

An overview of the microstructure and mechanical behaviour of nickel-based superalloys

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ABSTRACT

In modern gas-turbine engines, high temperatures and pressures, in addition to excessively lofty rotational speeds, are essential requisites as they play a determining role in engine efficiencies. However, high temperatures are known to significantly affect material mechanical properties, and tend to promote oxidation. An ideal material for this application, therefore, is not only expected to be fatigue-/creep-resistant but also to exhibit high mechanical-property stability against temperature changes and oxidation. This is because, typical operating conditions constitute mainly of excessively high thermal stresses, tempestuous mechanical stresses in prohibitively corrosive exhaust gases. Nickel-based superalloys, one of the materials widely employed for this purpose, are a unique class of materials, designed for the manufacture of components that form a backbone of gas turbine-engines. In addition to the significant influence microstructural features have on their mechanical behaviour, the synergetic influences between the characteristics of the operating conditions tend to be enormous. This paper presents an overview of the role microstructure plays on the mechanical behaviour of nickel-based superalloys. Differences in the chemical composition, in addition to the roles played by various elements, are discussed, including relevant, essential and important characteristics of the resulting nano- and micro-scale features. Mechanical behaviour under fatigue, creep and creep-fatigue loading conditions is investigated, particularly, how microstructure, temperature and loading regimes, affect fatigue, creep and creep-fatigue behaviour.

Keywords: Nickel-based superalloys; composition; processing; microstructure; deformation.

Una descripción general de la microestructura y el comportamiento mecánico de las superaleaciones a base de níquel

RESUMEN

En los motores de turbina de gas modernos, las altas temperaturas y presiones, además de velocidades de rotación excesivamente altas, son requisitos esenciales, ya que juegan un papel determinante en la eficiencia del motor. Sin embargo, se sabe que las altas temperaturas afectan significativamente las propiedades mecánicas del material y tienden a promover la oxidación. Por lo tanto, no solo se espera que un material ideal para esta aplicación sea resistente a la fatiga/deformación por fluencia (o “creep”), sino que también muestre una alta estabilidad de propiedades mecánicas contra los cambios de temperatura y la oxidación. Esto se debe a que las condiciones de funcionamiento típicas consisten principalmente en tensiones térmicas excesivamente altas, tensiones mecánicas tempestuosas en gases de escape prohibitivamente corrosivos. Las superaleaciones a base de níquel, uno de los materiales ampliamente empleados para este propósito, son una clase única de materiales, diseñados para la fabricación de componentes que forman la columna vertebral de los motores de turbina de gas. Además de la influencia significativa que tienen las características microestructurales en su comportamiento mecánico, las influencias sinérgicas entre las características de las condiciones de operación tienden a ser enormes. Este artículo presenta una descripción general del papel que juega la microestructura en el comportamiento mecánico de las superaleaciones a base de níquel. Se analizan las diferencias en la composición química, además de las funciones que desempeñan varios elementos, incluidas las características relevantes, esenciales e importantes de los aspectos resultantes a escala nano y micro. Se investiga el comportamiento mecánico bajo condiciones de carga de fatiga, fluencia y fatiga por fluencia, particularmente, cómo la microestructura, la temperatura y los regímenes de carga afectan el comportamiento de fatiga, fluencia y fatiga por fluencia.

Palabras clave: superaleaciones a base de níquel, composición, proceso, microestructura, deformación.

INTRODUCTION

Nickel-based superalloys are a unique class of materials, with superior mechanical properties at high temperature, designed for the manufacture of components (such as

turbine blades, guide vanes and discs) that form the backbone of gas aero-engines and other land-based turbine-engines. Typical operating conditions, for these components, constitute of excessive thermal stresses

(from high temperatures) and tempestuous mechanical stresses (from high rotation speeds) in extremely corrosive exhaust gases. As such, overall behaviour of these components is substantially affected by the synergy between high temperatures, corrosive environments and mechanical loadings, prevalent in turbine-engines [1, 2].

In modern gas-turbine engines (see figure 1a), these high operation temperatures and pressure ratios, in addition to the high rotational speeds, are essential, as they play a determining role in turbine-engine efficiency. However, high temperatures, in the range of 500°C to 1100°C, are known to significantly affect mechanical properties [2, 3] and tend to aggravate material oxidation [4]. An ideal material for this application therefore, is not only expected to be fatigue-/creep-resistant but also to exhibit high mechanical-property stability against oxidation. Additionally, the material needs to possess an inherently high surface continuity free from micro/nano-cracks and pores. Furthermore, other requirements include low tendencies to crack nucleation, along with high resistance to crack propagation. As these properties were not intrinsic in any pure metal, alloying was necessary. However, although alloying culminates in desirable properties, the large number of elements, in modern alloys, lead to property variations. Therefore, several elements are normally considered to achieve desired properties. Usually, characteristics of the intended application, such as loading conditions and the anticipated environmental conditions tend to influence this. Due to these harsh conditions in the intended applications, special types of alloys called superalloys are considered. Nickel-based superalloys, reviewed in this paper, are a type of superalloys with nickel as the major constituent. In these superalloys, chemical complexity (from the several elements added), diversity in the manufacturing and heat-treatment processes lead to behavioural specificity [5-9]. This often results from the variations in the resulting characteristic structures at micro- and nano-scales. Since different sections of turbine-engines tend to have different

levels of temperatures and stresses (figure 1a and b), different types of alloys, in terms of characteristic composition, are considered.

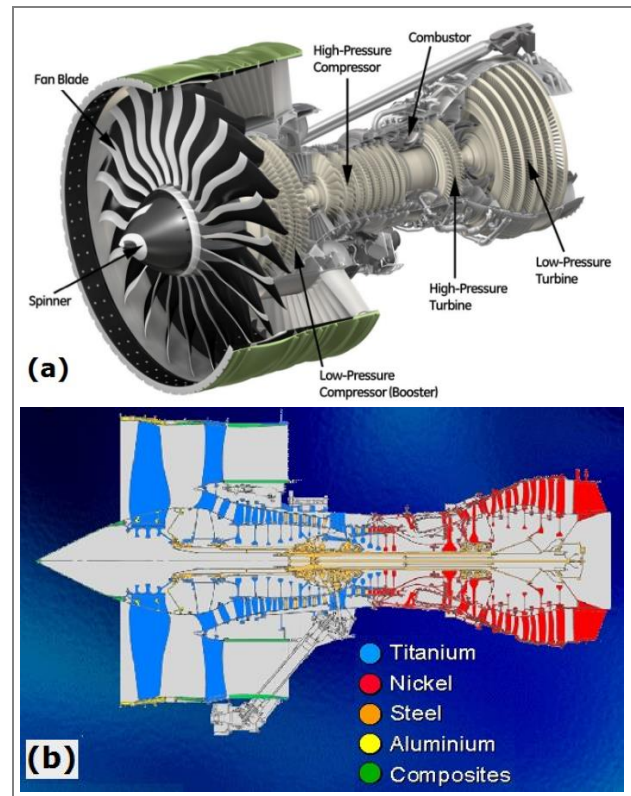


Fig. 1. Turbine engine: (a) parts of the turbine engine [10], (b) materials applied in the various parts of the turbine engine [11].

This paper presents a review of the mechanical behaviour of nickel-based superalloys. Firstly (Section 1: “Microstructural Features”), differences in the chemical composition, in addition to the roles played by the various elements, are discussed, including relevant and essential characteristics (and their importance) of the resulting nano- and micro-scale features. In the second section (Section 2: “Fatigue Behaviour”), cyclic deformation (fatigue) behaviour of nickel-based superalloys under various loading conditions is reviewed. In the third section (Section 3: “Creep Behaviour”), creep behaviour is studied. In both sections 2 and 3, influences of microstructure, temperature and loading are investigated. The paper ends with a review of the interaction between

creep and fatigue with particular emphasis on the influences of microstructure and temperature (Section 4: “Creep-Fatigue Behaviour”).

Microstructural Features.

For nickel-based superalloys to withstand high static/cyclic loads, exhibit high resistance to oxidation and crack initiation/propagation, a variety of elements are alloyed into nickel, resulting in variations in microstructure, mechanical and environmental properties [5, 7, 9].

Composition.

In its pure form, nickel is a silver-white transition metal/element with a relative atomic mass, based on reference to $^{12}\text{C}=12$, of 58.69, an atomic number of 28, a melting point typically of about 1,453°C and an average density of about 8.90 g/cm³ at room temperature, reducing to about 7.90 g/cm³ at melting point. It is ductile and tough and has an fcc crystal structure, a solid-solution austenite (γ), in which dispersoid and precipitate particles can form. The fcc matrix has the capability to dissolve extensive amounts of elements like Cr, Mo, W, Al, Ti and Ta, resulting in solution-hardening, improved corrosion and oxidation resistance [12].

Overall, several elements, at times exceeding 10, are alloyed into nickel. Constituents of nickel-based superalloys are commonly divided into three classes. First is a class comprising transition metals/elements such as Ni itself, Co, Fe, Cr and Mn. High temperature capabilities of nickel-based superalloys are enhanced by the second class of (heavy) metals such as W, Ta, Mo, Nb, Hf and Re. The third class is comprised of elements that impart high strength and improve other properties, such as oxidation-resistance which result from the formation of protective oxide layers. Elements, such as Al and Ti, result in the formation of secondary phases such as γ' -precipitates [13].

Group VI and VII elements, such as Cr, Mo, W, Fe, are referred to as *gamma (γ)-phase formers*. They form an

austenitic face-centred cubic (fcc) continuous phase known as *matrix* [14], which contains a higher percentage of solid-solution of elements, Mo, W, Co and Fe. On the other hand, group III, IV and V elements (like Al, Ti, Nb, Ta, Hf) are known as *gamma-prime (γ')-phase formers* [15]. This is a primary strengthening phase in nickel-based superalloys and is referred to as *precipitate* [2, 14, 16]. They have a highly unusual characteristic of becoming stronger and more creep-resistant with increasing temperature, a trait attributed to the relief of coherency stresses at the γ - γ' interface as dislocation accumulates. Furthermore, they act as barriers to dislocation motion [17]. Since the planes of their crystals are in accordance with those of the matrix, precipitates are coherent and have an ordered L1₂ intermetallic crystal structure.

In certain superalloys containing Nb, strengthening is by the *gamma double-prime (γ'') phase*, which is Ni₃Nb and is based on the D0₂₂ structure [17-20]. Formation of this phase occurs in a solid state as the supersaturated solid solution of γ -phase is cooled below its equilibrium solvus temperature, forming a basis for additional principal strengthening [21].

Carbon (C) when added, in quantities of 0.05-0.2%, combines with reactive and refractory elements such as Ti, Ta and Hf to form carbides such as TiC, TaC or HfC with fcc crystal structures. When subjected to operating conditions, these carbides normally react with each other and decompose to form other carbides such as M₂₃C₆ {e.g. (Cr, Mo, Fe)₂₃C₆}, M₆C and the rare M₇C₃ along grain boundaries. Ultimately, these carbides increase grain-boundary rupture strength at high temperatures. Generally, in conjunction with boron (B), C is added to enhance grain-boundary strength which further improves other mechanical properties. Examples of borides include M₅B₃ and M₃B₂. [2, 22].

Cobalt (Co) with a melting point of about 1640°C is a hexagonal close packed (hcp) metallic element. When added it improves high temperature capabilities apart from

strengthening of the matrix. Particularly, creep resistance is enhanced by making cross slip of dislocations more difficult [23], a critical attribute for high-temperature

applications [24]. Table 1 is a summary of the effects of the various alloying elements in common applications [2, 17, 19, 25].

Table 1. Summary of roles of alloying elements in nickel-base alloys.

Elements	Effect on superalloy
Co, Cr, Fe, Mo, Ta	Solid-solution strengtheners: γ (Ni_3Cr)
W, Ta, Ti, Mo, Nb	MC - type carbide stabilizer
Cr	M_7C_3 - type carbide stabilizer
Cr, Mo, W	M_{23}C_7 - type carbide stabilizer
Mo, W	M_6C - type carbide stabilizer
C, N	$\text{M}(\text{CN})$ - type carbide stabilizer
Al, Ti, Ta	Solid-solution strengtheners and stabilizes for γ' (Ni_3Al)
Co	Raises solvus temperature of γ'
Nb	Stabilizes γ'' (Ni_3Nb)
Al, Cr	Improves oxidation resistance
B, Zr, C	Grain-boundary strengthener
Cr	Improves sulphidation resistance

Macrostructure.

Various casting and solidifications techniques result in different microstructures, which are the single crystal, directionally solidified (with columnar grains) and traditional polycrystalline (with randomly sized and oriented grains), see figure 2 [25, 26].

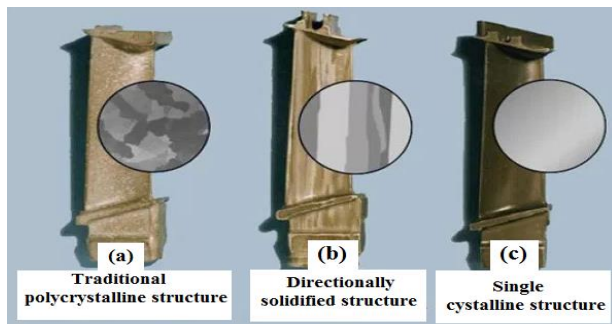


Fig. 2. Structure of a: (a) traditional polycrystalline (with randomly sized and oriented grains), (b) directionally solidified (with columnar grains), and (c) single crystalline nickel-based superalloys [25].

Generally, based on levels of temperature, gas turbine-engines are divided into three sections (see figure 1). In the high-pressure section, conditions are harsher than anywhere else. Due to their superior strength and oxidation-resistance, single crystalline materials are normally applied in the manufacture of components

(blades) for this section.

Directionally solidified alloys tend to have moderate strengths (as compared to single crystalline and polycrystalline) and are, hence, applied in the intermediate and low-pressure sections, where conditions are less harsh. Due to the presence of randomly sized and orientated grains, traditional polycrystalline alloys tend to be the weakest and, therefore, are used in the manufacture of discs which operate in the much less harsh environments.

Columnar grain structures and textures, obtained from directional-solidification casting processes, are known to significantly affect properties [2, 27-29] such as fatigue behaviour, although deviations due to inefficiencies of the solidifications/cooling processes [30, 31] tend to be detrimental [32]. Evidence from both experimental and modelling results [32], have demonstrated that columnar grain textures lead to orientation-dependent behaviour. Results revealed considerable reduced fatigue-life in specimens having grain boundaries transverse to the loading axis.

In comparison with specimens having grain boundaries aligned with loading axes, authors were of the view that

transverse grain boundaries acted as crack initialization sites, thereby leading to reduced fatigue-life. Similar behaviour is also observed in polycrystalline materials, where grain sizes and textures are determined by the solvus heat treatment temperature applied. Heat treatment under super-solvus temperature conditions lead to coarse grain-structures while subsolvus heat treatment conditions result in fine-grained structures [1, 33, 34].

Microstructure.

The presence, at microstructure level, of the $L1_2$ ordered γ' -precipitates, is known to lead to exceptional mechanical properties in nickel-based superalloys. These phases are embedded in the fcc γ -matrix phase. Hence, the duo-phase structure (γ/γ'), as shown in figure 3, enhances and accounts for the high temperature mechanical properties [35].

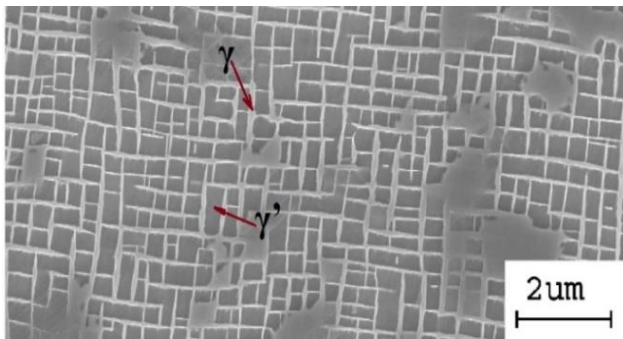


Fig. 3. SEM micrograph for the microstructure of a typical nickel-based superalloy having cuboidal morphology of γ' -precipitates [2].

According to Pollock and Tin [2], cooling rates in the range of $\sim 40^\circ\text{C}/\text{min}$ produce γ' -precipitate sizes in the range of 300-500nm, while slower rates result in a mixture of sizes from as small as <50 nm to as large as >500 nm.

These two phases (γ and γ') are typically solid-solution strengthened by refractory elements Co, Fe, Mo, W and Ta [27, 36], with improved oxidation-resistance and reduced γ/γ' mismatch [25, 35, 37]. Sizes and morphologies of the strengthening γ' -precipitates are normally controlled through varied manufacturing and heat-treatment processes.

In cast polycrystalline nickel base superalloys,

directionally solidified, carbon content ranges between 0.05 and 0.20% by weight. After the casting procedure, microstructure is normally dendritic in nature. The black spots in the regions between the dendritic structures, in figure 4a, are the primary MC carbides, where M stands for metals such as Cr, Mo, Ti, Ta or Hf but predominantly Ti [38, 39]. During heat treatment, however, γ' -precipitates gradually coarsen while primary carbides gradually degenerate into the form $M_{23}C_6$, according to the equation $MC + \gamma \rightarrow M_{23}C_6 + \gamma'$ and distribute along grain boundaries (see figure 4b and c).

In the secondary carbides, M is predominantly rich in Cr, Ti and Ta [26, 40, 39]. This was further confirmed by He et al. [41], who analysed both intra-grain and inter-grain (grain boundary) carbides using electron dispersion X-ray spectroscopy (EDS) analysis.

As shown by scanning electron microscopy (SEM), micrographs along with EDS spectra in figures 4d and 4e, the study established that carbides were mostly rich in Ti, Ta, C and W.

Ordinarily, other carbides such as M_7C_3 and M_6C are intermediately formed before the final formation of $M_{23}C_6$ [42]. On the other hand, the γ' -precipitate volume fraction increases significantly, due to the precipitation out of primary carbides (according to the equation $MC + \gamma \rightarrow M_{23}C_6 + \gamma'$).

Generally, most of the properties exhibited by nickel-based superalloys are a function of their chemical composition, in addition to the manufacturing route employed. The presence of several elements results in densities to vary between 7.50 and 9.00 g/cm^3 . Specific heat capacities increase with increasing temperature while melting points range between 1320°C and 1490°C . Thermal expansion mostly ranges between $8 \times 10^{-6}/^\circ\text{C}$ and $18 \times 10^{-6}/^\circ\text{C}$, while thermal conductivity doubles as temperature increases from room temperature to about 800°C .

Modulus of elasticity measurements conducted at various temperatures indicated a tendency to diminish with

increasing temperature, and typically dropped from 210 GPa at room temperature to about 160 GPa at 800°C [2, 21]. Usually, microstructure tends to significantly influence mechanical properties of nickel-based superalloys [43-46].

Fatigue Behaviour.

Fatigue loading has been cited as one of the main causes

of failure in engineering structures and components. Experimental studies have demonstrated that, this is so because materials subjected to alternating loads normally fail even at stresses that are way below the yield stress. Depending on loading conditions and material microstructure, cyclic-deformation behaviour is characterised by various features.

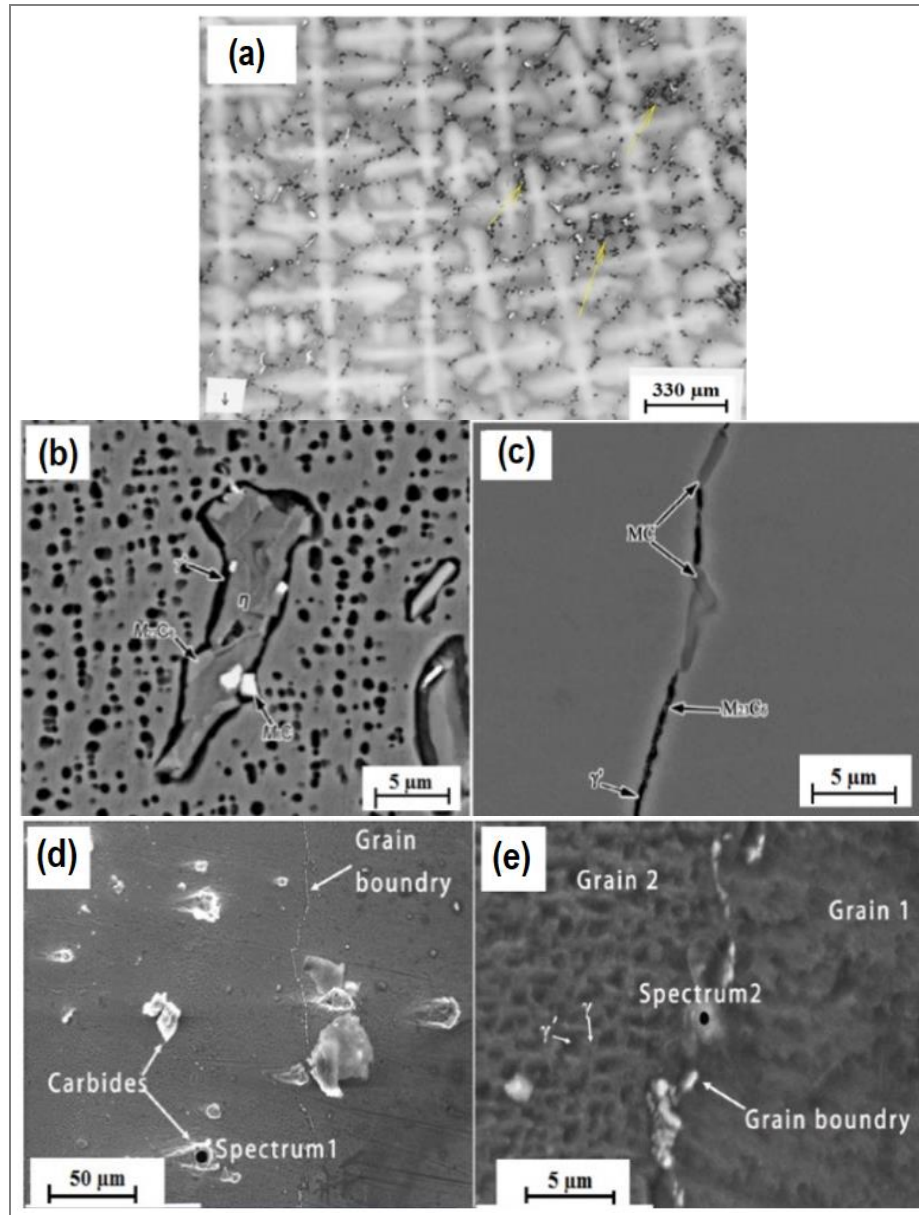


Fig. 4. (a) Structure of primary MC carbides in the inter-dendritic regions of a directionally solidified nickel-based superalloy [25], (b) and (c) Evolution from primary MC carbides to secondary $M_{23}C_6$ chain-like carbides and γ' -precipitates, through heat treatment of nickel-based superalloys [40], (d) and (e) Structure of secondary carbides in a directionally solidified nickel-based superalloy [41].

Earlier studies noticed that when metals are cyclically deformed, their elastic limits were not the same as those obtained when a monotonic load is applied.

It was acknowledged that softening and/or hardening characterises cyclic deformation. For nickel based superalloys, according to Yu et al. [47], cyclic hardening is a result of interaction of dislocations in the γ -matrix, while Ye et al. [48] noted that it was a result of interaction of dislocations with γ' -precipitates and grain boundaries. The study suggested that this leads to a reduction in dislocation mobility. Consequently, material resistance to plastic deformation significantly increases. On the other hand, cyclic deformation was reported to lead to formation of planar slip bands in the γ' -precipitates. This led to the shearing of γ' -precipitates due to dislocation motion, which resulted in increased dislocation mobility. Consequently, material resistance to plastic deformation and, hence, cyclic softening ensues [49]. Cyclic hardening and softening have been widely reported in nickel-based superalloys [50, 51]. Furthermore, a closer look at cyclic deformation shows that, in fully-reversed cyclic loading (with loading ratio, $(R_L = -1)$, compressive loading normally results in lower yielding stress level. This implies that compressive yielding occurs at a stress level less than that for tensile yielding. This normally is due to the unbalanced plastic deformation and leads to a change in the material's stress-strain characteristics. This deformation feature is known as the Bauschinger effect [52-54].

Effects of Microstructure.

This section reviews the effects of microstructural features on the cyclic deformation behaviour of nickel-based superalloys at nanoscale and microscale. At nanoscale, the influence of γ' -precipitates and γ -matrix is discussed, while at microscale, the influence of the grain-structure is reviewed. Nickel-based superalloys are inherently characterised by exceptional mechanical properties. This is due to the presence of the ordered γ' -precipitates at nanoscale. These phases are embedded in the γ -matrix

phase, which has an fcc crystal structure. These two phases (γ and γ') are typically solid-solution strengthened by the addition of refractory elements Co, Fe, Mo, W and Ta. This two-phase structure is responsible for the characteristic mechanical properties at high temperatures [55].

A study by Xia et al. [27] has investigated the influence γ' -precipitate sizes and morphologies have on the mechanical behaviour (creep) of nickel-based superalloys. This was achieved by considering three different γ/γ' microstructure variants of a directionally solidified alloy, designated SHT, HT1 and HT2. The SHT variant had regularly aligned cubic γ' -precipitate with sizes of 300 nm, HT1 had cuboidal 880 nm sized γ' -precipitates while HT2 had heterogeneous morphologies and sizes of γ' -precipitates. Creep tests were conducted with loading conditions of 1040°C/100 MPa and 1040°C/400 MPa. SHT and HT1 variants had identical creep behaviour while HT2 variant showed the poorest performance and this was attributed to the uneven deformation structure due to different γ' -precipitate morphology and sizes. This was a result of the ease with which dislocation broke up due to high stresses resulting from the uneven deformation structure. This culminated in short secondary stages and longer tertiary creep deformation stages in the loading condition 1040°C/100 MPa [27]. In general, other studies appeared to be in support of the view that nickel-based superalloys' cyclic/static deformation behaviour is strongly influenced by γ' -precipitate sizes [56], distribution [57], volume fraction [2, 58] and morphologies [59].

Cyclic deformation behaviour in single crystal nickel-based superalloys was recently studied by Segersäll et al. [60] who considered three different crystallographic orientations, i.e., $\langle 001 \rangle$, $\langle 011 \rangle$ and $\langle 111 \rangle$. Results showed substantial anisotropic behaviour. As compared to $\langle 011 \rangle$ and $\langle 111 \rangle$ where serrated yielding behaviour was observed, crystallographic orientation $\langle 001 \rangle$ exhibited

superior fatigue-life although with the lowest stiffness. Under strain-controlled loading conditions, more asymmetric deformation was reported for $\langle 011 \rangle$ and $\langle 111 \rangle$ orientations when compared to $\langle 001 \rangle$ orientation [60]. This asymmetric deformation behaviour was also strain-range dependent. Similar to the findings of Shi et al. [61], the creation of dislocation networks at the γ/γ' interfaces during deformation, coupled with the time-dependent coarsening of the γ' -precipitates, were the reasons cited for the observed asymmetric and cyclic softening deformation behaviour in $\langle 001 \rangle$ orientation. On the other hand, the cyclic hardening which characterized the deformation behaviour of $\langle 011 \rangle$ and $\langle 111 \rangle$ was attributed to the dislocation interactions occurring in the γ -matrix channels. As shown in figure 5 [61], it is clear that different crystallographic orientations lead to different fatigue-life behaviour. At the same strain range, fatigue life differences were substantial for the considered crystallographic orientations. The scatter band in fatigue life was about a thousand between $\langle 001 \rangle$ and $\langle 011 \rangle$, while it was about seven thousand cycles between $\langle 001 \rangle$ and $\langle 111 \rangle$. In general, nickel-based superalloys' properties such as stiffness (Young's modulus), shear modulus and Poisson's ratio were found to be highly dependent on the inherent microstructure, particularly, crystallographic orientations [62].

In directionally solidified alloys, having columnar grains, mechanical behaviour is highly dependent on orientation of the loading axis, with respect to the direction of solidification. This is the case when the principal-stress axis is changed from parallel to perpendicular, to the direction of solidification, and vice versa. Dong et al. [35] studied the tensile and cyclic deformation behaviour of a directionally solidified nickel-based superalloy and observed that deformation stiffness increased considerably when the loading axis was changed from parallel to perpendicular to the solidification direction. However, other studies noted that although this change in loading

axis orientation improved the strength, fatigue/creep-load bearing capabilities were substantially compromised [63, 64].

A more recent study [32], has demonstrated, based on both experimental and modelling approaches that this was due to the presence of grain boundaries normal to the loading axis. These grain boundaries acted as crack initialization sites, thereby leading to reduced fatigue-life in cases where the loading axis were perpendicular to the direction of solidification. This behaviour was apparent in a wide variety of DS alloys, such as nickel-based superalloys [35, 61, 65], Co-based alloys [66-68], Fe-based alloys [69] and Al-based alloys [70].

Through the control of heat treatment process parameters, grain-structures of polycrystalline alloys, manufactured through the powder metallurgy route are normally tailored. The choice of either supersolvus or subsolvus heat treatment temperature levels determines the resulting grain sizes. Supersolvus heat treatment temperatures normally yield coarse grained structures, while subsolvus temperatures result in fine grained structures [1]. Additionally, solution heat treatment temperatures and cooling rates are normally considered to obtain other microstructural variants such as sizes of γ' -precipitates [71].

Superior fatigue performance was reported in coarse-grained specimens as compared to fine-grained counterparts. The intensification of slip reversibility enhanced by planar slip, in addition to the reduced grain boundary area, were some of the attributes cited for the better performance in coarse-grained specimens [1]. This is similar to findings of other researchers [72-74]. A recent study has demonstrated that grain orientation has significant effects on crack growth behaviour [75]. However, other studies such as the one by Everitt et al. [76] cited the interaction between oxidation and fatigue, as the cause for superior fatigue performance in coarse-grained specimens as compared to fine-grained specimens.

Significant research efforts have shown that, in polycrystalline nickel-based superalloys, orientation plays a critical role in mechanical behaviour. It's noteworthy, however, that influence of grain-boundaries was not fully explored.

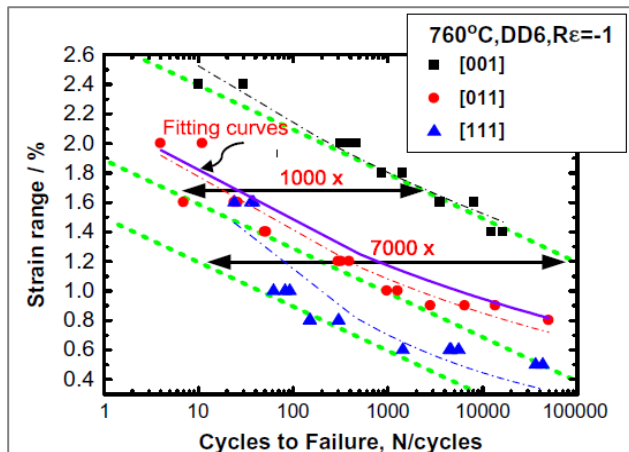


Fig. 5. Strain range vs fatigue life of a single crystal nickel-based superalloy, with $\langle 001 \rangle$, $\langle 011 \rangle$ and $\langle 111 \rangle$ orientations, tested at 760°C [61].

For instance, in the recent computer-based studies focusing on low-cycle-fatigue [32] and fatigue crack growth [75] behaviour, effects of grain-boundaries on the overall behaviour was completely neglected. Therefore, future research needs to explore and quantify the influence grain-boundaries have on mechanical behaviour.

Effects of loadings variables.

From the time Coffin [77] presented the first substantial evidence on effects of high-temperature oxidation on material behaviour, effects of loading variables on the mechanical behaviour of nickel-based superalloys have been continuously studied [78].

Studies such as those by Shi *et al.* [79] reported significant reductions in fatigue life for single crystal nickel-based superalloys as strain range increased. These observations were more pronounced for specimens with $\langle 011 \rangle$ and $\langle 111 \rangle$ orientation as compared to the $\langle 001 \rangle$ orientation, as shown in figure 5. For the four cyclic loading waveforms considered (figure 6), superior fatigue life was observed in

the specimen subjected to a load waveform without any dwell.

Imposition of dwells at maximum and/or minimum strain, as shown in figure 6b, c and d, gave almost identical results. Similar behaviour was also reported for the fatigue behaviour of a powder metallurgy nickel-based superalloy studied by Jiang *et al.* [1]. In the study, two trapezoidal waveforms of 1s-1s-1s-1s and 1s-20s-1s-1s, with a load ratio of $R_\epsilon = 0.1$, were considered. Results showed longer fatigue life for a shorter dwell (1 s) period. Similar results were reported elsewhere [80-82].

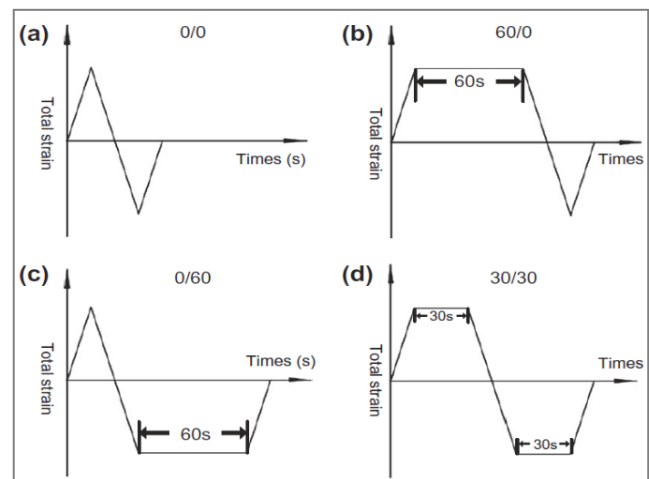


Fig. 6. Cyclic loading waveforms applied in the investigation of low-cycle fatigue behaviour for a single crystal nickel-based superalloy [79].

Highsmith and Johnson [83] studied the effect of load ratio on the fatigue behaviour of a directionally solidified nickel-based superalloy, and they showed that positive load ratio led to increased reduction in fatigue-life as compared to negative load ratio. Generally, it is widely acknowledged that the detrimental effects of fatigue become more pronounced with the decrease of loading rate (or loading frequency) and the increase of dwell periods at peak loads. This is also the case for increased load range, load ratio and stress intensity factor [53, 84]. Various studies have, over the years, investigated effects of various loading variables (e.g. load level, loading waveform, loading rates, temperature level, nature of

environment, microstructure, etc.) on the mechanical behaviour of nickel-based superalloys. Its noteworthy, however, that these studies mostly focused on the synergy of these loading variables. No study has made any attempts to quantify which of the variables is more prevalent than others.

Fracture Surface Analysis.

In ductile materials, failure results from the formation, growth and coalescence of voids at nano-scale, initiating mostly from boundaries between material bulk and inclusions or matrix and secondary phases, as shown in figure 7a. As such, fracture surfaces are normally characterized by “dimpled-like” features. These features

are known to result from the joining (coalescence) of micro-voids that initiate at the boundaries between material bulk and inclusions or between the matrix and secondary phases [85]. In brittle materials, however, failure is predominantly by cleavage fracture. This kind of fracture occurs along preferred planes which have the highest packing density. This is owing to the fact that, along those planes, there are fewer bonds to be broken and the spacing between planes is greater.

In polycrystalline materials, with randomly sized and oriented grains at microscale, the fracture mode is referred to as transgranular. This is because the crack path takes different directions each time it crosses a grain boundary, following the most favorably oriented cleavage plane in each grain.

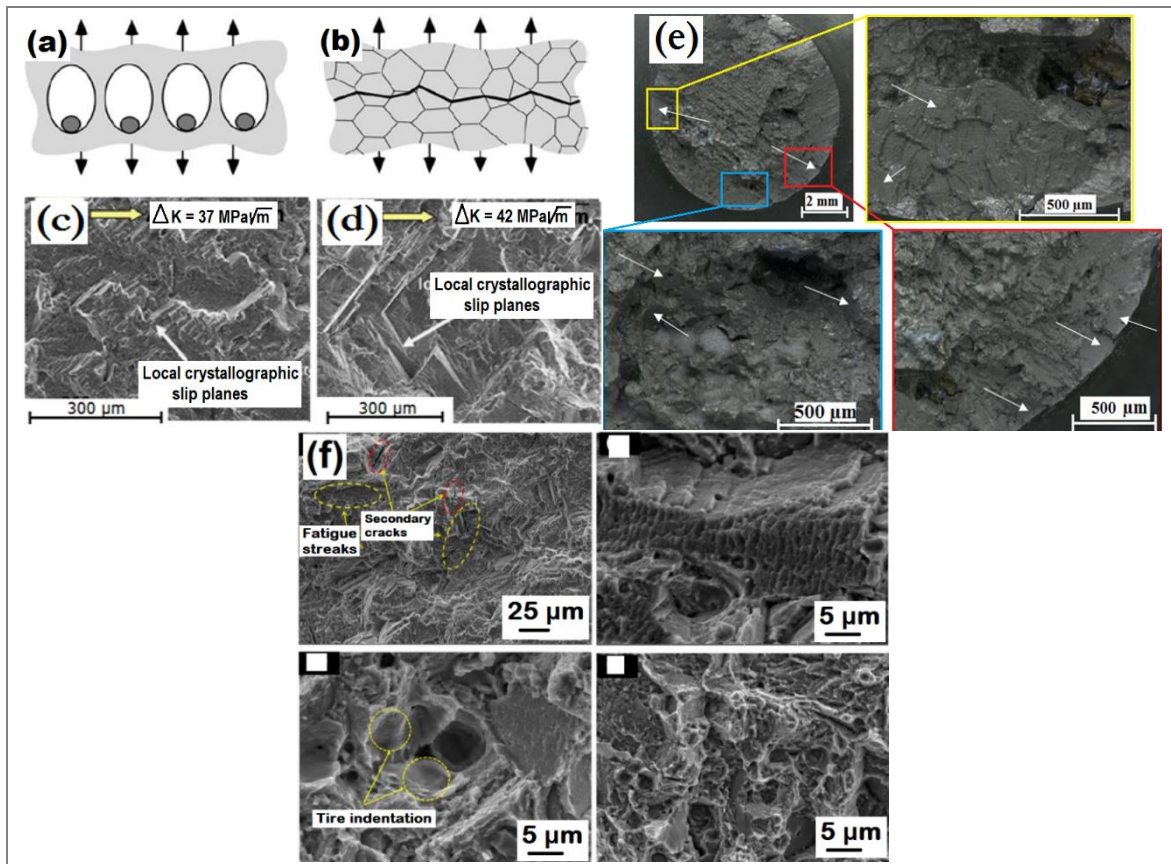


Fig. 7. Two micro-mechanisms of fracture in metals and alloys: (a) ductile fracture, (b) cleavage or trans-granular fracture [85]; (c), (d) [41] and (e) Characteristic (SEM) fractographs [32], showing multi-faceted fracture surfaces, reported in a DS nickel-based superalloy, (f) a combination of secondary cracks, cleavages, fatigue stripe, tire indentations and dimples [87]

As such, in polycrystalline materials, fracture surfaces are normally multi-faceted as shown in figure 7. In materials under mechanical loads, crack initiation normally occurs at and near stress-raisers, so called defects. These defect features include notches, holes, sharp edges, grooves, cracks and changes in cross-section area. In nickel-based superalloys, defects include carbides, inclusions and other microstructural abnormalities which may result in low-ductility sites.

Their size, shape and location primarily determine the extent to which the low cycle fatigue behavior will be controlled [26]. Ideally, under a mechanical load, interatomic bonds are stretched and the bonds which are near the stress-raisers tend to be stretched more. If the load is further increased, bond stretch increases too and the stress near the defects increases even further. Due to the presence of defects, local stresses may reach the fracture strength of the material. As a result, interatomic forces are overcome and bonds begin to break leading to separation along or across crystallographic planes, forming cracks whose growth rates depend on a myriad of factors [54, 86].

He et al. [41] studied the influence orientation of columnar grains and temperature on fatigue behaviour of a directionally solidified nickel-based superalloy. Fracture surfaces were analyzed to establish the underlying mechanism for fracture, using a combination of scanning electron microscopy (SEM) and optical microscopy (OM). SEM fractography notably showed similar features on the fracture surfaces at all the crack lengths considered (see figure 7c and d). As such, this implies that material fracture resulted from a similar fracture mode in all orientations considered [41].

Similar results were recently reported (see figure 7e) in a directionally solidified nickel-based superalloy which was subjected to loading along and perpendicular to the direction of solidification [32]. Elsewhere, SEM fractographic studies revealed a combination of secondary cracks, cleavages, fatigue stripe, tire indentations and

dimples, figure 7f [87].

Components such as turbine blades, guide vanes and discs are subjected to creep conditions i.e. excessive stresses under extreme temperatures. To give an acceptable level of safe-life analysis and assessment of such components, materials from which such components are manufactured are expected to exhibit a low propensity to crack initiation and a high resistance to crack propagation, under creep conditions. Many researchers have studied creep behaviour of nickel-based superalloys. These studies considered various aspects of creep, including influence microstructure and loading conditions such as temperature changes and variation in load levels have on the creep behaviour. In a bid to study the effect microstructural features have on the creep behaviour of nickel-based superalloys, Xia et al. [27] has investigated the influence variation of γ' precipitate morphologies have on the creep behaviour of directionally solidified nickel-based superalloys. In the study, three variant microstructures A, B and C (figure 8) were obtained from different heat treatment processes considered. Variant A had different morphologies and sizes of spherical γ' phases of sizes 150 nm between γ' raftings. Variant B had standard cubic morphologies which were regularly aligned, with sizes of 300 nm. Variant C had cuboidal γ' phases of about 880 nm. These features are shown in figure 8.

Creep behaviour was investigated by applying two different loading conditions. One set of specimens was subjected to a load level (stress) of 100 MPa at a temperature of 1040°C while another set was subjected to 400 MPa at 850°C. Results showed a heterogeneous creep deformation structure in specimens A, which was linked to the different morphologies of the γ' phases (figure 9).

This was said to be a result of the ease with which dislocation networks were breaking up due to high stress from the uneven deformation structure. Consequently, specimen A had short secondary creep stages and long tertiary stage at 1040°C/100 MPa. Conversely, specimens with a uniform distribution of microstructural variants (B

and C) had similar creep properties at to 1040°C/100 MPa, which were characterised by lower steady creep rates. Authors cited the regular dislocation networks in the γ/γ'

interface prevalent during creep deformation, in addition to the formation of perfect raftings, as the reason for this identical behaviour.

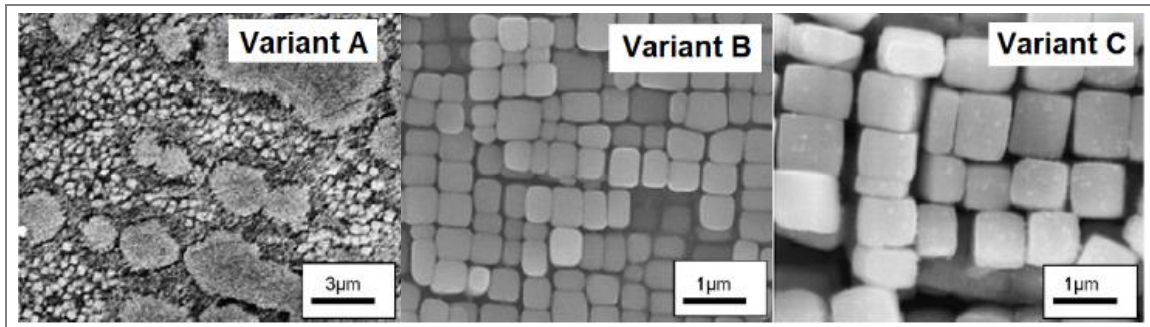


Fig. 8. Different morphologies of the γ' phase, obtained from the different heat treatments, employed to study their effect on the creep behaviour of nickel-based superalloys [27].

At 850°C/400 MPa, on the other hand, variant with standard microstructure (B) showed better creep properties than A and C (figure 9a). This was attributed to the tangling of dislocations, without formation of any regular networks. In variants A and C, however, inferior creep properties resulted from the inhomogeneous deformation structure during creep deformation (figure 9b).

Similar results were reported by [88]. Furthermore, another researcher, Nie et al. [36], used a computer-based modelling approach, to study the size effects of γ' precipitates on the creep properties of a directionally solidified nickel-based superalloy.

On the other hand, a study [58] to investigate the influence γ' precipitates volume fractions have on the creep behaviour of single crystalline material revealed superior mechanical behaviour in high volume fractioned specimens.

The study further reported similar behaviour even under different loading conditions such as variations in temperature and load levels. Generally, it is evident that microstructure [89], temperature [90, 91] and loading [91] have significant influence on the creep behaviour of nickel-based superalloys.

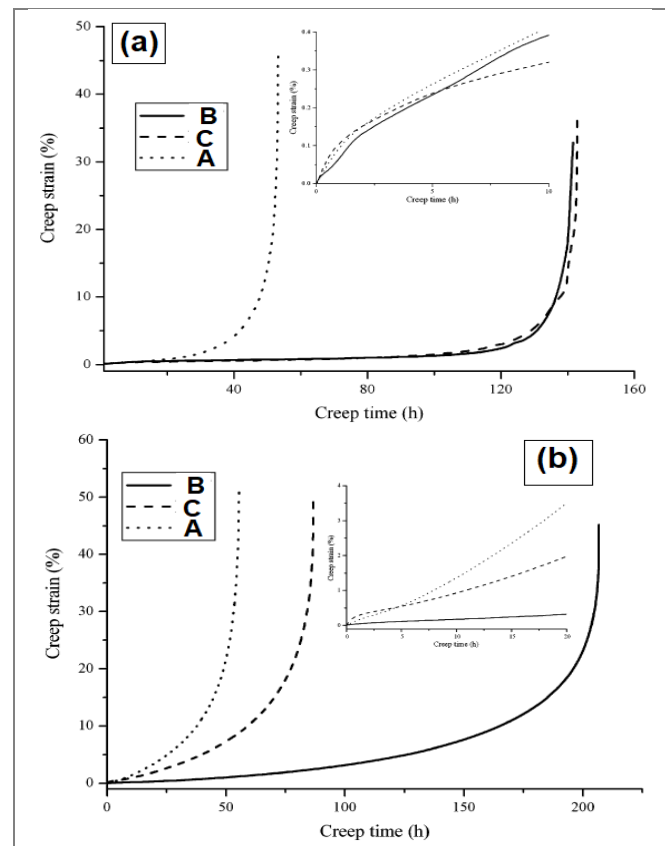


Fig. 9. Creep curves of a directionally solidified nickel-based superalloy with different morphologies of the γ' phase, employed to study their effect of on the creep behaviour at: (a) 1040 °C/100MPa, and (b) 850 °C/400MPa [27].

Creep-Fatigue Behaviour.

Interaction between creep and fatigue has been investigated extensively in nickel-based superalloys.

Variations in the load levels in addition to the variations in dwell times at various levels of maximum loads, at high temperatures, are common in service conditions of nickel-based superalloys. To investigate material behaviour under such loading conditions creep-fatigue studies are conducted.

Generally, inferior mechanical behaviour characterises increases in imposed dwell times, irrespective of any variations in loading rates. Variation of the duration of imposed dwell times at maximum loads is illustrated in figure 6 [79].

Reduced low cycle fatigue behaviour was reported in a directionally solidified [32, 92] and in a traditional polycrystalline [1] nickel-based superalloy, on increasing dwell times. Imposition of dwell times were found to bring about stress-relaxation [32, 93]. Similar results were reported elsewhere [80-82]. Other studies [75, 94] reported increased crack growth rates.

CONCLUSIONS

The role microstructure plays on mechanical behaviour of nickel-based superalloys has been reviewed. Differences in chemical composition, and roles of the various elements were considered, in addition to the characteristics of the resulting nano- and micro-scale features. Influences of loading conditions such as fatigue, creep and their interaction on the overall mechanical behaviour were investigated. Its noteworthy, however, that literature is rich in studies whose attention was mostly focused on the synergy of the loading variables (e.g. load level, loading waveform, loading rates, temperature level, nature of environment, microstructure etc). There appears not to be any attempts to quantify which of the variables is more prevalent than others, in a given study. Additionally, research needs to focus on how to improve materials' resistance to property deterioration due to high temperatures and damage due to fatigue and creep loading. Though, significant efforts towards providing quantification of the role orientation plays on low-cycle-

fatigue and fatigue crack growth behaviour in polycrystalline nickel-based superalloys, future research still needs to explore and quantify the influence grain-boundaries have on mechanical behavior.

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