

Available online at www.sciencedirect.com



Scripta Materialia 52 (2005) 603-607



www.actamat-journals.com

# Shearing of $\gamma''$ precipitates and formation of planar slip bands in Inconel 718 during cyclic deformation

L. Xiao <sup>a</sup>, D.L. Chen <sup>b</sup>, M.C. Chaturvedi <sup>a,\*</sup>

Department of Mechanical and Industrial Engineering, University of Manitoba, Room 356, Engineering Bldg., Winnipeg, Man., Canada R3T 2N2
Department of Mechanical and Industrial Engineering, Ryerson University, 350 Victoria Street, Toronto, Ont., Canada M5B 2K3

Received 25 October 2004; received in revised form 17 November 2004; accepted 19 November 2004 Available online 15 December 2004

#### Abstract

Fatigue of Inconel 718 at RT and 650 °C caused the formation of planar deformation bands and shearing of coherent and ordered  $\gamma''$  and  $\gamma'$  precipitates by paired dislocations. The paired dislocations could not cross-slip, resulting in planar slip and planar slip bands, whose spacing and width were almost independent of the cyclic plastic strain amplitude. © 2004 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

Keywords: Low cycle fatigue; Microstructure; Dislocation structure; Planar slip band; Nickel alloys

# 1. Introduction

Planar slip is a very common phenomenon in cyclically deformed concentrated solid solutions of copper alloys such as Cu-Al [1,2] and Cu-Zn [3], and in AISI 316, 302 type austenitic stainless steels [4,5]. The formation of planar slip bands in these alloys has been attributed to their low stacking-fault energy (SFE), which leads to the formation of wider extended dislocations with reduced ability of screw dislocations to cross-slip onto other slip planes. Therefore, the occurrence of planar slip in pure metals and alloys depends on their SFE. However, several authors [6–11] have demonstrated that a reduced SFE may not be the reason for the deformation to occur by planar slip in some solid solutions. Instead, they have suggested that short-range ordering (SRO) and short-range clustering (SRC) often play a decisive role in determining nature of slip in these alloys [6-11].

E-mail address: mchat@cc.umanitoba.ca (M.C. Chaturvedi).

In addition, there exists another class of alloys which exhibit planar slip characteristics [12–19]. These alloys belong to a group of precipitation-hardened materials, where finely dispersed second-phase particles are responsible for their high strength. These precipitates are usually coherent or semi-coherent, thus they can be cut by moving dislocations. Normally, the precipitation-hardened alloys show wavy slip in a way similar to that exhibited by single-phase alloys with a high SFE. However, when the precipitates have an ordered crystal structure the corresponding alloy has been observed to deform by planar slip. Merrick [12] reported that fine scale microtwinning of coarse  $\gamma''$  precipitate particles and dislocation looping around fine  $\gamma''$  and  $\gamma'$  precipitate particles occurred during creep of Inconel 718, and planar deformation bands were produced by the microtwins. Clavel and Pineau [14] studied low cycle fatigue (LCF) properties of Inconel 718 and showed that the precipitates were sheared during cyclic straining, and plastic deformation proceeded by the propagation of planar bands which were, however, identified as twins. However, Oblak and co-workers [15,16] observed numerous dislocation pairs in Inconel 718 deformed to 3% strain at room temperature, and reported that

<sup>\*</sup> Corresponding author. Tel.: +1 204 474 6675; fax: +1 204 261 6735.

shearing of  $\gamma''$  particles appeared to take place by the motion of a/2<110> dislocation pairs, and no evidence of stacking fault mode of deformation was found. Worthem et al. [17] also observed regularly spaced arrays of deformation bands on {111} slip planes during cyclic and monotonic deformation. They found that during cyclic mode of deformation the spacing of planar slip bands was nearly constant after different numbers of cycles at the same cyclic strain amplitude of 1.0%, however, it ranged from 0.80 µm down to 0.25 µm as the strain increased during monotonic deformation. They suggested that shearing of  $\gamma''$  precipitates occurred only during cyclic deformation, and no significant shearing of  $\gamma''$  precipitates was observed after the monotonic deformation. They did not identify any deformation twins either. Therefore, it seems that although planar slip bands in Inconel 718 have been observed by several investigators [12–22], however, conflicting observations on the shearing of  $\gamma''$  precipitates during cyclic and monotonic deformation have been reported. Furthermore, there does not seem to be an agreement on the mechanism of their formation either. Shearing of  $\gamma''$  precipitates during cyclic deformation of Inconel 718 was observed during present investigation, and is suggested to be the mechanism of formation of slip bands. Also, most of the studies of cyclic deformation of Inconel 718 seem to have been done at RT, therefore cyclic deformation was also studied at 650 °C and results are being also reported in this communication.

## 2. Materials and experimental procedures

Wrought Inconel 718 based alloy, with a chemical composition of (wt %) 18.45% Cr, 53.43% Ni, 18.83% Fe, 2.91% Mo, 4.86% Nb, was produced by the normal commercial practice for this investigation by Special Metals Corporation. The cylindrical low-cycle fatigue (LCF) specimens with a gage area of  $\varnothing$  6.35 mm × 10 mm were machined, and heat treated using a commercial heat treatment (CHT) schedule. It involved a solution treatment at 955 °C for one hour and then air cooled to room temperature, followed by aging at 720 °C for eight hours, cooled to 620 °C at a rate of 50 °C/h, and held at 620 °C for a total aging time of eight hours, then air cooled to RT.

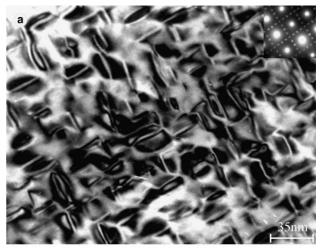
Symmetrical push-pull low cycle fatigue (LCF) tests were carried out under total strain control mode at room temperature (RT) and 650 °C, on a computerized servo-hydraulic Instron testing system, which was interfaced with Fast Track II software. Triangle waveform and a mean strain rate of  $3\times 10^{-3}~\rm s^{-1}$  were used. After fatigue failure, discs were cut from regions adjacent to the fracture surface and perpendicular to the loading axis. Thin foils for transmission electron microscopy (TEM) were prepared from these discs by twin-jet elec-

tro-polishing at a potential of 12 V in a 10% solution of perchloric acid in methanol at a temperature of  $-30 \,^{\circ}\text{C}$ , and examined by a JEOL-2000FX transmission electron microscope.

### 3. Results and discussion

A representative thin-film TEM microstructure of the heat-treated material is shown in Fig. 1a which is in [001] orientation as suggested by its selected area diffraction pattern (SADP), shown in the inset. Secondary reflections in the SADP were identified to be due to  $\gamma''$  and  $\gamma'$  precipitates, and Fig. 1b shows the dark field TEM micrograph obtained with a (010) reflection. These disc-shaped precipitates were estimated to be about 30 nm in diameter and 5 nm in thickness.

Fig. 2, which is a TEM micrograph with [001] orientation, shows two groups of planar slip bands, nearly



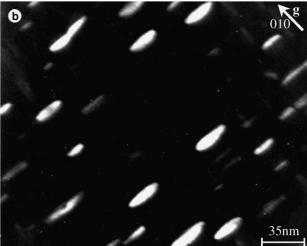


Fig. 1. TEM micrographs of heat treated IN 718 showing the presence of  $\gamma''$  and  $\gamma'$  precipitates: (a) Bright field image and SAD pattern with [001] orientation; (b) Dark field image with [001] orientation with (010) reflection showing  $\gamma''$  and  $\gamma'$  precipitates.

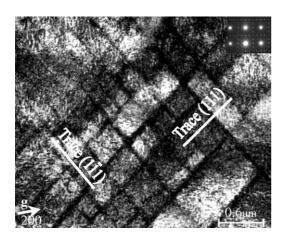


Fig. 2. Two groups of planar slip bands, nearly perpendicular to each other, seen in the specimen deformed at  $\Delta \varepsilon_p/2 = 0.58\%$ , at RT,  $(\tilde{\mathbf{g}} = 200; \text{incident beam} \parallel [0\,0\,1])$ .

perpendicular to each other, in a specimen fatigued to  $\Delta \varepsilon_{\rm p}/2 = 0.58\%$  at RT. The trace analysis indicated that two groups of bands lie on (111) and (111) slip planes, and slip was active simultaneously. Although precipitate particles were not very clearly seen in this specimen, however, the SADP with a [001] orientation, shown in the inset, revealed the presence of superlattice reflections, which are characteristic of  $\gamma''$  and  $\gamma'$  precipitates in age hardened Inconel 718 superalloy [15,16].

TEM microstructural examination of material fatigued at 650 °C revealed that the cyclic deformation at this temperature also occurred by planar slip, and multiple planar slip bands, intersecting on different {111} planes, penetrating the whole grains, were observed. An example is shown in Fig. 3, which is a thin film TEM microstructure of a specimen deformed to  $\Delta \varepsilon_{\rm p}/$ 2 = 0.13%. In the lower of the two grains, which is in [100] orientation with  $\tilde{\mathbf{g}} = 020$ , the traces of slip bands are parallel to the traces of  $(1\bar{1}1)$  and  $(1\bar{1}1)$  planes with (100) thin foil plane. However, the grain shown in the top part of the figure is in [111] orientation, and the trace analysis of the bands suggests them to be on  $(1\bar{1}1)$ ,  $(11\bar{1})$ ,  $(1\bar{1}\bar{1})$  planes which intersect the (111) foil surface. As shown in this figure, twinning was also observed to occur in this alloy. The activation of multiple planar slips is further illustrated in Fig. 4, where the slip bands are again seen to have formed on three primary {111} planes in a specimen with a [111] zone axis. This confirms that cyclical deformation of Inconel 718 at 650 °C also occurs by planar slip on different {111} planes. However, in comparison with the planar slip bands which formed in materials with low SFE or alloys with SRO, the distinguishing features of the planar deformation bands shown in Figs. 2–4 are: (1) an intense slip concentration occurred within the slip bands on all the slip planes, (2) the slip bands penetrated the entire grains, and (3) the multiple groups of slip bands on different slip planes intersected each other.

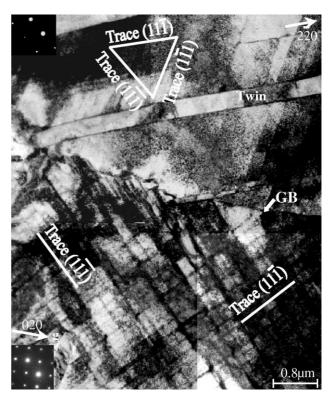


Fig. 3. Groups of planar slip bands observed in a specimen deformed at  $\Delta \varepsilon_p/2 = 0.13\%$  at 650 °C.

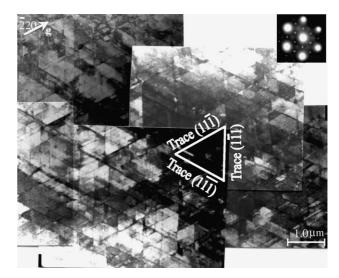


Fig. 4. Three groups of planar slip bands observed in a specimen deformed at  $\Delta \varepsilon_p/2 = 0.13\%$  at 650 °C, ( $\tilde{\mathbf{g}} = \bar{2}20$ ; incident beam || [111]).

The variation in the width and spacing of planar slip bands with cyclic plastic strain amplitude was measured. The values obtained at RT and 650 °C in foils at [100] orientation are shown in Fig. 5. It is seen that, the width and the spacing of planar slip bands remained almost constant as the cyclic plastic strain amplitude increased. The spacing was about 0.70  $\mu$ m, and the width 0.06  $\mu$ m at RT and 650 °C.

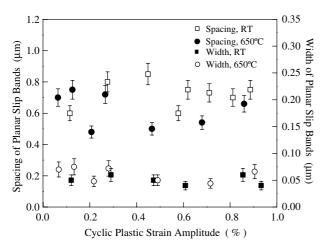


Fig. 5. Variation in the width and spacing of planar slip bands with the cyclic plastic strain amplitude in thin foils, in [001] orientation, of specimens deformed at RT and 650 °C.

The TEM dark field examination of  $\gamma''$  and  $\gamma'$  precipitates in fatigued specimens revealed that they were sheared during cyclic deformation, both at RT and at 650 °C. An example is shown in Fig. 6, which is the dark field micrograph with  $\tilde{\mathbf{g}} = [\bar{1}10]$  reflection in [110] foil in a specimen cyclically deformed to  $\Delta \varepsilon_p/2 = 0.6\%$  at 650 °C. Trace analysis showed that the shearing traces correspond to the intersection of {111} planes with the thin foil normal, indicating that  $\gamma''$  precipitates were sheared by the dislocations moving on the  $(\bar{1}11)$  primary slip planes. By comparing these microstructures (Fig. 6) with those of the non-fatigued specimens (Fig. 1b), it was found qualitatively that cyclic deformation reduced the size of  $\gamma''$  precipitates. A few precipitate-free deformation bands were also observed in the fatigued specimens, as seen in Fig. 7, which is the dark field micrograph taken with (100) superlattice of specimen

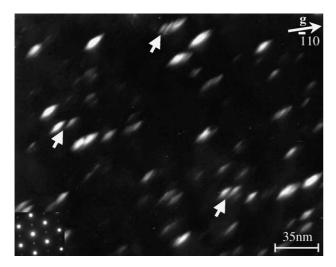


Fig. 6. Dark field micrograph with  $\tilde{\mathbf{g}} = [\tilde{1}10]$  reflection and the foil in [110] orientation, showing sheared precipitates in a specimen deformed at  $\Delta \varepsilon_p/2 = 0.6\%$  at 650 °C.

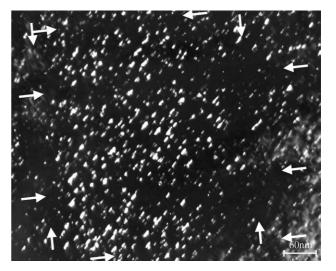


Fig. 7. Dark field micrograph with  $\tilde{\mathbf{g}} = [100]$  reflection and the foil in [001] orientation, showing precipitates-free bands in a specimen deformed at  $\Delta \varepsilon_p/2 = 0.6\%$  at 650 °C.

in (001) orientation. The precipitate-free bands, indicated by arrows, are seen to be parallel to the trace of  $\{111\}$  planes with the foil normal. The formation of precipitate-free planar bands has been reported to be closely related to the sharing of  $\gamma''$  particles by dislocations [22].

Under fully reversed fatigue condition, as the cyclic deformation proceeds, the strengthening precipitates,  $\gamma''$ , present in the slip bands are sheared by the leading dislocations [19]. After this initial cutting of  $\gamma''$ , the trailing dislocations in the same slip plane would shear the  $\gamma''$ precipitates again. The process would be repeated during each cycle and reduce the size of  $\gamma''$  particles to an extent that they offer very little or no resistance to the movement of dislocations. Thus, as the successive shearing of particles by dislocations continues, the stress required to shear the smaller particles is reduced. The preferential paths, which have a lower critical stress for the dislocation slip, would be established during the progressive deformation of the material. As a result, precipitate-free planar bands would be produced, and were observed (Fig. 7).

Long-distance movement of screw dislocations is possible along the slip planes in the first deformation owing to the planar character of slip in this alloy. Dislocation multiplication would continuously generate more dislocations on the same plane. Deformation can occur by a further emission of dislocations from the same sources or by new sources activated on the parallel planes. The width and spacing of planar slip bands were observed to be almost independent of the cyclic plastic strain amplitude (Fig. 5). These results demonstrate that slip was basically concentrated within these slip bands as the cyclic strain amplitude increased.

At the elevated temperature, the tendency to deform by slip along planar bands may be circumvented by thermally activated climb or by cross-slip, resulting in a homogeneous tangle of dislocations. However, planar slip persisted in Inonel 718 fatigued at 650 °C. This could be attributed to the fact that cross-slip is not a viable process for the paired dislocations produced in Inconel 718 by the shearing of coherent and ordered  $\gamma''$  precipitates. When the plastic deformation occurred via the motion of paired dislocations, deformation became localized on one plane on which they were positioned. The presence of coherent and ordered precipitates, therefore, suppressed the cross-slip of dislocations in Inconel at both the temperatures, resulting in planar slip.

### 4. Conclusions

- 1. A strong tendency to form planar deformation bands was observed in IN 718 fatigued at RT as well as at 650 °C. The planar deformation bands consisting of multiple groups of parallel bands formed via the motion of dislocations on {111} planes. The width and spacing of planar slip bands at both the temperatures were almost the same in specimens fatigued at different cyclic strain amplitudes.
- 2. The formation of planar slip bands is attributed to the shearing of coherent and ordered  $\gamma''$  and  $\gamma'$  precipitates by pairs of dislocations moving on the primary slip planes, which did not cross-slip during cyclic deformation. After the initial cutting of  $\gamma''$ , the trailing dislocations on the same slip plane repeatedly sheared the  $\gamma''$  precipitates, and the continued cyclic deformation reduced their size to such an extent that they offered very little or no resistance to the movement of dislocations, resulting in the formation of the precipitate-free planar bands.

# Acknowledgement

The authors would like to thank the Natural Sciences and Engineering Research Council of Canada and the consortium of Manitoba aerospace industries for the financial support.

#### References

- [1] Inui H, Hong SI, Laird C. Acta Metall Mater 1990;38:2261.
- [2] Laird C, Stanzl S, DeLaVeaux R, Buchinger L. Mater Sci Eng 1986;80:143.
- [3] Lukas P, Kunz L, Krejci J. Mater Sci Eng A 1992;158:177.
- [4] Li YF, Laird C. Mater Sci Eng A 1994;186:65.
- [5] Xiao L, Kuang ZB. Acta Mater 1996;44:3059.
- [6] Xiao L, Umakoshi Y. Phil Mag 2003;83:3407.
- [7] Xiao L, Umakoshi Y. Phil Mag A 2002;82:2379.
- [8] Gerold V, Karnthaler HP. Acta Metall 1989;37:2177.
- [9] Jouiad M, Clement N, Coujou A. Phil Mag A 1998;77:689.
- [10] Heino S, Karlsson B. Acta Mater 2001;49:353.
- [11] Steffens Th, Schwink Ch, Korner A, Karnthaler HP. Phil Mag A 1987;56:161.
- [12] Merrick HF. Metall Trans A 1976;7A:505.
- [13] Merrick HF. Metall Trans 1974;5:891.
- [14] Clavel M, Pineau A. Metall Trans A 1978;9:471.
- [15] Oblak JM, Paulonis DF, Duvall DS. Metall Trans 1974;5: 143.
- [16] Paulonis DF, Oblak JM, Duvall DS. Trans ASM 1969;62:611.
- [17] Worthem DW, Robertson IM, Leckie FA, Socie DF, Altstetter CJ. Metall Trans A 1990;21:3215.
- [18] Kalluri S, Rao KBS, Halford GR, McGaw MA. Superalloys 718, 625, 706 and Various Derivatives. The Minerals Metals and Materials Society; 1994. p. 593.
- [19] Sundararaman M, Mukhopadhyay P, Banerjee S. Acta Metall 1988;36:847.
- [20] Xiao L, Chen DL, Chaturvedi MC. Metall Mater Trans A 2004;35:347.
- [21] Sundaraman M, Kishore R, Mukhopadyay P. Metall Mater Trans A 1994;25:653.
- [22] He J, Fukuyama S, Yokogawa K. Mater Sci Tech 1995;11:916.