B. High Temperature Structural Materials

Symposium Organizers:

Qiang Charles Feng (Chair), University of Science and Technology Beijing, China; Shengkai Gong, Beihang University, China; Hyun Uk Hong, Changwon National University, Korea; Damon Kent, University of Sunshine Coast, Australia; Sammy Tin, Illinois Institute of Technology, USA; Hiroyuki Yasuda, Osaka University, Japan; Jun Zhang, Northwestern Polytechnical University, China

Monday PM Room: 305 (3rd floor)
August 19, 2019 Symposium: B

Power Generation

13:30-14:00 Keynote (1234455)
A New Ni-base Superalloy GH750 for 700 ℃ Advanced Ultra-Supercritical Power Plant application
Xishan Xie, University of Science and Technology Beijing, China

The world tendency of coal fired electrical power plant development is intended to reach high temperature up to 700 ℃ for raising thermal efficiency and decreasing CO2 emission. A new Ni-based superalloy Ni-Cr-Co-Mo-W-V-Nb-Ti-Al-C-B-Zr-La named GH750 has been designed and produced as tube product to fulfill the 700 ℃ A-USC boiler application.

GH750 is based on Ni-Cr-Co austenitic matrix for solid solution strengthening by addition of W and Mo and mainly strengthening gamma prime γ'- Ni3(Nb, Ti,Al) precipitation and combined with grain boundary carbide formation. The basic structure of GH750 after standard heat treatment is mainly about 15% γ' precipitated in γ-matrix and a certain amount of M23C6 carbide formed at grain boundaries. No any kind of harmful phases (such as Ni3Ti type η phase, σ phase and other TCP phase) formed after 10,000 hrs long time aging in the temperature range of 700-800 ℃. It shows excellent structure stability at high temperature application.

Long time stress rupture strengths for 105 hrs extrapolated by Larson Miller method at 700, 750 and 760 ℃ can reach 207MPa, 122MPa and 108MPa respectively. It clearly meet the requirement of 760 ℃/105 hrs stress rupture strength higher than 100MPa. GH750 not only characterizes with high stress rupture strength but also good long time stress rupture ductility , as example after 750 ℃, 180MPa, stress rupture tests ( longer than 15,000hrs), the elongation can keep in a high range of 15-18%.

The manufacturing performance of GH750 is excellent. It can be melted by VIM+ESR or VIM+VAR. Ingots are easy to forge, hot extrusion and cold rolling for required tube sizes.

GH750 has been evaluated as a candidate A-USC superheater / reheater materials for 700 ℃ A-USC test loop in China.

14:00-14:20 (1220272)
Shuangqun Zhao, Rui Fu, Yanfeng Wang, Department of Materials, Shanghai Power Equipment Research Institute, China

Ultra-supercritical coal-fired power generation technologies are developing worldwide for raising thermal efficiency and reduction of CO2, SOx and NOx emission. The research and development of high strength nickel-base superalloys for key and high temperature components of advanced ultra-supercritical power units have been gaining remarkable progress during the past years. Several nickel-base superalloys, such as Haynes 282, Inconel alloy 740H, Nimonic 263 and alloy 617B, have been selected as the main candidate materials in the developing project of advanced ultra-supercritical power plants in the world and were expected to be applied as the boiler tube or pipe, steam turbine rotor or casing, etc. In order to get the results of microstructure evolution and mechanical properties changes of Haynes 282, Inconel alloy 740H, Nimonic 263 and alloy 617B in the proposed long-term service temperature range, these four nickel-base superalloys, and a Chinese new superalloy GH750 developed for boiler tube, were heat-treated at 700 ℃, 760 ℃ and 800 ℃ for times...
up to 10000h, and its microstructural stability were examined by SEM, EDX and TEM. The experimental results show that the m phase forms in the alloy of Haynes 282 after aged at 760°C and 800°C for 3000h. In addition to the growth of g′ phase, no other phases form in samples of Inconel alloy 740H. The h phase forms in the alloy of Nimonic 263 after aged at 760°C for 5000h and after aged at 800°C for 3000h. The dense and uniform g′ particles precipitate in the sample of alloy 617B during aging at 700°C. However, the quantity of g′ particles decreases obviously during aging at 760°C and especially in 800°C. Microstructure observation indicates that no harmful and brittle TCP phases were found in the specimens of GH750 after long term exposure at 700~850°C except the coarsening of g′ particles. The test results of mechanical property show that the tensile strength keeps the high values and the impact ductility decreases firstly and then keeps stable during long thermal exposure. Furthermore, the influence of the main elements of chemical composition on the phase stability for these five nickel-base superalloys was discussed according to the thermodynamic calculation results.

14:20-14:40 (1222803)
Influence of lamellar structure on creep property of cellular precipitating Ni-38Cr-3.8Al alloy
Y Koyanagi, H Takabayashi, H Yasudai, R&amp;D center, Osaka University, Japan

A new precipitation-hardened Ni-Fe-base superalloy HT700 containing around 20 vol.% of γ ′ precipitates has been developed, which is evaluated as a candidate material for 700 °C A-USC boiler tube application. After a standard heat treatment and subsequently thermal aging for 8220 h at 750°C, the tensile behavior of HT700 is studied, and the evolution of dislocation structures with strain has been analyzed using transmission electron microscopy during tensile deformation at 750 °C. It is found that contrary to previous findings widely reported in Ni-base superalloy used for multiple critical components in aero-engines and land-based gas turbine, the initial plastic deformation of the experimental alloy is achieved mainly by the climb and cross-slip of dislocations together with Orowan looping process when the average size of γ ′ precipitates is around 140 nm. Additionally, the experimental observations reveal that as the tensile deformation proceeds, more and more a/2<110> matrix dislocations surround the same γ ′ precipitate, and stacking fault shearing also operates actively, which is rarely reported in the literature. Based on the experimental observations, it is deemed that the transition in the deformation mechanism with strain accounts for the variation of the flow stress with the strain. This study provides new insights into understanding the relationship between the operating deformation mechanisms and the deformation behavior of Ni-base/Ni-Fe-base superalloys.

14:40-15:00 (1225427)
Tensile deformation mechanisms in a new Ni-Fe-base superalloy at 750 °C
P Zhang, Y Yuan, Y Gu, J Yan, Y Dang, J Lu, H Yin, J Wang, Xi'an Thermal Power Research Institute Co., Ltd., China

The 700°C-class advanced ultra-supercritical (A-USC) boiler is a promising technology for new generation of high-efficiency coal-fired thermal power plants. A-USC technology will realize both higher thermal efficiency of coal-fired power plants and lower CO2 emission to prevent global warming. A new type cast & wrought Ni-Fe-based alloys with high strength and low cost has been developed recently. Considering the materials cost and workability for large component during application, high content Fe and no Co element were added into Ni-based alloys. The strength of the Ni-Fe-based alloy is much higher than some other existing Ni-Fe-based alloys and even some Ni-based superalloys. The cost of the Ni-Fe-based alloy is almost one half of that of In740 alloy. This Ni-Fe-based alloys have been evaluated as candidate materials for 700°C class A-USC boiler applications.

Creep property acts as one of the most important materials properties for boiler materials. After creep deformation at intermediate temperatures, microstructure analysis of the Ni-Fe-based alloys were performed by SEM, TEM and HRTEM-EDS. Alloying elements distribution behaviors were detected at nanoscale. Innovative deformation behaviors and strengthening mechanisms are presented that (1) double Orowan looping processes occurred in grain interiors, which could
improve the creep strength more effectively, (2) α-Cr precipitates formed and could act as effective obstacles to impede the dislocation gliding and thus increase the creep strength, and (3) the coarsening γ’ precipitates along grain boundaries contribute to strengthening the grain boundaries, resulting in the crack initiation and propagation in the relative soft region at the opposite side of the grain boundaries along the {111} slip planes.

The results presented are helpful in providing a new approach to design and develop novel high-performance alloys for A-USC boiler applications.

15:00-15:20 (1234394)
New generation of cast & wrought superalloy for A-USC boiler application beyond 700°C
F Sun, F Tadashi, Institute for Materials Research, Tohoku University, Japan

The 700°C-class advanced ultra-supercritical (A-USC) boiler is a promising technology for new generation of high-efficiency coal-fired thermal power plants. A-USC technoloy will realize both higher thermal efficiency of coal-fired power plants and lower CO2 emission to prevent global warming.

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The results presented are helpful in providing a new approach to design and develop novel high-performance alloys for A-USC boiler applications.

15:30-16:10 Tea Break and Poster Sessions

16:10-16:40 Keynote (1222226)
Development of Ni Base Superalloys for Future Energy Plant Application
Youngsoo Yoo, Hiwon Jeong, Seongmoon Seo, Daewon Yun, High Temperature Materials Center, Korea Institute of Materials Science, Korea

Increasing the operation temperature of energy plant system provides benefits of reduced cost and emission by enhancing the efficiency of the system. High temperature mechanical properties and environmental resistance of critical part should be increased to accomplish this goal mainly by enhancement of heat resistant structural materials like superalloys. Both solid solution strengthened and precipitation strengthened wrought Ni base superalloys are important in future energy plant systems such as advanced ultra supercritical (A-USC) steam turbines operating over 700°C. In this study we analyzed the effect of alloying elements on environmental property of solid solution strengthened superalloy by actual experiments and artificial neural network (ANN) method. In the first series of experiments Cr-W-Mo were considered and then the second series of experiments Mn-Si-Al were considered. Two NN models are combined to construct alloy design tool with six elements and through the NN calculations candidate alloys are suggested. Finally La-C-B effects are analyzed with similar procedure and the final alloys are designed for solid solution strengthened Ni Base superalloys. With the help of this model work new alloys with better oxidation resistance and creep property compared with commercial Ni base superalloys are developed. For the precipitation strengthened Ni base superalloys experimental design followed by
artificial neural network process are employed. Considering 7 elements Cr, Co, Mo, Ti, Al, Si, and C simultaneously 62 alloys are designed with Box- Behnken experimental design method. These alloys are actually produced and cyclic oxidation experiments are performed to construct database for artificial intelligence modeling. The database are trained with ANN to design new alloys with better creep and oxidation properties compared with a commercial precipitation strengthened Ni base superalloy. Microstructural analysis are performed to investigate the origin of the improved high temperature creep property of newly developed superalloys. The improvement of creep property of new solid solution strengthened superalloys seems caused by fine intragranular precipitation of M23C6 carbide. In the new precipitation strengthened superalloys the portion of planar slip increased considerably compared with target alloy.

16:40-17:00 (1222225)
Effects of Ti Addition on Mechanical properties of RAFM steel
S Y Im, J O Moon, C H Lee, H U Hong, Changwon National University; Korea Institute of Materials Science, Changwon, Korea

Reduced activation ferritic-martensitic (RAFM) steels are considered as structural materials for blanket module of nuclear fusion reactors because of their good thermo-physical properties with low activation capability, good high-temperature mechanical properties and long-life for nuclear applications. In this study, the effects of Ti addition on microstructure stability and mechanical properties of RAFM steel have been investigated. Ti addition of 0.01 wt% to conventional Eurofer 97 steel was intended to promote fine MX precipitation in order to enhance microstructure stability and mechanical properties. As a result, the Ti-added RAFM (Ti-RAFM) steel showed excellent high temperature tensile and creep properties. Eurofer 97 steel and the Ti-RAFM steel showed 592 h and 1823 h of creep life, respectively, after creep test at 550 °C, 200 MPa. From these results, excellent fatigue properties were also expected for the Ti-RAFM steel. Low cycle fatigue tests were therefore conducted under 550 °C, 0.0024 s-1 with total strain amplitudes of 0.4%, 0.8% and 1.2%, respectively.

Ti-RAFM steel surprisingly showed poor fatigue life of 2888, 720, 374 cycles in correspondence to each strain amplitude respectively, compared to Eurofer 97 steel with 3504, 1375, 502 cycles. This is thought to be related to the softening rate. Yield strength was higher in Ti-RAFM steel than in Eurofer 97 steel and stress reduction was also higher. In other words, it implies that resistance for recovery is very low. On the other hand, Creep-Fatigue Interaction (CFI) resembling the actual plasma on/off & operation conditions of the fusion reactor was conducted under 550 °C, 0.0024 s-1, Δεt=0.8%, tensile hold time = 600 sec. As a result, Eurofer 97 steel and Ti-RAFM steel showed 397 cycles and 578 cycles of life, respectively. Ti-RAFM steel therefore showed excellent properties in CFI compared to Eurofer 97 steel, indicating that creep is more effective in CFI. In conclusion, Ti addition effectively increases microstructure stability and mechanical properties of RAFM steels.

Monday PM Room: Room Vip (1st Floor) August 19, 2019 Symposium: B Intermetallics

13:30-14:00 Keynote (1338455)
Design Approaches and Properties of Novel Wrought TiAl Alloys for Jet Engine Applications
M Takeyama, Tokyo Institute of Technology, Japan

A five-year National project of “Structural Materials for Innovation (SM4I)” in Cross-ministerial Strategic Innovation Promotion Program (SIP) in Japan starting from 2014 is coming to an end. In this project of SM4I, a focus is placed on innovative structural materials applicable to LPT and HPC blades for jet engines, where the author at Tokyo Tech has played a key role as a technical leader to develop novel TiAl alloys, in collaboration with Hokkaido University and industries (Kobe Steel, Ltd and IHI Co.). Tokyo Tech has taken responsibility for alloy design based on phase diagram study and microstructure/mechanical property relationships. Hokkaido University has worked on oxidation behavior and mechanisms of the alloys proposed by Tokyo Tech. Kobe Steel is in charge of casting and recycling process technologies, and IHI is in charge of the
blade fabrication and forming process technologies. Eventually, we have successfully developed novel TiAl alloys applicable at 1073 K, with superior performance to the existing alloys. It is needless to say that the reliable phase diagrams are extremely important for the alloy design but commercially available existing database (DB) for the phase diagram calculations are not reliable, so that we firstly built up our own thermodynamic DB in Ti-Al-M1-M2 multi-component systems, to reproduce experimentally determined phase diagrams, by optimizing the interaction parameters among the elements in the following each phase of b-Ti, a2-Ti3Al, a-Ti and g-TiAl existing in the systems, with careful considerations of temperature, aluminum and M1/M2 concentration dependencies. The proposed alloys exhibit excellent hot workability even under faster strain rate of 10/s and with room temperature ductility of more than 1%. An introduction of bcc b phase and microstructure design using a unique phase transformation pathway of b+a→α→b+g in the multi-component systems makes it possible to develop the alloys with excellent properties in both process and service temperatures. The detailed microstructure control method to obtain the excellent mechanical/chemical properties will be presented.

Part of this study was carried under the research of SIP in JST (Japan Science and Technology Agency).

14:00-14:20 1220374
Mechanical properties of TiAl alloys with unique layered microstructure fabricated by electron beam melting
C Ken, S Masahiro, O Yeong, Y HiroyukiY, T Mitsuharu, I Ayako, U. Minoru, T Masao, N Takayoshi, Osaka University, Japan

The specific strength of TiAl alloys is much higher than that of other high temperature materials such as Ni-based superalloys. Moreover, the alloys exhibit excellent high-temperature strength and good oxidation resistance. Therefore, the alloys have been used for low-pressure turbines for aircraft jet engine. Electron beam melting (EBM) is a new technology for additive manufacturing, which is able to fabricate 3D products with high degree of complexity by adding layer-on-layer of materials. This process has attracted much attention as a fabrication process for TiAl alloys because of its high energy density and low residual stress as compared to other additive manufacturing processes.

In this study, microstructure and mechanical properties such as tensile and fatigue properties of the TiAl cylindrical rods fabricated by EBM were investigated focusing on an angle (theta) between the building and cylinder directions. As a result, we found that it is possible to obtain unique layered microstructure consisting of duplex-like regions and equiaxed gamma grains regions (gamma bands) under optimum process parameters. This layered microstructure is formed by repeated thermal effect from the melting pool. Therefore, the layered microstructure is always aligned perpendicular to the building direction. In other words, it is possible to control orientation of the layered microstructure by changing angle theta. We also found that mechanical properties of the alloy rods with the layered microstructure is strongly dependent on the angle theta. The rods fabricated at theta = 45 deg shows approximately 4 times higher elongation at room temperature than those fabricated at theta = 0 deg. Moreover, the theta = 45 deg rods exhibit better fatigue strength at room and high (750 OC) temperature compared to casting alloys even without hot isostatic pressing process. These excellent mechanical properties of theta = 45 deg rods are attributed preferential deformation at gamma bands. These results indicate that the alloys with the unique layered microstructure have a great potential for aerospace applications.

14:20-14:40 1240304
Effect of Zr on microstructure and mechanical properties of Nb-Si based multi-element alloys
Yongwang Kang, Fengwei Guo, Ming Li, AECC Beijing Institute of Aeronautical Materials, China

Nb-Si based alloys have the attractive characters such as higher melting points(>1750℃), relatively lower densities(6.6-7.2g/cm2) and excellent high-temperature strength in comparison with Ni based superalloys, which are greatly potential to serve in the condition with the temperature range of 1200~1400℃ as a family of ultrahigh temperature structural materials to replace Ni base superalloy for jet engines. Based on the previous development, the mechanical properties and oxidation resistance of
Nb-Si based alloys have been improved greatly. However, their comprehensive properties still did not fit the demand of engines. In order to improve the comprehensive properties, the influences of Zr on the microstructures and mechanical properties of Nb-Ti-Si-Cr-Al-Hf-Mo and Nb-Ti-Si-Cr-Al-V based multi-element materials were investigated. We found that Zr segregated in silicide and was able to induce the formation of γ-Nb5Si3 phase, which would accelerate the phase transformation of γ-Nb5Si3 to α-Nb5Si3 and refine the microstructure with the increase of Zr content during the high temperature heat treatment. In addition, the room temperature (H.T.) fracture toughness and hardness of the heat-treated alloys are higher than those of as-cast alloys. For heat-treated alloys, the room temperature fracture toughness and the hardness were improved with the higher addition proportion of Zr.

14:40-15:00 1230752
Microstructural evolution and hardness of rapidly solidified Nb-Si based alloys
Yueling Guo, Lina Jia, Hui Peng, Zhang Hu, Northwestern Polytechnica University, China

Nb-Si based alloys have shown great potential for future gas turbine and jet engine applications, because of their high melting temperatures (>1750 °C) and attractive creep resistance. The overall performance of cast Nb-Si based alloys has been significantly improved in the past three decades, but the low fracture toughness and insufficient oxidation resistance still impose restrictions on their practical application, owing to the coarse and complex microstructures. Microstructural refinement achieved by rapid solidification is an effective method to improve the mechanical property and the oxidation resistance of materials, which had been scarcely reported for Nb-Si based alloys.

In this context, we employed electron beam surface melting (EBSM), a typical rapid solidification method, to refine the microstructures of Nb-18Si-24Ti-2Cr-2Al (at.%) alloys. Prior to scanning, in order to prevent the cracking of brittle Nb-Si based alloys, each original substrate was preheated to 1200 °C by the high-energy electron beam. To determine their microstructure stability at high temperatures, the present work also investigated the microstructural evolution of Nb-Si based alloys processed by EBSM and subsequent heat treatment.

Results showed that a crack-free remelted layer on the original alloy produced by vacuum induction melting (VIM), with a significantly refined microstructure, was created by EBSM treatment. The grain size of silicides ranged from 15 to 113 μm for the VIM alloy, while it reduced to below 13 μm in the remelted layer. The Nbss and α-Nb5Si3 phases in the VIM alloys were transformed into Nbss and Nb3Si after EBSM upon rapid solidification. The eutectoid reaction of Nb3Si→Nbss+α-Nb5Si3 was triggered by HT at 1200 °C for 5h. A higher HT temperature at 1450 °C for 5h not only enabled the completion of the eutectoid decomposition of Nb3Si, but also promoted the growth and the coarsening of α-Nb5Si3 grains. The average hardness of the EBSM alloy was 829HV0.2 in the as-processed state, but reduced to 662HV0.2 and 578HV0.2, after the heat treatment at 1200 °C and 1450 °C for 5h, respectively.

15:00-16:10 Tea Break and Poster Sessions

16:10-16:40 Invited (1222611)
Good Compatibility of Ultrahigh-Temperature Strength and Room-Temperature Fracture Toughness for MoSiBTiC Alloy
Yoshimi Kyosuke, Tohoku University, Japan

Mo-Si-B-based alloys are promising candidates as ultrahigh temperature materials beyond Ni-based superalloys. Unfortunately, in general for structural materials, room-temperature fracture toughness comes at the expense of high-temperature strength, and the same is equally true of the Mo-Si-B based alloys. However, quite recently, the room-temperature fracture toughness of cast Mo-Si-B alloys has been well improved by TiC addition without impairing high-temperature strength. The TiC-added Mo-Si-B alloy, so-called MoSiBTiC alloy, shows a room-temperature fracture toughness value better than 17 MPa(m)1/2 after heat treatment and a heatproof temperature better than 1350°C which is defined as the temperature given by the creep lifetime of 1000 h under 137 MPa. The good compatibility between ultrahigh-temperature strength and room-temperature fracture toughness
is a big question for the MoSiBTiC alloy.

One possible mechanism responsible for the improved room-temperature fracture toughness is the ductile phase toughening with Mo solid solution (Moss). The volume fraction of Moss in the MoSiBTiC alloy is less than 50%, but Moss percolation in the microstructure is considerably improved compared with that in the ternary Mo-Si-B system. The microstructural improvement would come from the Mo/TiC eutectic reaction. The Mo/TiC eutectic reaction occurs at about 2175°C as a second solidification step, which is much higher than those of other solidification reactions involving constituent phases such as Mo5SiB2 (T2) and Mo2B as well as Moss and TiC. As a result, the Mo/TiC eutectic reaction appears to form the frame of the microstructure during solidification. This would be the reason why the Moss percolation is improved by the TiC addition.

The cast microstructure also largely affects tensile-creep behavior in the ultrahigh temperature region of 1400 – 1600°C. T2 formed during solidification has a plate-like shape on a several-tens micron scale. The plate surface is parallel to the (001) basal plane and the <100> directions of T2 preferentially grow along the cooling direction, and thus in a cast ingot, T2 has a strong texture though Mo and TiC show randomly-oriented distribution. During creep, T2 plates largely rotate and Moss works as sticky ligament in the small-plate-reinforced metal-matrix composites. This would be the reason why the MoSiBTiC alloy shows excellent creep resistance. Therefore, the unique microstructure formed during solidification is considered as the key of the good compatibility between ultrahigh-temperature strength and room-temperature fracture toughness for the MoSiBTiC alloy.

16:40-17:00 1222119
Effect of Ti content on microstructure and oxidation resistance of MoSiBTi2C alloys
H Tomotaka, Y Kyosuke, Tohoku University, Japan

TiC added MoSiB alloy (MoSiBTiC alloy) has a great attention as a promising candidate for next generation ultra-high temperature materials. This alloy mainly consists with Mo solid solution (Moss), Mo5SiB2 (T2), as well as TiC phase and exhibits outstanding high-temperature strength, creep strength, and room-temperature fracture toughness, yet it has poor oxidation resistance at elevated temperature. Recently, we developed a MoSiB-Ti-TiC (MoSiBTi2C) alloy consists with Moss, T2, TiC, and Ti5Si3 phase by the addition of 28% Ti and successfully reduced the density with improved oxidation resistance by keeping the mechanical properties. However, the oxidation resistance of the MoSiBTi2C alloy is still insufficient especially at intermediate temperature for the practical applications. The poor oxidation resistance of MoSiBTiC alloys mainly result from the continuous sublimation of volatile Mo oxide (MoO3), which disturbs the formation of continuous scales. In other words, the decline of activity of Mo in the alloy expected to improve the oxidation resistance. In this study, the increase in Ti concentration in MoSiBTi2C alloy was investigated for further improvement of oxidation resistance.

(74-x)Mo-xTi-14Si-6C-6B (x=28 to 50, mol%) were prepared by conventional arc-melting and homogenized at 1600 °C for 24 hours in argon atmosphere. Microstructure were analyzed by means of X-ray diffraction (XRD), Scanning electron microscope (SEM), and Energy dispersive X-ray spectroscopy (SEM-EDX). Isothermal oxidation tests were carried out at 800, 1100, and 1200 °C for up to 100 hours. Oxidized samples were also analyzed by XRD, SEM, and SEM-EDX. Microstructure of the alloys kept their original Moss-T2-Ti5Si3-TiC four phases even if Ti content reached 50 mol%. According to the EDX analysis, Ti was distributed in all phases by substituting Mo-site. The weight loss by oxidation became smaller as Ti content increased and 50Ti alloy exhibited the best oxidation resistance at 800 to 1200 °C due to the formation of dense TiO2 scale.

17:00-17:20 1222701
Microstructural evolution during ultrahigh-temperature tensile creep of MoSiBTiC alloy
Uemura Sojiro, Yamamuro Takateru, Yoshimi Kyosuke, Yamamoto Kamata Shiiho, Nakayama Shunichi, Eggeler Gunther, Maruyama Kouichi, Tsurekawa Sadahiro, Kumamoto University, Tohoku University, Ruhr-University Bochum, Japan

A MoSiBTiC alloy (65Mo-5Si-10Ti-10C(at. %)) has high potential for ultrahigh-temperature materials to
replace Ni-base superalloys, because the alloy has a low density, a high creep resistance showing the rupture time of over 1000 h under 137 MPa at 1360 °C and a high fracture toughness over 15MPa(m)1/2. The microstructure of the alloy is dominantly composed of the molybdenum solid solution (Moss), Mo5SiB2 (T2) and (Ti, Mo)Cx phases, in which the Moss phase would contribute to the good ductility and Mo5SiB2 and (Ti, Mo)Cx phases to high strength. In this study, we focused on the evolution of microstructure in the MoSiBTiC alloy during creep deformation at high temperatures. Tensile creep test was conducted under 137 MPa at 1500 °C in vacuum. Microstructure at creep strain of 0, 3, 12, 32, 53, 72% was observed by SEM, SEM-EBSD, TEM.

SEM observations revealed that the void formed at interface between Moss phase and other phases and the number of voids drastically increased just before the specimen was ruptured. SEM-EBSD and TEM observations revealed that the orientation dispersion and the KAM value of the Moss phase increased with increasing creep strain. In addition, fine (sub-)grains of dislocation free were formed due to the large Moss grains formed in cast ingots are divided, which suggested that dynamic recovery and recrystallization was occurred. The T2 phase, which has a plate shape of orientation (001) for the plate surface and <100] for side ones, rotated with increasing creep strain. The <100] strongly oriented the direction of the tensile axis and the plate surface of tensile specimen, and (001) oriented in the direction of the side of tensile specimen. Therefore, it is considered that the suppression for formation and concatenation of voids by the rotation of T2 phase and function of Moss phase like ligament is one of the reasons of MoSiBTiC alloy shows excellent high-temperature tensile creep resistance.

### 9:00-9:25 Invited (1232698)

**The effect of segregation of solutes at crystal defects on the mechanical performance of superalloys**

*P Kontis, L Lilensten, P Kürnsteiner, A Cervellon, J Cormier, D Raabe, B Gault, Max-Planck-Institut für Eisenforschung GmbH, Germany*

Development of new enhanced superalloys will allow operational temperatures of aero-engines and land-based gas turbines to increase, resulting in lower carbon emissions and improved performance. However, a better understanding of the deformation mechanisms at elevated temperatures is required.
for the development of smart material-design strategies. For instance, the mechanism controlling the complete or partial dissolution of γ' precipitates at elevated temperatures by the presence of high dislocation density is not well understood. We also have limited information on the deformation of secondary grain boundary phases, such as borides, which can control the mechanical performance of polycrystalline superalloys. Finally, it is still unclear how the undesirable topologically-closed packed phases forming in highly deformed regions during deformation at elevated temperatures. For all the above open questions we provide new fundamental insights into the role of crystalline imperfections, such as dislocations and stacking faults, on the lifetime of single and polycrystalline nickel-based superalloys. We have performed systematic high resolution characterization studies at the near-atomic scale by using atom probe tomography (APT) in nickel-based superalloys deformed under creep and fatigue conditions at various temperatures. We will present quantitative, near-atomic scale segregation of solutes at crystal defects in γ' precipitates and grain boundary borides. Direct observation of the segregation of particular solutes at crystal defects, allow us to elucidate the physical mechanisms controlling the mechanical performance of superalloys.

9:25-9:45 1222132

Interface structures in nickel-based single crystal superalloys during rafting at high temperatures
Dongqing Qi, Peng Zhao, Suyun He, Li Wang, Dong Wang, Langhong Lou, Yang Qi, Jian Zhang, Hengqiang Ye, Kui Du, Institute of Metal Research, Chinese Academy of Sciences, School of Materials Science and Engineering, Northeastern University, School of Materials Science and Engineering, University of Science and Technology of China, China

Single crystal nickel-based superalloys show excellent mechanical properties, especially high creep strength, and are widely used for turbine blades of aircraft engines and gas turbines. In recent years, with the increasing service temperature of turbine blades, the creep deformation behaviors above 1000 °C have been attracted strong attention. At such high temperatures, interfacial grooves from γ phase intruding into γ' phase were observed at γ/γ' interfaces during both creep and aging in nickel-based single crystal superalloys. It is worthy to note that there is often one dislocation at the tip of each groove. The types of dislocations at the tip of grooves have been comprehensively investigated. These grooves are determined being induced by the stress field of interfacial dislocations. In addition, some γ-forming elements, mainly Re, Co and Cr, are enriched around the dislocation at the tip of each groove. The enrichment of these elements with slow diffusion coefficients could impede the movement of dislocations along the interfaces. During cooling, the cooling rate has significant effects on the evolution of interfacial grooves, due to the swelling of primary γ' phases contending with the precipitation of secondary γ' phases in γ matrix. With a slow cooling rate, such as cooling in furnace, the swelling of primary γ' phases is more dominant. Therefore, the swelling of primary γ' phases during cooling could further increase the depth of grooves, especially with a slow cooling rate. While attentions were focused on the formation mechanisms of grooves before, there are many effects induced by grooves on the deformation mechanism, such as the evolution of γ/γ' interfaces and the evolution of γ' phases. In this work, the γ/γ' interface structures and their evolution during both creep and aging at 1120 °C are investigated in a third-generation single crystal superalloy with aberration-corrected scanning transmission electron microscopy in addition to energy dispersive spectroscopy.

9:45-10:05 1234773

The Tetrahedral-Octahedral Site Trajectories of Boron in L12 Ni3Al and Its (010) Antiphase Boundary
Yi Wang, Northwestern Polytechnical University, China, Pennsylvania State University, USA; K A Darling, U.S. Army Research Laboratory, USA; H Kim, S Shang, Pennsylvania State University, USA; L J Kecskes, Johns Hopkins University; Qiang Feng, Xidong Hui, University of Science and Technology Beijing, China; J Li, Northwestern Polytechnical
The development of superalloys has been driven by the more aggressive operating conditions of gas turbines in power plants (gasified coal, low-grade oil and gas with high sulfur content) and aircraft engines (high temperature and stress). To reveal the high-temperature strength, strengthening mechanism and the anomalous flow stress of superalloys, it is necessary to discuss their structural defects, such as stacking faults, anti-phase boundaries (APB) and dislocations. Being an important minor alloying element, boron (B) is commonly added to strengthen γ/γ′ Ni-based superalloys while dramatically improving the room temperature ductility of γ′, enhancing the tolerance for minor solidification defects and suppressing the intergranular fractures. Knowledge of site occupations of B in the γ′ Ni3Al phase is essential in revealing its interactions with key alloying elements. Furthermore, investigating site occupation of B in antiphase boundaries (APBs) of Ni3Al is necessary to study the role that APBs play in the anomalous yield behavior of Ni-based superalloys. In this work, we demonstrate the preferred octahedral site occupations of boron in L12 Ni3Al and its (010) type APB. Based on the electronic structures of (010) type APB, 12 initial tetrahedral sites for B are identified and reduced to 6 distinct configurations, which finally transform into 4 independent octahedral sites presented by the atomic trajectories. Far away from the fault layers of APB, the most energetically favorable octahedral site for B was dominated by energy differences associated to the tetrahedral-octahedral transition, which is attributed to a lower electron density of octahedral site than that of tetrahedral ones. This work provides an insight into the atomic and electronic basis for solid-solution strengthening mechanism of L12 Ni3Al and its (010) type APB.

10:05-10:20 Coffee Break

10:20-10:45 Invited (1231863)
Designing the microstructure of alloy 718
Sophie Primig, Felix Theska, Vitor Rielli, UNSW Sydney, Australia; Simon P Ringer, The University of Sydney, Australia

Nickel-based superalloys require critical properties such as high strength, low creep rates, wear, and corrosion resistance for operation under extreme environments. Alloy 718 is one of the most commonly used high temperature superalloys for contemporary aircraft engine disc applications. Conventional thermo-mechanical processing of alloy 718 turbine discs is carried out via multi-stage forging followed by solution annealing and double-ageing for controlled precipitation of secondary phases as precipitates and nanometer-sized clusters. In order to achieve higher yield strength, a ‘direct age process’ developed in the late 1970s [1] can be utilized instead. Even if this process is used for processing the majority of modern aircraft engine discs, the underlying microstructural mechanisms are not fully understood. Therefore, we developed a correlative microscopy approach [2] that allows imaging over multiple length scales covering the grain structure down to the atomic order to understand the corresponding processing-structure-property relationship during direct ageing. The chemical and structural similarities of precipitates and nanometer-sized clusters to the surrounding matrix material require high-resolution characterisation techniques including scanning electron and atom probe microscopy. We recently presented a model [3] for the microstructural evolution during direct versus conventional ageing in which we report a remarkable structuring within and between the nanoscale gamma’ and gamma" precipitation. Not only did we find an increase in both the volume fraction and size of the gamma" precipitates, we found a prevalence of gamma'/gamma" co-precipitation stacked in a variety of sequences depending on the nucleation condition. This finding opens up new degrees of freedom in the design of superalloy microstructure, raising fundamental questions as to which precipitate dispersions are the most effective, and how novel direct ageing-type processes might be devised that generate properly tailored microstructures.

10:45-11:05 1235001
Deformation behavior and strengthening-toughening of GH4169 alloy with multi-field coupling
Nickel-base GH4169 superalloy shows excellent combination properties including good fatigue property, good oxidation resistance and corrosion property. For satisfying high performance of aero-engine, both strength and ductility of GH4169 alloy at high temperature are required to be simultaneously improved for safety servicing. It is an effective method to strengthen alloys by adding alloying elements, which unavoidably leads to hard deforming and plasticity declining. Therefore, it is key to find method realizing strengthening-toughening and without any losing of hot-deforming ability. In present research, the multi-field (electric-pulse current (EPC)/temperature/stress) coupling was applied to GH4169 superalloy. The strengthening-toughening mechanisms and plastic deformation behavior of GH4169 alloy with multi-field coupling were investigated. The results show that deformation resistance decreases and plastic deformation ability increases of GH4169 alloy with multi-field coupling. It is found that the thermal vibration of atoms enhances leading to decreasing of Peierls force with multi-field coupling, which is the essential factor on deformation resistance decrease and plastic deformation coordinate ability increase. When the alloy aged with electric-pulse treatment (EPT)/temperature coupling, the ultimate strength, yield strength and fracture elongation increase simultaneously. The vacancy concentration increases of the alloy aged with EPT/temperature coupling. Vacancy induces ultrafine nm-sized γ’ phase to precipitate during tensile deformation at high temperature, which is the key factor on strength and ductility improvement.

11:05-11:25 1221093
Influence of Heat Treatment on Microstructures and High-Temperature Mechanical Properties of Selective Laser Melted Inconel 625
Jiwon Lee, Jinhyeok Kim, Sunyoung Jun, T Mathieu, C Etienne, L Philippe, H Hyunuk, Metal Material Science and Engineering, Changwon National University, Korea

Inconel 625 has been widely used for submarine, aerospace, power generation applications where superior mechanical resistance is required. Today, feasibility of manufacturing Inconel 625 by additive manufacturing (AM) is considered in the frame of near net shape fabrication. Although studies on AM are actively in progress world wide, microstructure as well as appropriate post heat treatment are not well understood yet. In this study, Inconel 625 is fabricated by selective laser melting (SLM) with proper manufacturing conditions (Power: 275 W, Scanning speed: 760 mm/s) ensuring 0.33 % of relative porosity fraction. On as-built sample, X-Ray diffraction residual stress measurement along the building direction resulted in normal and shear stress respectively of Min. 245 MPa average deviation 34 MPa and Min. 7 Mpa average deviation 5 MPa. Heat treatment is then applied to some of as-built samples with two different objectives. The first one is designed for recrystallization and homogenization; standard heat treatment. The second one is conducted to induce the grain boundary serration (GBS) as for strengthening the material and enhancing its creep properties; GBS heat treatment. The microstructure and the mechanical properties of as-built and heat treated samples are compared to that of standard wrought Inconel 625. The microstructure of as-built sample shows epitaxial grown columnar structures with interdendrite segregation dominantly composed with Nb and C due to rapid cooling and heating during fabrication. After standard heat treatment, recrystallization occurs on the whole sample, however NbC carbides still remain in inter and intra granular regions. Grain boundary serration is successfully obtained with over 70 % in volume fraction on wholly recrystallized grains by specially designed heat treatment. NbC carbides are precipitated on some parts of the grain boundaries and M23C6 carbides are also observed simultaneously in grain boundary serration heat treated samples. Based on these microstructural analysis, high temperature mechanical properties are evaluated and discussed.

11:25-11:45 1206153
Morphological Characterization of Multimodal Microstructure for Ni-Based Superalloy Udimet 720Li
Lei Wang, Northeastern University, China; Jinlan An, Northeastern University, Shenyang Aerospace University, China; Yang Liu, Xiu Song, Northeastern University, China
Ni-based superalloys are used for aerospace jet-engine components due to superior mechanical properties. It is important to control γ’ morphology for improving high-temperature strength in the superalloys. It is known that various γ’ morphology and γ’ sizes are exhibited during an isothermal aging for the alloys. Multimodal γ’ microstructure usually appears for the superalloys with higher γ’ volume fraction for disc applications. In recent years, researches have been attempted to the construct models predicting the high-temperature strength for Ni-based superalloys with multimodal γ’ microstructures. The aim of the present study is to evaluate the γ’ morphology quantitively by using the absolute moment invariants for Udimet 720Li.

Wrought Ni-based superalloy Udimet 720 is used in this study. The alloy was solution treated at the super-solvus temperature followed by various cooling methods; such as water quenching (WQ), oil quenching (OQ), air cooling (AC) and furnace cooling (FC). Isothermal aging was carried out at 1073–1173 K for up to 3000 h. Microstructure observation was conducted by using field emission scanning electron microscopy (FE-SEM) and transmission electron microscopy (TEM). The morphology of γ’ precipitates is quantitively evaluated by using the absolute moment invariants. Micro-Vickers hardness tester was used for hardness measurement. The secondary γ’ precipitates are observed in this study for every cooling condition, while tertiary γ’ precipitates are observed only for the FC alloy. That is, the slow cooling from the super-solvus temperature results in the multimodal distribution of γ’ precipitates. The size of secondary γ’ precipitates, d, increases continuously with the decrease of cooling rate, v, along with the following equation; $d \propto v^{-0.4}$. It is clarified that the morphology of secondary γ’ precipitates evolves from a spherical to a cubic and/or octo-dendrite shape with the decrease of cooling rate, while the aspect ratio remains unchanged. The effect of isothermal aging on the size and morphology of secondary γ’ precipitates are examined for the OQ and FC alloys. In the case of the OQ alloy, the value of d continuously increases with aging time at 1173 K, while the spherical shape remains unchanged during aging. The Ostwald ripening of secondary γ’ precipitates occurs for the OQ alloy. On the contrary, for the FC alloy, the morphology of secondary γ’ precipitates evolves from an octo-dendrite to a spherical shape with aging time under the almost constant value of d.

Tuesday PM Room: 305 (3rd floor) August 20, 2019 Symposium: B

Creep, fatigue and deformation behavior

13:30-14:00 Keynote (1220759)
Crack initiation mechanisms during VHCF of Ni-based single crystal superalloys
J Cormier, A Cervellon, F Mauguet, S Hemery, Physics and Mechanics of Materials Department, Institut pprime, France

Ni-based single crystal superalloys (SX) are widely used in gas turbine engines for the manufacturing of high pressure turbine blades due to their exceptional mechanical properties at high temperature. Service operations of blades may lead to fatigue controlled failure mechanisms due to the vibration introduced by the gas flow in addition to the centrifugal forces. These failures are difficult to forecast, as up to 90% of the fatigue life is spent in the crack initiation phase. To study crack initiation in these materials, an uniaxial ultrasonic fatigue testing system has been developed to operate at 1000°C, 20kHz and under different stress ratios. A wide range of SX superalloys having a <001> orientation have been tested to investigate the crack initiation mechanisms. CMSX-4 SX alloy cast using a Bridgman process is taken as reference. Bridgman cast MAR-M200, AM1 and CMSX-4 Plus alloys in addition to AM1 cast using a high rate solidification process have been used to expand the database. Crack initiation sites have been carefully characterized by SEM and EBSD to identify the role of their size and localization, how plasticity develops in their vicinity as well as the most detrimental casting defects. In this presentation, a critical analysis of the VHCF
life sensitivity to the casting pore size and gamma/gamma-prime microstructure degradation (i.e. gamma-prime rafting) will be performed, by using a specifically tailored crack initiation model. Moreover, for VHCF life in excess of 109 cycles, crack initiation occurs either through an internal Fine Granular Area (FGA) process or at the surface due to oxidation. By varying the casting process or the oxidation resistance among the different alloys studied, a map of crack initiation mechanisms in VHCF will finally be proposed for alloys. Moreover, the different stages leading to the formation of the FGA (including microstructure evolutions) and subsequent crack initiation will be detailed.

14:00-14:20 1233931
Vibration Characteristics of High Speed Rotating Blades:
Hua Wei, Zhejiang University, China

Turbine blades are one of the key high speed rotary components in aerocraft engines. It is very crucial to ensure the safe and reliable operation of engines. Many studies have shown that the vibration-induced blade failure is one of the important reasons resulting in blade failure. The study focused on the investigation of the natural vibration characteristics of blades coupled with the finite element analysis and experiments performed using the atomized liquid jet excitation technology for high speed rotary blades.

The atomized liquid jet excitation platform for high speed rotary blades was designed and constructed. The in-situ stress-strain testing system under the high rotation speed was set up. The high speed electric slip rings were used to transmit strain signals which were obtained through strain gauges attached to the root of blades. The vibration characteristics of blades, such as the modal frequency and resonance amplitude, was obtained by means of the in-situ stress-strain testing system under the specific rotation speed. Using the constructed platform, the vibration characteristics of blades was studied coupled with ANSYS software. The modal analysis of blades was carried out under static condition and high rotation speed, respectively. The influence of the centrifugal force caused by the high rotation speed on the inherent vibration characteristics of blades was investigated. The harmonic response analysis of blades were also made. The vibration characteristics of blades with or without damping blocks were finally compared under high rotation speed.

The obtained results showed that with the increase in injection excitation force, the peak strain of blades increased gradually but the change of injection excitation force could not affect the resonance frequency of blades. Because of the influence of centrifugal acceleration, the measured strain values of blades were greater in the deceleration stage than in the acceleration stage. In order to obtain the strain values of high speed rotary blades as accurately as possible, it was necessary to reduce the rotational acceleration of blades. The amplitudes of vibration strain of the damped blades were 50% lower than that of the undamped blades, and the inherent frequency of blades was reduced by 0.25%. It meant that the damping blocks had good vibration reduction effect.

14:20-14:40 1235799
Effect of secondary dendrite orientation on the low cycle fatigue behaviors of a third generation Ni-base single crystal superalloy
Li Wang, G Sun, Y Li, C Li, W Zheng, D Wang, L Lou, J Z Hang, Institute of Metal Research, Chinese Academy of Sciences, China

Effect of secondary dendrite orientation on the low cycle fatigue (LCF) behaviors of a third generation nickel-base single crystal (SX) superalloy was investigated in the present paper. SX bars were directionally solidified with high rate solidification technique. Then, those bars within 3° deviated from <001> were selected and full heat treated. LCF samples were machined with secondary dendrites about 0°, 26° and 45°, respectively, relative to the sample surfaces. To determine the role of holes on the LCF behaviors another group of samples were prepared with a hole in the center of the gauge section. LCF tests were carried out at 950 °C, with a strain ratio of R=0.1. It was found that for samples without holes LCF life decreases in the sequences of secondary dendrite around 45°, 0°, and 26°. When a hole presented in the center of the gauge section, samples with secondary dendrite around 0° relative to the sample surface exhibit the longest life, and the samples with secondary dendrite around
26experience the shortest life. It was discussed from micro to macro structures based on the oxidation, secondary dendrite orientation and the effect of the holes. Some of the LCF tests were interrupted to reveal the initiation and propagation of cracks during the process.

14:40-15:00 1221951
Re segregation at dislocations in the γ' phase of Ni-based single crystal superalloys
Xiaoxiang Wu, M Surendra, G Baptiste, E Gunther, Max Planck Institute für Eisenforschung, Ruhr-Universität Bochum, Germany

Ni-based single crystal superalloys have long been used as essential materials in gas turbines and power plants, due to their superior mechanical performance with respect to creep, fatigue and resistance to oxidation at elevated temperatures. The addition of a small amount of Re into Ni-based single crystal superalloys improves the creep properties significantly. However, the understanding of the role of Re is still limited and somehow unclear. Other researchers confirm that there is no observation of Re clusters at the γ matrix phase. To better understand the role of Re in superalloys and its role in creep, in the present work, we investigate the creep behavior of a second-generation Ni-based single crystal superalloy (ERBO1, equivalent to CMSX-4) at low temperature and high stress condition, using state-of-art transmission electron microscopy (TEM) and atom probe tomography (APT). We analyse the microstructure evolution in great detail for the interpretation of a peculiar double minimum creep behaviour at the primary creep stage.

We report evidence of Re and Mo segregation (up to 2.6 at.% and 1 at.% resp.) along with Cr and Co to the dislocations inside of γ' precipitates after 5% creep deformation at 750°C under an applied stress of 800 MPa in the [001] orientation. The observation of Re segregating to dislocations inside of γ' precipitates is new and can be utilised for a better understating of Re effect in superalloys. The observed segregation effects can be rationalized through bridging the solute partitioning behaviour across the γ/γ' interface and pipe diffusion mechanism along the core of the dislocation line. In combination with TEM investigations, the peculiar creep behaviour is further rationalised with the observed Re and Mo segregating to dislocations inside of γ' phases. The current results can improve our understanding with regard to the role of Re in Ni-based single crystal superalloys and provide new input for advanced alloy design.

15:00-15:20 1235155
Microstructure characteristics and tensile behavior of Ni-Co base superalloy manufactured by additive manufacturing
Ling Tang, Institute of Metal Research, Chinese Academy of Sciences, University of Science and Technology of China, China; Chuanyong Cui, Jingjing Liang, Institute of Metal Research, Chinese Academy of Sciences, China

A newly developed Ni-Co base superalloy with different contents of Co manufactured by additive manufacturing (AM) has been investigated. The microstructures were studied using OM, SEM, TEM, ICP-AES and EPMA. The results showed that the Ni-Co base superalloy made by AM showed a columnar microstructure. After heat treatments, primary γ¢, secondary γ¢ and tertiary γ¢ precipitated. The heat treatments also promoted the precipitation of η phase. The ICP-AES and EPMA investigation showed that loses and segregation of elements could account for the formation of η phase. Increasing of Co could promote the precipitation of γ¢ and decreased the segregation of Ti in the heat-treated state and was beneficial for decreasing the precipitation of η phase. The tensile tests were performed at temperatures ranging from room temperature to 800°C with a strain rate of 3×10⁻⁴. The yield strength, ultimate tensile strength, elongation, work hardening index of Ni-Co base superalloy were achieved. It has been found that yield and tensile strengths decreased and elongation showed an increasing trend with the rising of temperature. The tensile properties could be influenced by the strengthening elements and the volume of strengthening phases. In addition, columnar grain could also influence the tensile properties. Columnar grain had no transverse grain boundaries which could play an important role in strengthening at lower temperatures. What’s more, the adverse effect of transverse grain boundaries at higher temperatures could also be eliminated.
correspondingly in columnar microstructure. The fracture observations of Ni-Co base superalloys showed that the fractures were predominately cleavage.

15:30-18:00 Tea Break and Poster Sessions

Wednesday AM Room: 305 (3rd floor)
August 21, 2019 Symposium: B

Processing and alloy design

8:30-9:00 Keynote (1236125)
Microstructural evolutions of Ni-based superalloys during high temperature straining
Yinong Liu, The University of Western Australia, Australia; Haibo Long, Shengcheng Mao, Xiaodong Han, The University of Western Australia, Australia, Beijing University of Technology, China

In this talk I will present a very brief overview of the microstructural and compositional designs of Ni-based single crystal superalloys in the past decades and then focus our most recent work on the complex and multifaceted microstructural evolutions of these alloys during high temperature straining. The main aspects discussed include:

(1) Activities of dislocations. In this study we characterised the various movement manners of dislocations, such as sweep sliding, diagonal climbing and cross slip, their interactions with misfit of the g/g' interfaces, relationships between the full dislocations the g phase and the partial dislocation pairs the g' phases, formation and evolution of dislocation networks, and the modification of the Brooke formula.

(2) Formation of topologically close-packed (TCP) phases. In this study we clarified the morphologies and orientation relationships of the TCP phases with the matrix based on the minimum misfit criterion. We also discovered the progressive atomic shuffling for structure maturation after the formation of the TCP phases, revealing the atomistic processes of their formation.

(3) The evolution of secondary γ' phase precipitates. In this work we studied the continuation of secondary γ' precipitate formation during high temperature straining, and discovered two different types of secondary γ' precipitates formed inside the γ matrix and at nodes of dislocations networks, and explained their formation mechanisms and distinctive morphologies.

(4) Site preference of metallic elements in M23C6 carbide. In this study we used atomic resolution electron microscopy and super-EDS analysis to determine the atomic positioning of the alloying elements in M23C6 carbide precipitate in a Co-based superalloy.

(5) Effect of pre-existing anisotropic dislocation networks on creep behaviour. Formation of dislocation networks and g' phase rafting occur concurrently during creep testing. To delineate their effect on creep performance, we designed an innovative way to produce different anisotropic pre-existing dislocation networks without affecting the original g' cuboidal structure. This study characterised their influences on the morphology of the rafted g' phase and the creep properties of the alloys.

9:00-9:25 Invited (1351453)
Residual stress in precipitation hardening superalloys forgings
Hamish Fraser, Ben Georgin, Brian Welk, G B Viswanathan, The Ohio State University, USA

The use of powder metallurgy to produce gas turbine components provides the possibility for enhancement of local properties by using different alloys in various locations. In this way, the performance of a given component may be optimized without having to accept design compromises when using a single alloy. The present paper describes a study in which hot isostatic pressing (HIP) has been employed to produce samples consisting of two couples of differing Ni-base superalloy powders, one being CMSX-486 and CM247LC, and the other CMSX-486 and IN 718. The microstructures of the as-HIP’d couples, particularly the regions of the interfaces between the alloy combinations, have been characterized using optical metallography, scanning electron microscopy (SEM), and (scanning) transmission electron microscopy (S)TEM. Thus, the degree of compositional intermixing has been determined. Also, it has been observed that at the interfacial regions, there is coarsening of the gamma prime
Residual stress is inevitable to generate during the quenching process for precipitation hardening superalloys forging such as turbine disk. It is relaxed during the following aging treatment, but not eliminated due to superalloys’ low stacking fault energy and high creep resistance. Machining distortion could be caused by residual stress re-distribution and removed constraint, which may lead to significant re-working or additional operations. In addition, reduced but not negligible residual stresses of the final components plays an important role in determining service life. For these reasons, it has become increasingly important to know the mechanism of residual stress evolution and employ validated process models for prediction during and post manufacture, to optimize manufacture processes and improve the accuracy of lifing predictions.

In the present work, residual stress evolutions behavior during heat treatments of disk-shape forgings have been studied by advanced detections such as in-situ neutron diffraction. Various commercial precipitation hardening superalloys (Inconel 718, Waspaloy, Udimet 720Li…) were employed to study the effect of precipitation behavior on the residual stress induced by heat treatments.

It is shown that the quench-in residual stress is mainly generated by steep thermal gradients, which could reach the tensile strength level of the materials. The quench-in residual stress is partially relaxed during the ageing treatment as a result of plastic and creep deformation. The precipitation of γ’ or γ” plays an important role in the induction and relaxation of residual stress due to its significant effect on the plastic deformation behavior (such as tensile strength and creep rate) and expansion coefficient (volume shrinkage) at different temperatures. On the other hand, the precipitation behavior of γ’ or γ”, such as nucleation rate and variant selection could be influenced by residual stress condition. Based on considering the interaction between residual stress evolution and precipitation reaction, a residual stress prediction model is carried out with experimental validation.
investigated and the mechanical tested database for the alloy should be established with various test data. In this study, the hot tension and creep behaviors for Alloy 800H WM fabricated by a gas tungsten arc welding (GTAW) procedure were comparatively investigated with those of the BM through the hot tensile tests from room temperature to 900°C and a series of creep tests at 800°C. And the hot-tensile curves for Alloy 800H BM and WM were modelled in comparison with the two models of the Ramberg-Osgood (R-O) model known as a typically strong tensile model and the General Atomic (GA) model proposed as a proper model herein. In addition, the CCG behavior for Alloy 800H BM was investigated in terms of C*-fracture parameter through a series of CCG tests at 800°C. The equation for evaluating the CCG rate for Alloy 800H BM was proposed using the plot of da/dt vs. C*.

10:10-10:30 1221643
**Dependency of deformation behavior on γ’ precipitates formed by various heat treatments in Haynes 282 superalloy**
*Jin Hyeok Kim, Kyeong Yong Shin, Mathieu Terner, Byeong Ook Kong, Hyun Uk Hong, Changwon National University, Korea*

In this study, we investigated the microstructure features and the deformation mechanism of Haynes 282 superalloys in tension at 750 °C. The well-known strengthening phase in superalloy which is called γ precipitate was observed differently as various heat treatment conditions. We conducted the standard 2-step aging heat treatment (1010°C/2h + 788°C/8h) and modified 1-step aging treatment (800°C/4h) which is more economical. Moreover, three different cooling rates were conducted to study mass effect which can occur in large scale products. All of the samples subjected different heat treatments were observed similar microstructure. However, the size distribution of γ precipitates was significantly different from each other. In all samples, about 20% of γ precipitates were precipitated with an average size between 12 nm and 39 nm. The average γ precipitates size increased as the cooling rate from the solution temperature decreased. All four heat-treated alloys exhibited good mechanical properties at the high temperature of 750°C with a yield strength in particular well over 620 MPa. As it could be expected, the yield strength increased and the ductility decreased as the average γ’ particles size decreased. All the alloys exhibited a mixed mode of deformation characterized by shearing and bypassing. However, the dominant deformation mechanism depended on the γ characteristics resulting from different heat treatment conditions. Only 1-step aged specimens with the largest γ particles of 39 nm size, which was furnace-cooled from solution temperature, showed a plastic behavior typical of a dislocations shearing mechanism while the other alloys showed an Orowan dislocations bowing and looping mechanism behavior. To enhance theoretical approach, the strength increment was calculated to predict the major operative deformation mechanism based on the precipitation strengthening model. The theoretical calculation was well agreed with the results of observation.

10:30-10:45  Coffee Break

10:45-11:10 Invited (1234031)
**Materials Genome Initiative: Accelerated Ni-based single crystal superalloy design**
*Chengbo Xiao, Jingyang Chen, Science and Technology on Advanced High Temperature Structural Materials Laboratory, Beijing Institute of Aeronautical Materials, China; Xidong Hui, Zhenmin Du, University of Science and Technology Beijing, China; Xuejing Shen, Central Iron and Steel Research Institute, China*

Ni-based single crystal superalloys are key materials for hot section components of advanced aero engines and land based gas turbines. The successful experiences of General Electric (GE) and Pratt & Whitney (P&W) show that the use of Materials Genome Initiative (MGI) methods can accelerate the research and development of novel Ni-based superalloys. In the present study, the specific thermodynamic and kinetic database for Ni-based superalloys was founded preliminarily. The integrated design and screening of Ni-based single crystal superalloy composition were carried out combined with thermodynamic and kinetic calculations, machine learning technique and design experiences. High-throughput preparation
and characterization was carried out using techniques such as multitarget carousel oblique angle deposition, variable cross-section creep testing and original position statistic distribution analysis combined with laser-induced breakdown spectrometry (LIBS-OPA). An integrated multi-scale design method for microstructure and property of novel Ni-based single crystal superalloys was established based on the MGI techniques. The above work lays the solid foundation for changing the traditional developing pattern of Ni-based single crystal superalloys as well as shortening the developing period and reducing the developing cost. The 2nd generation Ni-based single crystal superalloy DD489 for gas turbines with high temperature capacity, good hot corrosion resistance and microstructure stability was developed successfully.

Precipitate-Mediated Dislocation Transformation in Superalloys

Yunzhi Wang, Longsheng Feng, Mike Mills, The Ohio State University, USA

High-temperature alloys in general and superalloys in particular are strengthened by ordered intermetallic phases that are relatively stable at elevated temperatures. Because of their low symmetry, these ordered intermetallic phases have rather complicated deformation mechanisms that are difficult to uncover by experiment alone. In this study we use a combination of ab initio calculation, phase field simulation and experimental characterization at individual dislocation level to illustrate how the interactions between dislocations and precipitates of a low-symmetry ordered intermetallic phase such as gamma” (D022, tetragonal), the primary strengthening phase in IN718 superalloys, breed new particle dislocations, which may initiate twinning and other deformation modes. For example, two unstable stacking faults exist in gamma” phase, APB-like and CSF-like. These unstable stacking faults, once created, will transform spontaneously into nearby stable stacking faults on the generalized stacking fault (GSF) energy surface and generate new Shockley partial dislocations that do not exist in the system before the shearing events. Thus, the gamma” precipitates serve as a “dislocation-transformer” that transform incoming <110>/2 dislocations into Shockley partials (<116>/6) that are not part of the incoming full dislocations. By passing the same <110>/2 dislocation consecutively on one slip plane from an active Frank-Reed source, we found that, for each type of <110>/2 full dislocations, only one specific Shockley partial prevails in the system. This could be one of the reasons why extensive micro-twinning is observed in these alloys. Understanding this precipitate-mediated dislocation transformation mechanism can further spark new ideas on alloy design, especially for tailoring the twinning mode. This work is supported by US NSF under grant the DMREF program (Grant No. DMR-1534826).

Phase field method (PFM) has achieved increasing prominence in recent years for microstructure simulation. However, the PFM has not been utilized directly by designers for engineering applications since it has difficulties in being quantitative when applied to multi-component alloys. Any alloy-specific and quantitative phase field simulations require thermodynamic and phase equilibrium information as input for millions of different chemical compositions. Direct calculations of the required thermodynamic quantities for each composition from thermodynamic models are prohibitively expensive computationally, especially for multi-component and multi-phase alloys when the number of components is greater than four. To overcome this computational bottleneck, a thermodynamic data management system, PanDataNet, is developed to provide PFM with the required thermodynamic quantities. PanDataNet manages required properties by PFM on a net of temperature and composition grids. Properties on each grid are
calculated only once from PanEngine (thermodynamic and phase equilibrium calculation engine) and only those grid points needed by PFM will be calculated. PanDataNet can save all calculated properties for future use. The speed to access PanDataNet is several orders of magnitude faster than the direct access to PanEngine. An example will be given for the direct coupling of PanDataNet and PFM in microstructure simulation of a multi-component alloy and the results demonstrate the computational efficiency of PanDataNet, a high-speed high-throughput thermodynamic data management system.

11:55-12:15 1222133

Machine learning accelerates component design of 2nd generation long life Nickel-based single Crystal superalloys

Qianyu Huo, Jinbin Chen, Xidong Hui, State Key Laboratory for Advanced Metals and Materials, University of Science and Technology Beijing, China

Jingyang Chen, Science and Technology on Advanced High Temperature Structural Materials Laboratory, Beijing Institute of Aeronautical Materials, China

Nickel-based single crystal superalloys are widely used in the gas turbines blades because of their excellent high-temperature properties. However, the complex interaction of the multi-element also brings many possibilities for component design and optimization. In order to develop a 2nd generation nickel-based single crystal superalloys with longer life, it is usually necessary to test creep rupture life of many possible components as an important parameter to evaluate their stress rupture property. To obtain this parameter, a lot of time and cost are needed, and the traditional "trial-and-error" method is difficult to obtain better components quickly. Therefore, it is necessary to use the existing experimental and computational data, combining the material genome and machine learning data mining methods to study, in order to speed up the component design of the 2nd generation long-life nickel-based single crystal superalloys.

Our work is based on the 2nd generation long-life nickel-based single crystal superalloys designed by Beijing Institute of Aeronautical Materials. Thermo-Calc thermodynamic software is used to calculate the information of γ, γ' and TCP phase content of the alloy in the range of component. The calculated data and other parameters are used as input features, and the creep rupture life is taken as the target parameter, and the model between the appropriate features and the target is established by using Support Vector Regression algorithm (SVR). In this work, we use the better training model to predict the creep rupture life of the untested alloys in the component range, and choose the components with higher creep rupture life or lack of experiment to carry out experiments, so as to form a positive cycle for database, performance prediction and experiments, thus accelerating the component design process of 2nd generation long life nickel-based single crystals superalloys for gas turbines blades.

The results show that the machine learning method can still accelerate the research and development of components for the problem when there are only a few training samples in our initial work. The relationship between different features and creep rupture life is established by support vector regression (SVR) algorithm, and the suitable components can be found more quickly by experiments based on the predicted results of the model. This work combines thermodynamic calculation, data mining and experiment with the idea of material genome, and develops the traditional design idea of nickel-based single crystal superalloy from component to performance to quickly screen the suitable components range of the alloys according to performance requirements, which greatly reduces the research cost and cycle of the 2nd generation nickel-based single crystal superalloys.

Wednesday PM Room: 305 (3rd floor)
August 21, 2019 Symposium: B

Novel concepts

13:30-13:55 Invited (1234281)

Gamma–Gamma Prime based Precipitation Strengthenable High Entropy Alloys

R Banerjee, B Gwalani, S Dasari, A Jagetia, R Mishra, University of north Texas, USA
While high entropy alloys (HEAs), based on single phase concentrated solid solutions, have attracted a lot of worldwide attention, their potential application as real engineering alloys is rather restricted, especially for high temperature applications. Furthermore, often the experimentally observed single phase HEA is the result of second phase precipitation constrained by thermodynamic and kinetic factors. This presentation will focus on designing HEAs strengthened using a second (or more) phases. Al0.3CoCrFeNi will be discussed as a model HEA, to investigate second phase precipitation as a function of different thermomechanical treatments. This includes refined L12 (γ′) precipitation as well as B2 precipitation in this alloy leading to substantial improvements in yield and tensile strength while maintaining substantial ductility. Subsequently, using a CALPHAD based solution thermodynamic approach, another novel precipitation strengthened fcc based HEA has been developed with the objective of maximizing the phase fraction and solvus temperature of L12 (γ′) precipitates. Detailed microscopy using transmission electron microscopy (TEM) and atom probe tomography (APT) was carried out to characterize the alloy in various conditions. Hall-Petch strengthening coupled with precipitation resulted in a high tensile yield strength of 1600 MPa with a reasonably good ductility of ~15%. These results hold promise for the development of precipitation strengthened HEAs for high temperature applications.

13:55-14:20 Invited (1219962)
Design of γ' -strengthened Co-base alloys based on multi-scale characterization
Choi Pyuck-Pa, Im Hyeji, Yoo Boryung, Korea
Advanced Institute of Science and Technology, Korea

In this research we have explored Co-Ti-based alloys forming a two-phase gamma/gamma' microstructure with cuboidal gamma' precipitates of 60 ~ 70% volume fraction and 0.5 ~ 1.0% lattice misfit. Such a microstructure is well known from modern Ni-based superalloys, used in aerospace and land-based gas turbine applications, and is reported to be beneficial for high-temperature strength and creep resistance. However, the simple binary Co-Ti system cannot meet the above-mentioned criteria for the gamma/gamma' microstructure, and hence, the aim of this work is to identify novel multicomponent Co-Ti-based alloys forming the desired microstructures. We have investigated the binary Co-Ti reference system as well as ternary Co-Ti-X (X=Cr, V, Mo, W) and quaternary Co-Ti-X-Y (X=Mo, W, Y=Cr, Al) alloys with respect to their microstructure and high-temperature properties. Aged alloys were characterized using scanning electron microscopy, X-ray diffraction, and atom probe tomography (APT). APT analyses were performed to elucidate the elemental partitioning and site-occupation behavior of specific alloying elements and to understand the changes in gamma' volume fraction and morphology upon alloying. We found that Co and Cr partition to gamma, whereas Ti, Mo, W, and Al partition to gamma'. Additions of third elements to the reference Co-Ti system lead to significant increases in gamma' volume fraction. Elemental spatial distribution maps of the L12-ordered gamma' phase reveal that the third elements preferentially replace Ti on its sub-lattice. Some of them also affect the Ti solubility within gamma. The remaining excess Ti is available for formation of additional gamma', resulting in enhanced gamma' volume fractions and phase stability. In addition to microstructural characterization, we performed differential scanning calorimetry measurements to determine phase transition temperatures such as gamma-prime solvus, solidus, and liquidus temperatures. By combining the above-mentioned characterization techniques, we could elucidate the role of the alloying elements on the gamma/gamma' microstructure and the stability of the gamma' phase.

14:20-14:40 1233104
Thermal stability and strengthening effects of nano-precipitated Ni3Al phase in CrCoNi alloys
Ning An, Jianjun Tian, Yongji Niu, Beijing Beiye Functional Materials Corporation, China

The single-principal alloy is mainly constituted of one metal or intermetallic compound, then optimized by alloying and processing to achieve optimum performance. However, the arisen multi-principal alloy design subverts the classic design. Multiple principal elements could lead to more composition
design choices, together with reasonable heat treatment and deformation treatment, which can meet different engineering requirements. CrCoNi alloy is a single-phase solid solution with FCC structure. If there are only Cr, Co, and Ni components, CrCoNi alloy do not have precipitates; However, the addition of other microalloying elements, there can precipitate some super-lattice structure phases in CrCoNi alloy. It is well known that the proper addition of Al and Ti elements can facilitate to precipitate a large number of L12 type γ' phase (Ni3Al) and DO24 type Ni3Ti phases in Ni-based alloys. With the increase of Al and Ti content, these precipitates can grow rapidly from nanometer to micron in size. That could significant improve high temperature creep properties.

In this study, the effects of Al and Ti additions on the microstructure and properties of CrCoNi alloys were investigated by means of X-ray diffraction analysis, differential scanning calorimetry, scanning electron microscopy, transmission electron microscopy and Thermo-calc; And the microstructure and strengthening and toughening mechanism of Ni3Al phase precipitation strengthening CrCoNi multi-principal alloy were explored too. The results presented that the addition of Al and Ti significantly reduced the solidus and liquidus temperatures of CrCoNi multi-principal alloys, and enlarged the solidification interval. The Al/Ti ratio has a significant effect on the type and precipitation order of precipitated phases. The results of tensile and microscopic analysis presented that the deformation-induced three-dimensional structure of nano twins could play a significant role in strengthening and toughening. The multi-layer nanostructures with nanocrystalline, nano twin nanocrystalline, nano Ni3Al precipitates and microcrystals can be regulated and controlled by adjusting the composition and process. The microstructure stability and toughness of CrCoNi multicomponent alloy can be improved significantly. There are superior oxidation resistance and corrosion resistance in CrCoNi alloy. With improvement of creep resistance at high temperature, it is very promising to broaden the application range of CrCoNi alloy, which can be more widely used in high temperature structural parts.

Studies of discontinuous precipitation in Ni-Co-Al ternary alloy system
Yang Zhou, Jun Wang, Zhou Fei, Shanghai Jiaotong University, China; Philip Nash, Illinois Institute of Technology, USA

Discontinuous precipitation (DP) produces precipitate lamellars that have a distinct orientation relationship with the matrix and nanoscale widths and spacings. The major effects of discontinuous precipitation on the microstructure and mechanical properties in Ni-Co-Al alloy system were investigated in this work. 1) We proposed a novel technique for the large-scale synthesis of aligned-plate nanostructures that are self-assembled and self-supporting, which involves developing nanoscale two-phase microstructures through discontinuous precipitation followed by selective etching to remove one of the phases. This technique is demonstrated in Ni-Co-Al ternary alloy system using the discontinuous precipitation of a γ’ phase, (Ni, Co)3Al, followed by selective dissolution of the γ matrix phase. The method could be applied to any alloy system in which the discontinuous precipitation transformation goes to completion. The resulting structure may have many applications in catalysis, filtering and thermal management depending on the phase selection and added functionality with the retained phase. 2) The mechanical properties of Ni-Co-Al alloy samples with DP structure were investigated. The results imply that the presence of large volume fraction of DP transformed structure increases the hardness, ultimate tensile strength and yield strength significantly while decreasing the ductility. Higher aging temperature favors the occurrence of continuous precipitation and decreases the driving force for discontinuous precipitation.

Effect of L21-Co2Al(Ti,V) precipitates on deformation behavior of Fe-Al-Co-Ti-V single crystals
Yasuda Hiroyuki, Yasunishi Yuki, Kobayashi Ryota, Cho Ken, Osaka University, Korea

Recently, ferritic heat-resistant alloys strengthened by the intermetallic precipitates such as B2-type NiAl
and L21-type Ni2AlTi have attracted much attention due to high strength at high temperatures. The CoAl precipitates with the B2 structure are also found to be effective in increasing high temperature strength of ferritic Fe-Al based alloys. The CoAl phase is precipitated in the bcc matrix with cube-on-cube orientation relationship with small misfit strain. If the precipitates are sheared by the dislocations in the bcc matrix, the precipitates strongly suppress the dislocation motion due to the difference in primary slip system between the matrix and the precipitates, resulting in high strength. We call the precipitation hardening mechanism "slip frustration hardening". However, if the B2 precipitates dissolve into the bcc matrix above 973 K, the yield stress decreases steeply. Small amount of Ti addition leads to the formation of the Co2AlTi precipitates with the L21 structure. The L21 precipitates demonstrates a high dissolution temperature, which is favorable for high temperature strength. However, the dislocations in the bcc matrix bypass the coarse L21 precipitates with large misfit strain, resulting in low strength. On the other hand, if small amount of V is doped, the Co2AlV phase with the L21 structure is precipitated in the bcc matrix with small misfit strain. The precipitates are sheared by the dislocation in the bcc matrix, resulting in high strength. However, V doping is not effective in increasing the dissolution temperature of the precipitates. Co-doping of Ti and V leads to the formation of the L21-Co2Al(Ti,V) precipitates and is effective in decreasing misfit strain between the bcc matrix and the precipitates and increasing the dissolution temperature. As a result, the L21 precipitates can be cut by the dislocations, resulting in high yield stress above 500 MPa even at 1023 K. The precipitation hardening mechanism was examined using the Fe-Al-Co-Ti-V single crystals.

15:20-16:10 Tea Break and Poster Sessions

16:10-16:35 Invited (1348517)
**β-NiAl based protective coatings for advanced single crystal superalloys**
Hongbo Guo, Jian He, Liangliang Wei, Hui Peng, Shengkai Gong, Beihang University, China

β-NiAl based coatings have seen promising potential application as protective coatings over superalloys or as the bond coats in thermal barrier coating (TBC) system due to their capability of forming an α-Al2O3 oxide scale above 1150 °C. However, the oxide scale on the β-NiAl based coating spalls readily during high-temperature cyclic oxidation. In this work, more than 10 reactive elements (REs) were investigated to improve the cyclic oxidation performance. The effects of different REs on the adherence of Al2O3/NiAl interface were investigated by first principles theory calculations and experiments. We find that the solubility of the REs in NiAl alloy arrive at an order of Hf >Zr>Dy>Y>La. All the REs exhibit an affinity for sulfur, with an order of La>Dy>Y>Zr>Hf, whereas effects of the REs on the Al2O3/NiAl interface exhibit an order of Hf>Y>Hf>Zr>clean interface>La. Combined with experimental results, we provide some suggestions on how to choose appropriate REs. Meanwhile, co-doping of congeneric REs such as Hf+Zr or Dy+Y is more effective than single RE doping in improving the oxidation behavior due to the synergistic effect of co-segregation of RE ions. Some strategies on inhibiting interdiffusion between the β-NiAl coatings and advanced single crystal superalloys were discussed to constrain the formation of secondary reaction zone (SRZ) and needle TCP phases in the superalloys.

16:35-17:00 Invited (1235820)
**Application of Pt-Ir Paste Coating for Ni-based superalloys**
Hideyuki Murakami, National Institute for Materials Science, Japan

Ni-based single crystal superalloys with good high temperature strength and creep resistance can be used in the hottest section of gas turbines such as turbine blades. For further protection against oxidation and hot corrosion, thermal barrier coating (TBC) systems were developed. Basically, TBCs consist of a ceramic top coat which provides thermal insulation and a metallic bond coat, which not only supports the bonding between the TC and a substrate material but also works as an Al reservoir to form a TGO layer during high temperature exposure. Recently, a Pt diffusion coating with the γ-γ’ microstructure, which is similar to that of the substrate has been proposed as a BC. It has been reported to reduce rumpling and suppress the
precipitation of brittle TCP phases. To further improve the performance of bond coats, addition of iridium was focused, because it exhibits the highest melting temperature, high tensile strength at room temperature, excellent chemical stability and lower diffusivity into Ni-based alloys than Pt. It can thus be expected that the addition of Ir into Pt diffusion coating may result in better thermal properties and prolong the life of coated materials. The authors’ research group has investigated Pt-Ir-modified aluminate coatings and reported that Ir addition can improve the high temperature oxidation resistance of the Pt-modified aluminides. Then the next step is to further improve the oxidation resistance of Pt diffusion coating by the Ir addition. In this study, the microstructure and oxidation resistance of Pt-Ir diffusion coating on single crystal Ni-based superalloy were investigated. Indeed, Yasui et. al. reported that in the electrodeposition process, addition of Ir to Pt-diffusion coating suppresses the formation of voids in the substrate but oxidation resistance of the coating is dependent on the quality of Pt-Ir films deposited, which is affected by substrate surface finishing and composition. To minimize this problem, a new route to fabricate Pt and Pt-Ir coating, i.e., a paste coating, was introduced in comparison with the electroplating method. The phase transformation and cyclic oxidation behaviors of the Pt and Pt-Ir coatings at 1423 K by both paste and electroplating methods were discussed. Some Ni-based single crystal superalloys were used as the substrate materials. The Pt and Pt-Ir films were deposited on the surface of specimen by two methods: electroplating and paste coating. All the electroplated coatings were diffusion heat treated for 1 hour at 1373 K in flowing Ar to protect coatings from oxidation, whereas paste-coated specimens were eat treated at 773 K for 2.5 hours to evaporate the binding elements and diffusion heat treated for 1 hour at 1373 K in flowing Ar.

The cyclic oxidation tests were carried out in a programmable muffle furnace at 1423 K in still air. Each thermal cycle consisted of three periods: i) putting the specimens into the furnace to rapidly increase the temperature of specimens to 1423 K (the temperature of furnace), ii) holding the specimens in furnace at 1423 K for 1 hour, and iii) removing the specimens from the furnace for 20 minutes air-cooling. The surface and morphologies and element concentration profiles along the cross section of the coating were analyzed by an FE-SEM equipped with EDS. In addition, the crystal structure of the coatings was identified by an x-ray diffractometer.

The paste coating by a simple spray method was successfully applied to synthesize Pt and Pt-Ir diffusion coatings. It was found that Ir addition retards the formation of voids, and the pasted Pt-20Ir coating shows the promising features as the bond-coat: with no cracks, less voids and stable protective Al2O3 formation on the surface.

17:00-17:20 1234536

Research on the Interface Reactions and Wettability between a Y-containing Superalloy Melt and Ceramic Materials

Yun Zi, Chinese Academy of Sciences, University of Science and Technology of China; Jie Meng, Yizhou Zhou, Institute of Metal Research, Chinese Academy of Sciences, China

In this paper, the interface reactions and wettability between a Y-containing superalloy melt and ceramic materials were studied by means of sessile drop tests. The microstructural characterization of the alloy-ceramic interface and the elemental composition of reaction products were performed by SEM/EDS analysis and EPMA analysis, the phases of reaction products were identified by XRD analysis, and the surface analysis of reaction products was carried out by XPS. The results showed that Al and Hf in the alloy melt reacted with SiO2 in the ceramic mould when Y content was 0.011 wt%, and the reaction products were Al2O3 and HfO2, with little change in the wetting angle. However, when Y content was higher than 0.011 wt%, Y and Al reacted with SiO2 in the ceramic mould, and the wetting angle decreased significantly. The reaction products of 0.1Y alloy were Y3Al5O12 and Y4Al2O9, and the reaction products of 0.5Y alloys were double reaction layer composed of Y2O3 and Y3Al5O12. For the alloy-ceramic core system, when Y content was 0.011 wt%, the reaction products were Al2O3, HfO2 and ZrO2, and the wetting angle dropped slightly. However, when Y content was more than 0.011 wt%, Y, Al, Hf and Ta both reacted with SiO2 in the ceramic core, the reaction products of 0.1Y
alloy were Y3Al2(AlO4)3, Y4Al2O9, HfO2 and ZrO2, and the reaction products of 0.5Y alloy were Y2O3, TaO, HfO2 and ZrO2. ZrSiO4 in the ceramic core partially decomposed to form ZrO2 at high temperature.

17:20-17:40 1235763
Experimental and numerical investigation of compressive creep in 3D-woven Ni-based superalloys
Hoon-Hwe Cho, Dinc Erdeniz, Keith W. Sharp, David C. Dunand, Hanbat National University, Korea

Micro-architected Ni-based superalloy structures, with Ni-20Cr-3Ti-2Al (wt.%) composition and r/r'-microstructure, are created by a multi-step process: (i) non-crimp orthogonal 3D-weaving of ductile, 202 mm diameter Ni-20%Cr wires, (ii) gas-phase alloying with Al and Ti, (iii) simultaneous transient-liquid phase (TLP) bonding between wires and homogenization within wires via interdiffusion, (iv) solutionizing to create a single-phase solid solution, and (v) aging to precipitate the r' phase. The creep behavior of these 3D-woven r/r' nickel-based superalloys is studied under uniaxial compression via experiments at 825 oC and via finite element (FE) analysis, using a 3D model of the woven structures obtained through X-ray micro-tomography. The creep strain rate for the woven Ni-based superalloy is higher than that for the bulk superalloy due to the lower solid volume fraction of the woven structure, while the creep exponents are identical. The compressive creep behavior is sensitive to the geometry of the woven structures: fewer wires perpendicular to the load and fewer bonds between wires cause lower creep resistance of the woven structure, due to a reduction in load transfer from the longitudinal wires (which are primarily load-bearing) and the perpendicular wires. Creep buckling of longitudinal wires drastically reduces creep resistance of the woven structure, confirming the importance of maintaining longitudinal wires vertical and parallel to the uniaxial compression direction. Finally, reducing wire cross-section, e.g., via oxidation, reduces creep resistance. The oxidation kinetics of the wire structures at 750, 825, and 900 oC displayed parabolic rate constants comparable to commercial Ni-based superalloys, but indicates that up to 35% of the wire cross-section is oxidized after 7 days at 825 ºC, such that oxidation-resistant coatings are needed for long-term use in oxidative environment.

Thursday AM Room: 305 (3rd floor)
August 22, 2019 Symposium: B
Casting and manufacturing

8:30-8:55 Invited (1235420)
Research statutes and progress of solidification structure and grain defects in nickel-based single crystal superalloys
Lin Liu, Jun Zhang, Hengzhi Fu, Northwestern Polytechnical University, China

With the alloy development and size and structure diversification of engine blades, two aspects of single-crystal superalloy development have increased the likelihood of solidification defects forming in industrial practice. First, refractory element additions, to enhance the creep performance and alloy stability in service, have a detrimental effect on their casting characteristics. This has led to difficulties in controlling the single crystal orientation and to an increased occurrence of stray grains and freckle chains. Second, thermal conditions in producing large castings for industrial gas turbines are quite different from those experienced in the manufacture of turbine blades for aero-engines. In particular, the critical solidification parameters, temperature gradient and solidification rate are significantly reduced and the solidification front tends to be macroscopically curved, rather than planar. These factors combine to produce conditions where solidification defects are more likely to occur.

The present presentation initially introduces some typical casting defects frequently encountered in casting practices in single crystal superalloys, in order to establish physical basis for defect formation, solidification characteristics, dendritic orientation
and growth competition for single crystal superalloys are analyzed. The grain selection process during single crystal casting both in with bottom seeding and spiral grain selection is simulated by a macro-scale ProCAST coupled a meso-scale Cellular Automaton Finite Element (CAFE) model. The microstructure evolution of grains with different orientations during directional solidification is investigated also by the phase-field method. An in depth analysis is presented of formation mechanisms, effect of processing parameters/composition, and possible control technique for distinct defect. We emphasis on the effect of solidification characteristics of alloys, crystal growth orientation, thermal and solutal fields on the defects, try to reveal nature of defect formation. The key developments and future challenges in this field are summarized. The overall objective is to establish defect formation criterion and a relationship between alloy chemistry, process and defect-free single crystal superalloy components.

8:55-9:20 Invited (1233438)
A Phenomenological Analyses of Freckling in Directional solidification of Ni-base Alloys
Hongbiao Dong, University of Leicester, UK; Fu Wang, Xi’an Jiao Tong University, China; Zihui Dong, University of Leicester, UK; Dexin Ma, Shenzhen Wedge Central South Research Institute, China

Freckles have been of interest for many researchers for around 50 years, due to their deleterious detrimental effects in the advanced directional solidification (DS) and single crystal (SC) castings of superalloys. Freckles appear as long trails of equiaxed grains aligning roughly parallel to the direction of gravity. And they were often found on the surface of DS/SC superalloy castings. It is now generally agreed that freckles are associated with thermosolutal convection originating in the interdendritic liquid during solidification, which is driven by a density inversion occurring in the mushy zone as a result of interdendritic segregation. Besides the alloy chemistry the occurrence of freckles is known to be mainly dependent on the solidification parameters and casting size. A lower temperature gradient G and solidification velocity V result in the larger dendrite spacing and higher permeability. The occurrence of freckles was rarely taken geometrical factor of castings into account. In this work, the freckle formation in complex geometry superalloy components was analysed. According to new features of freckle appearance, freckle is subject to edge and the curvature effects. In polygonal casting sections, freckles are formed preferably on the convex edges. In the components with curved contour, freckles were exclusively found on the outward curving surface having positive curvature. Two effects can be attributed to the overlapping or divergence of the surface effect, which can more effectively affect the freckle formation than the local thermal conditions.

9:20-9:45 Invited (1232355)
Formation and evolution of casting defects in single crystal nickel based superalloys
Jian Zhang, Institute of Metal Research, Chinese Academy of Sciences, China

Single-crystal (SX) nickel-based superalloys have been widely used in turbine blades for their excellent high-temperature capabilities. However, casting defects, such as slivers, freckles, and micro-pores, limit the performance of SX superalloys. The formation and evolution of sliver and micro-pores in SX superalloy are discussed in the present talk. Ex-situ X-ray computed tomography (XCT) characterization combined with microstructure observation and electron backscattered diffraction (EBSD) revealed that sliver generally generated from one or two deformed dendrites. The misorientation between sliver and matrix did not change during directional solidification, although the orientation of both sliver and matrix may rotate according to the geometry of the casting. The role of micro-pores during solidification, heat treatment, creep and fatigue tests was also discussed. For example, large casting pores generated during directional solidification may induce deformation of regular dendrite arms. Sliver developed from the deformed dendrites. Some of the casting pores grew up in situ during solution heat treatment. New pores also generated during solution. It is believed that the growth behaviors of both solidification-pores and solution-pores can be related to the vacancy formation and diffusion during heat treatment. Size and volume fraction of most micro-pores was
unchanged during low cycle fatigue (LCF), but micro-pores near the surface may play an important role in LCF.

9:45-10:05 1222340
High temperature mechanical behavior of ceramic core for directional solidification of turbine blades
Jiangwei Zhong, Zilin Xu, Qingyan Xu, Baicheng Liu, Tsinghua University, China

The high temperature mechanical behaviors of SiO2-based cores for the directional solidification of turbine hollow blades were investigated. Isothermal uniaxial compression tests of ceramic core samples were conducted on a Gleeble-1500D mechanical simulator with an innovative auxiliary thermal system. This thermal system was established to measure the high-temperature mechanical properties of non-conductive materials. The measured temperature can reach 1600 °C. The temperature control accuracy is ±4 °C. The stress-strain results, macro- and microstructures of SiO2-based ceramic cores were investigated experimentally. The microstructures were characterized by the scanning electron microscope (SEM). The elastic moduli of ST25, ST700, ST1100, and ST1400 are 2726.39 MPa, 2259.19 MPa, 2316.82 MPa, and 1441.87 MPa, respectively. With the increase of the temperature, the elastic modulus of the SiO2-based ceramic core shows a decreasing trend. There is a small change in the range of 25~1100 °C, while the elastic modulus decreases rapidly at the range of 1100~1400 °C. The stress-strain curve of ST1400 at the viscoplastic stage is narrow, indicating that the high-temperature experimental result achieves high repeatability and reproducibility. However, the overall high-temperature mechanical property of the sample ST1400 decreases significantly. The SiO2-based ceramic core samples are all brittle fractures, but when the temperature exceeds 1400 °C, there are almost no cleavage fractures in SiO2 particles of the ST1400 fracture, and there are some dimple-like ZrSiO4 sections. The mechanical behavior of the SiO2-based ceramic cores is characterized by thermo-viscoelastic and viscoplastic property. In the high temperature environment of 1400 °C, the main reason for the viscous slip of the SiO2-based ceramic core samples is that the surface of the fine SiO2 particles is initially melted, which plays a role in lubrication between large particles. The SiO2, which is initially melted at a temperature of 1400 °C, adheres to the surface of the large SiO2 particle. When the temperature drops further to the room temperature, it combines with the large particles to form a unitary body.

10:05-10:25 1220599
Application of combined HIP and high pressure heat treatment for superalloy post processing
Chuanlong Hao, Anders Eklund, James Shipley, Quintus Technology, China

Since the middle of the 20th century, due to the increase of the operating temperature of the aero-engine, high temperature properties of the turbine blade and turbine disk alloys have been further demanded. High temperature properties like fatigue, creep, fracture toughness, damage tolerance and reliability become more and more important. The mechanical properties of superalloys depend strongly upon the state of microstructure, which, in turn, is controlled by the chemical composition and the processing conditions. The volume of strengthening phase like gamma prime, double gamma prime phase etc. of the service alloys has been increasing as alloying continuously increased. Meanwhile, forming problems arise as well as the data scatter increases. Investment casting and additive manufacturing in place of forging becomes a tendency for those critical components. These forming methods may have defects such as Porosity, segregation, shrinkage, and microcracking. Hot Isostatic Pressing (HIP) is a technology that has been around for 60+ years. By using high temperature and high gas pressure, defects like porosity, shrinkage and microcracks will be eliminated. Later developments include rapid cooling and rapid quenching to enable higher productivity and high pressure heat treatment (HPHT). A modern HIP system today has a great flexibility and can cool with up to 4500 °C/min under pressures up to 200 MPa. These capabilities make it possible to heat treat a great array of different materials and alloys. In this presentation, the combination of hot isostatic pressing and high pressure heat treatment with fast quenching in one cycle for the alloy 718 and
ERBO-1 shows defects like porosity, shrinkage has been eliminated, microstructure and mechanical properties improved. In the case of alloy 718, Inconel 718 was fabricated by selective laser melting (SLM) and subsequently subjected to different heat treatments. The one with HIP plus treatment eliminate the porosities and satisfied the properties required by the specification of their corresponding forged and casted parts. In the case of ERBO-1, the application of hot isostatic pressing for heat treatment of the single-crystal Ni-base superalloy ERBO/1. An integrated heat treatment consisted of solutioning and aging in the HIP incorporation of quenching, was successfully applied. It led to smaller γ'-particle sizes and narrower γ-channels compared to the conventionally heat-treated material and almost no porosity. It is thus proved that the combination of hot isostatic pressing plus heat treatment is a good way for post processing for those alloys.

10:30-10:45 Tea Break and Poster Sessions

10:45-11:10 Invited (1232673)
Microstructural evolution of Ni-based K403 alloy during thermal exposure
Jiehua Li, Montanuniversität Leoben, Austria

The microstructure evolution of Ni-based K403 superalloy as a function of thermal exposure temperature and time has been investigated by a correlative characterization method, including scanning electron microscopy, electron backscatter diffraction, transmission electron microscopy, high-angle angular dark-field scanning transmission electron microscopy (HAADF-STEM) imaging and electron energy loss spectroscopy (EELS) as well as atom probe tomography. The as-cast microstructure shows a typical dendritic structure and consists of a γ solid solution, γ' phase, (γ + γ') eutectic phase and a metal carbide (MC) phase. After thermal exposure at 800 °C or 950 °C for up to 200 h, the typical dendritic structure was still observed. However, the MC carbides were partially decomposed and further transformed to the M6C phase and M23C6 phase. Furthermore, the size of γ' was increased from 302.88 ± 20.49 nm to 374.75 ± 29.76 nm (800 °C, 50 h) and 751.73 ± 123.14 nm (950 °C, 50 h), respectively. The morphology of γ' was changed from cubic to triangular or round. A topologically close-packed (TCP) σ phase was observed after thermal exposure at 800 °C for 100 h or 950 °C for 50 h. More interestingly, an in-situ phase transformation of the σ phase to other TCP phase (i.e. P phase) was also observed after thermal exposure at 950 °C for 50 h. The present investigation provides a better understanding on the high temperature performance of the Ni-based K403 superalloy, which is essential to predict the failure and thereby enhance the reliability and service life of the K403 Ni-based superalloy.

11:10-11:30 1235847
Design of Ni-Base Superalloys for Additive Manufacturing
Sammy Tin, Illinois Institute of Technology, USA

Additive manufacturing techniques can now be utilized as innovative tools that provide unlimited design flexibility for the fabrication of geometrically complex metallic structures. For production of Ni-base superalloy components used in advanced gas turbine engines, these techniques may enable transformational design concepts and contribute to the development of ultra-efficient power systems for aerospace propulsion, space exploration and power generation. One of the major challenges associated with additively manufactured Ni-base superalloy components is that the extreme temperature gradients encountered during processing negatively impact the underlying microstructure and mechanical properties of the material. Although the macroscopic shape and chemistry of the additively fabricated part may be identical to the conventionally manufactured part, the resulting properties are usually compromised. In an effort to make Ni-base superalloys more amenable for processing via additive manufacturing, varying levels of benign inoculants that promote heterogeneous grain nucleation were blended into IN718 powder feedstock and used for powder bed selective laser melting (SLM) trials. For nominally identical processing conditions, varying levels of metal, intermetallic and reactive oxide particles comprised of elements common to Inconel 718 were systematically blended with the powder feedstock and used to additively build structures in a directed metal deposition (DMD) system. The effectiveness
of these inoculants on modifying the resulting grain structure of the fabricated structures was evaluated using advanced characterization techniques. Selected inoculants were found to reduce the average grain size and grain size distributions in as-fabricated specimens. The effectiveness of these inoculants on modifying the resulting grain structure during additive manufacturing will be discussed.

**11:30-11:50 1220563**

Microstructure-Mechanical Property Relationship of Additive Layer Manufactured Ni-Based Superalloys

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The aerospace and gas turbine-based power generation industries have become increasingly interested in the potential use of Additive Layer Manufacturing (ALM) methods for the beneficial production of high-temperature components. Ni-based superalloys are being widely used as the small batches of complex components in the above-mentioned industries. Over the past several years, new superalloy processing and fabrication routes have been explored by ALM utilizing laser and electron beam melting technologies. Recently, we have established an international collaboration team to enlighten the scientific community towards profound understanding of Inconel 625 processed by selective laser melting (SLM) method: Politecnico di Torino (Italy) is in charge of SLM fabrication, Institut Clément Ader at IMT Mines Albi (France) equipped with a SLM machine as well is in charge of residual stress measurement, Changwon National University (Korea) is responsible for mechanical/microstructural characterization. Additionally, a collaboration with KITECH is being conducted to develop a prototype vane component by means of the electron beam melting (EBM) method, and an initial study of EBMed Inconel 718 will be discussed. Microstructures and tensile properties were characterized for as-fabricated and heat-treated SLMed Inconel 625 samples. The as-fabricated microstructure of the alloy consisted in columnar grains with a very fine dendritic structure exhibiting high density of tangled dislocations. This highlights the high degree of residual stress developed during SLM processing, characterized by a repetition of rapid heating and cooling. A solution treatment led to recrystallization by which equiaxed grains with bimodal size distribution in their size (average size of 90 and 10 μm) were observed. After aging treatment, very fine (10-30 nm) discoidal γ" phases as well as the discontinuous formation of elongated Cr-rich M23C6 carbides at grain boundaries formed. The tensile properties of SLMed Inconel 625 will be discussed with respect to their typical microstructures. Moreover, cylindrical Inconel 718 samples (diameter 15 mm and length 100 mm) were successfully fabricated by EBM. A relative density higher than 99.9% was obtained under the optimized energy density of 2.0~3.5 kJ/mm². Hatch scanning resulted in <100>-oriented columnar grains strongly aligned along the building direction. The focus offset significantly affected the cooling rate and accordingly the development of the microstructure. Even in the as-fabricated condition, considerable amount of γ" yielded excellent tensile properties, higher than those of fully heat-treated wrought Inconel 718. The microstructure-mechanical property relationship of ALMed superalloys will be discussed with a comparison between SLM and EBM methods. Furthermore, we will address our on-going project to fabricate Rene80 by EBM

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Characterization of high-entropy-TLP joints of a single crystal superalloy:

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When the misorientation angle of base metal was more than 10°, there would be second-phase particles distributed in grain boundaries, and thus resulting in poor mechanical property of joint; moreover, the characteristics of joint did not change with base metal misorientation in this case. In order to reduce the detrimental effect of base metal misorientation on the performance of a nickel-based single crystal TLP joint, the insert metal composition
and bonding process for the purpose of obtaining high-entropy-TLP joints were designed. Microstructural and compositional analyses were performed using a scanning electron microscopy (SEM). Electron back scattering diffraction (EBSD) approach was applied to determine the crystallographic orientation of the joints. The results show that, there are also high-angle grain boundaries and compounds at the joint center. Under the condition of random base metal misorientation, with the newly designed insert metal and bonding process for joining, joints with significantly improved performance can be obtained. The high entropy component reduces the formation of the compound to a certain extent, and significantly weakens the detrimental effect of grain boundary and compounds on joint performance. Mechanical property tests at elevated temperatures have been done to determine the joints’ quality. High temperature creep rupture strength (for 100h at 980℃) could achieve no less than 150Mpa, and tensile strength (at 980K) of the joints could attain 670MPa.