LCF behaviour of a single crystal nickel-base superalloy

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Abstract

Low cycle fatigue behaviour of a single crystal nickel-base superalloy designated as DD8 has been investigated using both scanning electron microscope (SEM) and transmission electron microscope (TEM). The results are presented and discussed in this paper. Variation of stress amplitude with number of cycles for samples fatigue tested with a strain of 0.66% in fully reversed total strain control mode shows slight difference in the behaviour at ambient temperature and at 850°C. SEM examination of fractured surfaces shows initiation of fatigue crack at the external surface. Planar slip and cleavage facets are observed at both the temperatures. TEM examination reveals dislocation bands containing tangles, loops and dipoles. Substructure at higher temperature indicates beginning of the cell formation. An interesting feature observed is phase transformation induced by strain both at room temperature and at 850°C.

Riassunto

Vengono presentati e discussi i risultati di uno studio al microscopio elettronico, sia di scansione (SEM) sia di trasmissione (TEM), del comportamento a fatica oligociclica di una superlega monocristallo a base di nickel, denominata DD8. È stata osservata, fra il comportamento alla temperatura ambientale e quello agli 850°C, una modesta differenza della variazione dell'ampiezza della sollecitazione in funzione del numero dei cicli per i campioni testati allo 0.66% di deformazione secondo le modalità del controllo totale della sollecitazione completamente invertito. L'analisi al SEM ha dimostrato che la cricca da fatica inizia alle superfici esterne. A tutte le due temperature sono presenti lo scorrimento planare e sfaccettature di clivaggio. L'analisi al TEM invece ha rivelato fasce di dislocazione contenenti grovigli, anse e dipoli. La sottostruttura alla più alta delle due temperature indica l'inizio della formazione di celle. Di particolare interesse sarebbe l'osservazione della trasformazione di fase indotta dalla tensione, sia alla temperatura ambientale sia agli 850°C.

INTRODUCTION

Single crystal nickel-base superalloys are being used in advanced gas turbines and jet engines because their mechanical properties are superior to those of polycrystalline alloys [1]. Elimination of grain boundaries in single crystal alloys imparts excellent creep properties to them. So elements like, B, C, Hf and Zr used to strengthen grain boundaries are not required for single crystals. This results in increase in melting temperature which allows higher and longer heat treatment and consequently better control over gamma prime size and distribution. Casting defects such as inclusions and pores which may act as initiation sites for fatigue cracks are much less in single crystals and hence single crystals have better fatigue resistance. Growth of single crystals along low modulus <001> direction endows them with better thermal fatigue resistance. Low cycle fatigue (LCF) has a significant effect on life of a component of a machine, and LCF is an essential limiting factor for design life of components.

Investigation of LCF behaviour of single crystals is thus essentially required and some results of such studies have been reported in the literature [2-4]. Some single crystal nickel-base superalloys have been developed at the Institute of Metal Research (IMR), Shenyang, China, and their fatigue properties have been investigated [4,5]. In the present paper experimental results on LCF behaviour of one of these single crystal alloys are presented and discussed.

EXPERIMENTAL

The single crystal alloy investigated is designated as DD8 and it is one of the single crystal alloys developed in IMR. It has composition (in wt pct) of 15.85 Cr, 8.40 Co, 5.85 W, 3.91 Al, 3.98 Ti, 1.03 Ta and balance Ni. The single crystals of <001> orientation were produced by using casting method with crystal selector and high solidification rate [4]. The heat treatment given to the alloy is as follows:
1100°C/8 h AC + 1240°C/4 h AC + 1090°C/2 h AC + 850°C/ 24 h AC
This heat treatment produced uniform distribution of cuboidal γ' particles with volume fraction of 54.9%.
Samples used for fatigue tests had dimensions as shown in Fig.1. Fatigue tests were done on a servohydraulic 100 kN fatigue testing machine in fully reversed (R= -1) total strain control mode using triangular wave form of frequency 0.1 Hz. Fatigue tests were performed in air at ambient temperature and at 850°C with a total strain of 0.66%.
Fractured surfaces were examined in a scanning electron microscope. Slices were then cut and ground to about 50-60 µm. 3mm dia specimens were then punched out of the slices. Twin jet electropolisher was used to prepare thin foils for examination in a JEOL transmission electron microscope. Electrolyte was composed of 7% $HC10_4$ in ethanol and used at -30°C.

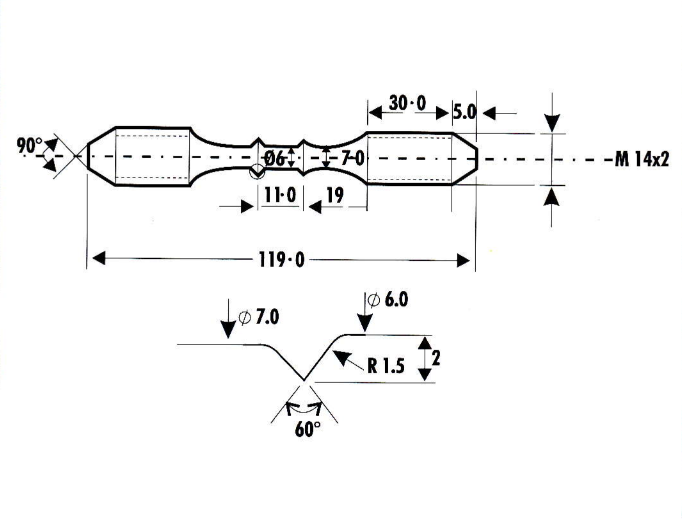

Fig. 1: Geometry of samples used in fatigue test. All measurements are in mm

RESULTS AND DISCUSSION

Fatigue Test

Variation of stress amplitude with number of cycles for samples fatigue tested with the same strain (0.66%) at ambient temperature and at 850°C is shown in Fig.2. At room temperature stress amplitude remains constant for the first four cycles indicating that to and fro motion of dislocations under cyclic load may be such that neither hardening nor softening is produced. From the 5th cycle upto about 60 cycles, there is a slight hardening. A slight and broad secondary hardening peak is observed around 400 cycles before the sample fractures.
At higher temperature (850°C) the stress amplitude is lower than that at room temperature. It is due to the fact that at higher temperature dislocations are thermally activated and hence lower stress is needed to produce same strain. At higher temperature the behaviour is slightly different. For the first three cycles, there is no hardening or softening but then there is softening. Again there is slight secondary hardening before the sample fractures. Reasons for the observed behaviour of stress amplitude are discussed in the section on Dislocation Structure.

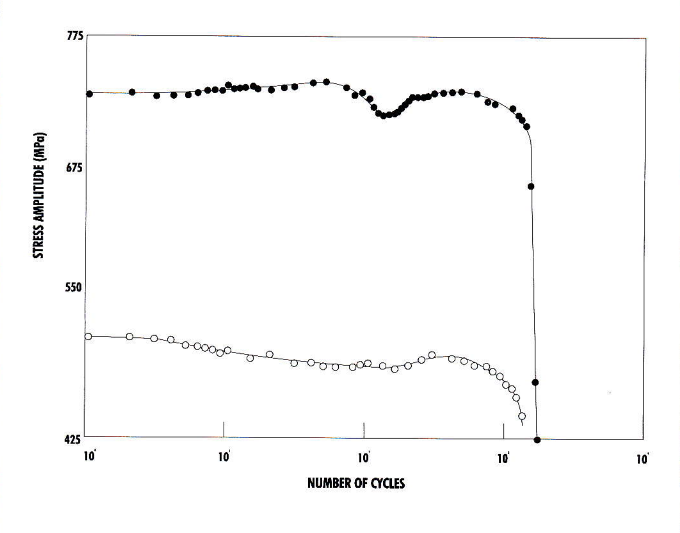

Fig. 2: Variation of stress amplitude with number of cycles.
• RT ○850°C

Fractography

Fig.3 shows low magnification SEM micrograph of fractured surface of sample tested at room temperature. The fatigue crack initiates at the surface from the bottom of the micrograph and all along the periphery of the sample. Same type of features are observed along the periphery. Fig.4 shows morphology at the vicinity of the boundary between the fractured and the free surface. There are slip bands on the free surface and then to the left is the fractured surface. This micrograph indicates that at the boundary, crack along the slip band opens up and serve as a crack initiation site, then crack propagates along {111} planes. These cleavage facets indicating stage I crack propagation are shown in Fig.5. Stage I cracking involves weakening of the slip planes by cyclic deformation and glide plane decohesion which occurs under the influence of the local shear and normal stresses at the crack tip [6]. At room temperature major portion of crack propagation is by cleavage (Stage I) which is in agreement with the conclusions of Anton [2] and other workers [7]. Striations indicating stage II mode are observed in the centre of the sample. Other features observed are secondary cracks and dimples.

Similar features are obeserved on the fractured surface of sample tested at 850°C. There are multiple initiation sites (Fig.5) again with faceted crack growth (Fig.6) followed by the area which is flat. The major portion of the fracture surface is flat and stage II crack propagation is dominant. No

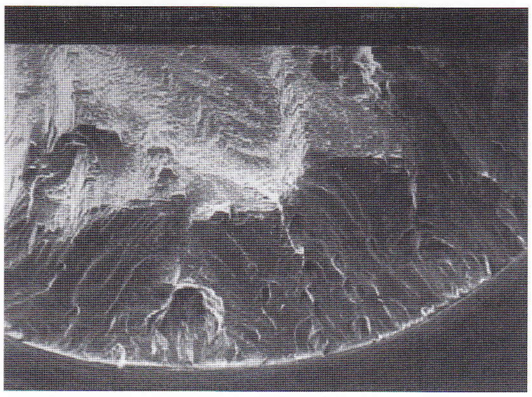

Fig. 3: SEM micrograph of fractured sample tested at room temperature showing crack initiation sites

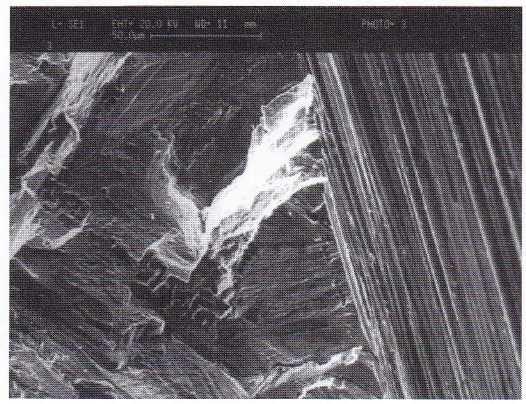

Fig. 4: Morphology at the boundary of fracture and free surface

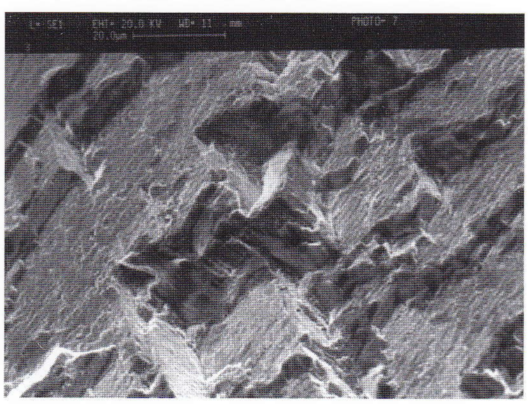

Fig. 5: Cleavage facets in room temperature tested sample

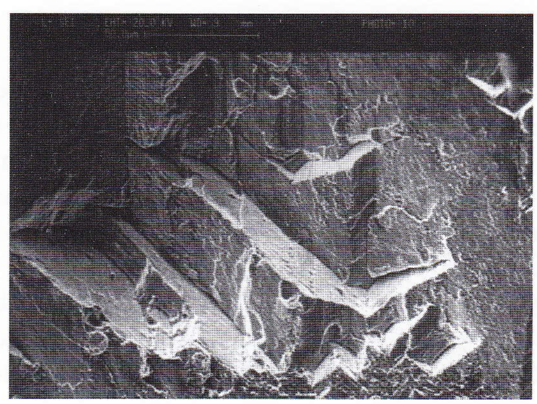

Fig. 6: Facet growth in sample fatigued with 0.66% strain at 850°C

striations are observed. These are perhaps hidden under the oxide layer as the sample surface is oxidised. Oxide layer is observed to crack because of the influence of crack tip stress field.

Dislocation Structure

Transmission electron microscopy of undeformed material shows that the single crystal has cuboidal gamma prime. The dislocation structure is found to be non-uniform and a few dislocations are observed (Fig.7). Transmission electron microscopy studies of fatigued samples were carried out in order to investigate the operative deformation mechanism which occurred during fatigue.

During deformation most of the dislocations tend to move and accumulate around γ/γ' interface within γ matrix as shown in Fig.8 because γ/γ' interface is a strong barrier for dislocation motion. Slight hardening observed at room temperature (Fig.2) may be due to dislocation multiplication. As deformation continues, more and more dislocations are produced. Energy of the crystal increases. It is well known that the crystal will tend to lower its energy. The energy of the crystal with numerous dislocations would be lowered if re-arrangement of dislocations in the form of low energy structure takes place. Cyclic deformation in push-pull straining provides ideal conditions for achieving low-energy dislocation structure because large cummulative strains give rise to high dislocation densities and the to and fro motion of dislocations enhances entrapment probabilities [8]. This sort of rearrangement may be the cause of softening observed at room temperature. TEM

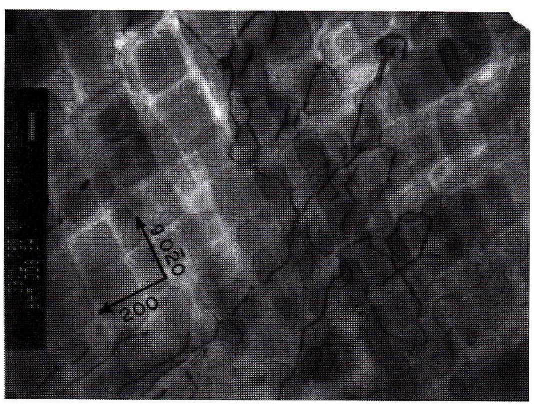

Fig. 7: Dislocation structure in undeformed sample

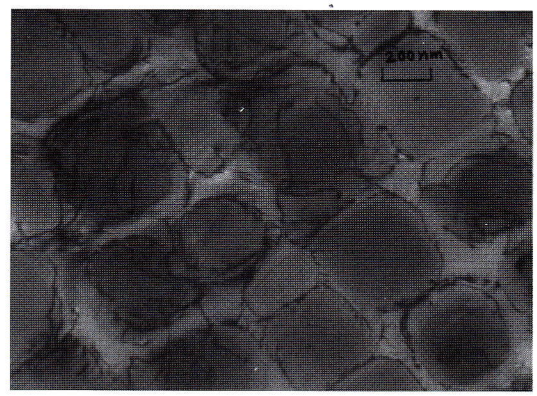

Fig. 8: Accumulation of dislocations around γ' precipitates

investigations reveal that at room temperature, dislocations rearrange themselves into planar bands of dislocations (Fig.9) which are intersecting. Dislocation bands are composed mainly of entangled dislocations (Fig.10). Some dislocation loops and dipoles are also observed. Intersecting bands indicate that multiple slip occurs.

Another form of low energy dislocation structure is dislocation cell which is formed by individual gliding dislocations as these accumulate at γ/γ' interface and then react to form

Fig. 9: Intersecting planar bands of dislocations in room temperature tested sample

Fig. 10: Dislocation structure within bands in room temperature tested sample

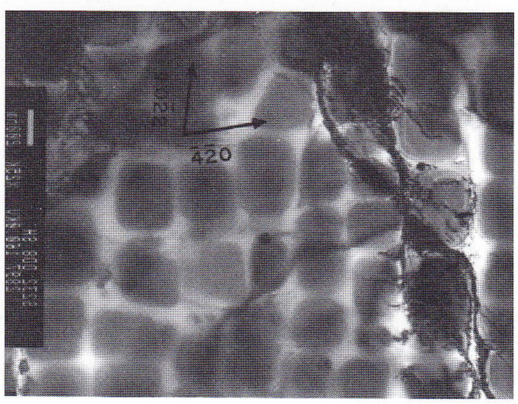

Fig. 11: Sub-structure in sample tested with 0.66% strain at 850°C

cells. Once a cell is formed it offers resistance to the motion of dislocations and absorbs more dislocations. At 850°C, no full fledged cell formation was observed, yet there is indication of tendency to form cells as shown in Fig.11. As mobility of dislocations enhances at 850°C, cell formation is expected at this temperature. As shown in Fig.2 softening takes place which may be due to rearrangement of dislocations in the form of bands, walls, and cells.

Another feature observed at 850°C is 3-D network of dislocations (Fig.12). Similar structure has been observed in single crystals of DD8 nickel base superalloy at 760°C and has been shown to form because of interaction of two sets of parallel dislocations arising from misfit interface between matrix and γ' precipitates [4]. Cracking is also observed to occur along different directions (Fig.14). Such a cracking has also been observed in single crystals of nickel base superalloy PWA 1480E [9].

Small globular particles are observed in the matrix both at ambient temperature and at 850°C as shown in Fig.13. These are most probably formed due to phase transformation induced by strain as reported at 760°C [4]. These particles are efficient barrier to the movement of dislocations in the matrix. This may be the plausible cause of secondary hardening observed around 400 cycles.

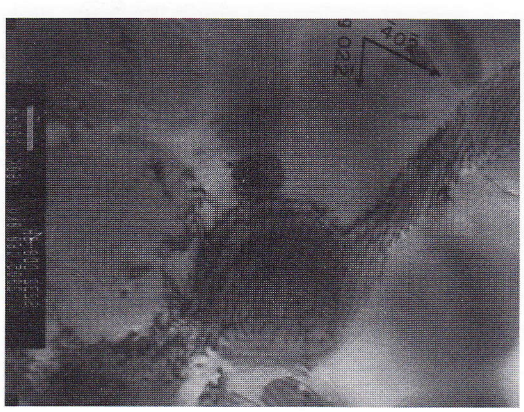

Fig. 12: 3-D dislocation network in sample tested with 0.66% strain at 850°C

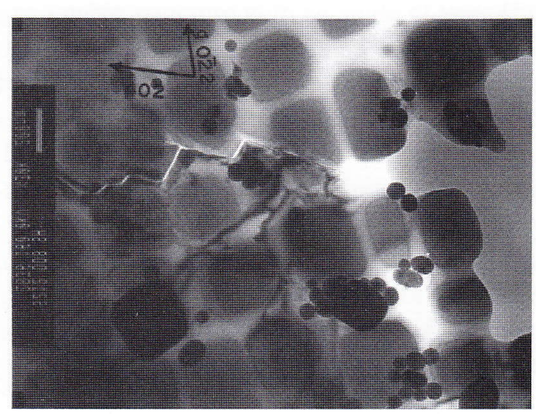

Fig. 13: Fine precipitates and cracking induced by strain during fatigue

CONCLUSIONS

1. LCF behaviour of single crystals of DD8 nickel base superalloy is slightly different at 850°C from that at room temperature. After a few cycles softening is observed at 850°C compared to slight hardening at room temperature.
2. Both stage I and stage II propagation modes are observed at both room temperature and at 850°C, but stage I is dominant at ambient temperature and stage II at 850°C.
3. Planar bands resulting in cleavage fracture are observed at both temperatures.
4. Substructure at higher temperature indicates beginning of cell formation.
5. Small globular precipitates induced by strain are the most probable cause of secondary hardening observed around 400°C at both the temperatures.

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