Mechanisms of Fatigue Failure of Nickel-Base Alloys at Room and Elevated Temperatures in the Very High Cycle Regime

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Fatigue failure at very high number of cycles ($N > 10^7$) does not follow the same principles of damage mechanism as is observed in the range of conventional fatigue limit. With the applied global strain amplitude being well below the elastic limit, life is dominated by heterogeneously distributed and localized plastic deformation in the microstructure. Hence, the dislocation/particle interaction in precipitation hardened alloys, such as Nimonic 80A, plays a major role during VHCF cyclic deformation.

The nickel-base superalloy Nimonic 80A was tested in the peak-aged and overaged condition with spherical agglomerated precipitates and cuboidal regularly distributed precipitates, respectively. Under room temperature conditions planar single slip and at elevated temperatures partially wavy dislocation arrangements were found in failed and in run-out samples. The overaged Nimonic 80A showed a slightly superior fatigue behaviour compared to the peak-aged condition. Isothermal cyclic deformation at elevated temperatures revealed a pronounced decrease of cyclic life at 800°C for the overaged condition.

1. Introduction

Nickel-base alloys are widely used as material for turbine blades or discs because of their excellent mechanical properties at room and at elevated temperatures. In the present study the precipitation-hardening nickel-base alloy Nimonic 80A was analyzed regarding its fatigue behaviour in the very high cycle fatigue (VHCF) range, where from a macroscopic point of view specimens are subjected to a purely elastic strain. There is a consensus that in the VHCF range the fatigue life is dominated by the crack initiation phase rather than the crack growth period [1-3]. Hence, a strong influence of the microstructure on damage evolution must be expected.

Extensive research work has been done with regard to the influence of microstructure, loading type and amplitude as well as temperature on the fatigue behaviour of precipitation-hardened nickel-base alloys for the LCF and HCF regime [4-10]. A comprehensive study on high-temperature fatigue behaviour of nickelbase alloys was elaborated for Nimonic 75 and 105 [11]. With regard to the VHCF behaviour recent studies concentrate on the influence of heat treatment conditions and predeformation [12, 13]. The study presented tries to confirm the earlier findings and aims at an extension of the existing knowledge for the VHCF range, where the relation between heat treatment condition of precipitation hardened alloys and cyclic deformation at elevated temperatures has not yet been analysed.

2. Material and Experimental Methods

The nickel-base alloy Nimonic 80A was investigated in two different heat treatment conditions, peak-aged and overaged. The chemical composition and details about the heat treatment are given elsewhere [12]. After solution annealing with subsequent water quench, ageing at 710 °C for 16 hours and 850 °C for 500 hours to establish the peak-aged and the overaged condition, respectively, was applied. The peak-aged condition was defined on the basis of maximum hardness at room temperature. Vickers hardness tests resulted in a value of 362 HV 30 for the peakaged and 261 HV 30 for the overaged condition. Microstructural studies were performed by means of transmission electron microscopy (TEM) as well as scanning electron microscopy (SEM). The TEM specimens were mainly cut parallel to the longitudinal axis of the specimens and prepared by ion thinning in the last preparation step. The microstructure of the peak-aged condition consists of spherical γ' precipitates with a mean size of 20 nm (Fig. 1a). The γ' precipitates changed into a cuboidal shape and an almost linear structured arrangement reaching particle sizes of around 250-300 nm during overaging heat treatment (Fig. 1b). The volume fraction of the γ ' precipitates has not yet been measured precisely but is assumed to be between 10 to 20% according to respective literature [14-16].



Fig. 1. TEM micrographs of a) peak-aged and b) overaged Nimonic 80A.

After heat treatment, specimens for fatigue testing were machined from bars as round samples with a diameter of 3-6 mm depending on the fatigue test system being used. Prior to testing the specimens were mechanically ground and electrochemically polished. Tests were conducted in load-controlled constant-amplitude cycling in laboratory air at room temperature, 400°C, 600°C and 800°C under symmetrical push-pull conditions (R = -1). Test frequencies varied according to the test system used: around 130 Hz for a resonance electromechanical device, 760 Hz for a servohydraulic system and around 20 kHz for an ultrasonic fatigue test system. The type of testing device also defined the number of cycles, which classified a sample as run-out. During testing at room temperature the specimens were cooled by means of compressed air. For the high-temperature fatigue tests the servohydraulic and the resonance system were equipped with an induction high-frequency heating. All tests were executed in ambient air.

3. Results

3.1 Fatigue Testing

The influence of the microstructure, which was established by means of heat treatment, on the VHCF behaviour of the precipitation-hardened alloy Nimonic 80A as well as the influence of elevated temperatures during fatigue testing was studied. Fig. 2 depicts the results obtained at room temperature showing a slightly superior fatigue behaviour for the overaged condition. Failure occurred up to a number of load cycles of N = $5,5 \times 10^7$. Fractographic analysis in the SEM revealed striations on the crack surface but did not show any pronounced features giving evidence of the precise crack origin (surface or interior). Transgranular crack growth dominated in the VHCF range. Beyond the limit of N = $5,5 \times 10^7$ no appreciable crack or specimen rupture was observed at room temperature. Nonetheless, the formation and growth of slip markings in single grains could be observed at the surface of run-out specimens.



Fig. 2. Fatigue results for peak-aged and overaged Nimonic 80A at room temperature (run-out specimens are marked with arrows).

Cycling at elevated temperatures led to failure at far lower stress levels than at room temperature (Fig. 3). This effect was most pronounced for the overaged condition at 800°C, where even at a stress amplitude of 200 MPa a sample failed after $9,53 \times 10^7$ cycles. For the peak-aged condition a clear decrease in fatigue strength at 800°C was observed for the VHCF regime but by approaching the conventional fatigue limit (N $< 10^7$) passed into the S-N curve for room temperature. The fatigue results for the tests at 400°C and 600°C do not exhibit an apparent tendency yet and additional tests have to be executed to either confirm a superior fatigue behaviour at 600°C, or to prove that the results just demonstrate the large scatter in the fatigue life results at the VHCF range. The latter assertion would imply that for the VHCF regime no temperature influence up to 600°C (a likely operating temperature range for this type of alloy) can be observed for Nimonic 80A. With respect to a possibly superior fatigue strength at 600°C, the effect of dynamic age hardening, which was observed during tensile testing at 400°C and 600°C, will have to be discussed in connection with the load controlled test procedure. Also the effect of oxide layers being formed on the crack surface during testing at elevated temperatures and a possible frequency effect requires further consideration.



Fig. 3. Fatigue results for peak-aged and overaged Nimonic 80A at elevated temperatures (run-out specimens are marked with arrows).

3.2 Microstructural Analysis

As fatigue life in the VHCF range is dominated by the crack initiation phase and heterogeneously distributed cyclic deformation processes take place, the reason for the specific fatigue behaviour must be closely related to microstructural features. For Nimonic 80A the interaction between dislocation formation and mo-

bility and the size, shape and distribution of the γ' precipitates is of utmost importance. Therefore, fatigue tests were accompanied by TEM studies investigating the dislocation arrangements after cyclic deformation (Fig. 4). Nimonic 80A shows planar dislocation arrangements in single grains with no interaction between the activated slip bands at room temperature both for the peak-aged and the overaged condition. Fig. 4b illustrates that even for a run-out sample (no crack initiation at N = 4,58 x 10⁸) dislocations are arranged in dislocation pile-ups against isolated precipitates, and at the same time dislocation free zones exist. Typical of the VHCF microstructure is the coexistence of virtually dislocation free grains and those with the dislocation arrangements as described before.



Fig. 4. TEM micrographs comparing the dislocation arrangements in Nimonic 80A after fatigue testing at room temperature in a) the peak-aged and b) the overaged condition.

The TEM studies of the dislocation/particle interaction at elevated temperatures revealed the formation of Orowan loops around isolated precipitates and proved the existence of particle cutting for the overaged condition (Fig. 5). The dislocation arrangements are still dominated by a planar slip character and limited to single slip bands. However, as compared to the dislocation arrangements formed at room temperature, a first trend towards a more wavy slip character (Fig. 5a) and a slight interaction between the activated slip bands is indicated (Fig. 5b). Additional TEM investigations during the continuation of the ongoing work are necessary to clarify, whether at 800°C a change into a more pronounced wavy slip character takes place. Figure 5a indicates a slight increase of the dislocation density compared to the dislocation arrangements is still limited to isolated grains.



Fig. 5. TEM micrographs of dislocation particle interactions in overaged Nimonic 80A after fatigue testing up to $N = 1.4 \times 10^7$ at 600°C showing a) Orowan loops and b) particle shearing.

4. Discussion

The investigations upon the fatigue behaviour of the precipitation-hardened alloy Nimonic 80A in the VHCF range revealed that the active damage mechanisms cannot be envisaged by means of conventional prediction models. It has been shown that the heterogeneous character of cyclic deformation dominates the formation of dislocation arrangements and its consequences for damage evolution and crack initiation. Hence, the specific mechanisms must be analysed in connection with the microstructural changes during cyclic deformation at very low stress amplitudes.

Comparing the dislocation/particle interaction of the peak-aged and the overaged condition at room temperature, the difference in length of the active slip bands is particularly noticeable. In the grains of the peak-aged condition single slip bands find their way through the whole grain and are only stopped by the barrier effect of the grain boundaries (Fig. 6a). As a consequence of their specific size and shape, the γ' precipitates can easily be cut by the dislocations. In contrast, the dislocation movement in the overaged condition is dominated by pile-ups at the larger and cuboidally shaped particles (Fig. 6b).

Assuming that the same shear stress in favourably oriented grains is active in both heat treated microstructures, the shear stress will lead to a stress concentration at the grain boundaries for the peak-aged condition and to a competing effect of dislocation pile-ups at precipitates and the formation of new dislocation slip bands for the overaged condition. As a consequence, the formation of new dislocations deploys a more homogeneous cyclic deformation in the favourably oriented grains in the overaged condition. Therefore, the peak-aged condition leads to an earlier formation of microcracks at lower shear stresses than the overaged condition. Additionally an earlier change from single to multiple slip resulting from the lattice rotation of the single slip bands may also be a likely reason for the weaker fatigue strength of the peak-aged condition.



Fig. 6. Illustration of the dislocation particle interaction for the peak-aged and the overaged condition of Nimonic 80A.

Following the idea of the competing effect of dislocation pile-ups at γ' precipitates and formation of new dislocations, the additional effect of thermally activated overcoming of obstacles gains importance. In the case of thermal activated dislocation climb, the energy being introduced into the system by means of an elevated temperature must be higher than the barrier strength of the obstacle. This might explain the pronounced decrease of fatigue strength at 800°C and the lesser effect at 400°C and 600°C. However, this does not justify the stronger effect for the overaged condition compared to the peak-aged condition. Again the question arises whether possible microstructural effects are responsible for this effect or whether it is just a consequence of the large scatter of fatigue results in the VHCF range. The observed phenomena give rise to the assumption that the cutting process absorbs a higher amount of energy than the climbing process. The higher fatigue strength at 400°C and 600°C might be related to dynamic strain ageing processes promoting planar slip, which is confirmed by the Orowan loop and the particle cutting in Fig. 5. Moreover, the influence of the specific coherency stresses between matrix and precipitates for the different heat treatment conditions as well as the effect of the particle shape might provide further reasons for the unexpected fatigue behaviour in the VHCF range.

Possible particle growth effects evoked by the elevated temperatures might be the reason for the decreasing influence of the original heat treatment condition on the fatigue strength in the VHCF regime, as is illustrated in Fig. 3 for $N > 2 \times 10^7$. At 800°C the surface oxidation may contribute significantly to crack initiation at very high number of cycles ($N > 10^8$). The damage mechanism at elevated temperatures in the VHCF range will therefore mainly be dominated by the time and also by frequency dependant diffusion-controlled climb and surface oxidation processes. Hence, the influence of fatigue testing conditions and in particular possible frequency effects will have to be taken into closer consideration.

5. Conclusions

The influence of particle shape and size on the dislocation motion and arrangement during the cyclic life of the precipitation-hardened nickel-base alloy Nimonic 80A in the VHCF regime was studied under room and elevated temperature conditions. The resulting dislocation/particle interaction during cyclic deformation was analysed in order to develop an understanding of the dominating damage mechanisms.

Fatigue experiments were carried out up to $N = 10^{10}$. Failure occurred up to $N = 5.5 \times 10^7$ for the tests at room temperature and up to $N = 1.9 \times 10^8$ for those at elevated temperatures. The run-out samples exhibited the formation and growth of slip markings in single grains as a consequence of irreversible dislocation movements. The damage mechanism was generally dominated by the heterogeneity of the cyclic deformation. Dislocation slip bands were limited to isolated grains and showed pile-ups against the grain boundaries and at cuboidal particles for the peak-aged and the overaged condition, respectively. This led to a superior fatigue strength of the overaged condition at room temperature. At 800°C a pronounced decrease of cyclic life could be observed for both heat treatment conditions. The dislocation/particle interaction showed the formation of Orowan loops as well as particle shearing at 600°C. The possible influence of the testing conditions on the fatigue results at 800°C connected with diffusion controlled dislocation climb processes and surface oxidation will be the subject of further investigations.

6. Literature

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