THE DEVELOPMENT OF γ/γ' INTERFACIAL DISLOCATION NETWORKS DURING CREEP IN NI-BASE SUPERALLOYS

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Abstract

Differences in lattice parameter between the γ and γ' phases in Ni base superalloys lead to the formation of interfacial networks of edge dislocations upon thermal exposure. These networks accommodate the lattice mismatch between the two phases. In this paper, the development of these lattice mismatch networks during creep is considered. The mismatch nets develop as a result of local reactions between creep dislocations gliding on stressed octahedral slip systems. No long range climb or Orowan looping of precipitates is required. Models which demonstrate the local reactions and the resultant nodal networks are given. It is concluded that variations in the observed networks are the result of combinations of these reactions, which are dependent upon the local dislocation content in the γ' channels. Examples of both equilibrium and transitional interfacial dislocation arrangements from interrupted creep specimens are presented.

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Introduction

Ni base superalloys possess high temperature strengths which far exceed those of the individual constituents (ie. γ and γ' phases). Of considerable importance in determining the mechanical properties of this class of alloys is the γ/γ' interface. The nature of this interface, as well as the morphology of the γ' phase, is strongly influenced by the difference in lattice parameter between the two phases: the lattice mismatch.

The difference in lattice parameter between the γ matrix and coherent γ' precipitates in a Ni base superalloy creates very high misfit stresses. During high temperature exposure, the misfit can be relieved by the formation of networks of edge dislocations at the interfaces between the two phases. The dislocations arrange themselves such that their extra half planes are in the phase with the smaller lattice parameter, thus accommodating the lattice parameter mismatch between the phases. Larger mismatch alloys require more closely spaced dislocations to relieve the mismatch, and measurement of the dislocation spacing in the nets can yield a measure of the lattice mismatch [1]. These networks generally form concurrently with the rafted γ' structure. It is believed that the presence of these networks, like rafting in general, can either be beneficial (acting as barriers to dislocation motion) or detrimental (serving as dislocation sources for γ' cutting) to creep strength, depending on the alloy and the stress and temperature conditions [2].

The dislocations within the nets are thought to originate from slipping matrix dislocations captured on the interface. However, the line directions of dislocations deposited on the interfaces during slip through the "channels" of γ phase are not in the proper orientation to efficiently relieve the mismatch. As dislocations move from "slip" to "mismatch" orientations, there is a reduction in misfit energy as well as a considerable decrease in dislocation line energy, due to the more efficient accommodation of misfit [3]. Thus, the line directions of the dislocation segments at the interfaces give an indication of how well established the mismatch accommodating networks have become in the specimen. The "slip" and "mismatch" orientations for dislocations slipping on $\{111\}$ planes in a $[001]$ oriented specimen are given in Table I.

The nature of these nets is well established [1,4], but there is still some question as to how they form from the slip deposited dislocations. One mechanism which has been proposed involves the formation of Orowan loops around the γ' particles [5]. These loops must subsequently rotate by 90° along the γ/γ' interface, by a combination of glide and climb, in order to orient themselves to most efficiently relieve the mismatch strains. There are three major problems with this mechanism. First, it involves a considerable amount of movement of large dislocation loops. Second, actual studies of dislocation arrangements during the early stages of creep in high volume fraction γ' alloys indicate that the dislocations do not tend to form Orowan loops around individual γ' particles, but travel long distances through the γ channels [3]. Third, this mechanism does not account for the presence in the nets of dislocations with Burgers vectors such as $1/2[110]$ and $1/2[\bar{1}10]$, which would not be expected to be slipping in an $[001]$ oriented specimen. This last objection has been addressed by considering reactions between the dislocations [6]. For example, the reaction: $1/2[10\bar{1}] + 1/2[0\bar{1}1] \rightarrow 1/2[1\bar{1}0]$ results in a reduction in $|b|$ from a^2 to $1/2a^2$ (where a is the lattice parameter), and thus would be energetically favorable. However, the mechanism for the realignment of slip oriented dislocations into their mismatch orientations is still thought to involve long range slip/climb of large loops.

Table I: Comparison of slip deposited and mismatch accommodating dislocation line directions on (001) γ/γ' interfaces for a [001] oriented creep specimen.

<u>Burgers Vector</u>	<u>Slip/Mismatch Plane*</u>	<u>Line Direction</u>
011	$\bar{1}\bar{1}1$	$\bar{1}\bar{1}0$
011	$\bar{1}\bar{1}1$	$\bar{1}10$
011	001_m	100
$01\bar{1}$	$\bar{1}11$	$\bar{1}\bar{1}0$
$01\bar{1}$	111	$\bar{1}10$
$01\bar{1}$	001_m	100
101	$\bar{1}11$	110
101	$\bar{1}\bar{1}1$	$\bar{1}10$
101	001_m	010
$10\bar{1}$	111	$\bar{1}10$
$10\bar{1}$	$\bar{1}\bar{1}1$	110
$10\bar{1}$	001_m	010

* The subscript "m" indicates a mismatch orientation

All of the difficulties described above can be overcome if the nets are assumed to form by nodal reactions between slip deposited dislocations and localized rearrangements. In this paper, the formation of rafted γ' precipitates with fully developed interfacial networks during high temperature creep will be discussed. Dislocation reactions which can result in the formation of the equilibrium mismatch network configuration will be described. In addition, results from TEM investigations of interrupted creep specimens for several different single crystal superalloys will be presented which illustrate both fully developed and transitional interfacial dislocation networks.

Experimental Procedure

Specimens of several different directionally solidified and single crystal alloys were tested in tensile creep in the [001] orientation and interrupted at various stages of creep life for TEM examination. All of the alloys were high γ' volume fraction compositions. Lattice mismatch values were determined by measuring dislocation network spacings established during over aging at 1080°C. This technique is described elsewhere [1]. All of the alloys displayed similar values of lattice mismatch ($\delta=(a_{\gamma'}-a_{\gamma})/a_{\gamma}$), between -0.2 and -0.3%.

TEM specimens were cut with foil normals parallel to the stress direction (ie. transverse cross-sections) and thinned using standard electrochemical techniques. The formation of interfacial networks was concurrent with rafting in the specimens; therefore, networks lying on (001) planes (perpendicular to the stress axis) were chosen, and line directions determined from micrographs taken with [001] beam directions. Due to the numerous short segments of dislocations comprising the networks, detailed line direction analyses of individual segments were not performed. Burgers vector analysis was performed using the double extinction invisibility criterion, combined with analysis of nodes in the networks.

Results and Discussion

Models for Mismatch Network Formation

An example of a process by which the equilibrium mismatch network configuration can be developed by reactions between slip oriented dislocations is shown in Figure 1. In this example, two perpendicular sets of $1/2[0\bar{1}1]$ dislocations (Figure 1a), one resulting from slip on (111) and the other from slip on $(\bar{1}\bar{1}1)$, react to form a single set with an average line direction of $[100]$, as shown in Figure 1b. In this manner, the "mismatch" configuration is achieved without long range climb and/or glide. The addition of $1/2[10\bar{1}]$ dislocations, resulting from slip on the $(1\bar{1}1)$ plane, and subsequent reactions result in a net containing $1/2[0\bar{1}1]$, $1/2[1\bar{1}0]$, and $1/2[10\bar{1}]$ dislocations, all oriented in optimum mismatch accommodating configurations (Figures 1c and d). $1/2[101]$ dislocations, resulting from slip on the $(\bar{1}\bar{1}1)$ plane may also be added and knitted into the net, as shown in Figures 1e and f. Subsequent reactions of the type: $1/2[101] - 1/2[0\bar{1}1] \rightarrow 1/2[110]$ and $1/2[101] - 1/2[1\bar{1}0] \rightarrow 1/2[011]$ result in the net shown in Figure 1g, the equilibrium mismatch configuration. Alternatively, the $1/2[101]$ and $1/2[10\bar{1}]$ dislocations can combine to form the $[100]$ Burgers vector, as shown in Figure 1h. Further dissociation of the $[100]$ segments once again yields the standard (001) net.

Figure 1) Schematic representation of dislocation reactions leading to the formation of mismatch accommodating nets on a (001) γ/γ' interface during creep. Line directions and Burgers vectors are marked on the figures. Crystallographic directions are shown b). See text for details.

The formation of the mismatch nets may also be aided by the extensive cross-slip known to occur in these alloys. The "zig-zag" morphology of Figure 1a and b, for example, could result from cross-slipping of a single set of $1/2[0\bar{1}1]$ dislocations. Another example is shown in Figure 2. Here, cross-slipping dislocations with several different Burgers vectors are deposited on the γ/γ' interface. Diffusional rounding of the corners at the cross-slip locations then occurs, along with climb of dislocation segments along the interface and reactions between these segments to form the mismatch network.

Figure 2) Schematic representation of an alternate mechanism for mismatch net formation on γ/γ' interfaces. Corners on cross slipped dislocations are rounded through diffusional processes, followed by nodal reactions similar to those described in Figure 1.

Many permutations of these basic themes are possible, all of which rely only on local dislocation rearrangements within the nets, without the necessity of long range climb and/or glide around the interfaces. Of course, the actual sequence of dislocation knitting into the nets will vary within the interface according to the local dislocation content, resulting in many different intermediate configurations within a given net. This is particularly true for creep specimens, in which dislocations continue to enter and leave the nets, so that the steady state configuration will deviate from that of the unstressed condition. Also, the scale of these interactions will not necessarily be that of the equilibrium mismatch spacing, particularly early in creep, when the dislocation densities in the interfaces are low. At this stage, longer climb/glide distances will be required, as governed by the actual dislocation spacing in the interface.

Experimental Results

An example of extensive cross slip resulting in a "zig-zag" morphology in slip deposited dislocations is shown in Figure 3. This specimen was interrupted after being creep tested for only two minutes at 825°C , resulting in negligible strain, and illustrates the initial movement of dislocations through the γ matrix. Note that, rather than the formation of Orowan loops around

individual γ' particles, the dislocations extend long distances in the specimen through the γ channels.

Figure 3) TEM micrograph showing initial dislocation movement within the γ matrix after creep testing for two minutes at 825°C. (reference 3)

The specimen shown in Figure 4 was tested to 0.4% creep strain (235 hours, ~10% expected life) at 982°C. Several doubled $b=1/2[011]$ dislocations, with line directions approximately parallel to $[\bar{1}10]$ (thus lying on $(\bar{1}\bar{1}1)$ slip planes) are visible in the micrograph. Reverse g experiments showed them to have the same sign Burgers vector. The fact that they are doubled implies that they have cut through the γ' precipitates, even though they are in the γ phase in this micrograph. Dislocations like these were common in this specimen, as well as dislocations within the γ' , indicating that considerable cutting of dislocations through the γ' is occurring. The γ' has rafted in this specimen; however, the interfacial networks are not fully developed, and many dislocations are still in slip orientations. An example of a dislocation with both mismatch

Figure 4) Weak Beam TEM micrograph of an interrupted creep specimen tested to 0.4% creep strain (235 hours, ~10% expected life) at 982°C, showing incomplete mismatch networks with some slip oriented dislocations within the nets (example shown with arrows is a $b=[011]$ dislocation in both mismatch (m) and slip (s) orientations) as well as paired $b=1/2[011]$ dislocations in slip orientation (line direction = $[\bar{1}\bar{1}0]$) within the γ matrix. (transverse cross-section)

and slip oriented segments is shown in the micrograph. This configuration appears to have resulted from cross slip within the γ channel, as depicted in Figure 2. The combination of a rafted microstructure with poorly developed interfacial nets may be responsible for the extensive γ' cutting observed in this specimen. It is difficult for dislocations to loop around the rafts, and the networks, which can act as barriers to dislocation movement through the γ' , are not yet fully established.

Figure 5) TEM micrograph (a) and accompanying schematics (b and c) of interrupted creep specimen tested to 400 hours ($\sim 10\%$ expected life) at 982°C . showing dislocation networks on the γ/γ' interfaces (overlay of two micrographs to show all of the dislocations within the net, some double images of dislocations appear due to tilt between images). Most of the dislocations in the upper left are slip oriented (schematic in b), while the dislocations in the lower right hand corner of the micrograph are forming a distorted mismatch network (schematic in c). The arrows provide common reference points in the micrograph and schematics. All $\langle 110 \rangle$ Burgers vectors have a magnitude of $1/2\langle 110 \rangle$. (transverse cross-section)

Figure 5 shows a transverse cross section of a specimen tested to 400 hours (~10% expected life) at 982°C. The interfacial dislocation nets in this specimen are in distorted mismatch configurations in some areas; however, a large number of the interfacial dislocations are in slip orientations as seen in the upper left corner of the micrograph. Several slip oriented dislocations are seen entering into a distorted mismatch network, demonstrating the transition from slip deposited dislocation structures to mismatch accommodating networks. For example, many of the $\mathbf{b}=1/2[10\bar{1}]$ dislocations in the lower right region of the micrograph (and depicted schematically in 5c) are in slip orientation outside of the mismatch net and curve into the mismatch orientation as they enter the net.

As illustrated in Figure 1, $\mathbf{b}=\langle 100 \rangle$ dislocations can also accommodate mismatch within the networks. The reaction: $1/2[101] + 1/2[10\bar{1}] \rightarrow [100]$, depicted in transforming between the network of Figure 1g to that shown in 1h, involves no change in the value of $\mathbf{b}^2$, and thus is not expected to result in a significant change in total line energy. A few examples of $\mathbf{b}=\langle 100 \rangle$ dislocations are found in Figure 5.

An example of an interfacial dislocation network in which all of the line segments are close to their mismatch orientations is shown in Figure 6. This TEM micrograph was taken from an interrupted creep specimen tested to 0.5% creep strain (10 hours, ~5% expected life) at 1093°C. This net is a somewhat distorted version of the equilibrium mismatch configuration. Comparison with Figure 1 reveals that many of the dislocations possessing Burgers vectors capable of slip (eg. $1/2[10\bar{1}]$) are bowed away from their ideal mismatch accommodating line directions towards

Figure 6) TEM micrograph (a) and accompanying schematic (b) of an interrupted creep specimen tested to 0.5% creep strain (10 hours, ~5% expected life) at 1093°C, showing dislocation networks on the γ/γ' interfaces. Area of schematic is marked in the micrograph (overlay of two micrographs to show all of the dislocations within the net, some double images of dislocations appear due to tilt between images). (transverse cross-section)

slip orientations. Thus, dislocations within the networks are responding to the applied stress. Note that the $1/2[110]$ and $1/2[1\bar{1}0]$ dislocations, which have no resolved stress for slip in this specimen, tend to be relatively straight and in their mismatch orientations.

In Figure 7, an example of a well established mismatch network is shown. This specimen was interrupted after 0.5% creep strain (20 hours, ~5% expected life) at 1093°C. The arrangements of dislocations in the ideal mismatch network shown in Figures 1 and 2 can be easily recognized in this micrograph, although some distortion is still present. As in the previous example, the $1/2[110]$ and $1/2[1\bar{1}0]$ dislocations, which have no resolved stress for slip in this specimen, tend to be relatively straight and in their mismatch orientations, while dislocations having other Burgers vectors tend to be slightly bowed. A few $b=\langle 100 \rangle$ can also be seen in this network.

Figure 7) TEM micrograph (a) and accompanying schematic (b) of an interrupted creep specimen tested to 0.5% creep strain (20 hours, ~5% expected life) at 1093°C, showing dislocation networks on the γ/γ' interfaces. Arrows in (a) and (b) show equivalent positions. (overlay of two micrographs to show all of the dislocations within the net, some double images of dislocations appear due to tilt between images). (transverse cross-section)

The examples presented above show the evolution of the interfacial network structure during creep. Dislocations deposited on the γ/γ' interfaces are in slip oriented segments; however, extensive cross slip results in an average line directions closer to those required for the most efficient accommodation of lattice mismatch. In addition, local reactions between deposited dislocations can lead to properly oriented mismatch networks. The observations of loose nets of slip oriented dislocations and dislocations bending from slip to mismatch orientations as they enter mismatch nets are consistent with this model. The previous model, involving the slip/climb rotation of individual loops by 90° around the γ' precipitates, would be expected to result in long segments of dislocations in intermediate orientations (between slip and mismatch line directions). These were not observed.

Areas of predominantly slip oriented dislocations and other areas of reasonably well developed mismatch networks exist in the same specimen, as in Figure 5 where two such regions are in close proximity. Thus, slip oriented dislocations do not necessarily begin to reorient themselves

immediately after being deposited on the interface. This indicates that a cooperative transformation may occur in a relatively short time. A critical density of interfacial dislocations, with the proper Burgers vectors, may be necessary to initiate the interactions and rearrangements required to form the mismatch nets. Once these criteria are met, the transformation may proceed rapidly.

Once the mismatch nets are formed in a stressed material, their maintenance is a dynamic process, involving the knitting of slipping dislocations into, and out of, the networks. The bowing of dislocations with Burger vectors capable of slip, observed in the micrographs, may be a precursor to the process by which these dislocations escape from the nets. However, clear examples of dislocations actually entering and leaving the nets were not observed. This process probably occurs very rapidly in these specimens, making the probability of direct observation of such an event in an interrupted creep specimen rather low.

Conclusion

In this paper, we have suggested that the formation of interfacial mismatch dislocation networks in Ni base superalloys occurs by local reactions between line segments of slip oriented dislocations, as opposed to the previous model involving long range slip/climb of individual Orowan loops. Experimental observations are consistent with this mechanism.

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