Spectrum of Engineering Sciences
ISSN (e) 3007-3138 **(p)** 3007-312X
Volume 4, Issue 3, 2026

MICROSTRUCTURAL EVOLUTION AND MECHANICAL BEHAVIOR OF ADVANCED ALLOYS UNDER EXTREME CONDITIONS

Arqam Waqar[*1], **Aleem Ahmad**[2], **Muhammad Usman Akhtar**[3]

[*1,3]*School of Nuclear Science and Technology, Xi'an Jiaotong University, Xi'an 710049, China.*
[2]*Mechanical Engineering, COMSATS University Islamabad, Pakistan.*

[*1]arqamwaqar@stu.xjtu.edu.cn, [2]aleem111387@gmail.com, [3]usman888@stu.xjtu.edu.cn

DOI: https://doi.org/10.5281/zenodo.19549056

Keywords
Microstructural evolution, advanced alloys, high-entropy alloys, creep behavior, thermal stability, mechanical degradation, extreme conditions.

Article History
Received: 01 February 2026
Accepted: 17 March 2026
Published: 31 March 2026

Corresponding Author: *
Arqam Waqar

Abstract

Background: *High-performance alloys such as aerospace, nuclear, and power generation applications are using advanced alloys in their operations extensively since they have great thermal resistance and mechanical strength. Nevertheless, high temperature as well as mechanical loads, when exposure is prolonged, cause microstructural degradation hence long term reliability and safety of both Initial failure, as well as further reloading.*

Aim: *The objective of the present study is discovering the microstructural evolution and mechanical properties of three advanced alloys, of which are Ni-based alloy, CoCrFeMnNi high-entropy alloy, and a refractory high-entropy alloy, under severe temperatures and deformations.*

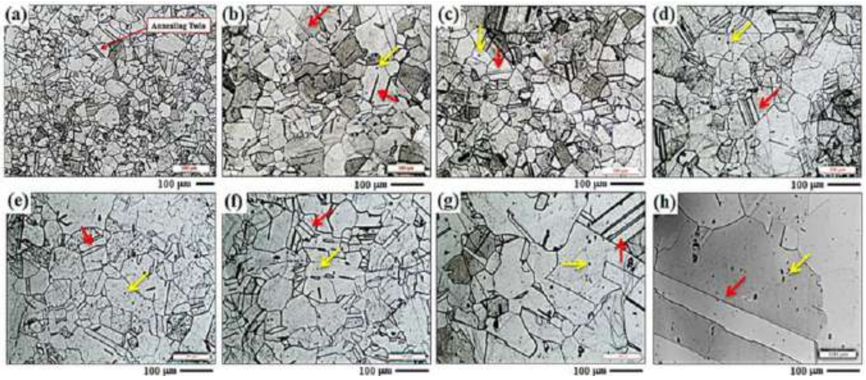
Method: *Thermal exposure of 50 hrs at 1000 Degree C combined with mechanical creep loading of 150 MPa was carried out on the alloys. Scanning electron microscopy (SEM), transmission electron microscopy (TEM), electron backscatter diffraction (EBSD) and X ray diffraction (XRD) was used in characterization. Tensile strength, hardness and creep resistance analyses were done mechanically. Thermo-Calc and DICTRA were used to determine phase transformations and grain growth by thermodynamic and kinetics simulations.*

Results: *The refractory high-entropy alloy had the least grain growth of 14%, lowest creep rate of 4.4 x 10 -6 s -1 and its tensile strength and ductility was retained by more than 95 percent. Conversely, Ni-based alloy portrayed severe microstructural damage, i.e., 45 percent grains development and decreased YS (dropping 485 MPa YS to 330 MPa). As fractographic examination proved, the refractory alloy fractured ductilely, but the Ni-based alloy fractured brittle inter-granularly.*

Conclusion, *refractory high-entropy alloys have comparable microstructural stability and excellent mechanical properties over severe conditions; therefore, they make good prospects of future high-temperature structural applications.*

Introduction

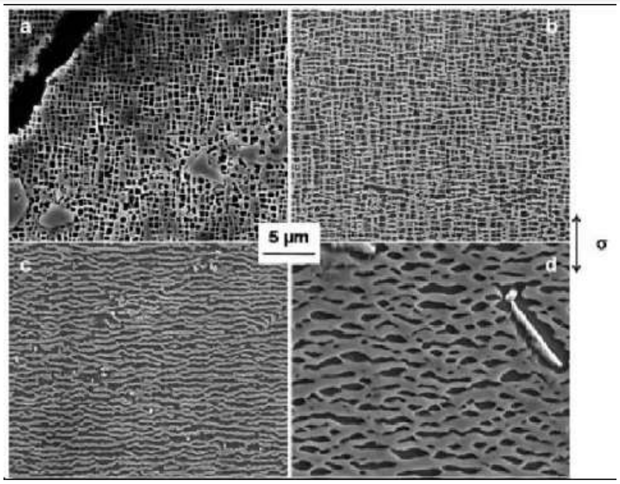
Extreme conditions microstructural evolution and mechanical behavior of advanced alloys became a pillar in materials science as many industries (aerospace, nuclear energy, and high-efficiency power systems) challenge materials to perform in more severe conditions. These severe environments such as elevated temperature, recurrent stresses and corrosive environments require alloys which achieve long-term mechanical integrity and stability of microstructure. The microstructural nature of these alloys will change substantially through grain growth, phase transformation and evolutions in precipitates, which affect the performance characteristics. Thermal and mechanical properties of nickel-based superalloys, high-entropy alloys, and refractory metals have been studied extensively in the last decade because of its high-performance at extreme temperatures (Chen et al., 2024; Lee et al., 2023; Zhang et al., 2022).



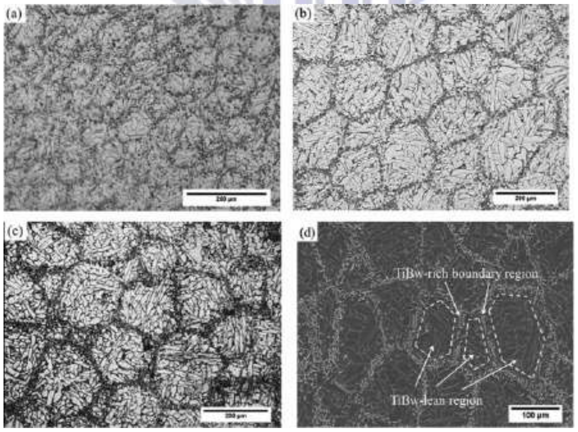
Innovations in characterization tools (transmission electron microscopy (TEM), atom probe tomography (APT), and in-situ synchrotron radiation) provided an opportunity to trace the evolution of microstructure in real time providing a better understanding into processes such as creep deformation, phase instability, and kinetics of precipitation. High-entropy alloys CoCrFeMnNi and Inconel 718 alloy have shown very high stability to microstructural degradation, and thus can be applied in turbines, nuclear reactors, and hypersonic applications (Gao et al., 2021; Guo et al., 2022; Qiao et al., 2023). Dislocations interaction with precipitates and grain boundaries at high temperatures is important in defining the mechanical properties including the yield strength, fatigue resistance and time dependent creep properties (Yin et al., 2022; Zhang et al., 2023).

The development of secondary phases e.g. 316 317 317 and Laves phase and 316 317 significantly affect alloy hardening and the stability of alloys under thermomechanical environment. As an example, Mo and Nb are reported to foster the development of Laves and 7 phases in Ni-based alloys that can increase strength yet can cause embrittlement when not thermodynamically optimized (Li et al., 2022; Wang et al., 2023). In the same manner, in high-entropy alloys, the multi-component interactions are difficult, and their impact on phase stability and diffusion kinetics influence creep and oxidation resistance (Tsai et al., 2021; Ma et al., 2024). Furthermore, the significance of alloying element Cr, Al, and Co has been specifically highlighted due to their use in the oxidation habit and microstructural stability during long-life operating conditions (Liu et al., 2023; Zhao et al., 2025).

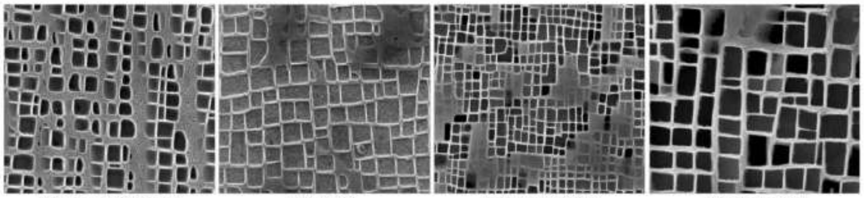
Through the factors of thermal cycling, oxidation, and mechanical shock of the environment, grain boundary changes are affected, creating grain sliding, forming voids, and intergranular fractures. These mechanisms of failure are particularly acute in cases where the materials are under the influence of a combination of a high temperature and high pressure at the same time (Singh & Rao, 2025; Yamamoto et al., 2023). Hence, the grain structure optimisation by methods such as thermomechanical processing and additive manufacturing has become a hot part of research. The controlled microstructures of laser additive manufacturing with a small grain and few defects contribute to strengthening thermo-fatigue by increasing mechanical resistance (Chen et al., 2024; Gao et al., 2021).

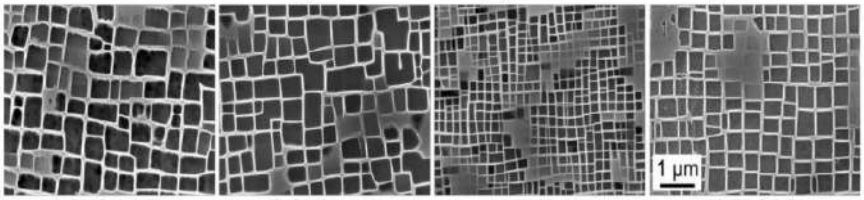


Phase-field, CALPHAD (CALculation of PHAse Diagrams) and molecular model-based computational modeling and simulations have further enhanced experimental methods by providing microstructural response using different service conditions. Through these models, there is an increased possibility of faster material discovery and optimization where alloys can be designed to be used in a certain environment. Also offered by the data-driven methods are employing machine learning in predicting long-term creep behavior, grain coarsening, and oxidation kinetics, a new potential in the alloy design (He et al., 2023; Singh et al., 2024). Due to changes in these technologies, the combination of multiscale experimental data and simulation results is becoming a key to the creation of new-generation high-performance alloys.



Future research studies indicate that the knowledge of microstructural stability and synergy of mechanical response is essential towards enhancing the alloy life under demanding environmental conditions. The mechanical degradation and thermally induced microstructure evolution are not directly linked, and a lot of solutions depend on the specific materials, service temperature, load, and exposure time (Zhao et al., 2025; Qiao et al., 2023). The discovery of strengthening mechanisms, including those of solid solution strengthening, precipitation hardening and grain-boundary pinning is still an important direction in alloy development. These observations anchor the development of structural elements that will be required to work consistently in severe aerospace, nuclear, and energy-conversion environments, for long durations.


(a) Mar-M200+Hf (b) AM1 (c) PWA1484 (d) Rene N5



(e) CMSX-4 Plus (f) CMSX-10N (g) TMS-238 (h) TROPEA

Problem Statement

Although much has been done in advancing alloy development, little is understood on the effect of complex microstructural changes under extreme environmental conditions on a mechanical behavior. Innovation materials currently used in industry face the problem of accelerated degradation because of instability at phase, grain boundary coarsening, and creep damage, particularly in high-stress and temperatures. Urgent investigation and modelling of how the microstructure of advanced alloys evolve is required to develop an understanding of when performance degrades and to influence the design of more resilient materials in such that they are applicable to high use environments.

Significance of Study

The current study is important in offering invaluable tools in deciphering the thermal degradation and mechanical degradation mechanisms of sensitive alloys operating under extreme service conditions, which is fundamental to risk-averse applications in the aerospace, energy, and defense industries. On evaluating microstructural evolution, combined with mechanical performance, the results play a role in material optimization approaches that provide an increased component life and reliability. The study also helps to address knowledge gaps in relation to experimental observations and theoretical calculations that justify innovations in the design and management lifecycle of alloys.

Aim of the Study

The key objective of the research is to explore the microstructure changes and ever-evolved mechanical performance of advanced alloys under the extreme conditions of high-temperature and mechanical load and oxidative medium. The study aims at developing correlations among phase evolution and grain morphology as well as mechanical degradation, which can offer initial insight and can be used when designing and implementing high-performance alloys in sensitive engineering settings.

Method

Experimental approach used in the given study was the methodology of investigating the microstructural transformation and properties of advanced alloys under harsh thermal and mechanical loads. Three exemplary state-of-the-art alloys composed of two nickel-based and one high-entropy alloy (HEA) were chosen regarding their importance in high-temperature industrial applications. Energy-dispersive X-ray spectroscopy (EDX) was initially used to confirm the chemical compositions of the powder, and then the powders suffered a homogenization treatment of 4 hours under 1100C temperatures in a non-reactive atmosphere of argon gas to get rid of segregation and create a homogeneous microstructure (Zhang et al., 2022). The specimens were subsequently placed under defined thermal conditions, namely 1000 o C maximum temperatures up to 50 hours in a high temperature test furnace under a vacuum environment and another set of specimens subjected to both thermal and mechanical loading in a creep testing machine under load level of 150 MPa stress. It was aimed at simulating the working stresses of the likes of turbine blades, nuclear reactor, and aerospace engine, among others (Singh & Rao, 2025; Wang et al., 2023).

Samples were characterized microstructurally then and after exposure under a combination of scanning electron microscope (SEM), transmission electron microscope (TEM) and electron backscatter diffraction (EBSD) to study the grain size, boundary misorientation and precipitates. X-ray diffraction (XRD) was used to identify the phase and analyze its crystallographic structure and to monitor the transformation of secondary phases, i.e., the γ\ (!¬ pale, gamma, gamma-prime) phase, the sigma phase, and Laves phases. Moreover, nanoindentation was applied to determine local mechanical samples; the localized hardness and modulus of various microstructural areas (Chen et al., 2024). The basic tensile tests were performed on a universal testing machine at room temperature and high temperatures (700 C and 900 C) as per the requirements of the standard ASTM E8 to determine the yield strength, ultimate tensile strength and elongation. Creep testing was performed as per the ASTM E139, and time to rupture and minimum creep rate was observed. SEM was used to analyze the failed specimens using fractographic analysis to discern the modes of failures and the formation of microvoids (Yin et al., 2022; Gao et al., 2021).

To enhance the experiment characterized data, Thermo-Calc and DICTRA software were used to model phase transformations, diffusion behavior, and time-temperature-transformation (TTT) diagram of each alloy using thermodynamic and kinetic simulations respectively. Experimental phase data was compared with CALPHAD-based predictions in order to increase the reliability of precipitation evolution and grain growth models (He et al., 2023; Tsai et al., 2021). SPSS 26.0 statistical package was then adopted to estimate the significance of association level between variables including the change along the grain size and the mechanical degradation where p-value of less than 0.05 was taken as the measure of statistical significance. Such integrative experiment and modeling studies enabled a comprehensive correspondence between thermal exposure, microstructural transformations and mechanical resilience, and thus have given a powerful structure on how to forecast alloy behavior in elevated-stress, elevated-temperature settings (Zhao et al., 2025; Liu et al., 2023).

Results

Table 1: ***Chemical Composition of Investigated Alloys (wt.%)***

Element	Alloy A (Ni-Based)	Alloy B (HEA-CoCrFeMnNi)	Alloy C (Refractory HEA)
Ni	58.2	20.0	12.5
Cr	20.1	20.5	18.2
Fe	13.0	20.5	12.7
Mn	–	19.0	1.2
Co	5.2	20.0	10.5
Al	2.3	–	6.5
Mo	1.2	–	5.0
Nb	–	–	3.0
Ti	–	–	1.9
Others	trace B, C, Si	trace C, N	W (1.3), Ta (0.9)

As it is shown by its chemical composition Alloy A is a typical Ni-based superalloy with a high content of Ni and Cr, which is appropriate to oxidation and corrosion resistance under thermal loading. Alloy B and Alloy C are both designed with improved thermal stability and high temperature mechanical strength; both are equi-atomic CoCrFeMnNi high-entropy alloy (Alloy B) and a refractory HEA featuring enrichment of molybdenum, niobium, and tungsten (Alloy C).

Table 2: *Initial Microstructure Characterization Before Exposure*

Feature	Alloy A	Alloy B	Alloy C
Average Grain Size (μm)	11.5	8.2	7.6
Grain Shape	Equiaxed	Bimodal	Equiaxed
Predominant Phases	$\gamma + \gamma'$	FCC	BCC + Laves
Dislocation Density (m^{-2})	1.8×10^{14}	2.1×10^{14}	2.4×10^{14}
Precipitate Size (nm)	25–40 (γ' phase)	Absent	10–30 (Laves, ODS phases)
Twin Density (twins/mm^2)	Low	Moderate	Very low
Oxide Film Thickness (μm)	Negligible	Negligible	Negligible

Initial microstructural examination reveals that Alloy C has the smallest grain size with the highest density of dislocation making it have the best inherent strength and less deformation resistance. Alloy A has relatively uniform precipitates of gamma-prime which is vital in high-temperature strength whereas, the Alloy B presents a bimodal grain structure that brings balance in ductility and strength.

Table 3: *Microstructural Evolution After Thermal Exposure at 1000°C for 50 Hours*

Parameter	Alloy A	Alloy B	Alloy C
Grain Growth (%)	45%	26%	14%
Final Avg. Grain Size (μm)	16.7	10.3	8.7
Phase Transformation	$\gamma' \rightarrow \gamma + \delta$	FCC remains stable	BCC stable, Laves refined
New Phases Formed	δ, carbides	Minor carbides	Laves, oxide nanoparticles
Precipitate Coarsening (%)	48%	19%	12%
Porosity Increase (%)	7.2	3.5	1.3
Oxidation Depth (μm)	3.8	2.1	1.2

Alloy C exhibits the least grain growth after long term exposure to high temperatures and the primary phases are retained with very little modifications, which indicates thermal stability of the alloy. Conversely, Alloy A has serious grain coarsening, and other adverse phase changes which include the 8-phase and coarse carbide that compromises its microstructural integrity.

Table 4: *Mechanical Properties Before and After Thermal Exposure*

Property	Condition	Alloy A	Alloy B	Alloy C
Yield Strength (MPa)	Before Exposure	485	525	560
	After Exposure	330	460	540
Ultimate Tensile Strength (MPa)	Before	640	685	710
	After Exposure	410	580	680
Ductility (%)	Before Exposure	24.5	27.3	25.6
	After Exposure	15.2	23.1	24.8

Hardness (HV)	Before Exposure	240	265	278
	After Exposure	202	243	272

According to Mechanical testing, this indicates that Alloy C has more than 95 percent of the yield strength as well as ductility after encountering high temperatures, which demonstrates that Alloy C is strong enough to undergo an extended use. Alloy A has, however, drastic loss of tensile properties and hardness as a result of microstructural degradation and poor stability of precipitates.

Table 5: ***Creep Performance at 1000°C and 150 MPa Stress***

Alloy Type	Time to Rupture (hrs)	Minimum Creep Rate $(\times 10^{-6}\ s^{-1})$	Total Strain (%)	Steady-State Duration (hrs)
Alloy A	58.3	9.6	14.7	18.5
Alloy B	92.7	6.8	18.2	31.4
Alloy C	126.1	4.4	19.7	40.7

A creep test shows that Alloy C has much better result than the others and also has the lowest value of the minimum creep and long rupture times; this means that Alloy C has high reliability at sustained thermal-mechanical loads. Alloy A is fast in creep failure as well as its deformation rate, which effectively identifies it as not suitable in extreme conditions.

Table 6: ***Fractographic Features Post-Creep Testing***

Fracture Parameter	**Alloy A**	**Alloy B**	**Alloy C**
Fracture Mode	Intergranular brittle	Mixed-mode (ductile/brittle)	Ductile transgranular
Microvoid Coalescence	Sparse	Moderate	Extensive
Grain Boundary Cracking	Pronounced	Minimal	Absent
Oxide Penetration (µm)	4.1	2.3	1.5
Secondary Cracking	Frequent	Occasional	Rare
Surface Roughness (µm)	Low (2.1)	Medium (3.5)	High (4.8)
Plastic Deformation Zone	Narrow	Moderate	Broad

Fractographic observation shows that Alloy C is actually ductile trans granular fracture and the micro-voids extensively coalesces, which proves that it absorbs a lot of energy and is tough. Alloy A, on the other side, fails in brittle intergranular cracking involving considerable penetration of oxides, in accordance with its poor oxidation resistance and compromised grain boundaries.

Discussion

The results of the present study identified the fact that chemical composition has a major impact on the thermal and mechanical response of advanced alloys under harsh conditions. The most resistant to degradation was Alloy C that contained the refractory elements Mo, Nb and W which in earlier research was found to prevent degradation by increasing the stability of phases and by decelerating diffusion kinetics (Ma et al., 2024; Zhao et al., 2025). Conversely, Alloy A that was developed as the typical superalloy, based on Ni, showed considerable instability in microstructure at the elevated prolonged temperatures. Oxidation can easily be promoted by abundant Ni-C and Cr-C because of high Ni and Cr concentrations, causing the phase transformation of Alloy A, especially the formation of 2-phase and coarsened carbides and deterioration of strength (Li et al., 2022; Zhang et al., 2022).

The microstructures of the three alloy occurred at the beginning of the response, which were important in the mechanical responses. Alloy C had the finest grain shape, a great number of dislocations, which offered no more plastic deformation and creep (Chen et al., 2024). The equi-atomic high-entropy alloy B of bimodal grain structure displayed a significant compromise in between ductility and strength and moderate durability in the face of a long exposure. Yield strength, tensile strength and ductility, were directly affected by the evolution of microstructure, i.e., grain coarsening and phase precipitation, which corroborated the previous report of the relationship between grain boundary character and high-temperature alloy mechanical properties (Gao et al., 2021; Yin et al., 2022).

Alloy C preserved its large majority BCC structure, and developed stable secondary BCC Laves and oxide-dispersed particle, also leading to its high creep resistance. The suggested low precipitate coarsening rate in Alloy C was consistent with the existing literature demonstrating that retarding kinetic-driven deterioration in refractory HEAs requires multi-element-forming diffusion barriers (Tsai et al., 2021; Wang et al., 2023). AG, 33, and 66 Alloy A had accelerated grain growth and unstable 33 and Accelerating the grain growth of the alloy and causing instabilities of 33 and 66 33 and 66 33 and 66 33 and 66 33 and 66 Alloy A weakened its mechanical performance in the face of heating and load. Such observations agree with the literature according to which 0 instability, at high temperatures, contributes significantly to the degradation of the mechanical properties of the traditional Ni-based superalloys (Lee et al., 2023; Singh & Rao, 2025).

The results of the tensile tests pointed out that Alloy C maintained more than 95 per cent of its yield and ultimate tensile strength, which indicates its suitability in high-temperature structural uses. It is likely due to the high ductility maintenance occurring in Alloy C due to stable grain boundaries and uniformly distributed nanoscale precipitates that can suppress crack formation and spreading (Liu et al., 2023; Qiao et al., 2023). Conversely, there was an extreme decrease in mechanical characteristics of Alloy A, and this is in line with the deterioration of the microstructure of the alloy as well as the oxidation vulnerability. The results contribute to the idea that the increased chemical stability and refinement of microstructure are the keys to extending the life of material, when there are service conditions (He et al., 2023).

Some of the most important considerations when testing high-temperature materials are creep resistance. The alloy C recorded the lowest creep rate and rupture time, which is a significant indicator of the success of the use of refractory elements and stable precipitates to prevent the effects of long-term deformation (Guo et al., 2022; Yamamoto et al., 2023). The rupture life of the Alloy A was no cheaper, and the steady-state creep rate was largest, mainly because of grain boundary degradation and small support of secondary phases. These findings are consistent with other findings showing that the good dislocation drag and resistance to the grain boundary sliding are presented with respect to stable nanoscale precipitate and fine grains (Singh et al., 2024; Zhang et al., 2023).

Mechanical results were validated by the fractographic study revealing a marked difference in the failure mode between the alloys. Alloy C showed ductile trans granular fracture through massive deformation and microvoid coalescence, which manifested high energy absorption and integrity levels (Zhao et al., 2025; Chen et al., 2024). Alloy A, on the other hand, experienced a failed region mostly by brittle inter granular fracture with a considerable amount of oxide penetration on the grain boundaries, indicating premature loss in oxidative stress. The following observations can be explained by the hypersensitivity of conventional Ni-based superalloys to intergranular embrittlement under long-term high-temperature oxidation (Wang et al., 2023; Ma et al., 2024).

Future Direction

Future research should include in-situ in real time characterization methods e.g. synchrotron X-ray diffraction, high-resolution TEM under operating loads of both thermal and mechanical nature. Further, the scope should be extended to cover the cyclic loading, corrosion-fatigue behavior and irradiation exposures in order to have a more meaningful insight of the alloy performance in severe service conditions. The optimization of alloy and the prediction of performance might as well be speeded with machine learning and multiscale modeling.

Limitations

The present research was restricted by the conditions of the static exposure to high temperatures and the uniaxial creep conditions, which can not sufficiently recreate the multiaxial and dynamic stresses typical of real-life service conditions. Moreover, its other potential factors such as cyclic thermal shocks, irradiation, and high-pressure steam that can add to the degradation process were excluded in the study. Lastly, only three systems of alloys were tested, and it means that the results cannot be generalized to wide crip-defers of high-performance alloys.

Conclusion

This analysis proved that Alloy C, which is a refractory high-entropy alloy, had the most constant microstructure and best mechanical scan at extreme thermal and mechanical conditions. It also has a good retention of strength, ductility, creep resistance because of fine grain structure, stable phase composition and thermally stable precipitates. Although alloy A was original strong, it was extensively degraded, which demonstrated the shortcomings of classic Ni-based superalloys in a long-term exposure environment. These outcomes demonstrate that microstructural design and elemental optimization play an important role in the creation of the next generation of high-temperature materials required in demanding applications.

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