular fracture [2]. The study of stress relaxation cracking (SRC) in laboratory conditions has been performed in previous works by introducing tensile residual stresses via compression of CT like samples made cold rolled material [2,3], in order to reproduce mechanical properties observed in the HAZ. It was shown that SRC could only be observed in 316L(N) for a specific residual stress threshold [2]. However, the properties of samples obtained thereby had a homogeneous microstructure and did not account for real metallurgic and mechanical heterogeneities found for welded material.

Therefore, the aim of this work was to investigate SRC in welded 316L(N) and compare it with non-welded samples. An experimental procedure was established to get insight into SRC phenomenon: the final compression at room temperaturewaspreceded by cold rolling or welding. The conditions of compression were carefully chosen using preliminary finite element analysis, to ensure that the anticipated threshold level of residual stresses required to induce SRC was achieved during compression. The studied samples presented different levels of residual stresses and plastic strain. They were also more or less prone to carbide precipitation. After stress relaxation tests carried out at 575 and 600 °C for 580 and 1470 h, the microstructure and grain boundary damage were analyzed using observations carried out by SEM.

Damage in samples in cold rolled samples appeared as creep-like cavities on grain boundaries (Figure 1) exclusively nucleated on M23C6 carbides. Results show longer relaxation times induce higher number and size of cavities, with 600 °C being the most damaging temperature. In as-welded samples,very few damage was observed after relaxation in temperature. In welded samples with additional stress introduced by compression a high number of cavities was found, mostly present in the HAZ regions where the highest levels of accumulated strain, tensile residual stresses and precipitation density were originally found. These first results show the residual stresses introduced solelyby a single pass welding are insufficient to lead to SRC. Further investigations on SRC susceptibility of multi-pass welded samples are foreseen, assumed to induce higher levels of residual stresses.

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Effect of heat treatment on the creep response of an AlSi10Mg alloy produced by additive manufacturing

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Additive manufacturing (AM) is a relatively new technology that can significantly affect product development in mechanical industry. The extremely fine microstructure of AM components provides unique tensile properties, in terms of high strength. In the case of the AlSi10Mg, in particular, the microstructure of the as-deposited material is formed by columnar Al grains several microns in diameter. On a finer scale, the columnar grains are fragmented in smaller cells, few hundreds of nm in diameter, separated by eutectic regions rich in Si (and Mg). The long cells, in turns, exhibit a substructure of equiaxed sub-cells, again few hundreds of nm in diameter, again separated by eutectic regions rich in Si. This peculiar microstructure greatly influences the creep response of the alloy. The material behaves as a sort of composite, in which the sub-micrometric Si particles act as reinforcements. In this context, prior heat treatments, such as stress relieving or annealing, affect the initial microstructure and, as a consequence, greatly influence the creep response. The present papers aims at investigating the effect of a stress relieving treatment carried out at 205°C for 2 h on creep samples produced by AM with the following deposition parameters: substrate temperature of 150°C, laser power 350 W, spot size 80 □m, scan speed 1.15 m/s, hatch spacing 170 □m, layer thickness 50 □m. The growth direction was parallel to the sample axis (loading direction). Constant load creep experiments were carried out on annealed samples at 150 and 175°C. The minimum creep rate dependence on applied stress was then compared with the data obtained by testing the as-deposited material in similar experimental conditions. The experiments clearly confirm

that stress relieving leads to a notable increment of the minimum creep rate. In particular, at 150°C, the increase in minimum creep rate is close to 2 orders of magnitude, while is somewhat lower at 175°C. This behavior was rationalized by considering the strengthening effect of Si particles, and the coarsening phenomena occurring during stress relieving. The material response was also described by a physically based constitutive model.

14:00–15:00 Abstract talks VIII

Microstructure changes and creep behaviour of P92 steel processed by severe plastic deformation

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Blum, Wolfgang, Friedrich-Alexander University of Erlangen-Nuremberg, Inst. f. Werkstoffwissenschaften, Erlangen
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Introduction/purpose
High pressure torsion of the austenitic steel 304 at room temperature generates ultra-fine ferritic grains. Creep at 923 K leads to phase transformation to austenite, grain coarsening and increase of deformation strength.

Methods
Coarse-grained austenitic steel 304L with mean grain size of about 30 µm was processed by HPT at room temperature using one rotation under an applied pressure of 6 GPa. Tensile creep tests at constant load were carried out in argon atmosphere at 923K and engineering stresses ranging from 50 to 150MPa. The microstructure was analyzed in a Tescan Lyra 3 XMU scanning microscope equipped with a NordlysNano EBSD detector and a transmission electron microscope Jeol 2100F operating at 200 kV.

Results
HPT deformation induces transformation of austenite into ferrite. At high temperatures the UFG ferritic microstructure is unstable and transforms back to austenite. Short term annealing and creep testing at 923 Kleads to formation of σ phase. It was observed that creep leads to dynamic recrystallization and grain coarsening. The misorientation distributions reveal that high-angle grain boundaries (~78%) with high fraction of Σ3 boundaries dominate after creep. The stress exponent $n=d\ln\dot{\varepsilon}/d\ln\sigma$ of the minimum creep rate is about 3. All tests start with a period of primary creep which shortens with decreasing stresses. The fracture strains increase significantly with decreasing creep load up to nearly 1 (100%) at engineering stress of 50 MPa, justifying the term superplastic behaviour. The largest part of creep strain occurs in the period where the relative increase of the creep rate with strain slows down. This feature is interpreted in terms of changes of the grain structure.

Conclusions
The HPT of 304L austenitic steel at room temperature led to the formation BCC crystal lattice with nearly random misorientation distribution. The creep exposure at 923 K led to the coarsening of austenitic grains while the mean grain size does not exceed the expected stationary size of subgrains. UFG steel exhibits significantly higher minimum creep rates than reported for CG steels.

Acknowledgments
The authors acknowledge financial support from the Czech Science Foundation (grant No. 19-18725S). The work was supported in part by a grant-in-aid from MEXT, Japan, for scientific research (A) (No. 19H00830). The HPT process was carried out at the International Research Centre for Giant Straining for Advanced Materials (IRC-GSAM) at Kyushu University, Fukuoka, Japan.

Low cycle fatigue modeling of a single crystal nickel-based superalloy at different temperature ranges

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Hartmaier, Alexander, Ruhr-University Bochum, Micromechanical and macroscopic modelling, Bochum

During the operation of turbines in jet engines or in power plants, high thermal and intermittent mechanical loads appear, which can lead to high-temperature fatigue failure or to thermomechanical fatigue. High performance superalloys with thermal barrier coating and advanced internal cooling system are the key materials of turbine blades, which are able to endure a wide range of stresses and temperatures. Since fatigue is a complex and time-consuming process, it is important to develop realistic numerical models to predict fatigue behavior and to extrapolate the limited experimental results into a wider range of thermomechanical conditions [1].

To accomplish this, a reference volume element (RVE), mimicking the typical γ/γ′ microstructure of a nickel-based single crystal superalloy is introduced. This cubic RVE consists of one central cubic γ′ precipitate and surrounding six half-width channels of γ matrix, which represents the simplest self-repeating structural unit of the microstructure. With the help of this RVE, the temperature and deformation-dependent internal stresses in the microstructure can be taken into account in a realistic manner, which proves to be essential in our understanding of the fatigue behavior of this material. The material behavior in the elastic regime is described by temperature-dependent anisotropic elastic constants. The flow rule for plastic deformation is governed by the thermal activation of various slip systems in the γ matrix, the γ′ precipitate and also by cube slip along the γ/γ′ microstructure. This phenomenological crystal plasticity/creep model takes different mechanisms into account, including thermally activated dislocation slip, the internal stresses due to inhomogeneous strains in different regions of γ matrix channels and in γ′ precipitates, the softening effect due to dislocation climb, the formation of ⟨112⟩ dislocation ribbons for precipitate shearing, the Kear-Wilsdorf locks.

This constitutive law is parameterized based on experimental data for CMSX-4 single-crystal superalloy by applying an inverse analysis to identify the material parameters based on many low cycle fatigue tests in the intermediate temperature and high stress regime. The identified material parameters could predict low cycle fatigue behavior at different temperatures. The model does not only reliably reproduce the experimental results along different crystallographic loading directions, but it also increases our understanding of the relative importance of the different deformation mechanisms for the fatigue behavior under various conditions.The main conclusion is that kinematic hardening, which is responsible for the shape of the hysteresis loops is completely described by the internal stress within the γ/γ′ microstructure.

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Phase-field coupled strain gradient crystal plasticity model to study high temperature creep in Ni-based superalloys

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Creep resistance of superalloys at high temperature is one of the most important parameters defining the range of applicability of superalloys. A combination of thermodynamics and material diffusion with elasto-plasticity within the framework of phase-field allows us a depth and systematic analysis of creep behavior of superalloys. In order to investigate the creep properties of single crystal Ni-based superalloys during the evolution of microstructure, a dislocation-based strain gradient crystal plasticity model [1] is implemented. The model is calibrated against the experimental results of a creep test at a high temperature and low stress. Then, it is used to predict the kinetics of the microstructure up to 1% creep strain, in which diffusion is assumed to be controlled by the slowest diffusing element Rhenium Re [2]. It is demonstrated that the loss of coherency between the matrix and the precipitate is crucial for the coalescence of the γ precipitate and initiation of rafting [3,4,5] and rotation of the γ matrix. It is further observed that highly localized shear bands were formed under high stresses and a tendency of rafting direction toward 45 degrees [6]. Finally, sensitivity of microstructural topology and evolution kinetics towards creep properties of superalloys was analyzed [4,5]. The effect of pre-strained matrix on the evolution of γ precipitates is highlighted.

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Numerical simulation of the damage and fracture of a cylindrical specimen subjected to thermal shock by using a gradient damage model

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In this paper, we report a study on initiation and propagation of cracks in brittle materiels subjected to thermal shock. In the context of the variational approach to the mechanics of fracture [1,2], a damage model was formulated and implemented into a finit element code. This damage model verifies the conditions of irreversibility, stability and total energy conservation. Using this numerical model, the modeling and numerical simulation of brittle fracture phenomena of a thin cylindrical specimen quenched with cold water were carried out. The numerical simulations allow a presentation on whole the process of crack initiation and propagation. It has been shown that there are three phases during the evolution of the damage. First an elastic response without any damage, then a phase of homogeneous damage, followed by several bifurcations where cracks distributed periodically. In this work, the spacing between cracks and the length of cracks is studied as a function of the intensity of the thermal and as a function of materiel parameters in particular the characteristic length. We show that we can predict the nucleation of a complex pattern of cracks and its propagation in good agreement with the experimental results of the literature [3]. This study leads to an understanding of the formation and evolution of thermal shock cracks observed during experiments on ceramics.

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15:30–16:30 Abstract talks IX

Two-scale simulation of plasticity in bcc metals: combination of atomistic simulation and dislocation dynamics
Starikov, Sergei, Ruhr-University Bochum, ICAMS, Bochum

Plastic deformation of metals is a complicated phenomenon that links behaviour of crystal defects with macroscopic change of a sample shape. It is known that one of the basic mechanisms of plasticity is a motion of dislocations under applied stress. In this work, on the example of Mo and Nb, the study of plastic deformation in bcc metals was performed with multi-scale modelling. The temperature-dependent mobility functions of screw and edge dislocations were calculated from molecular dynamics simulation. The simulations of screw dislocation movement under applied shear stress revealed that the process can proceed in two different regimes: through thermally activated motion and athermal motion. Hence, the dislocation velocity depends on the shear stress in a non-trivial way. The calculated data were implemented in the dislocation dynamics model. Such model allows to simulate plastic deformation taking into account temperature effect on the dislocation mobility. The changes of yield stress predicted by the dislocation dynamics simulation at variation of the input parameters are analysed in details.

In situ investigation of recovery strain in nanocrystalline Ni at ambient temperature
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Blum, Wolfgang, Friedrich-Alexander University of Erlangen-Nuremberg, Department of Materials Science, Institute I, Erlangen

Dynamic recovery enables quasi-stationary (steady state) creep. The strain associated with recovery (recovery strain) has been investigated in tension of nanocrystalline Ni at ambient temperature. During the tensile deformation the line width of X-rays has been measured in situ in the synchrotron beam of the Swiss light source (Villigen, Switzerland). In absence of grain coarsening the line width is a measure of the distribution of deformation-induced internal stresses caused by dislocations and local intra- and intergranular inhomogeneities of inelastic strain. Stress changes are performed to follow the transient evolution from an initial to a final quasi-stationary state. After an initial large stress reduction dynamic recovery dominates over defect production as seen from the decline of X-ray line width with time and strain. With stepwise increasing stress generation and storage of defects sets in again as seen from the increase of X-ray line width. All over the transient the creep rate varies only in a small window. The in situ test proves that dynamic recovery (i.e., recovery under stress) generates strain at a non-negligible rate from about 100% after a large stress reduction to about 20% in the quasi-stationary state. The nature and kinetics of recovery strain and its significance for quasi-stationary deformation are discussed.

The creep behavior of additively manufactured inconel 625
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Son, Kwangtae, University of Southern California, Chemical Engineering and Materials Science, Altadena

Creep tests of additively manufactured (AM) Inconel 625, both hot isostatically pressed (HIP) and and non-HIPed were performed at various stresses at 650 to 800oC and compared with the behavior of (conventional) wrought alloys. Tests were performed over a 24 hour period and also after exposure to the test temperature of 650oC for one year. The one year tests consisted of a time-temperature profile similar to Inconel 625 service conditions in jet engines. We found that the creep strength of AM 625 has equal or superior creep strength at 650 to 800oC compared to the wrought alloy, even after one year exposure to the test temperature. The HIP AM alloys had significantly superior ductiliy over the non-HIP AM Inconel 625, as expected. However, the ductility of the HIP AM Inconel 625 is still substantially lower than the wrought alloy, particularly at the longer exposure times to the test temperatures. The explaination for the dramatic loss of ductility is not clearly idenified, but embrittlement by sulfur is a possibility.

Anisotropic Biaxial Creep Behavior of CWSR and R_x Niobium Modified Zircaloy-4

Hawary, Mahmoud, North Carolina State University, Nuclear Engineering, Raleigh
Murty, K.L., North Carolina State University, Nuclear Engineering, Raleigh

Zr-based alloys are commonly used as thin-walled tubing to clad the radioactive nuclear fuel in light water reactors (LWRs). ZIRLO®, a niobium alloyed Zircaloy-4, is developed to enhance the deformation and degradation characteristics of Zircaloys. Because of its crystal structure (HCP), Zircaloys possess limited slip systems leading to preferred orientations (or textures) due to cold working. This texture affects the performance of Zircaloys in many ways including creep deformation. Thermal creep behavior of ZIRLO® tubes is investigated in its as-received (CWSR) and recrystallized (Rx) conditions at a temperature of 500oC. This investigation is carried out using uniaxial and biaxial creep tests performed on internally pressurized tubing superimposed with axial load under varied hoop-to-axial stress ratios from 0 to 2. Both the axial and hoop strains are monitored using a linear variable differential transducer and a non-contact laser telemetric extensometer respectively. The ratios of the steady-state creep rates along hoop and axial directions under varied stress ratios yielded the anisotropy parameters (R and P) in Hills formulation for the generalized stress. The creep loci are derived at a constant energy of dissipation while the crystallographic textures will be evaluated using electron backscattered diffraction (EBSD) from which crystalline orientation distribution functions (CODF) will be generated for predictions based on appropriate slip CODF-Creep models. This work is supported by the National Science Foundation grant CMMI1727237.

Scientific Programme – 16 June 2021

09:00–10:00 Invited talks VII

TEM investigation of the relevant parameters controlling the creep behavior in the Ni-based polycristalline AD730TM at 700°C

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Vultos, Winnie, University of Toulouse, CEMES-CNRS, Toulouse
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Cours, Robin, University of Toulouse, CEMES-CNRS, Toulouse
Douin, Joël, University of Toulouse, CEMES-CNRS, Toulouse
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Cormier, Jonathan, Institut Pprime, Physics and Mechanics of Materials Department, Futuroscope Chassenneuil

The enhancement of efficiency in gas turbine requires the design of new superalloys capable to withstand higher temperature with a reasonable cost. In this context, the superalloy AD730TM has been developed by Aubert & Duval to be a cost effective superalloy for high temperature (up to 700°C) rotating components. Its development was based on thermodynamical calculations made for similar alloys (Udimet 720Li, René 88DT), as the design of new superalloys often relies on incremental changes in compositions of existing alloys. As the γ/γ' Ni-based superalloys are designed for aeronautic applications, efforts have been made to give a better understanding of their behavior in the case of real service conditions. It is well known that, in the case of polycrystalline Ni-based superalloys, the thermo-mechanical treatments and service conditions may be at the origin of microstructure evolutions which are essential to be identified, as the mechanical properties are highly dependent on microstructural parameters. The effect of the microstructure on the creep behavior, and more generally, on strengthening mechanisms, has been extensively studied in the case of Udimet 720Li and René 88DT superalloys, as well on other similar polycrystalline superalloys such as N18 and NR3. These papers have pointed out that the γ' precipitation is the main creep controlling parameter of this type of superalloys in the temperature range 650°C-800°C. Due to its recent development, the creep deformation micromechanisms in the range 600 MPa-850 MPa at 700°C and the microstructural stability after heat treatment at higher temperature, in the case of the AD730TM nickel based superalloy has not been widely studied. A recent work carried out on this superalloy has pointed out the increase of the ductility and the decrease of the time to failure during creep at 600 MPa/700°C or 850 MPa/ 700°C in a fine grain microstructure superalloy in comparison with a coarse grain microstructure. The major difference between both microstructures is the presence of primary γ' precipitates in the fine grain microstructure. These primary γ' precipitates are usually considered as a stable phase and not considered to be key in controlling the creep deformation mechanisms, their usefulness being to keep a fine grain size during forging operations and subsequent sub-solvus heat treatments. This paper is aimed at analyzing the influence of these primary γ' precipitates on the creep deformation mechanisms at 700°C and to check the stability of these primary γ' precipitates using transmission electron microscopy (TEM) characterization.

Processing and creep of polycrystalline Ni-based superalloys

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Polycrystalline Ni-based superalloys (PS) are used for critical high-temperature components which have to withstand mechanical loads at temperatures exceeding 900°C. In the present work, we investigate the microstructure and the mechanical properties of PS which were produced using vacuum induction melting followed by thermomechanical treatments (including hot and cold rolling). Different alloy compositions were screened with the objective to find a composition with better creep resistance than the commercial alloy C-263. For this purpose, more Al and W were added while Ti-level was slightly decreased to increase the solvus temperature of the γ" phase and improve the level of solid solution strengthening. A parametric study was then performed to investigate the influence of precipitation temperatures (where the γ" phase forms) on the room temperature hardness to identify the optimum peak-aged condition for each alloy and select the alloy with the most promising properties (new alloy C-264). The effects of alloying on the peak-aged microstructure, creep behavior, and the microstructural evolutions after either creep or long-term anneals were investigated for both C-263 and C-264 alloys, combining uniaxial tensile creep testing with analytical scanning and transmission electron microscopy. Moreover, atom probe thomography was used to identify the effect of alloy composition on chemical partitioning between the γ-matrix and γ'-particles in the peak-aged state and to identify phases that resulted from internal oxidation during creep or long-term anneals. For two alloys, namely the commercial C-263 and the newly developed C-264 alloys, the creep activation parameters n (stress exponent) and Q (apparent activation energy for creep) were determined and discussed in the light of literature data for similar alloys. Based on these results, the new alloy C-264 was shown to have superior creep properties compared to its predecessor and for this reason, this newly designed alloy is currently on its way to be patented.

Evolution of microstructure under complex stress states during creep of single crystal Ni-base superalloy CMSX-4

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The behavior of high-temperature superalloys under complex loading scenarios is particularly significant for practical applications e.g. in aeroengine turbine blades [1]. Caccuri et al. [2] have designed and produced monocrystalline CMSX-4 specimens of a specific V-shaped geometry (Figure 1a) in order to examine the morphological evolution of the alloy during creep at 1093 °C. Due to the specimen geometry, complex stress states ranging from compressive to tensile arise within the same specimen during creep in [001] direction.

Analysis of the crept γ/γ" microstructure in the SEM shows wide variations in its morphology especially with respect to directional coarsening. It changes direction from P- to N-type in the region of transition from compressive to tensile stress states (Figure 1b) [3]. However, after the same creep time of 5 h, the morphological degradation is much less advanced in the region which has experienced compressive stresses, where distinct rafting is only visible after 24 h. Lamellas for investigation in the TEM have been extracted from these specimens at different locations and in specific crystallographic orientations ([100], [001]) to allow for a more thorough examination of the microstructure including crystal defects. It is evident that the activity of crystal defects and thus the stage of creep have advanced much further in the tensile (Figure 1e) than the compressive region (Figure 1c) after the same creep time, necessitating more complex comparisons between these states involving microstructural information obtained in the TEM. On the compressive side, isolated clusters of dislocations localized around their sources indicate an early incubation period. A dislocation network at a γ/γ" interface in an intermediate region after 5 h (in the center of Figure 1d) is shown to be in an early "slip" orientation, i.e. no rearrangement to a more misfit-relieving configuration has taken place at this stage [4]. After a creep time of 24 h, isolated γ" precipitate shearing is found in the compressive region.

Further investigations of this kind are conceivable, e.g. with other specimen geometries or under creep in the low-temperature high-stress regime where more planar defects are expected to form. Since many planar defects reside on {111} planes, lamellas

extracted in this orientation are of particular interest, allowing the in-plane examination of these defects including their leading and trailing partials. To this end, a workflow for the extraction of lamellas in {111} orientation from [001]-oriented specimens in the FIB/SEM environment has been devised.

Acknowledgement: The authors gratefully acknowledge financial support by the DFG via the collaborative research center SFB-TR 103. They further acknowledge Dr. S. Makineni from MPIE Düsseldorf for help with preliminary ECCI studies.

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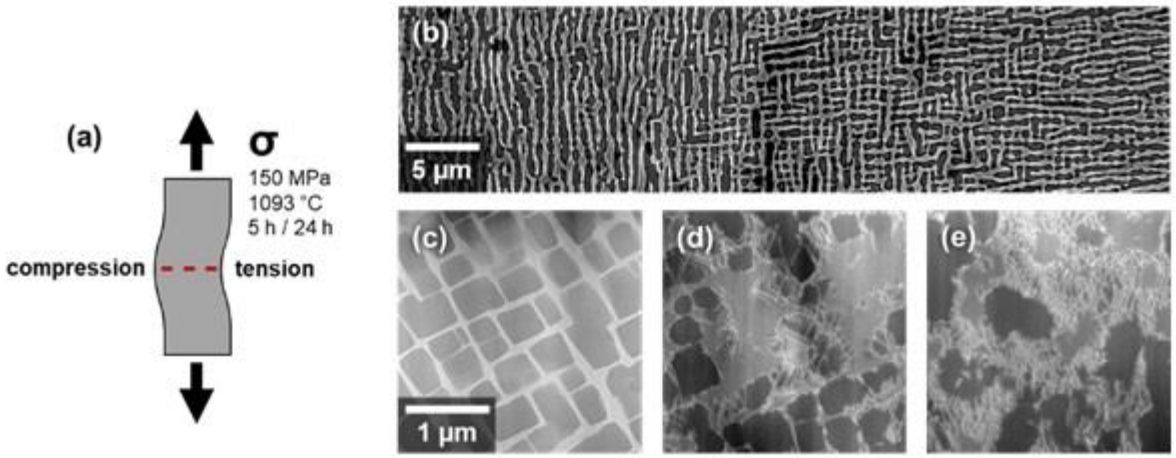

Figure 1. (a) Schematic drawing of the geometry of the V-shaped creep specimens and parameters of the [001] creep experiments. The red rectangles represent the compressive, intermediate, and tensile regions from which the lamellas depicted in (c-e) have been extracted as well as their orientations. (b) SEM micrograph of the region of transition between P- and N-type rafting in the specimen crept for 24h. (c-e) ADF-STEM micrographs of lamellas in [001] orientation extracted from the compressive, intermediate, and tensile regions, respectively, of the specimen crept for 5h. The apparent non-directional coarsening is due to the formation of plate-like rafts parallel to the foil plane.

10:30–11:30 Abstract talks X

How do deviations from <100> target tensile directions affect creep in the low temperature high stress creep regime (750°C, 800 MPa)

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Bonnekoh, Carsten, Ruhr-University Bochum, Material Science, Bochum
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Critical components made of directionally solidified Ni-base superalloy single crystals (SX) are often designed on the basis of tensile creep data obtained for nominal <100> tensile loading directions. During directional solidification, the <100> direction is a natural growth direction. But it is difficult to cast ingots in precise crystallographic directions, and angular deviations up to 15° from a <100> target direction can occur. The present study investigates how deviations of 5, 10 and 15° from <100> towards <110> and <111> directions affect creep behavior at 750°C and 800 MPa. Creep data are presented and analyzed. In addition, transmission electron microscopy is used to find out whether angular deviations of 15° (from <100> towards <110> and <111>), which differ in creep rates, result in different dislocation substructures as compared to high symmetry <100> orientations. For this purpose, dislocation substructures are investigated in material states, which were creep deformed up to 1% strain.

Modelling of non-steady state creep behaviour using cantilevers in bending

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With the advancement in testing and modelling tools in the recent times, there is a demand to design structures against creep with improved safety and reliability. As most of the structures in engineering applications are subjected to bending loads during service, it becomes important to understand their creep response directly under bending. However, bending is peculiar due to the linear stress distribution along beam thickness in elastic regime, which combines with non-linear dependence of creep strain rate on stress and leads to stress redistribution to satisfy Bernoullis assumption of section planarity at a given cross-section. A numerical methodology is developed to calculate the redistribution in stress which is based on the hypothesis that the total strain is maintained linear at every instant by a combination of non-linear creep strain and non-linear elastic strain as shown in the figure below (Fig. 1).

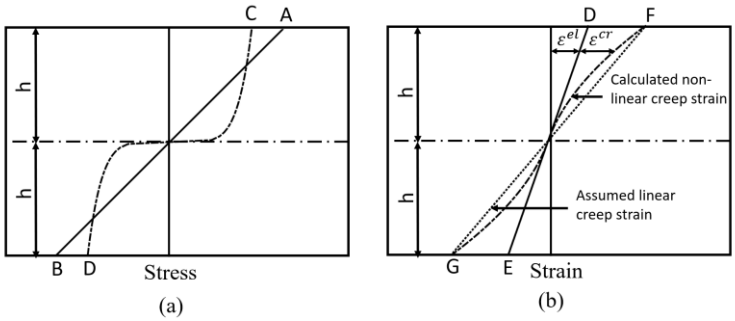

Fig. 1: Evolution of (a) stress and (b) strain along the thickness of a cantilever sample. Lines AB in (a) and DE in (b) represent the stress and strain distributions under elastic condition, respectively. CD in (a) is the non-linear stress profile that is established to satisfy the linear strain profile, FG in (b), along with a non-linear constitutive relationship.

The model has been validated for a variety of materials ranging from Al and ferritic steels to titanium alloys. The creep response of ferritic steels, commonly used in boiler tubes, shows primary as well as secondary stages whereas room temperature creep response of Ti-6Al, relevant to dwell-fatigue in aero-engines, shows only primary stage. It is observed that stress saturates at large creep strains when elastic strain becomes negligible and saturated stress profile is dictated by the stress exponents. The linear stress profile under elasticity saturates first to a profile dictated by stress exponent in primary followed by secondary. This stress evolution is non-linearly superimposed on microstructural evolution and the resulting behavior cannot be treated as equivalent to a constant stress test. The stress redistribution time can be as high as 100 hours in case of ferritic steel at an effective stress of 0.5 σydue to slow creep rates at low loads whereas it takes more than 30 hours in Ti-6Al alloy even at σydue to large elastic strains of 0.7% and extremely low creep rates of the order of 10-8 s-1. Therefore, stress redistribution time is a complex function of material constants along with the applied load which makes its estimation non-trivial. Moreover, stress gradient along the beam length makes every cross-section to evolve independently. The model holds great significance under such conditions by accurately capturing the transition between different creep stages at every section and obtain the overall creep response of the beam. It also aids experimentalists in designing their experiments to achieve steady state through the estimation of redistribution time and thereby avoiding ambiguity in the use of analytical expressions which are defined only for steady state. Therefore, this work serves as a milestone in understanding the interaction between geometry, material and loading under the bending creep and establishing bending as the standard mechanical testing technique to obtain equivalent uniaxial properties.

Creep-recovery behavior of low density polyethylene films under different applied stresses.
Benmiloud, Nourelhouda, Amar Telidji university, Engineering process, Laghouat

Creep behavior of low-density polyethylene films was investigated. The creep experiments were carried out at different stresses. The minimum strain rate has been found to increase with increasing applied stress, and obey to Norton's law with parameters
A1 =1.02289E-14 and n1 = 8.37. The isometric curves were used to determine the life time of each deformation. The isochrones curves were used to determine the linear- non- linear transition of the material. It was concluded that the creep behavior of all the films was basically controlled by the deformation of the amorphous phase and, specifically, dependent upon the density and physical state of tie-chains in the amorphous region.

12:30–13:15 Invited talks VIII

On the rhenium problem
Panfilov, Peter, Ural Federal University, Institute of Natural Sceince, Yekaterinburg

Introduction
According to experience, an HCP-metal should be ductile, but rhenium (Tmelt=3186°C; c/a=1,614 would not be machined at room temperature. This puzzle is unsolved until now, despite deformation mechanisms in rhenium are known (they are inherent for an HCP-metal). Rhenium is the prospective material for ultra-high-temperature applications; however, its almost zero malleability makes this perspective foggy. The cause of the unusual deformation behavior of this refractory HCP-metal is discussed.

Methods
Both electron-beam-melted rhenium (coarse-grained metal) and rhenium compacted sponge (fine-grained metal) were used as the model materials. The samples' deformation behavior was examined under bending, rolling, drawing, shearing, and high-pressure torsion (HPT) at room temperature.

Results
It was shown that the plasticity of rhenium depends on the share of the tensile stress in the deformation scheme and changes from zero under bending to high under HPT. The grain-boundary sliding, which takes place in a coarse-grained HCP-metal, is the cause of this effect. The malleability of rhenium under rolling could be considerably increased by using thin (2D) work-pieces, where the plane stress state is realized (figure1).

Conclusion
It means that the deformation behavior of rhenium obeys the empirical rule for HCP-metals and, hence, this refractory metal can be processed at room temperature.

The work was supported by the Russian Science Foundation (#18-19-00217).

Figure 1. Electron-beam-melted rhenium workpiece under the cold rolling: (i) initial state; (ii) rolling on 21%; (iii) rolling on 65% (after annealing 30 minutes at 1400oC).

On the role of elementary dislocation processes in medium temperature (T<800°C) creep of single crystal Ni-base superalloys

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During low temperature and high stress creep, dislocation ribbons consisting of four dislocation segments jointly move through the γ/γ'-microstructure of single crystal Ni-base superalloys. This results in the formation of planar faults in the ordered γ"-phase and has been discussed in the literature. In the present study we investigate how these planar defects nucleate in the early stages of creep. We show that two <110> dislocation families must react in the γ-channels at γ/γ'-interfaces. We provide mechanical and microstructural proof for the importance of this γ-channel dislocation reaction. Using shear creep testing, we show that <112>{111} specimen creeps significantly faster than a <110>{111} specimen (Fig. 1), because the two dislocation families which are required for this reaction are both activated with equivalent resolved shear stresses. We also provide microstructural TEM evidence which identify the elementary dislocation reactions which govern <112>{111} shear creep deformation. We also show, that the formation of planar faults in the macroscopic <110>{111} shear system, where only one dislocation family is strongly promoted, is hampered (Fig. 2). The results are discussed in the light of recent interpretations of double minimum creep and confirm a 2D discrete dislocation model, which was published recently.

12:30–13:30 Abstract talks XI
Room Meeting 2

High temperature creep study of uranium dioxide: three-point bending experiments, finite element simulation and microstructural characterizations

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Iltis, Xavière, CEA, DEC, Saint-Paul-Lez-Duranc
Antou, Guy, University of Limoges, IRCER, Limoges

Uranium dioxide is the main fuel in Light Water Reactor (LWR) in France. Operating temperatures range between 400°C and 1000°C over timescales of years. During power transients, fuel temperatures rise substantially and the material undergoes viscoplastic deformation processes. It is therefore essential to a safe and sustainable operation of nuclear fuels to obtain high temperature creep behavior laws that are physically based. However, uranium dioxide shows a wide range of non-stoichiometry, and very few if any tests have ever been carried out under controlled oxygen partial pressures [1].

In recent years, we have adapted two high temperature test devices (a compression device and a three-point bending one) in order to control the oxygen partial pressure during the mechanical tests. We essentially report here results obtained in a three point bending mode.

Experiments involve thin rectangular samples of uranium dioxide, obtained by a powder metallurgy route and sintered at 1700°C under a mixture of Argon and Hydrogen. Tests are carried out at CEA Cadarache, in a device designed to conduct high temperature bending tests, while controlling precisely the oxygen partial pressure under a flowing mixture of humidified Argon and Hydrogen. The purpose of these tests is to identify the creep law for UO 2 , in which the creep rate would depend upon stress, temperature and also the oxygen partial pressure, i.e. the stoichiometry deviation. For this purpose, constant crosshead speed experiments have been carried out under different conditions (crosshead speed, temperature and oxygen partial pressure).

Moreover, a F-E model has been developed to describe the three-point bending device and to simulate these various tests using a kinematic hardening law used with some degree of success to describe compression tests [2] and bending tests on UO 2 [3]. In this work, we demonstrate the material is subject to recovery creep and discuss the dependence of the secondary creep rate to the oxygen partial pressure.

In addition to the macroscopic bending tests, complementary SEM/EBSD [4] examinations are carried out with the aim of identifying specific microstructural mechanisms associated with recovery creep.

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[3] Creep of uranium dioxide: bending test and mechanical behavior. C. Colin. PhD 2003. Ecole des Mines de Paris
[4] Influence of strain conditions on the grain sub-structuration in crept uranium dioxide pellets. M.Ben Saada et al. Journal of Nuclear Materials, Volume 518, May 2019, Pages 265-273

High throughput creep tests in bending using digital image correlation

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Conventionally creep tests performed by bending of specimens in 3,4-point and cantilever configurations have been used to obtain steady-state creep parameters. These tests are employed to obtain one strain-rate- stress data point from a single beam by using the average creep response from load point deflection; thus, several tests are required to calculate the stress exponent and steady state pre-exponential constant. Therefore, multiple strain-rates -stress pairs available as a result of the gradient stress state in a bending test are not used for mechanical characterization. The state-of-art advancement in high temperature instrumentation and techniques, especially non-contact strain measurement like digital image correlation can be adapted to perform bending creep tests and obtain hundreds of potential strain-rate stress pairs from a single specimen. In this work digital image correlation was employed to record 2D strain field from one face of the cantilever. Apart from the instrumentation and data acquisition, analytical equations were developed to correlate strain/strain-rate information from each location on a cantilever with known geometric parameters and finally arrive at the corresponding stress at that location. Firstly, the strain rate-stress pairs were used to accurately determine the stress-exponent of the material (e.g., Al, Pb, etc.) by using geometric relationships between creep strain and stress exponent. The stress exponent was further used to calculate stress from analytical equations. Thus, steady-state creep-rates and stress exponent corresponding to a range of stresses were calculated from a single cantilever tested at a constant load and at constant temperature. Further, specific locations across the cantilevers are demarcated where the stress does not redistribute as a function of creep deformation. These locations enable obtaining primary creep parameters which is otherwise impossible because of the stress redistribution in beams. Stress redistribution in bending creep occurs as a consequence of maintaining strain linear across the beam thickness. Accordingly, the potential of cantilever bending creep tests in obtaining hundreds of strain-rate-stress pairs from a single specimen, thus enabling power-law creep characterization in primary and secondary creep regime from a single specimen is demonstrated as a high throughput creep testing technique.

Extraction of power-law creep parameters and residual life assessment of steels by performing creep tests in bending

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Studying creep of materials by bending cantilevers is an alternate method for obtaining power-law creep parameters. Not only are the fabrication and testing of cantilever samples using conventional creep machines relatively easier as compared to the standard uniaxial tensile samples, the use of digital image correlation (DIC) for obtaining the strain field across the thickness and length of the sample may allow extraction of multiple "stress-strain rate" pairs using a single cantilever sample. These associated advantages have renewed interest in the bending creep of metallic materials recently. In this work, we have firstly validated the bending creep methodology by comparing the steady-state power-law creep parameters of three steel alloys used in power plants (namely, T11, T22 and 9-12 % Cr steels) obtained from uniaxial compression and bending tests. Further, we extended the application of bending creep to estimate the residual life assessment (RLA) of the boiler tube steels that have been in service in power plants for various lengths of time. Tests were performed on cantilevers of thickness > 1 mm and aspect

ratio (defined as length to thickness ratio) of > 6, so that they qualify as Bernoulli's beams, at 600 ºC by applying an effective stress in the range of 100 – 200 MPa. Strain was measured by both DIC and using standard analytical equations which rely on the steady-state deflection rate of the loading end. Herein, the utility of measurement of strain field using DIC has also been established. For all samples, a stress exponent, n ~ 6 was observed under the test conditions from both uniaxial and bending tests; however, the steady-state strain rates were widely different. The RLA depicted an additional 4 years of residual life in the in-service steel on top of its original design-life of 27 years. These steels being specifically ferritic-bainitic, have been microstructurally characterised and a structure-property correlation has been established between as-fabricated and in-service steels to rationalize the experimental observations.

Extraction of uniaxial creep parameters from indentation experiments- an artificial neural network-based approach to the Inverse problem.

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The conventional creep property extraction method requires a large number of uni-axial tests at different loads, which consumes an intensive amount of time and material resources. It has been a while since indentation experiments have been posed as an alternative to the uni-axial experiments for creep parameter extraction. The theoretical understanding of the indentation behavior under non-linear elastic, plastic, and creep conditions is still far from completion to have an analytical solution to this inverse problem. Artificial Neural Network (ANN) has been successfully employed on numerous inverse problems but has not been explored much in the context of uniaxial creep property extraction from indentation experiments. In the current study, we use fully connected sequential multi-layered ANN trained on Finite Element indentation creep simulations to map the observables (displacement, time) of indentation creep experiments to the corresponding uniaxial creep parameters.

In indentation, the stresses range from a low-stress level well within the elastic regime to high stresses well beyond the material's yield stress in the plastic regime. And as the indentation experiments last for a short time, it is likely that some volume of the material does not reach the steady-state creep regime and creeps in the primary stage for the entire duration of the experiment. Thus, we choose a suitable constitutive creep law ($\varepsilon_{creep}=B\sigma^{n}t^{\alpha}$), such that it can capture the entire creep behavior in the wide range of stresses. We then generate training sets by conducting FE simulations with varying creep parameters (B,n,α), keeping the elastic and plastic flow properties of the material fixed. Fixing the elastic and plastic property makes learning with fewer examples easier for the ANN and simplifies the problem of uniqueness (different combinations of material properties (elastic, plastic and creep), giving the same indentation creep behavior: displacement vs. time plot).

The displacement-time curve, the output of constant load and hold FE indentation creep simulation, is fitted (eg. dcreep=d0 Atb), and the pre-processed fitting parameters (eg. d0,A, b) are provided as inputs to the ANN. We train the ANN using a back-propagation algorithm based on a stochastic gradient descent to map these inputs to the corresponding creep parameters used in the FE simulations. Performance of the ANN is improved by optimizing its architecture using the Bayesian optimization approach and varying the fitting function, the parameters of which are the inputs to the ANN. We evaluate the performance of ANN-based on its prediction on the validation set (size - 10% of the training set) and continue thelearning till the mean squared error of this prediction is in the order of 10−3. Finally, we test this trained ANN on indentation creep experiments on Lead and compare its prediction with creep parameters obtained from the uniaxial creep experiments.

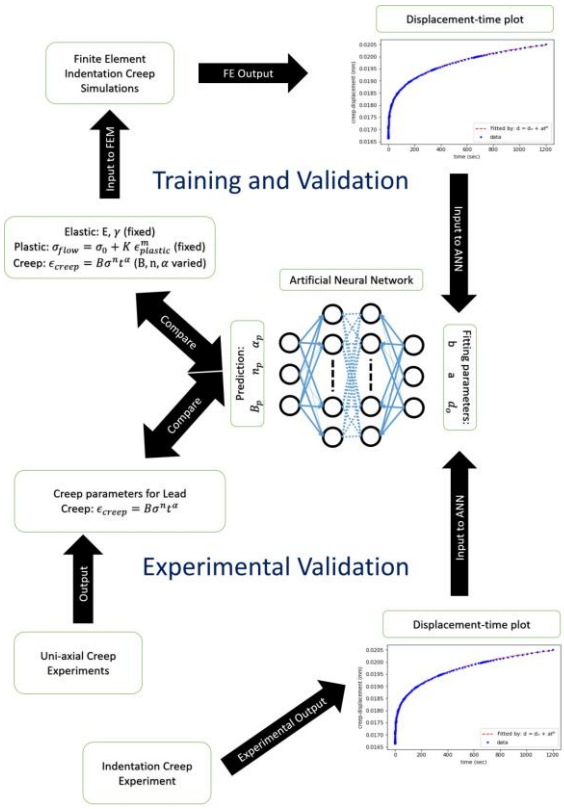

Fig1:Schematic of the ANN-based approach

14:00–15:00 Abstract talks XII

Creep response of a Ti-6Al-4V alloy produced by Additive Manufacturing annealed at 740°C

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The creep response of a Ti-6Al-4V ELI alloy produced by Additive Manufacturing (AM) was investigated after annealing at 740°C for 2 h. Even after heat treatment, the fine microstructure produced by the ultra-fast cooling rates typical of AM processes, maintained a high tensile strength (UTS=1015 MPa on samples tested with the surface in as-deposited state). Dog-bone creep samples were tested, after mechanical polishing of the surface, at 500 and 600°C, by a combination of constant load (CL) and variable load (VL) experiments, covering a range of 4 orders of magnitude of strain rates. The minimum creep rate dependence on applied stress was described by a power law, whose stress exponent was observed to increase from 4-5 under low stresses, to 9-10 in the high stress regime. Under many regards, the creep response of the AM annealed Ti-6Al-4V was remarkably similar to that of similar alloys, produced by conventional technologies, such as forging, and with a duplex structure of equiaxed α grains and lamellar grains (α laths separated by β phase). In particular, for long test durations, the AM and the duplex alloys exhibited similar strain rates if tested under the same stress. Conversely, in the high strain rate regime at 500°C, the AM alloy maintained a noticeable advantage in term of creep response, with strain rates almost one order of magnitude lower than its conventional duplex counterpart.

Creep tests of thin pre-oxidized Ti–6Al–4V alloy samples. Influence of oxygen content on creep rate between 450 and 600°C.

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Owing to excellent corrosion resistance and high specific modulus, titanium alloys are widely used for intermediate temperatures [1-2] in the aerospace, chemical, petrochemical marine and medical fields. However, titanium tends to oxidize rapidly when heated in air and, with more harmful impacts on its mechanical properties; it has the capacity to be highly enriched in oxygen and nitrogen. So titanium alloys are then limited in their operating temperatures due to their oxidation kinetics and to the significant modification of the sub-surface microstructure [2-3].

In this study, we focus on alloy Ti-6Al-4V and creep behaviour of thin components.

Pre-oxidation leads to different depths affected by oxygen enrichment. These composition gradients are first characterized and then creep tests are conducted.Oxidations were carried out under air between 450 and 600°C for durations of up to 6816 hours.

Creep tests were carried out under moderate stresses and up to total creep deformations of less than 1% in order not to initiate cracking. Scanning electron microscopy observations were made at the end of the tests to verify that no cracks had initiated in the alloy in order to have strain curves unmodified by damage.

Results obtained evidenced that an oxygen-enriched zone occupying 5% of the cross-section of a specimen is sufficient to induce detectable changes in its creep behaviour. The behaviour in the temperature and stress levels tested can be modelled by a Norton's law with parameters depending on the local oxygen content.

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Tension/compression asymmetry during creep of advanced Co-base superalloys

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The creep properties of an advanced γ´-strengthened Co-base superalloy were investigated in tension and compression, since it is known from established Ni-base superalloys that different material responses can occur under the same creep conditions by inverting the loading direction. Additionally, fundamental insights into the deformation mechanisms can be achieved by keeping the test parameters constant while changing the direction of the applied load. Three sets of parameters were chosen to determine these properties in the high, medium and low temperature regime. Therefore, creep tests were conducted at 1050°C/125MPa, 900°C/250MPa and 750°C/800MPa. It was found that this Co-base superalloy reveals a pronounced tension/compression asymmetry, especially at high temperatures, decreasing with decreasing temperature. While the minimum creep rate at 1050°C and 125MPa is almost one order of magnitude higher in tension compared to compression, the minimum creep rates at 750°C and 800MPa are nearly identical. As expected, SEM investigations revealed a pronounced P-type rafting in tension and N-type rafting in compression at 900°C and 1050°C, since the γ/γ’ lattice misfit is known to be positive. At 750°C no rafting occurred, indicating that the different types of rafting might be one reason for the difference in the creep properties at the higher temperatures. Additionally, TEM analysis showed a significant difference in the deformation behavior depending on the test parameters as well as the load direction.

Creep properties of L12 hardened γ/γ' Co-Ni base Superalloys with low W content

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Pyczak, Florian, Helmholtz-Zentrum Geesthacht, Metal Phsics, Geesthacht

Creep properties of polycrystalline L12 hardened γ/γ' Co-Ni base superalloys based on Co-35Ni-15Cr-5Al-5Ti-0.1B with additions of Mo, W and Ta are presented. By reducing the content of Mo and W, a new alloy series with reduced mass density was designed. In parallel, it was tested whether Ta addition can be used to compensate for some of the negative effects of Mo and W reduction as for example reduced γ' solvus. To do this in a systematic way it is necessary to understand and assess the influence of Mo, W and Ta on alloy properties in general and especially on creep behavior. The mean radius and volume fraction of γ' was analysed. In addition, the influence of Mo and W on the lattice parameters of γ and γ' and the yield strength (0.2 % offset flow stress) of the alloys at different temperatures was determined. This was done with the aim to interpret the creep strength of those alloys (850ºC, 480MPa and 750 ºC, 620MPa) in dependence of those other alloy properties. The most important findings which will be presented are the following: (1) The γ' solvus temperature is increased by adding more Ta but the solidus and liquidus temperatures decrease. (2) The yield strength increases with Ta, W or Mo addition compared with the baseline composition Co-35Ni-15Cr-5Al-5Ti-0.1B. (3) The mass density of the alloys increased with Ta and W addition. (4) Alloying of Ta and W improves creep life dramatically but Mo containing alloys exhibited accelerated creep rates which can maybe explained by its effect on the lattice misfit. To Conclude, the investigated novel compositions provided insights to design novel polycrystalline Co-Ni base superalloys with decreased density and improved creep strength.

14:00–15:00 Abstract talks XIII

Tensile creep testing of NiAl single crystalline miniaturized specimens

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Heilmaier, Martin, Karlsruhe Institute of Technology, Institute for Applied Materials, Karlsruhe

Dog-bone shaped NiAl single crystalline miniaturized specimens were tested with different gauge lengths (700 µm and 1400 µm), widths and thicknesses (200, 300 and 400 µm) in [110] soft and [100] hard orientation up to 1000 °C. A contactless strain measurement based on the Digital Image Correlation (DIC) method was used in a customized setup, which provides a strain accuracy better than 10-4 at 1000 °C and simultaneously enables the analysis of the spatially resolved strain field on the miniaturized specimens [1].

The creep behavior of the miniaturized specimens in both orientations showed an influence of the specimen geometry on the ratio of length to the square of thickness L/h2 in the gage section. Smaller creep strain rates were observed in specimens with larger L/h2 ratios. For specimens with smaller L/h2 ratios, the creep behavior of the miniaturized specimens agreed well with conventional (bulk) specimens [2]. For specimens with larger L/h2 ratios, an increasing shear strain localization was observed in the strain distribution analysis with the DIC method and corresponding EBSD measurements revealed a larger density of geometrically necessary dislocations (GNDs). Using a simplified cantilever model it is verified that the GND density is proportional to the setup misalignment and, thus, strongly dependent on the L/h2 ratio. Discussion of the influence of GNDs on the creep behavior yields the following: For specimens tested in [110] soft orientation, the GNDs obstruct the <100> dislocation glide resulting in decreased creep strain rates. For specimens tested in [100] hard orientation, the <110> dislocations decompose into <100> dislocations [3]. The edge part of these dislocations is sessile and can move only via <100> dislocation climb. Therefore, the influence of the GNDs on the creep behavior is less pronounced as compared to specimens tested in [110] soft orientation.

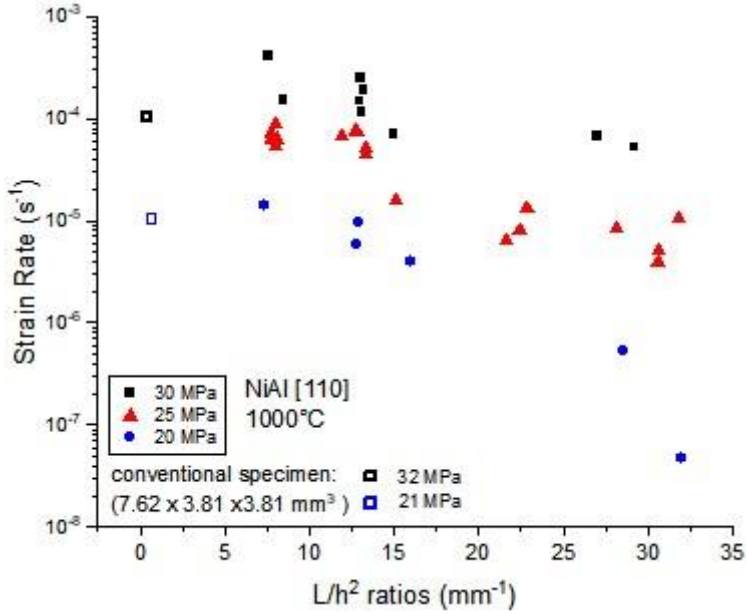

Figure 1: Steady-state/min creep strain rates vs. the ratio of gage length to the square of thickness L/h2 of micron-sized miniaturized specimens and conventional millimeter-sized specimens (7.62×3.81×3.81 mm3) in [110] soft orientation [2].

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Microtensile creep of freestanding MCrAlY bond coats

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Bond coats are essential in gas turbine technology for oxidation protection. The creep properties of these bond coats have large influence on their lifetime and durability since they have to withstand high thermal and mechanical loads. The investigation of creep properties of thin coatings is nearly impossible by conventional testing methods which is why advanced nanomechanical testing techniques have to be applied. In this study a new method of microtensile creep testing in a thermomechanical analyzer (TMA) was validated for the Ni-base superalloy Waspaloy in combination with conventional macroscopic tensile creep tests and used to investigate the creep properties of freestanding MCrAlY bond coats at elevated temperatures. Furthermore, the influence of different annealing atmospheres on the creep properties was examined.

Besides Waspaloy, three types of MCrAlY, a Ni base bond coat Amdry 386, a Co base bond coat Amdry 9954 and Amdry 9954 2wt.% Al2O3 (ODS) produced by low pressure plasma spraying, were analyzed. The microtensile creep specimens had a diameter of 450µm and were produced by a high precision grinding and polishing process. The experiments were conducted with a constant load at temperatures between 900 and 950°C.

Creep rupture of the MCrAlY bond coats was mainly due to void nucleation along the β-γ interfaces and grain boundaries since the two-phase miocrostructure of the bond coats consists of a fcc γ-Ni/Co solid solution and a B2 β-NiAl phase. The time to failure is larger in Ni-based Amdry 386 compared to that in Co-based Amdry 9954 due to a higher fraction of the high-strength β-NiAl phase at test temperatures. The addition of ODS-particles in the Co-based bond coat Amdry 9954 resulted in a better creep resistance, but lower ductility in comparison to ODS particle-free Amdry 9954.

Additionally, the influence of annealing these three bond coats in Ar-O2, Ar-H2O and Ar-H2-H2O atmosphere on the creep properties was examined. The annealing in the different atmospheres had no significant influence on the microstructure and on the creep strength of the Co base bond coats Amdry 9954 and Amdry 9954 ODS. The creep properties of Amdry 386 at 900°C were much more affected by the annealing in different atmospheres and the annealing in Ar-H2O led to a significantly lower creep rate than the annealing in Ar-O2 and Ar-H2-H2O.

In summary, this new method of microtensile creep testing in a micromechanical analyzer allows to investigate precisely the high temperature creep properties on a small scale. Thus, the creep properties of freestanding MCrAlY bond coats could be determined without an influence of the substrate.

Heterogeneous stress relaxation in thermally-cycled Sn thin films at early stages

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For tin and other metal films, Coble and Nabarro-Herring creep are frequently suppressed by the presence of a surface oxide. Under these conditions, stress relaxation mechanisms of whisker and hillock formation and power-law creep can dominate. Tin whiskers grow from shallow surface grains by a process involving local grain-boundary-sliding-limited Coble creep. A gap in the understanding of whisker formation is how shallow grains form and why only some become whiskers. Our recent results suggest an important role for near-surface yielding, sub-grain formation, and grain boundary formation in creating shallow grains that ultimately become whiskers. This paper focuses on quantifying the evolution of local microstructure, and crystallographic and surface orientation, including the formation of shallow grains, during thermal cycling of large grain sized Sn films on Si substrates. Utilizing time-dependent characterization and phase field models of local plasticity as a function of local crystallography, we have developed a model that allows for the separation of the processes leading to shallow grain formation and out-of-plane whisker growth.

Decoding the structural origin of creep in colloidal gels by machine learning
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Question:
When subjected to a sustained load, jammed colloidal calcium–silicate-–hydrate (C–S–H) gels—the binding phase of concrete—tend to exhibit delayed viscoplastic creep deformations. However, the structural mechanism of creep in C–S–H gels (and driving force thereof) remains only partially understood. This partially arises from the fact, due to the long timescale of creep, its physical modeling has remained challenging.

Methods:
Here, based on a mesoscale model of C–S–H gels, we present an accelerated simulation method—based on stress perturbations and overaging—to model creep deformations in C–S–H [1]. Based on these simulations, we adopt Support Vector Machine (SVM, a supervised machine learning classification algorithm) to decode the structural mechanism of creep in C–S–H gels [2]. This allows us to find Needles in haystacks, that is, to pinpoint the key structural features enabling grain reorganizations and creep deformations within the gel.

Results:
Our simulations yield a very good agreement with nanoindentation creep tests, which suggests that concrete creep occurs through the reorganization of C–S–H grains at the mesoscale. We show that the creep of C–S–H exhibits a logarithmic dependence on time—in agreement with the free-volume theory of granular physics. Further, we demonstrate the existence of a linear regime, i.e., wherein creep linearly depends on the applied load—which establishes the creep modulus as a material constant. Based on the machine learning analysis, we define a new structural metric that is capturing the propensity of a gel to exhibit creep deformation.

Conclusions:
These results establish machine learning as a promising pathway to decipher the relationship between materials structure and creep response under sustained load. These insights offer a new physics-based basis for nanoengineering colloidal gels featuring minimal propensity for creep [3].

References:
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[2] Liu, H., Fu, Z., Yang, K., Xu, X., & Bauchy, M. (2019). Machine learning for glass science and engineering: A review. Journal of Non-Crystalline Solids: X, 4, 100036.
[3] Liu, H., Du, T., Krishnan, N. A., Li, H., & Bauchy, M. (2019). Topological optimization of cementitious binders: advances and challenges. Cement and Concrete Composites, 101, 5-14.

15:30–16:40 Invited talks IX and conference closing

Microstructure evolution and enhanced creep property of a high Nb containing TiAl alloy with carbon addition
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Titanium aluminides are used for turbine blades of aero engines, which operate up to 750 °C. Under those conditions, creep strength is one of the limiting factors during service and there is an ongoing urge for improved creep resistance of TiAl alloys. The introduction of carbon to benefit from carbide precipitation to harden the material is an attractive way to achieve this. Unfortunately, the small carbide particles can undergo significant changes during creep service. The microstructure evolution and carbide precipitation in a Ti-46Al-8Nb-0.7C alloy as well as its creep properties at intermediate temperatures are investigated by high-energy X-ray diffraction and electron microscopy. The alloy with a nearly fully lamellar microstructure exhibits excellent creep resistance, which could be attributed to the good microstructural stability and strengthening effects from both P- and H-carbides. The creep parameters have different effects on the precipitation of the carbides. The overall volume fraction of the carbides shows a positive correlation with the creep temperature and time. However, the thermal

stability of P-carbides in the γ grain interior decreases at a higher creep temperature. The creep stress hardly affects the precipitation and morphology development of the P-carbides. On the contrary, a higher stress can promote the H-carbide formation at the γ/α2 interfaces via α2 lath decomposition in lamellar colonies.

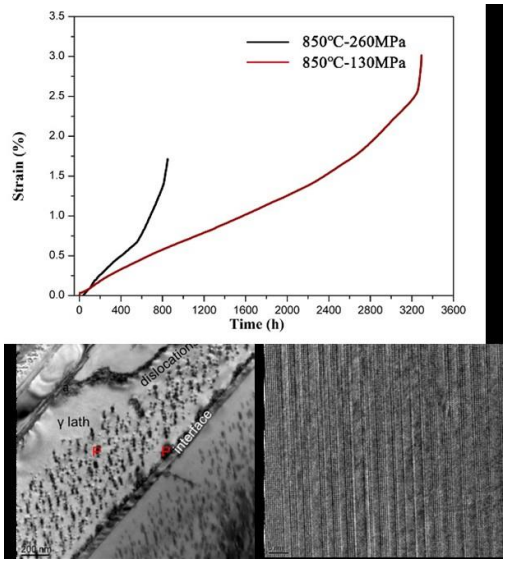

Fig. 1: Creep curves of the samples tested at 850 °C under tensile stresses of 130 and 260 MPa (top); carbide precipitates in the lamellar microstructure pictured by TEM (bottom left); decomposing α2 lamella pictured by HRTEM (bottom right)

Understanding creep mechanisms and modeling creep deformation in dispersion-strengthened alloys

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Historically, creep deformation in dispersion-strengthened (DS) alloys has been modeled using a variety of different approaches starting from the simple power-law model to the phenomenological threshold stress model that accounts for the abnormally high stress exponents and activation energies obtained in the power-law model. Later, physics-based models that focus on the kinetics of the rate-controlling process were introduced, which include the local climb model, general climb model, and the Rösler-Arzt model (dislocation detachment-controlled creep). These models have been applied interchangeably to a wide variety of DS alloys, and typically, the model selection is only based on the knowledge of the size and volume fraction of the dispersoids, the nature of the particle-matrix interface, and some ex-situ transmission electron microscopy evidence that shows dislocation-particle interactions. Difficulty often arises when the selected model fails due to the fact that the hypothesized subprocess is not actually rate-controlling. Furthermore, there is little connection between the different models resulting in seemingly isolated approaches. Therefore, it is highly desirable to verify the rate-controlling process by creep data prior to modeling the creep behavior of dispersion-strengthened materials.

In this work, we mechanically identify the rate-controlling process of creep by performing both steady-state and stress change creep experiments on four DS alloys: GlidCop-Al15 and GlidCop-Al60 (Cu-Al2O3), GRCop-84 (Cu-Cr2Nb), and FVS0812 (Al-Al12(Fe,V)3Si). Data acquisition in stress change experiments was conducted with microstrain and microsecond resolution so that the creep response to the stress change can be assumed to be measured at constant microstructure. This condition is critical for the determination of the operational activation area and activation energy of creep. Combining the creep results from both experiments, the rate-controlling processes for the selected DS alloys were determined to be dislocation detachment, local climb, and forest dislocation interactions, respectively. Furthermore, we find that the threshold behavior in detachment-controlled creep can be successfully modeled by considering the back jump of just-detached dislocations from particles. We therefore propose a new, modified Rösler-Arzt model to accommodate the physical origin of the threshold stress and the dependence of creep on dislocation density and inter-dislocation forces. This model helps the understanding of creep mechanisms in different DS systems using an integrated approach.

Accelerated Creep Testing of Inconel 718 - Properties and Microstructure

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With the recent advancements in additive manufacturing, it is now possible to optimize processing parameters in order to tailor the phase composition and microstructure. Further heat treatment can regularize the microstructure, nucleate additional hardening phases, and increase the overall strength of superalloys. To qualify a new material for service in the industrial gas turbine (IGT) industry, multiple 10,000 hour conventional creep tests are required. This time bottleneck is a serious inhibitor to the adoption of new materials in the IGT industry. The Stepped Isostress Method (SSM) is an accelerated creep test where a specimen is subjected to a constant temperature while stress is stepped increased after a dwell period until rupture. It is important to consider the deformation mechanism maps and time-temperature-transformation (TTT) diagrams to ensure the failure mechanisms and microstructure of the material at the initial stress level of the SSM tests replicate similar behaviors observed during a conventional creep test.

The objective of this study is to develop an accelerated creep testing program in order to rapidly characterize and qualify designer materials, specifically superalloys. The subject material in this study is Inconel 718 (IN718) at 649℃. Experiments begin with an initial stress based on the deformation maps for Nickel-Chromium alloys and TTT diagrams for IN718. After a set duration has passed the stress is increased and held for another set duration This process is then repeated until creep rupture occurs. The data from the SSM experiments is then accelerated using creep-strain-rate (CSR) and creep-strain-intercept (CSI) matching. The accelerated creep tests are compared to conventional creep test data. It was observed that a six-month accelerated creep test can resemble the minimum-creep-strain-rate and predict creep rupture of a conventional creep test lasting three years. Material characterization techniques including: optical microscopy, backscattered electron imaging (BSE), secondary electron imaging (SE), and Energy Dispersive X-Ray Spectroscopy (EDS) are conducted to observe the microstructure of SSM testing and compare what is observed in literature.