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The recrystallization of nickel-base superalloys

A. PORTER, B. RALPH
Department of Metallurgy and Materials Science, University of Cambridge, Cambridge, UK

The effects of recrystallization on the γ' distribution in four nickel-base superalloys of varying γ' volume fraction (Nimonics PE16, 80A and 115, and Udimet 720) have been studied by transmission electron microscopy. These effects are explained in terms of high solubility and diffusivity in the recrystallization interface, and it is suggested that high diffusivity assumes greater importance as the amount of solute dissolved in the boundary increases. Some attention is given to the nucleation of recrystallization. It is shown that in one of the alloys (Udimet 720), nucleation at grain boundaries involves subgrain coalescence. Subsequent growth of the nucleus occurs by strain-induced boundary migration.

1. Introduction

Although recrystallization of multiphase materials is an important metallurgical process, most work to date has concentrated on relatively simple alloys. Extensive investigations into the recrystallization of materials with a low volume fraction of a second phase have been performed; but little is known about the effects of a second phase present to more than a few per cent (e.g. [1]).

Nickel-base superalloys are good examples of technologically important multiphase materials. The ordered intermetallic, γ', may be present to a volume fraction of between 0.05 and 0.7, and distributions of, for example, carbides and nitrides are also incorporated into the microstructure. The complexity of superalloys has meant that little investigation into their recrystallization has been performed. In addition, few studies to date have attempted to relate the results obtained to the mechanisms of recrystallization which have been proposed for simpler systems.

One approach to the study of superalloys has been to use model systems to investigate certain features of the recrystallization behaviour of commercial materials. Such a method has been used with Ni–Al binaries [2, 3] and the Ni–Cr–Al ternary system [4] to investigate the effects of recrystallization on γ', without the influence of other distributions. Other published work has tended to adopt a more technological approach involving phenomenological observation of commercial materials. Such studies (e.g. [5–9]) have investigated single alloys, with no attempts to establish the general features of recrystallization in a range of materials. Secondary recrystallization, important to dispersion-strengthened materials, has also been examined (e.g. [10–12]), but again these investigations have involved only one material. A recent review summarizes these studies [13].

This present paper results from work which attempts to move away from the "single-alloy" approach by investigating more than one material. Four wrought nickel-base superalloys have been studied, in order to examine the influence on the γ' distribution of the passage of a recrystallization interface, in superalloys of varying γ' volume fraction. In addition, some attention has been given to the mechanism by which grain-boundary nucleation of recrystallization occurs in one of the high γ' alloys.

2. Experimental procedure

The four nickel-base superalloys used in this investigation are all commercially available (for details see, for example, [14]). In the fully heat-treated state, Nimonics* PE16, 80A and 115, and Udimet* 720 have γ' volume fractions of approximately 0.08, 0.15, 0.5 and 0.6 respectively. The

*Nimonic and Udimet are trademarks.

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TABLE I The initial and recrystallization treatments received by the alloys

Alloy	Initial heat treatment	Cold roll (% reduction)	Recrystallization annealing T(K)	γ' solvus T(K) (e.g. [14])
Nimonic PE16	4 h/1313 K/AC* (solution treatment) + 1 h/1173 K/AC + 8 h/1023 K/AC	50	1123	1153–1173
Nimonic 80A	8 h/1353 K/AC (solution treatment) + 16 h/973 K/AC	50	1193	1233–1253
Nimonic 115	1.5 h/1463 K/AC (solution treatment) + 6 h/1373 K/AC	50	1373	1423
Udimet 720	4 h/1443 K/AC (solution treatment) 4 h/1353 K/AC 24 h/1116 K/AC 16 h/1033 K/AC	30 (the maximum reduction which could be obtained by cold rolling)	1373	1423

*AC = air cooled.

recrystallization treatments received by the alloys are summarized in Table I. They were given the heat treatments recommended for commercial use (e.g. [14]), deformed by rolling, and given partial recrystallization anneals (typically 600 sec) at approximately 50 K below the γ' solvus. The specimens were water quenched from the recrystallization temperature.

Specimens were electrolytically etched for light microscopy in a solution of 10% phosphoric acid in water, at 3 V. Thin foils for transmission electron microscopy (TEM) were produced by standard twin-jet polishing techniques. The solution used was 3% perchloric acid in 2-butoxyethanol.

3. Results

3.1. Nimonic PE16

The initial heat treatment results in the precipitation of a uniform distribution of small spherical γ' particles, typically 10–15 nm in diameter. These particles are cube–cube related with the matrix.

A partial recrystallization treatment (typically 600 sec at 1123 K) reveals two basic facts: γ' particles may still be imaged by TEM in the unrecrystallized regions of the specimen but the recrystallized areas contain no such particles, and selected area diffraction patterns contain no superlattice reflections in specimens quenched from the recrystallization temperature. The fact that the γ' particles are still visible in the unrecrystallized areas means that dissolution of γ' occurs during recrystallization, rather than as a result of deformation.

Fig. 1 is a bright-field image of a partially recrystallized sample, which although clear of γ' precipitates of imageable size has a mottled appearance which may be attributed to the early stages of γ' reprecipitation.

3.2. Nimonic 80A

The initial microstructure of Nimonic 80A is very similar to that of Nimonic PE16, except for a higher volume fraction of γ'. As with PE16, there is only one size distribution of γ', of the order of 20 nm diameter, produced during the initial ageing. The effects of partial recrystallization on the microstructure of 80A are recorded in Fig. 2. This shows that γ' is present in the recrystallized regions of this alloy, but that the size of these particles falls as the recrystallization interface is approached. The γ' particles are found to be cube–cube related with the matrix which sur-

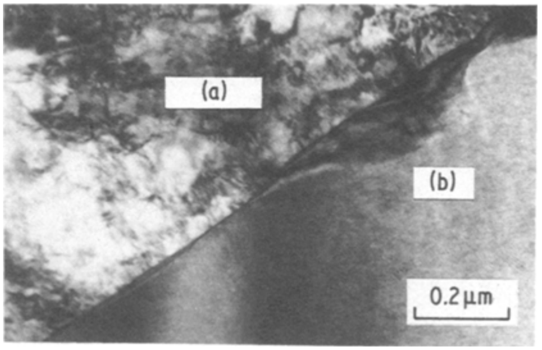


Figure 1 TEM bright-field image of partially recrystallized Nimonic PE16. No γ' particles can be imaged in the recrystallized region (b), although the mottled appearance of the γ-phase may be attributable to the early stages of γ' re-precipitation.

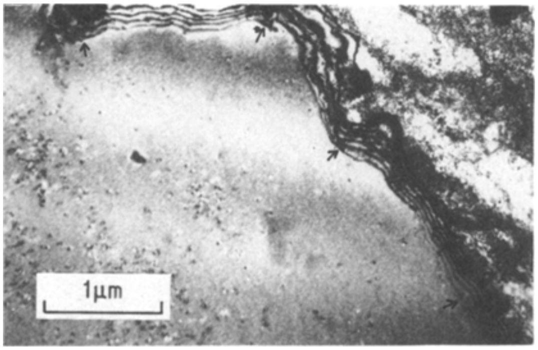


Figure 2 Bright-field image of partially recrystallized Nimonic 80A. The recrystallization interface is marked. The passage of the recrystallization interface dissolves all the γ' particles, and they reprecipitate immediately behind it.

rounds them, before and after recrystallization. The obvious explanation of these observations is that the recrystallization interface is responsible for the complete dissolution of γ', which then reprecipitates immediately behind the moving boundary, and coarsens with time at the recrystallization temperature.

3.3. Nimonic 115

This alloy has a more complex microstructure than the first two, and also has a much higher volume fraction of γ'. Its standard heat treatment (Table I) results in a tri-modal distribution of γ', as shown in Fig. 3a. The large γ' particles are formed on cooling after the first stage of the heat treatment, and coarsen to their final size (about 0.5 μm diameter) during the second part of the process. The secondary precipitates form and grow during the second stage of the heat treatment; those which form near to primary γ' particles are smaller than those elsewhere, because of the depletion of γ'-forming elements in these regions. Finally, on cooling from the second stage of the ageing treatment, very fine tertiary γ' particles precipitate from the supersaturated solid solution. As with the previous two alloys, the γ' in Nimonic 115 is cube–cube related with the matrix, and even the large primary particles are fully coherent.

The changes occurring on recrystallization, however, are different from those in the previous two alloys. TEM studies reveal that the tri-modal distribution of γ' has been replaced after recrystallization by much larger and less regular primary γ' (Fig. 3b), still cube–cube related with the surrounding matrix, and fine γ' which precipitates on cooling. The large γ' particles are partially coherent with the matrix. Fig. 3c demonstrates that these irregular γ' particles are produced by discontinuous precipitation at the recrystallization interface.

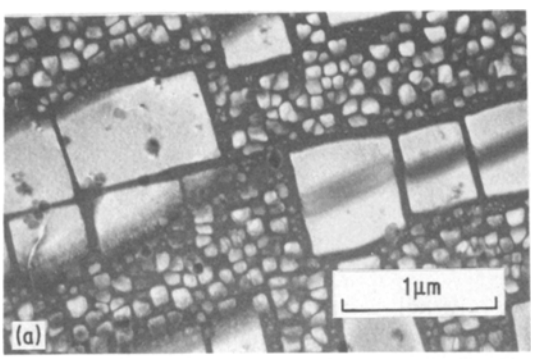
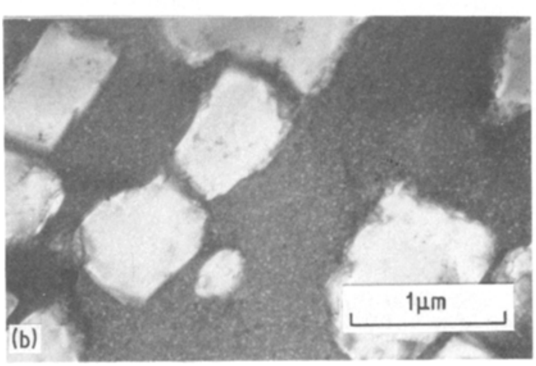
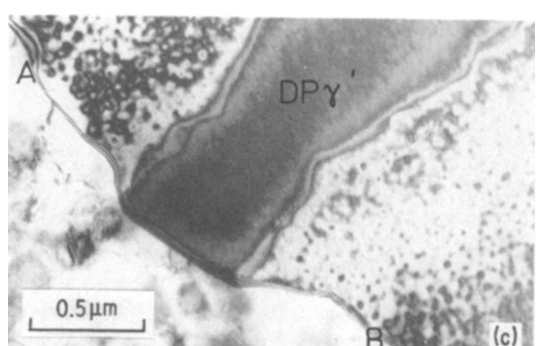


Figure 3 (a) γ' dark-field image of Nimonic 115, after the heat treatment recommended for commercial use (Table I). (b) γ' dark-field image of recrystallized Nimonic 115. Recrystallization results in the discontinuous precipitation of large, irregular, primary γ' particles. Fine γ' particles precipitated on cooling are also visible. (c) Production of a discontinuous precipitate of γ' (marked DP γ') at the recrystallization interface (AB) in 115.





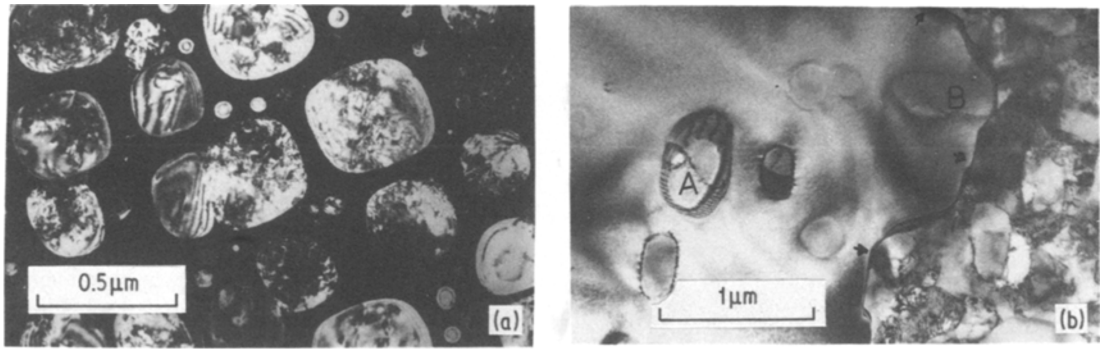


Figure 4 (a) γ' dark-field image taken from a γ' extraction replica of Udimet 720, after the initial heat treatment described in Table I. (b) The early stages of recrystallization in Udimet 720 (bright-field image). The γ' particle (A), with dislocation arrays at its interface with the matrix, is 2–3° misoriented from the cube–cube relationship. Recrystallization has proceeded beyond the nucleation stage, as evidenced by the migration of the high-angle boundary (arrowed). The early stages of discontinuous precipitation of γ' associated with this migration (e.g. at B) can be observed.

3.4. Udimet 720

This material has the highest γ' fraction of the four materials studied. Its heat treatment results in large primary γ' particles, up to 0.5 μm in diameter, which are approximately spherical (implying very low γ/γ' mismatch). Fig. 4a shows a γ' dark-field image of Udimet 720 in this state.

The effects of recrystallization on the γ' dispersion are similar in this material to those in Nimonic 115. There is, however, one difference; at the early stages of recrystallization, some γ' particles are found in recrystallized areas, which are up to 3–4° misoriented from the cube–cube relationship with the matrix, and which are of similar shape and morphology to the γ' in the starting material. Examples of such particles are shown in Fig. 4b, and their origin is discussed in Section 4.2.

4. Discussion

4.1. The influence of recrystallization on γ'

It has previously been demonstrated that diffusion along, and solubility in, moving grain boundaries is high compared with stationary interfaces (e.g. [15, 16]). It is clear that such enhanced diffusivity and solubility are responsible for the observations reported above.

In Nimonic PE16, high solubility appears to be the important property associated with the recrystallization interface. Recrystallization causes complete dissolution of γ', and reprecipitation is slow to occur. Thus it may be said that the boundary moves at a rate at which it can dissolve γ' and then leave solute behind, distributed throughout the matrix. The first stages of homogeneous renucleation of γ' are later observed by TEM to be associated with mottled contrast in the matrix. Nimonic 80A behaves similarly to PE16, except that the higher fraction of γ'-forming elements causes the high diffusivity of the recrystallization interface to attain some importance. Renucleation of γ', either at or immediately behind the recrystallization interface, is rapid. The implication of this is that the moving boundary dissolves γ', and then leaves solute behind in locally supersaturated regions, equivalent to disordered γ'. As well as leaving all the elements for γ' renucleation, the moving boundary leaves enough vacancies for the γ' nucleation barrier to be removed, and rapid reprecipitation of γ' takes place. This reprecipitation and growth of γ' behind the recrystallization interface is similar to previous observations in a Ni–Al binary system [3].

In the two high γ' fraction alloys, the γ' distribution in the cold-rolled material is essentially similar (though "pancaked") to that which results from the prior heat treatment. The microstructural changes found in this investigation are, again, directly attributable to the high solubility, and, more importantly, high diffusivity, associated with a moving boundary. In this case, the recrystallization interface takes nearly all the γ' it encounters into solution. However, the high fraction of γ'-forming elements means that the boundary quickly becomes supersaturated with solute, and this supersaturation is relieved by the production of large, discontinuous γ' particles. Quantification of the changes in γ' distribution occurring during recrystallization in Nimonic 115 has been made [17]. The site at which a dis-

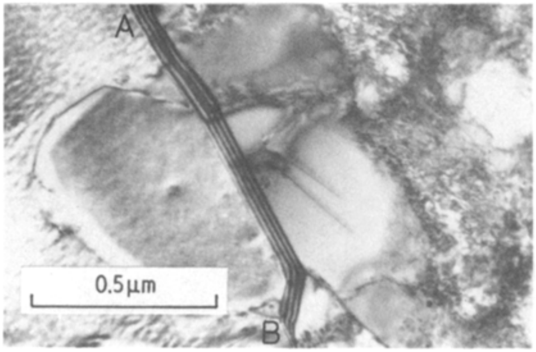


Figure 5 "Cut-through" of a γ' particle at the recrystallization interface (AB) in Nimonic 115, to form the early stages of a discontinuous γ' precipitate. The recrystallization interface was moving from left to right.

continuous precipitate of γ' nucleates may be determined by the "cut-through" of a primary precipitate by the recrystallization interface. Such a situation is shown in Fig. 5, where the moving boundary is changing the orientation of a γ' particle in an equivalent way to the change of orientation of the matrix. In terms of the previous discussion, this means that the high local solute concentration associated with a large dissolving γ' particle provides sufficient supersaturation for the immediate nucleation of a discontinuous γ' precipitate, before substantial diffusion of solute along the boundary to other precipitation sites occurs. Growth of such a precipitate occurs by the feeding of solute, from other dissolved γ' particles, along the recrystallization interface.

High solubility and diffusivity in moving interfaces may therefore account for all the changes in γ' distribution found in the four superalloys investigated. However, it is also clear that these two properties are of varying importance as the γ' fraction of the material is changed. In the lowest γ' alloy (Nimonic PE16), high solubility is the feature of overriding importance. In Nimonic 80A, high solubility and diffusivity are both important in the production behind the interface of local regions supersaturated with γ'-forming elements. The discontinuous precipitation found in the highest γ' fraction superalloys is strongly dependent on the high diffusivity of the moving boundary. Thus, in broad terms, while high solubility and diffusivity in a moving boundary are both important, it may be said that high diffusivity is of increasing relative importance as the amount of solute dissolved in the interface increases.

Interfacial coarsening, where large γ' particles coarsen during recrystallization without orientation change, has been reported by other workers [9]. It has not been observed in the superalloys examined in this investigation, and this may be attributed to the relatively uniform primary γ' size produced during the heat treatment of wrought materials. Interfacial coarsening has only been found to occur during the recrystallization of powder-produced superalloys, where the γ' size distribution is broader. It may therefore be suggested that for interfacial coarsening to occur, a γ' particle must be large compared with its neighbours, and too large to be dissolved by the recrystallization interface.

4.2. The nucleation of recrystallization in Udimet 720

The γ' particles misoriented by several degrees from the cube–cube relationship with the matrix, after the early stages of recrystallization of Udimet 720, offer evidence as to the process by which recrystallization is nucleated. In general, in wrought superalloys, nucleation occurs in the vicinity of grain boundaries, although large intragranular carbides may also act as nucleation sites [17]. In the case of Udimet 720, grain boundary nucleation is observed, and the misoriented γ' particles suggest that this nucleation occurs by a subgrain coalescence process involving subgrain rotation.

The subgrain coalescence model for the nucleation of recrystallization was first proposed by Li [18]. He suggested that adjacent subgrains rotate into the same orientation, and then coalesce. Eventually, the boundary angle of the subgrain increases to a value where a mobile recrystallization interface is formed. The present trend is to consider a composite theory, in which a high-angle boundary at which a subgrain has formed (by growth or coalescence) migrates by strain-induced boundary migration to produce the recrystallization nucleus (e.g. [19]).

When examined prior to the onset of recrystallization, deformed Udimet 720 is observed to contain well-recovered arrays of dislocations in the matrix, while relatively few dislocations are found within the γ' particles and at the γ/γ' interfaces. The γ' particles are of the right size and distribution to define the subgrain size in the γ, and the formation of a viable recrystallization nucleus involves the coalescence of some of these γ subgrains. The low dislocation density within the γ' particles means that nucleation of recrys-

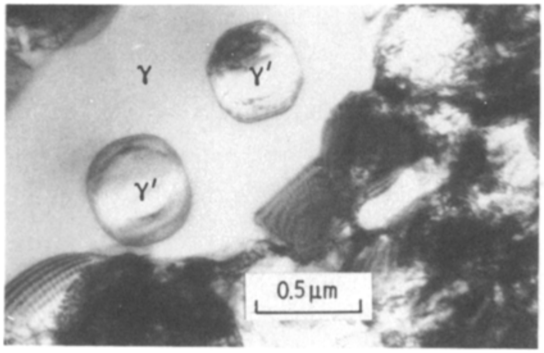


Figure 6 Bright-field image of nucleation of recrystallization in the vicinity of a grain boundary in Udimet 720. A large, dislocation-free γ subgrain is indicated, and the γ' particles within it are slightly misoriented from the cube–cube orientation relationship with the matrix.

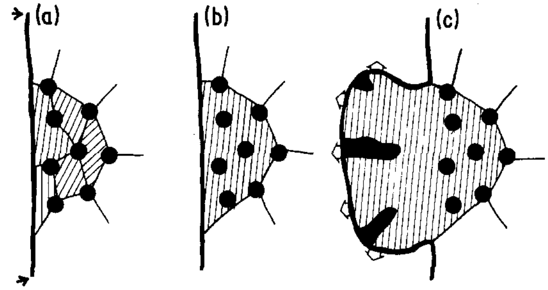


Figure 7 Schematic representation of the nucleation of recrystallization in Udimet 720. (a) γ' particles (black) define the subgrain size. A high-angle grain boundary is indicated. (b) Subgrain coalescence of the γ-phase occurs, with γ/γ' interfaces acting as dislocation sinks. The orientations of the γ' particles are unchanged by the coalescence process. (c) Growth of a recrystallization nucleus occurs by strain-induced boundary migration of the high-angle grain boundary, leading to dissolution and discontinuous reprcipitation of γ'.

tallization essentially involves the rearrangement of the matrix dislocations. The γ' particles act as a "graticule" by retaining the orientation they possessed in the as-deformed material, since the γ/γ' interfaces act as sinks for dislocations during the rotation of the γ subgrains. Fig. 6 is an example of a large recrystallization nucleus which has formed by subgrain coalescence. Dark-field electron microscopy demonstrates that the orientations of γ' particles within such subgrains are unaffected by the coalescence of γ around them.

The reason for nucleation of recrystallization at grain boundaries being favourable, is that the original grain boundary can act as a mobile interface once a nucleus of sufficient size has formed by subgrain coalescence. It is therefore commonly observed that the γ' particles, which are slightly misoriented from the cube–cube relationship with the γ, also have no rational relationship with material the other side of the recrystallization interface in partially recrystallized material. More evidence for supposing that it is the original grain boundary which acts as the mobile interface is supplied by the fact that misorientations of up to only 3–4° from cube–cube are observed. Much larger γ/γ' misorientations would be required before the coalescing subgrain itself developed a mobile recrystallization interface.

Low-angle grain boundaries are sometimes observed to occur within larger γ' particles (as in Fig. 4b). This may be assumed to be due to recovery of the few dislocations found within γ' into a well-defined cell structure. Hence, although the deformation level in γ' is much lower than in the γ, a small amount of independent recovery occurs.

The basic elements of the preceding discussion are given schematically in Fig. 7, which indicates the formation of a viable recrystallization nucleus by subgrain coalescence, and its subsequent growth by strain-induced boundary migration of an associated high-angle grain boundary. This moving interface results in dissolution and discontinuous precipitation of γ', as discussed previously.

5. Conclusions

The effects of recrystallization on the γ' distribution in nickel-base superalloys may be attributed to high solubility and diffusivity in the recrystallization interface. In low γ' fraction materials, high solubility causes complete dissolution of γ' at the moving boundary, followed by reprecipitation behind it. In high γ' fraction materials, high diffusivity in the recrystallization interface assumes greater importance, and reprecipitation occurs discontinuously.

Nucleation of recrystallization at grain boundaries in Udimet 720 occurs by subgrain coalescence. The γ' distribution is not changed during the coalescence process, but the γ/γ' interfaces act as dislocation sinks during coalescence of γ subgrains. Subsequent growth of the recrystallization nucleus occurs by strain-induced boundary migration of the grain boundary at which recrystallization nucleates.

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