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Surface recrystallization in a single crystal nickel-based superalloy

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It is shown that, at temperatures above the γ' solvus, rapid recrystallization occurs in the surface layers of superalloy single crystals which have been cold worked either by surface grit-blasting or by indentation. At temperatures below the γ' solvus, cellular discontinuous recrystallization may be observed. By applying a recovery treatment insufficient to cause discontinuous recrystallization, it is possible to suppress the recrystallization of grit-blasted specimens when subsequently heated above the γ' solvus.

1. Introduction
During high temperature operation, there may be deleterious effects in a superalloy arising from the presence of grain boundaries in the material. These problems have been at least partially overcome in the case of turbine blades by the use of directionally solidified or single crystal material. Single crystal components were initially produced from superalloys which were originally developed for use in the polycrystalline form and which thus contained grain boundary strengthening additions such as boron, hafnium, zirconium and carbon.

A new generation of single crystal superalloys is being developed in which such additions are omitted. This has the effect of raising the solidus temperature of the alloy, thus opening a heat-treatment "window" permitting a homogenization anneal above the γ' solvus temperature. This leads to a more uniform microstructure and thus enhanced mechanical properties in the product.

After removal from the mould in which they are grown, cast single crystals may receive a normal grit-blasting surface treatment. The object of the present work was to explore the effect of such working treatment upon the recrystallization behaviour of these crystals during the homogenization anneal. The recrystallization mechanisms in nickel-based superalloys have been reviewed by Porter and Ralph [1], and it has been pointed out by Doherty [2] that a dispersion of coherent particles will be at least four times more effective than incoherent particles in restraining grain-boundary motion. Thus at temperatures below the γ' solvus, inhibition of recrystallization might be expected, whereas above that temperature the recrystallization process might be expected to be relatively unimpeded.

2. Experimental procedure
2.1. Alloy composition
The alloys employed differed from a conventional polycrystalline superalloy (such as MAR M002) by the absence of the trace elements boron, hafnium and zirconium, and by a substantial decrease in carbon content. The composition is given in Table I.

The alloy was available in the form of as-cast single crystals of dimensions 10 mm × 45 mm × 150 mm, from which blocks were cut for the experimental investigation.

2.2. Specimen deformation
As well as having a number of crystals with grit-blasted surfaces, a number of crystals were also deformed by a technique of surface indentation, in order to provide a reproducible surface strain. For this purpose a tool of the form illustrated in Fig. 1 was employed to produce a surface groove of V-shaped profile, as indicated in Fig. 2. The applied compressive load was calculated to give a chosen load per unit length of indentation, using an Instron testing machine.

TABLE I Composition of alloy

Element	Atomic percentage
Al	5.5
C	0.015
Cr	8.5
Co	5.0
Ta	2.8
Ti	2.2
W	9.5
Ni	balance

2.3. Annealing treatments

The specimens were annealed in an air furnace, followed by an air cool. The measured time for the specimen to attain the annealing temperature was not greater than 3 min.

2.4. Metallography

Metallographically polished specimens were electro-etched in a solution of 10 vol% orthophosphoric acid in water at a potential of 3 V. It was found that contrast between individual recrystallized grains and the matrix crystal was achieved only by employing polarized light.

The depth of recrystallization was measured both by optical microscopy and also by means of microhardness traverses in the region beneath the indentation.

3. Experimental results and discussion

3.1. Indented specimens

V-shaped indentations were produced in specimens by means of the indenting tool shown in Fig. 1. Fig. 3 shows the microhardness profile below a groove formed by a load of 150 kg mm^{-1}. An annealing treatment of 4 h at 1300° C was applied

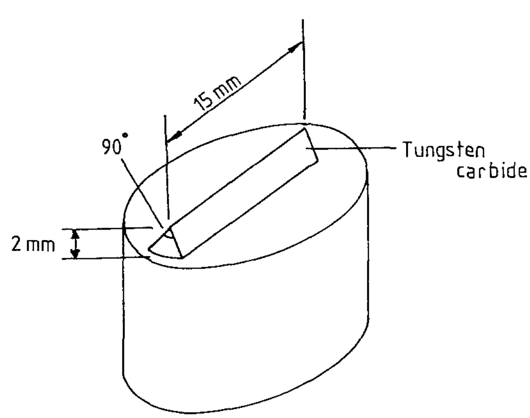

Figure 1 Diagram of tool used for producing surface indentations on crystals.

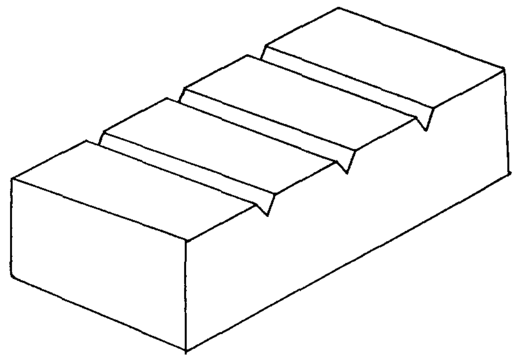

Figure 2 Diagram of specimen after indentation by tool illustrated in Fig. 1.

to this specimen, which corresponds to a solution heat-treatment for this material, and it was observed that recrystallization had occurred below the indentation. It was found that no recrystallization occurred below indentations formed by loads of less than 80 kg mm^{-1}, and it was only occasionally found below indentations from applied loads up to 120 kg mm^{-1}.

3.1.1. Annealing at 1300° C

The kinetics of recrystallization were studied by measuring the depth of the recrystallized layer formed at 1300° C at annealing times between 2 min and 4 h. For each time of heat treatment, the depth of recrystallization was measured for at least six indentations, and the results are shown in Fig. 4. A simple model may be developed to relate the rate of advance of the recrystallization front, V, with the change in microhardness, as follows.

The driving force for recrystallization, P, may be taken as approximately $G\mathbf{b}^2(\rho_1 - \rho_2)$, where G is the shear modulus, $\mathbf{b}$ the Burgers vector, and ρ_1 and ρ_2 the dislocation density in the deformed and recrystallized matrix, respectively. The velocity, V, of the recrystallization front will be given by

$$V = mP$$

where m is a mobility term.

Since, according to the forest theory of dislocations, the flow stress (and hence the hardness) is proportional to $\rho^{1/2}$, we might expect that

$$(\text{change in microhardness})^2 \propto \text{rate of advance of recrystallization front.}$$

The change in hardness for a given point may be taken as the value of microhardness, H, less the

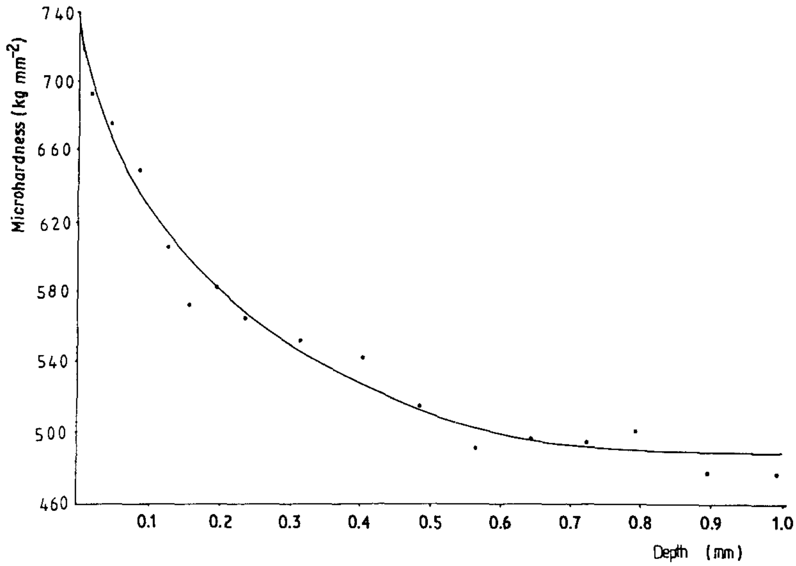

Figure 3 Microhardness profile beneath the base of a groove (150 kg mm^{-1} indentation).

microhardness of undeformed material (480 kg mm^{-2}), as evident in Fig. 3. If the gradient of the curve of Fig. 4 is plotted against $(H - 480)^2$ as in Fig. 5, it may be seen that a linear relationship does indeed exist over the range investigated.

3.1.2. Annealing at 1200° C

Fig. 6 illustrates the effect of varying the temperature of isothermal anneal. 15 min treatments were carried out at a series of temperatures decreasing from 1307° C in approximately 10° C intervals, and the depths of recrystallization were measured.

The absence of recrystallization in specimens treated below about 1250° C arises from the retardation effects due to the presence of the γ'-phase in the alloys. Below 1220° C, the volume fraction of γ' is 0.6, and the radius of the γ' particles is of the order 0.5 μm. If the boundary

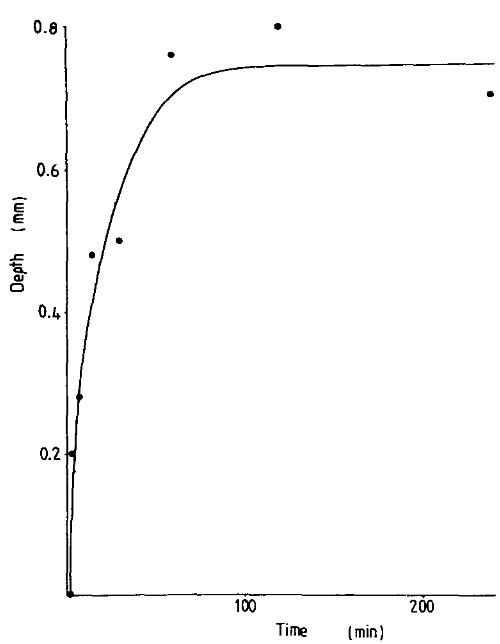

Figure 4 Depth of recrystallization against time of exposure to 1300° C (150 kg mm^{-1} indentation).

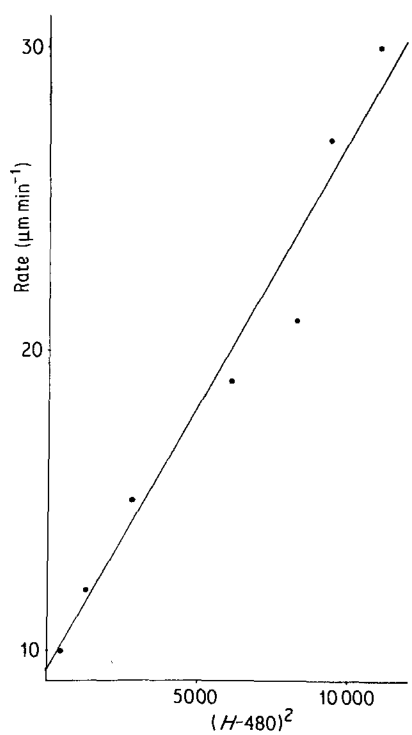

Figure 5 Rate of advance of recrystallization front at 1300° C against the square of the microhardness change.

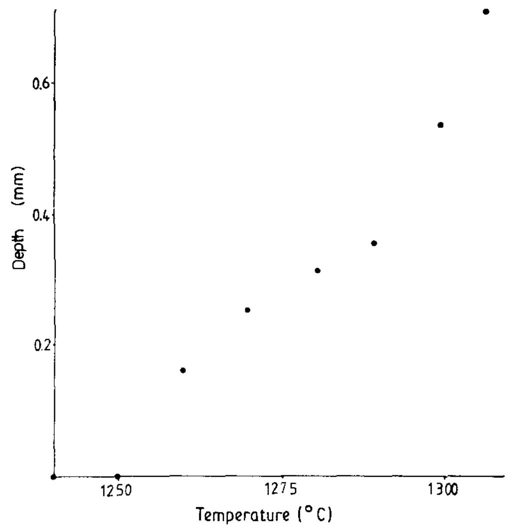

Figure 6 Depth of recrystallization against temperature of heat treatment (150 kg mm^{-1} indentation), for 15 min anneals.

energy is taken as 1 J m^{-2}, then a retarding force of the order 10^7 N m^{-2} would be produced, according to the expression due to Doherty [2]. In the present alloy the γ' begins to go into solution above 1220° C until at 1300° C it has all dissolved. It is clear from Fig. 6 that the volume fraction of γ' in the specimens investigated falls to a value at which recrystallization will commence at temperatures in the region of 1250° C. This was the lowest temperature at which any recrystallization was observed, and so a series of experiments was carried out at 1200° C in order to establish if the stored energy in the deformed crystals could be reduced by recovery mechanisms.

It was found that, in all specimens treated at 1200° C for more than one hour, recrystallization occurred by the cellular process of discontinuous precipitation, as illustrated in Fig. 7. The results of these treatments are shown in Fig. 8, where it can be seen that the depths of recrystallization due to the discontinuous process are much less than those occurring at 1300° C (Fig. 4). It is suggested that this decrease in the rate of advance of the recrystallization front arises since the dislocation density is reduced by recovery mechanisms during the long heat treatment.

Specimens which had been previously exposed to 1200° C for one of a range of periods were then subjected to a second standard treatment of 15 min at 1300° C. These final depths of recrystallization are shown in Fig. 9 as a function of the time of exposure to 1200° C. The observed reduction in the depth of recrystallization after exposure to 1300° C for 15 min suggests that significant recovery mechanisms are operating at 1200° C, although it seems unlikely that recrystallization at 1300° C could be completely suppressed simply by a prior treatment at 1200° C.

3.1.3. Annealing at 1000° C

Indented specimens were exposed to 1000° C for 300 h in the hope that recovery might occur at this temperature without any forms of recrystallization occurring. It was in fact found that cellular recrystallization did take place, and furthermore that subsequent treatment at 1300° C for 15 min produced an average recrystallized depth of 0.5 mm.

It would appear, therefore, that at 1000° C, even after 300 h, there has been little, if any, reduction in the driving force for recrystallization. Thus for a significant decrease in driving force of recrystallization by recovery within a commercially acceptable period, a higher temperature is needed in this material.

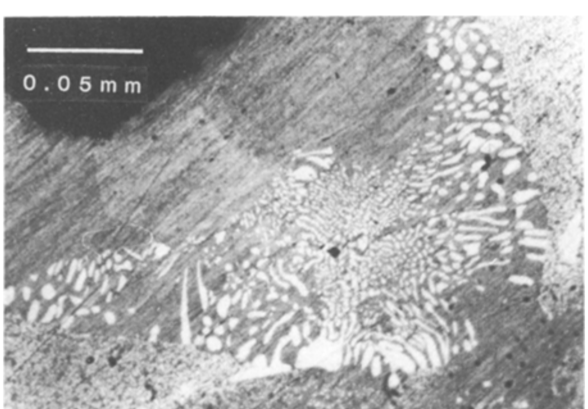

Figure 7 Cellular recrystallization after 4 h at 1200° C.

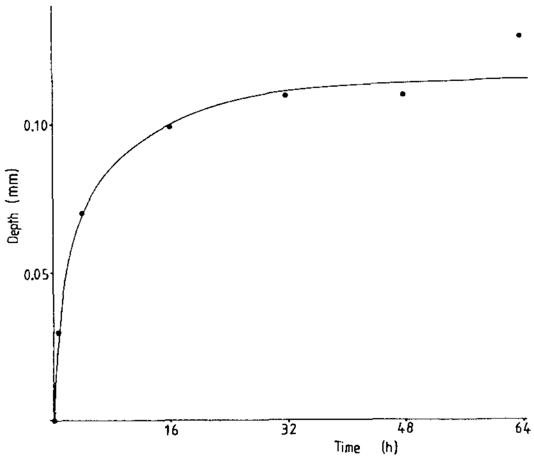

Figure 8 Depth of cellular recrystallization against time of exposure to 1200° C (150 kg mm^{-1} indentation).

3.2. Grit-blasted specimens

Grit-blasting of as-cast single crystals will cause work hardening of the surface layers, and this can be detected as before by means of a microhardness traverse. The microhardness profile below the surface of grit-blasted material is illustrated in Fig. 10, where it may be seen that a cold worked layer of depth 0.2 mm is present, the maximum (surface) hardness being about 580 kg mm^{-2}.

During production of single crystal components from the present alloy a solution treatment of 1300° C for 4 h is carried out, and this was observed to result in the formation of a recrystallized layer of depth approximately 0.17 mm. If the grit-blasted specimen is exposed to 1000° C for 300 h it was found that during subsequent treatment at 1300° C recrystallization behaviour was unaffected. This again suggests that a higher temperature is required if the stored energy is to be relieved by a recovery treatment.

The results of Section 3.1 above indicate that a temperature in the region of 1200° C is needed to bring about significant reduction in the driving force for recrystallization. At 1200° C, however, cellular recrystallization is observed after 1 h, so a shorter treatment time than this must be applied. As shown in Fig. 8, the use of short periods of exposure to 1200° C did not prevent the indented specimens from recrystallizing, although after longer periods of exposure the depth of recrystallization is reduced from 0.5 to 0.2 mm.

Referring to Fig. 3, it may be seen that when recrystallization of the indented specimens occurs to a depth of only 0.2 mm, then no recrystallization has taken place in regions where the as-deformed microhardness was less than approximately 580 kg mm^{-2}. Consideration of Fig. 10 reveals that the microhardness before heat-treatment of the grit-blasted material does not exceed 600 kg mm^{-2}. It was therefore considered that exposure of grit-blasted specimens to 1200° C for 30 min might reduce or eliminate recrystallization during subsequent treatment at 1300° C, without itself causing cellular recrystallization.

Grit-blasted material was accordingly given 30 min at 1200° C, air cooled and then given 15 min at 1300° C. No recrystallization was observed below the blasted surface, which suggests that this forms the basis of an appropriate heat-treatment to suppress surface recrystallization in grit-blasted crystals.

4. Conclusions

1. Exposure of grit-blasted specimens to

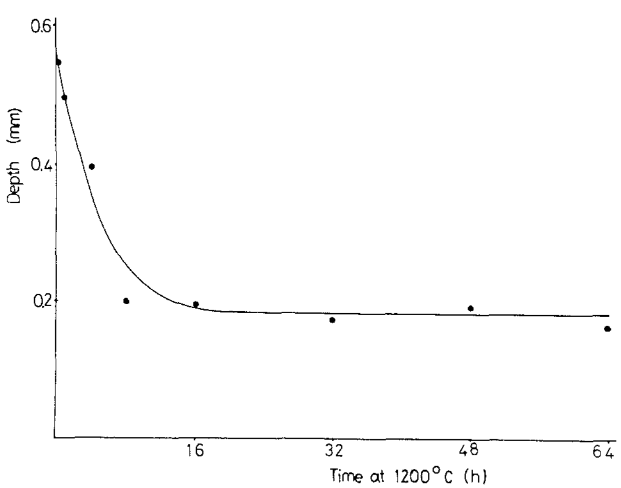

Figure 9 Depth of recrystallization after 15 min at 1300° C against time of exposure to a prior treatment at 1200° C (150 kg mm^{-1} indentation).

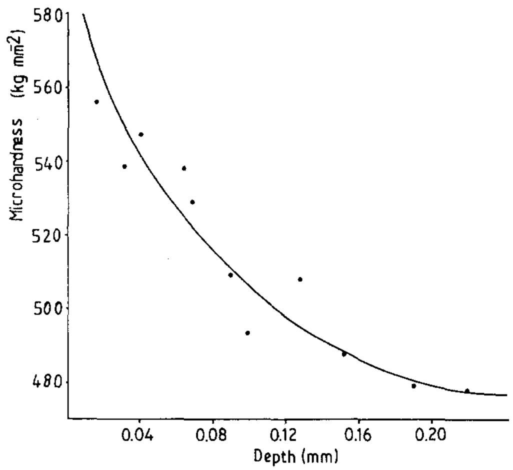

Figure 10 Microhardness profile below a grit-blasted surface.

1300° C for 4 h results in surface recrystallization to a depth of 0.17 mm.

2. At 1300° C the rate of advance of the recrystallization front beneath a surface indentation is proportional to the square of the change in microhardness occurring during recrystallization.

3. Below 1250° C the retardation effect of γ' particles is sufficient to prevent recrystallization, although specimens exposed to 1200° C for more than 1 h display cellular recrystallization.

4. Recrystallization of grit-blasted material during exposure to 1300° C may be prevented by a prior treatment at 1200° C for 30 min.

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