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Phase Transformations in Nickel base Superalloy Inconel 718 during Cyclic Loading at High Temperature

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Abstract

Nickel base superalloys are hi-tech materials intended for high temperature applications. This property owns a complex microstructure formed by matrix of Ni and variety of precipitates. The type, form and the amount of these phases significantly affect the resulting properties of these alloys. At sufficiently long exposure to high temperatures, the transformation phase can occur, which can lead to degradation of properties of these alloys. A cyclic plastic deformation can accelerate these changes, and they could occur at significantly lower temperatures or in shorter time of exposure. The aim of this study is to describe phase transformation, which can occur by a cyclic plastic deformation at high temperatures in nickel base superalloy Inconel 718.

1. Introduction

Inconel 718, age hardenable nickel base superalloy, is the most widespread material in its class. It was introduced in the 1960s, and it is commonly used in cast, powder metallurgy and a mainly in the wrought form. Inconel 718 has many industrial applications, as a chemical, petrochemical, energy and aviation industry. For many years, Inconel 718 has been the standard material for the turbine discs of gas turbine engines manufacturing. Inconel 718 exhibits outstanding mechanical properties in a wide range of working temperature, from -250°C, as a material for to liquid gas storages, up to 700°C for turbine disc application. This alloy also has good technological properties and excellent corrosion resistance. Great properties of this alloy are given by the unique microstructure, consisted of matrix γ , precipitates γ'' , γ' , δ and carbides. The matrix γ is a solid solution of alloying elements like Cr, Fe, Mo in Ni, and it has a FCC crystal lattice. Gamma double prime (γ'') is the metastable phase (Ni_3Nb), with tetragonal, space centered crystal structure (D0_{22}). It is the main strengthening phase in Inconel 718, and the volume fraction of γ'' in the structure is typically 15-20%. When exposed to high temperature for sufficient time, gamma double prime transform to the stable δ phase. Delta phase (δ) is the stable form of Ni_3Nb with orthorhombic crystal structure. It is known that the presence of δ phase in large quantity is undesirable, but precipitated at the grain boundaries, δ prevents the grain growth, thus,

in this form it has a positive effect on the mechanical properties. In microstructure of Inconel 718 γ' phase is also present, but due to low volume fraction of this phase, γ' has only minor effect on the properties of this alloy. At high temperatures (over 700°C) and at sufficient long exposure, the γ'' tends to transform to the δ phase, so this transformation sets the upper temperature limit for the operational condition of Inconel 718 (DESVALÉES Y. ET AL. 1994, DONACHIE M. J. ET AL. 2002, KALLURI S. ET AL. 1994, RADAVICH J. F. 1989, BELAN J. 2015). In this study is analyzed microstructure of Inconel 718 after cyclic loading at 700°C, which is the highest possible operating temperature, with an aim to describe the phase transformation that can occur.

2. Experiment

Nickel base, precipitation hardenable, superalloy Inconel 718 was used as an experimental material in this study. Rods with diameter 12mm were used, from which the specimens for fatigue tests were machined. The rods were heat treated by the standard heat treatment used for Inconel 718, which consists of solution annealed at 920°C for 1 hour, followed by rapid quenching and subsequent two-stage precipitation hardening at 720°C for 8 hours and 620°C for 8 hours. Chemical composition of testing material and the basic mechanical properties are shown in the Tab.1 and Tab.2.

Table 1. Chemical composition of the tested material

In 718	C	Fe	Cr	Ni	Mo	Nb	Ti	Al	Si	Co
wt. %	0.03	18.04	18.5	53.21	3.04	5.3	1	0.52	0.06	0.13

Source: own study

Table 2. Basic mechanical properties of the tested material

	Ultimate tensile strength [MPa]	Yield strenght Rp0.2 [MPa]	Elonga-tion [%]	Hardness HBW
20°C	1474	1238	26	450
650°C	1211	1030	13.5	-

Source: own study

The fatigue tests were carried out on the electromagnetic pulsator ZWICK ROELL AMSLER HFP 5100 equipped with a high temperature chamber. During the entire test, temperature 700°C was maintained. Tests were carried out in the load-driving regime, so the driving parameter was the stress amplitude. Specimens were loaded by the sinusoidal, symmetric, tension-compression loading (stress ratio R=-1). During test, the resonant frequency of the specimens was recorded, which can provide valuable information about processes in the material. On the specimens broken by the cyclic loading at 700°C were then carried out microstructural analysis in the areas near the fracture surfaces, with the aim to observe the phase transformation which can take place in material during testing. Samples were prepared by the standard procedure for the metallographic preparation of sample and subsequently samples were electrolytically etched in the solution of 10g CrO₃ in 100ml H₂O at constant current 5V. Microstructural observations were supported by the measurements of the microhardness (Vickers) carried out on the Zwick ZHμ, microhardness testing machine.

3. Results and discussions

Microstructure observation shows significant changes, which took place after cyclic loading at 700°C. Figure 1 and 2 shows the microstructure in initial state and the microstructure after cyclic loading at 700°C, where the duration of loading was 7.5 hours. In microstructure of initial state, particles of the δ phase are localized on the grain boundaries, where, as was stated before, they have positive effect on the properties of the alloy. In microstructure after cyclic loading at 700°C occurred a massive increase of amount of the δ phase particles, which is now present even in the grain interiors. Figure 2 observation shows, that precipitation of the δ phase particles take place on some preferential crystallographic planes. Measurements of microhardness in the areas with increased amount of delta phase shows decrease of values by the 10%. Although there are different, often opposing opinions (RADAVICH J.F. 1989, LIS. ET AL. 1994, RUIZ C. ET AL. 1992) on the influence of the

δ phase itself on the mechanical properties, least the decrease of microhardness values in the delta-phase regions indicates a decrease of mechanical properties in these areas.

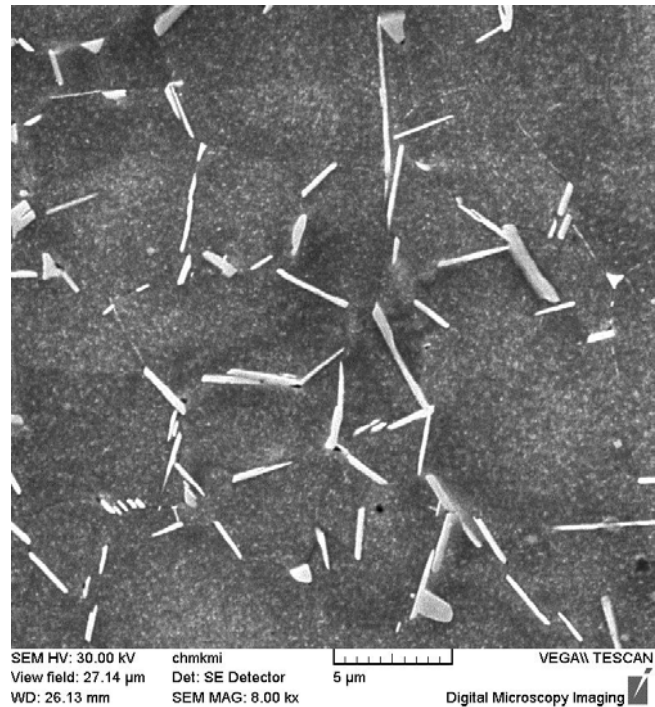


Fig. 1. Microstructure of initial state

Source: own study

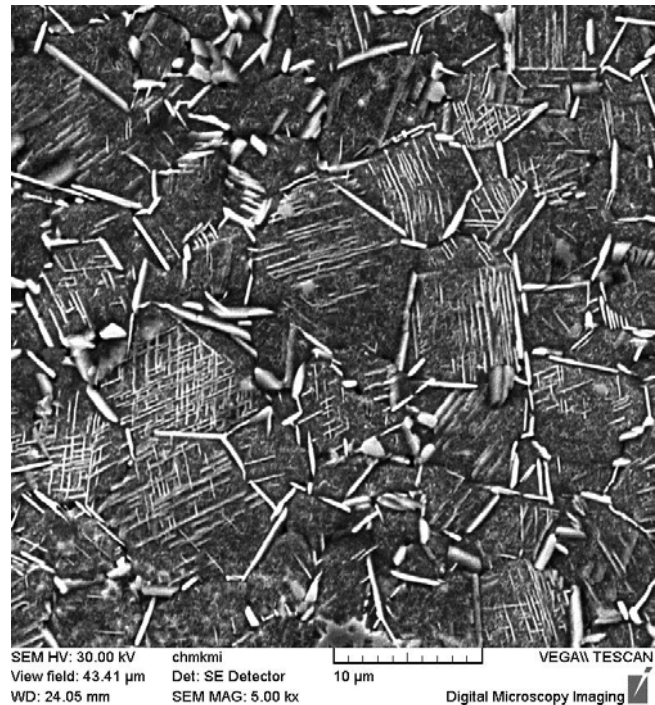


Fig. 2. Microstructure after cyclic loading at 700°C for 7.5 hours

Source: own study

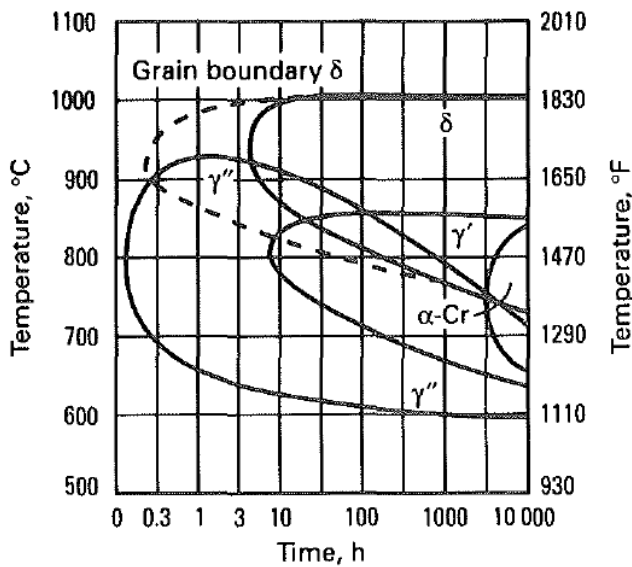


Fig. 2. TTT diagram for In 718

Source: (CHANDLER H. 1996)

Study of published TTT diagrams of for Inconel 718 (Fig.3) shows, that no significant microstructural changes should take place at exposure to 700°C for such a short time (7.5h). Based on this fact, observed changes must be caused by superposition of other factors, as only a temperature exposure. To understand these changes it is necessary to understand mechanisms of plastic deformation and the interaction of precipitates with the dislocations in the precipitation hardenable materials, like the nickel base superalloy Inconel 718. During cyclic plastic deformation in Inconel 718, in the first stage increase the dislocation density, which causes a slight increase of resonant frequency of the tested specimen. Dislocations are pinning on the precipitates, where the increase of dislocation density causes the increase of the stress required for the further formation and movement of the dislocations. When dislocation interacts with the precipitate, there are known two basic phenomena, depending on the size of the precipitates. The first case, when precipitates are small enough, the dislocation can shear them and the second case, typical for larger precipitates, a so called Orowan looping takes place (HULL B. 2005, PINEAU A. 2009, SIMS S. H. 1987). In Inconel 718, the particles of γ'' has a disc shape, with the diameter of 30-60nm and thickness 5-9nm, so shearing is the preferred interaction of dislocation with the precipitates γ'' . Gamma double prime is an ordered precipitate with the D022 crystal structure and for its shearing are necessary super dislocations with the double Burger's vector, than a regular dislocation in FCC matrix (REED C. 2006). Superdislocations are high energy configuration state, so their formation begins in the second stage of cyclic plastic deformation. At the beginning of the second stage, movement of the free dislocations is exhausted, so any other plastic deformation can be accommodated only by the shearing of the precipitates by super dislocations, whose formation begins as a consequence of increasing critical stress for dislocation movement. This process will show up in the decrease of resonant frequency of the tested specimen. During shearing

precipitates by super dislocations, some phenomena can occur (mechanism of shearing of the ordered precipitates is complicated process, due to decrease of energetic demands, which typically splits into to a super partial dislocations etc. This mechanism is described in the work in detail (HULL B. 2005). At first, shearing of γ'' can result in creation so small particles of γ'' , so they are not thermodynamically stable, and they dissolve in the matrix, which result in an increase of local concentration of Nb in the matrix. The second very important phenomenon is the creation of stacking fault, between leading and trailing super partial dislocations. In the area of stacking fault, a small area with the similar atomic configuration as in the δ phase in certain atomic planes is formed, from which, due to relatively high temperature and the fact, that a part of Nb content is dissolved in the matrix as a residue from dissolved γ'' particles, particles of δ phases can nucleate and grow. These claims are supported by observation of a manner, how the δ phase precipitates, where from the Fig.2 it can be concluded that the δ phase precipitates at some certain crystallographic planes, and these planes are considered to be $\{111\}$ planes, which are the primary slip planes in the FCC metals.

4. Conclusion

Based on the microstructure observation of Inconel 718 after cyclic loading at 700°C, it's obvious that major phase transformations occur. In the microstructure significant increase of amount of the δ phase was observed, especially in the interior of grains. This phase transformation can cause degradation of mechanical properties of Inconel 718, either by the presence of δ phase particles, or by the absence of the γ'' precipitates, which is associated with the δ phase content increase.

Formation of δ phase particles is caused by superposition of cyclic plastic deformation and the high temperature during the cyclic loading.

Based on the carried out experiments, it can be concluded that the phase transformations can occur even at lower temperatures or in shorter time as is shown in the TTT diagrams, when this alloy is exposed to a superposition of high temperature and cyclic loading.

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镍基超合金铬镍铁合金718在高温循环加载期间的相变

关键词

镍基超合金
 铬镍铁合金718
 相变
 高温疲劳

摘要

镍基超合金是用于高温应用的高科技材料。该属性具有由Ni基体和各种沉淀物形成的复杂微结构。这些相的类型、形式和数量都显著影响了这些合金的性能。在足够长时间暴露于高温下，可能发生转变阶段，这可能导致这些合金的性能降低。循环塑性变形可以加速这些变化，并且它们可以在显著较低的温度或较短的暴露时间内发生。本研究的目的是描述相变，这可以通过镍基超合金Inconel 718在高温下的循环塑性变形而发生。
