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The Effect of Heat-Treatment and Nitrogen Addition on the Critical Current Density of a Worked Niobium 44 wt % Titanium Superconducting Alloy

C. BAKER*
Central Electricity Research Laboratories, Leatherhead, Surrey, UK

Mechanical working of a single phase superconducting alloy leads to an increase in the critical current density, J_c. After cold-rolling J_c exhibits anisotropy as the angle of the magnetic field to the rolled surface is changed.

This paper studies the effects of interstitial addition and heat-treatment on J_c and its anisotropy in both cold-rolled and wire-drawn alloy. The variations in J_c are correlated with the observed changes in the microstructure.

1. Introduction

Narlikar and Dew-Hughes [1] have shown that dislocations introduced by cold-work produce increased critical current densities in type II superconductors. They interpret the dislocations as regions of varying κ (the Ginsburg-Landau parameter [2]) and these interact with and pin flux threads. At high deformations the dislocations interact to form a substructure of low dislocation density cells surrounded by highly dislocated walls. Narlikar and Dew-Hughes [1] have shown that the strongest pinning arises from a non-uniform dislocation distribution.

When metals are cold-rolled the cells are elongated in the rolling direction and compressed transverse to the rolling direction [3]. Embury, Keh and Fisher [3] have shown that for bcc metals the cell size in the rolled face shows little variation with deformation whereas the cell width in the edge face is progressively reduced with deformation.

The critical current density of a cold-rolled superconducting alloy exhibits anisotropy as the angle of the transverse field to the rolling plane is varied [4]. The maximum value of J_c occurs when the field is parallel with the rolling plane and the Lorentz force on the flux threads is perpendicular to the rolling plane. Hence the dislocation distribution yielding the maximum pinning is that observed in the edge face [5].

In this investigation, a niobium 44 wt % titanium alloy was studied both cold-rolled and wire-drawn after various treatments. The critical current densities measured were correlated with the three dimensional dislocation distribution.

2. Experimental Details

The alloy, of composition niobium/44 wt % titanium, was supplied by Imperial Metal Industries, Kynoch Ltd, as forged ingot. The interstitial composition was 645 ppm oxygen, 50 ppm nitrogen and 405 ppm carbon. A small amount of this alloy was remelted using RF heating on a water cooled copper hearth, with a weighed quantity of titanium nitride powder, sufficient to give an additional 1100 ppm nitrogen. To ensure homogeneity the ingot was turned over six times and remelted. The interstitial analysis after this treatment was 930 ppm oxygen, 480 ppm nitrogen and 380 ppm carbon. This nitrogen-doped ingot was heated in vacuum for 1 h at 1100° C and then quenched into water. The two forms of starting material were (a) forged and (b) nitrogen-doped and quenched.

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*Present address: Centre for Materials Research, University of British Columbia, Vancouver 8, British Columbia, Canada.

These were cold-rolled 95% to 0.05 cm thick sheet and 99.3% to 0.0075 cm thick sheet and wire-drawn to 0.025 cm diameter (99.93% reduction in area). An ingot was also made with nominally 2200 ppm nitrogen but this edge-cracked severely on rolling and was discarded. Samples of the forged ingot and the 0.0075 cm thick forged, cold-rolled sheet were reheated to 1100° C and were water-quenched or slowly cooled.

Sheet specimens for critical current measurements were punched from the sheet. They were 9 cm long, 0.75 cm wide with a central gauge length 0.5 cm long and 0.15 cm wide. Their long axis was parallel with the rolling direction. The critical current anisotropy was measured when specimens were rotated about their long axis so that the field changed from perpendicular to parallel to the rolled surface. Current contacts for both sheet and wire were made with copper blocks under pressure and the field was always transverse. The critical current was measured as that current which produced a voltage of 5 μV across the gauge length.

The sheet specimens were tested in an Oxford Instrument Co Ltd 40 kG niobium-zirconium super-conducting magnet, and the wire specimens in an RCA 100 kG Nb_3Sn tape superconducting magnet.

All measurements were made at 4.2° K. Thin foils for transmission electron microscopy were prepared in three ways:

(i) By chemically polishing the sheet in a hot mixture of equal volumes of hydrofluoric, nitric and sulphuric acids;

(ii) From sections cut transverse to the rolling direction for the 0.05 cm thick sheet. These were mechanically polished until they were $\sim$0.005 cm thick and then chemically polished to yield foils thin in the rolling direction [5];

(iii) From the 0.025 cm diameter wire by mechanical polishing until it was $\sim$0.0025 cm thick. This was then chemically polished.

3. Metallography

3.1. Optical

The starting ingots were examined prior to working. The forged ingot (fig. 1a) showed slightly deformed crystal grains, an etched structure associated with the forging, and a second phase precipitate. The precipitate was present in the grain-boundaries and inside the grains. The alloy is in the β single-phase region [6] which precludes the possibility of α-titanium precipitates. Extraction replicas taken from the ingot and examined in the electron miroscope indicated that the precipitate had a face-centred cubic crystal structure and a lattice parameter of $\sim$4.45 Å. The carbides and nitrides are face-centred cubic and have parameters TiC — 4.3, TiN – 4.23, NbN – 4.37 and NbC – 4.46 Å [7].

The forged and quenched alloy (fig. 1b) was fully recrystallised with equiaxed grains, sharp boundaries and no precipitation. The interstitials have been retained in supersaturated solid solution.

In the alloy doped with nitrogen and quenched, only part of the titanium nitride had dissolved, the remainder being in the form of massive crystals (fig. 1c). The analysing technique (a vacuum fusion procedure using an iron bath at 1800° C; performed at the National Physical Laboratory, Teddington) would not be expected to detect these large crystals hence the 480 ppm nitrogen is the nitrogen in solution. Because 1100 ppm nitrogen was added it is obvious that only half this amount has dissolved. The β-grains were again fully recrystallised.

3.2. Electron Microscopy

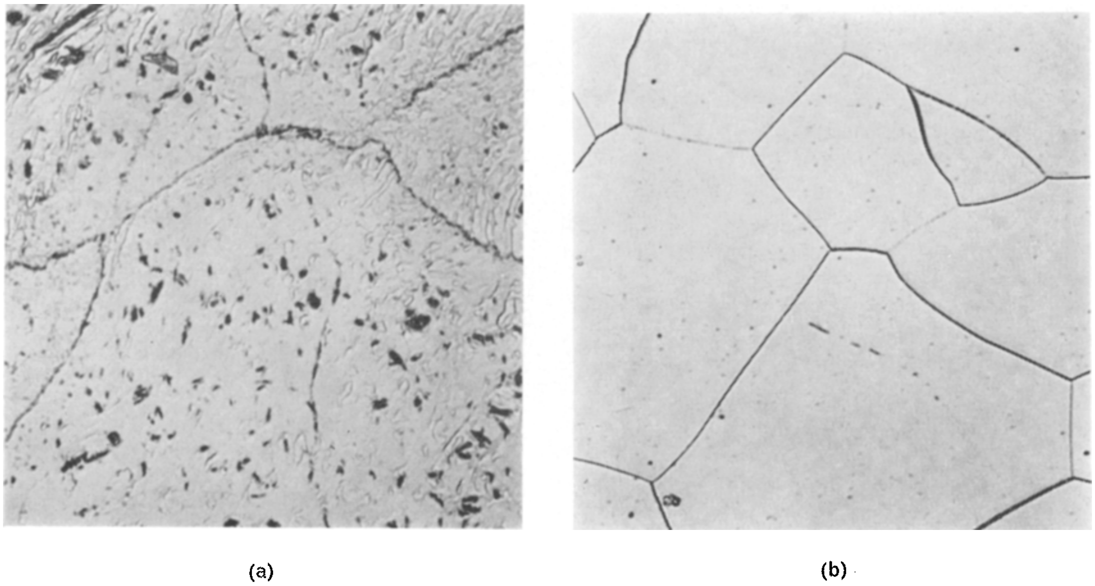
3.2.1. Unworked

A thin slice was cut from the forged ingot and this was thinned and examined. The forging had introduced a substructure in the form of small grains 1000 to 2000 Å in diameter (fig. 2).

The 0.0075 cm cold-rolled, forged alloy which had been water-quenched from 1100° C had large grains with no precipitate or substructure and the grain-boundaries were sharp (fig. 3a). However on ageing in the range 400 to 700° C a fine precipitate formed (fig. 3b). The mean precipitate diameter for 1 h ages was 125 Å at 400° C, 150 Å at 500° C, 300 Å at 600° C and 2000 Å at 700° C. After 1 h at 500° C the precipitate spacing was in the range 1000 to 2000 Å. The precipitates exhibited strain contrast in the ageing range 400 to 600° C and at 700° C were observed as plates lying on $\{100\}$ planes on the β-matrix.

3.2.2. Wire Specimens

In the wire-drawn condition, both forged and nitrogen-doped wires had a fibrous structure with the fibre axis parallel to the wire axis (fig. 4). There was a preferred orientation of $\langle 100 \rangle$ parallel to the wire axis. The dislocation density inside the fibres was high and individual dis-



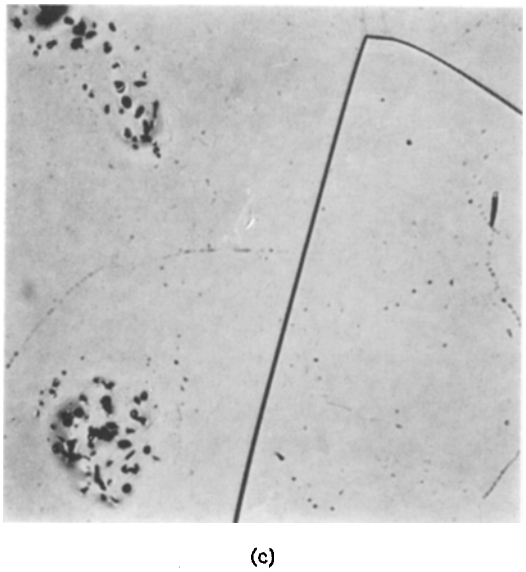


Figure 1 Optical micrographs of (a) forged ingot (b) forged ingot, reheated to 1100° C and water-quenched and (c) forged, nitrogen-doped and water-quenched from 1100° C (× 345).

locations could not be resolved. It was also noticeable visually that the overall dislocation density in the nitrogen-doped wire was not uniform. On ageing, the fibres coarsened and the boundaries became sharper. At high ageing temperatures, the fibres began to break up into more equiaxial grains as recrystallisation commenced. Table I lists the mean fibre diameter for 1 h anneals in the range 300 to 600° C and fig. 4 shows representative micrographs.

In the forged alloy the fibre size progressively increased whereas in the nitrogen-doped alloy, it at first decreased. This minimum is somewhat dubious in that the fibre boundaries in the as-worked, nitrogen-doped alloy were not too clearly defined.

The dislocation density within the fibres was

Figure 2 Electron micrograph of substructure in the forged ingot (× 27 500).

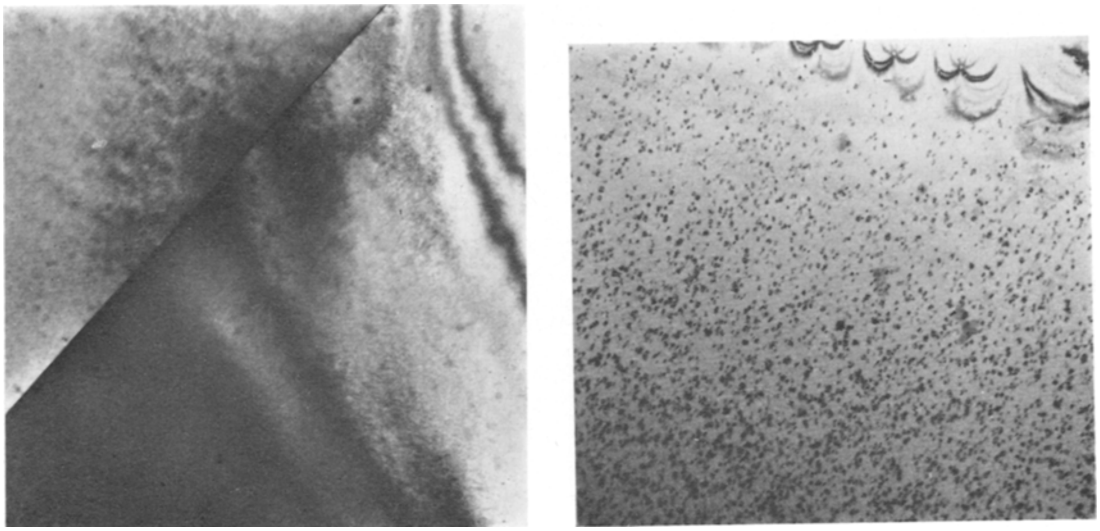

(a) (b)

Figure 3 Electron micrographs of forged ingot (a) water-quenched from 1100° C and (b) water-quenched from 1100° C and aged 1 h at 500° C (×27 500).

TABLE I

Heat-treatment	Mean fibre diameter	
	Forged alloy, Å	Nitrogen-doped alloy, Å
As-worked	500	750
+ 1 h at 300° C	700	650
+ 1 h at 400° C	1100	750
+ 1 h at 500° C	2500	1100
+ 1 h at 600° C	10^4	3000

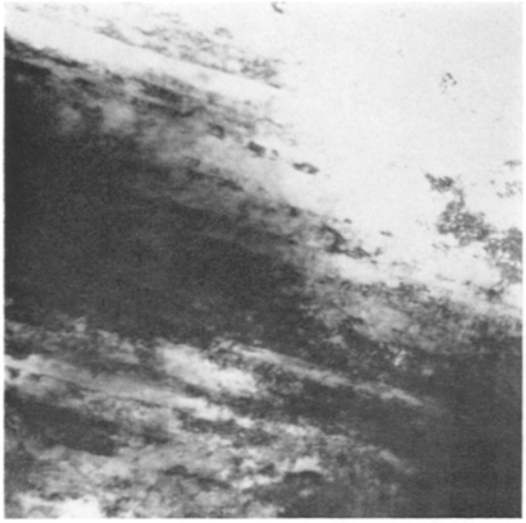
reduced with increasing ageing temperature. Individual dislocations became resolvable after 1 h at 400° C in the forged alloy and after 1 h at 500° C in the nitrogen-doped alloy. Indeed the most striking feature in comparing the two sets of micrographs is that the fibre appearance and diameter in the forged alloy is very similar to the nitrogen-doped alloy aged for 1 h at a temperature 100° C higher. The interstitials' main effect seems to be in lessening the dislocation mobility. With the high dislocation densities present they will be in the form of Cottrell [8] atmospheres around the dislocations. In none of the micrographs was there visible a consistent and uniform precipitate, nor was there any evidence of one on the diffraction patterns.

It is interesting to note the similarity of fibre diameter in the two alloys. If the fibres in the forged alloy are considered as arising from deformation of the original grain or substructure then this would yield fibres 5 μm or 50 Å diameter respectively. The fibre size is therefore more probably associated with a spacing between precipitates or some other inhomogeneity, as was found by Embury, Keh and Fisher [3].

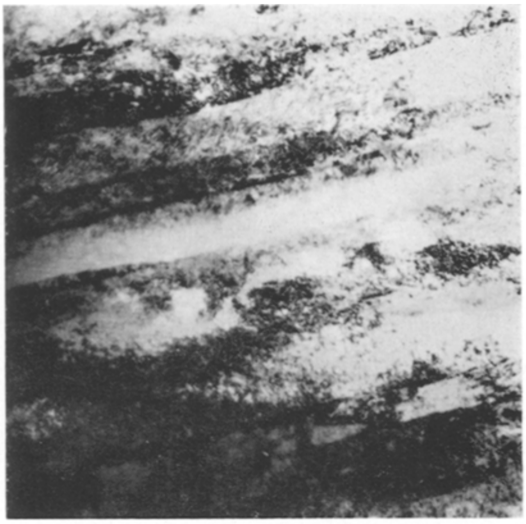
3.2.3. Worked Sheet

The forged alloy had a fairly uniform dislocation distribution in the rolled surface (99.3% reduction) with, in some regions, indistinct cells ⩾ 0.25 μm diameter of much lower dislocation density (fig. 5c). The end section structure (95% reduction, fig. 5c) showed clear elongated cells ~ 1000 Å wide, similar to the fibres observed in the wires (fig. 4). The nitrogen-doped alloy had a much more uniform dislocation distribution in the rolled surface (99.3% reduction) with no evidence at all of cells (fig. 5b). Even in the end face (95% reduction) the only regions with clear elongated cells were those close to the large undissolved titanium nitride particles (fig. 5d).

On ageing the dislocation structure in the rolled surface became less uniform, the dislocations formed into networks and at higher temperatures small recrystallised grains formed. In the end sections the cells behaved as did the fibres in the wires, at first coarsening as internal dislocations annealed out, then at higher temperatures becoming equiaxed as recrystallisation com-



(a)



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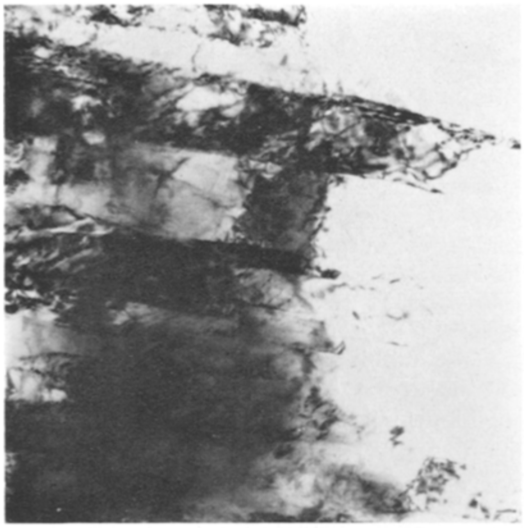
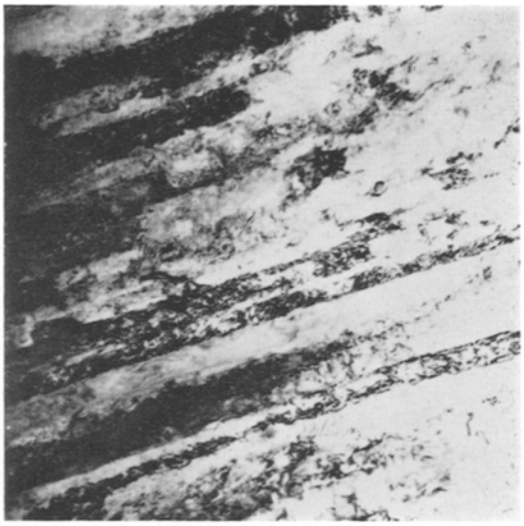
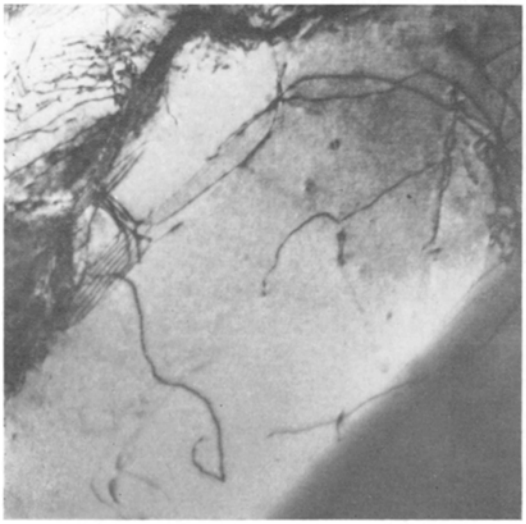


(e)

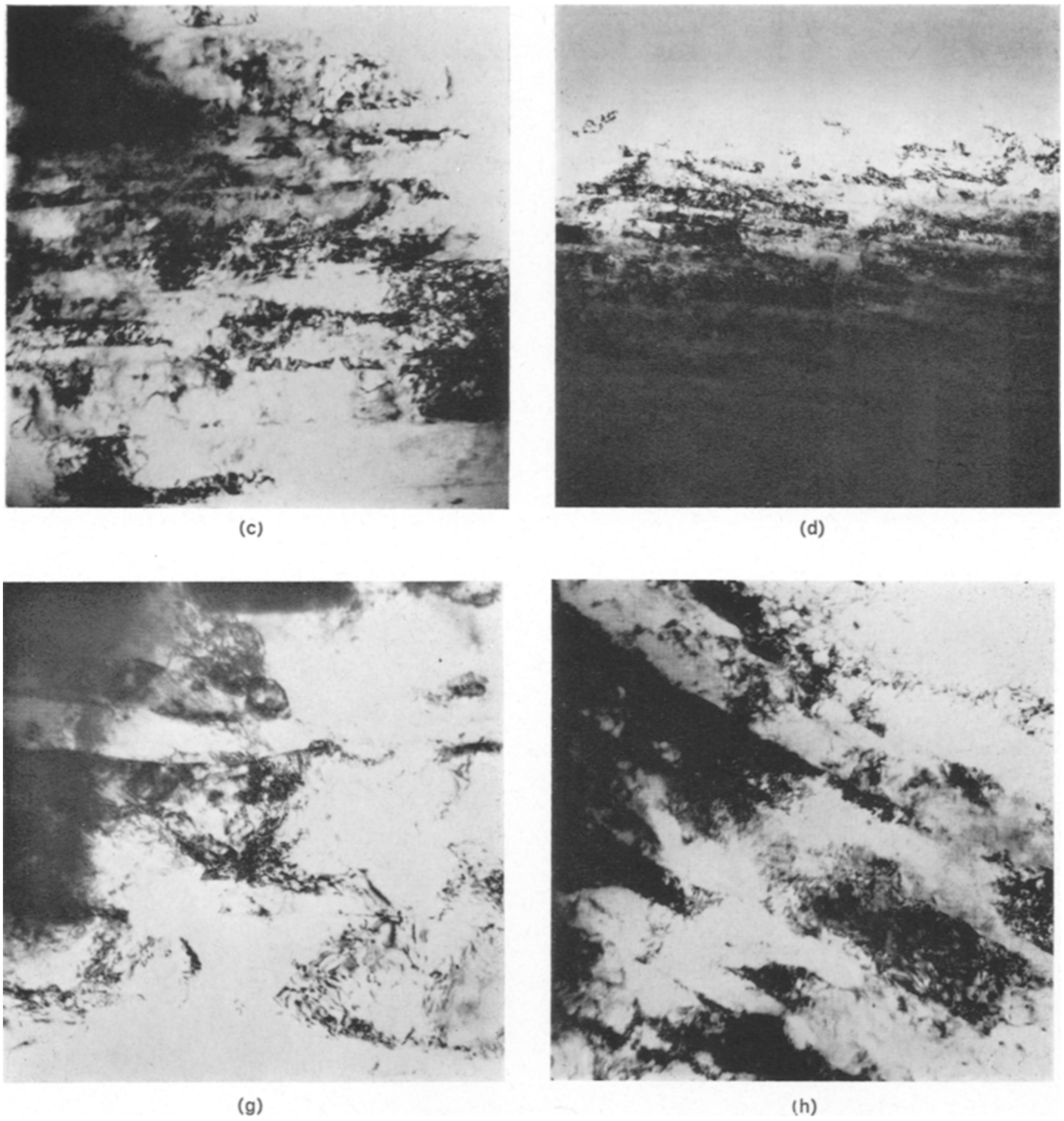


(f)



(i)

(j)



(c) (d)

(g) (h)

Figure 4 Electron micrographs of wire specimens (a) forged alloy, as-worked (b) nitrogen-doped alloy as-worked (c), (a) + 300° C; (d), (b) + 300° C; (e), (a) + 400° C; (f), (b) + 400° C; (g), (a) + 500° C (h), (b) + 500° C; (i), (a) + 600° C; (j), (b) + 600° C; 1 h ageing time (× 55 000).

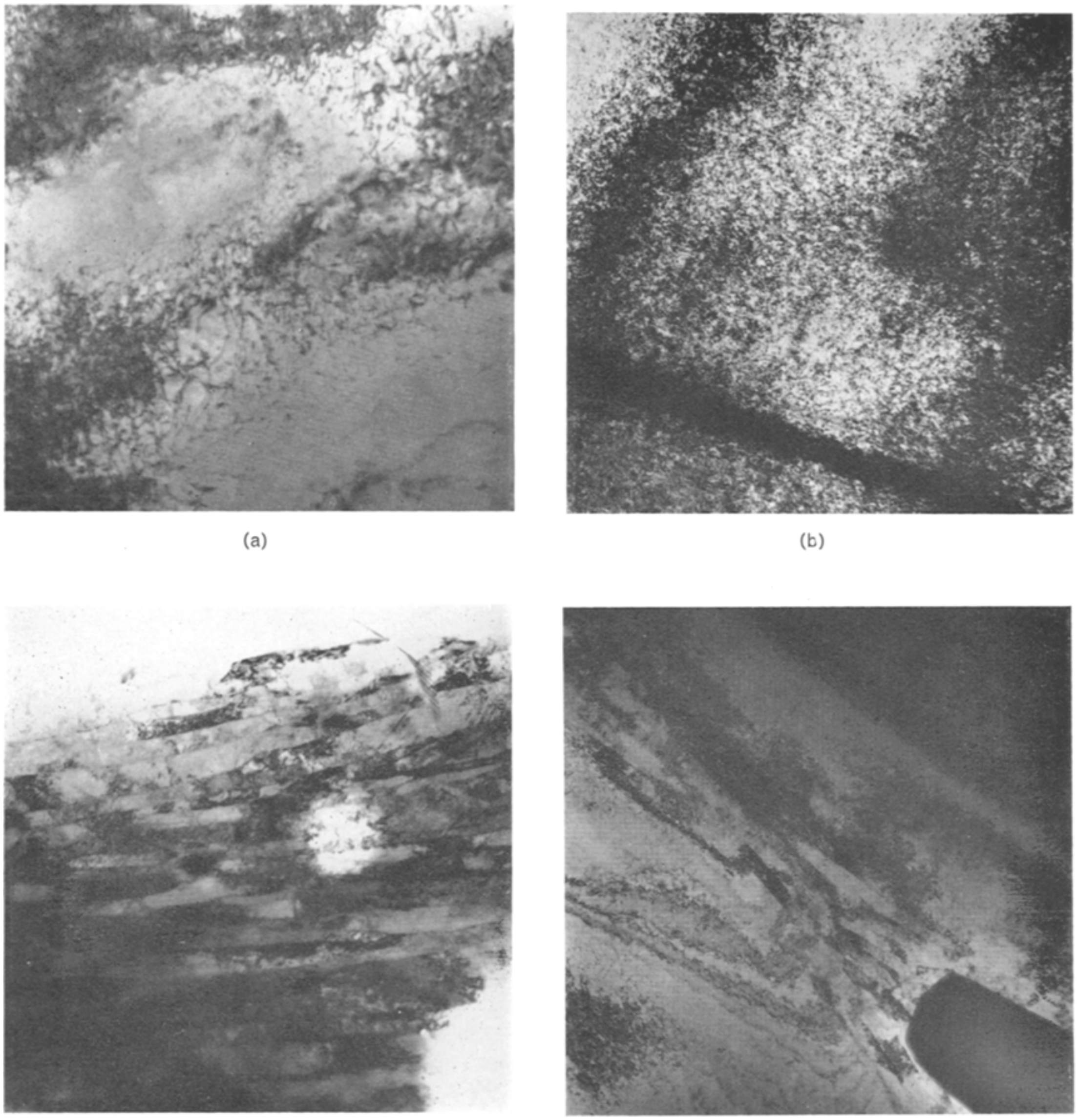
menced. At ageing temperatures up to 500° C the observed structure, particularly in the nitrogen-doped alloy, could vary considerably from area to area. Ageing for 1 h at 600° C (fig. 6) shows fully recrystallised grains in the rolled surface and recrystallised but slightly deformed grains in the end face for the forged alloy, but in the nitrogen-doped alloy the rolled surface structure is only partially recrystallised and the end face shows coarse fibres beginning to break up into equiaxed grains.

Again the dislocation mobility in the nitrogen-doped alloy was impaired and structures in this alloy were comparable with those in the forged alloy at a 100° C lower ageing temperature.

3.3. Critical Current Measurements

3.3.1. Recrystallised Sheet Specimens

The 0.0075 cm thick sheet of forged alloy re-quenched from 1100° C was tested in the as-



(a) (b)

(c) (d)

Figure 5 Electron micrographs of as-worked sheet specimens. (a) Forged alloy structure observed in rolled surface. (× 55 000). (b) Nitrogen-doped alloy structure observed in rolled surface (× 27 500). (c) Forged alloy structure observed looking along the rolling direction (× 27 500). (d) Nitrogen-doped alloy structure observed looking along the rolling direction (× 27 500).

quenched and aged conditions. The anisotropy in the critical current density, J_c, as the field was changed from parallel to perpendicular to the rolled surface had a maximum value of 2.5 : 1 indicating that most of the effects of cold-work had annealed out. The variation of J_c with ageing at 30 kG and with the field parallel to the rolled surface is shown in fig. 7. J_c (H || RP) reached a maximum after 1 h at 600° C with a value 20 times that in the as-quenched condition. At 500° C the material exhibited training and both the trained and untrained values are shown.

The 0.0075 cm sheet, slowly cooled from 1100° C had a measured anisotropy of 1.75 : 1 and a J_c (H || RP) of 2.2×10^3 amps cm^{-2}. J_c increased by only 10% on ageing.

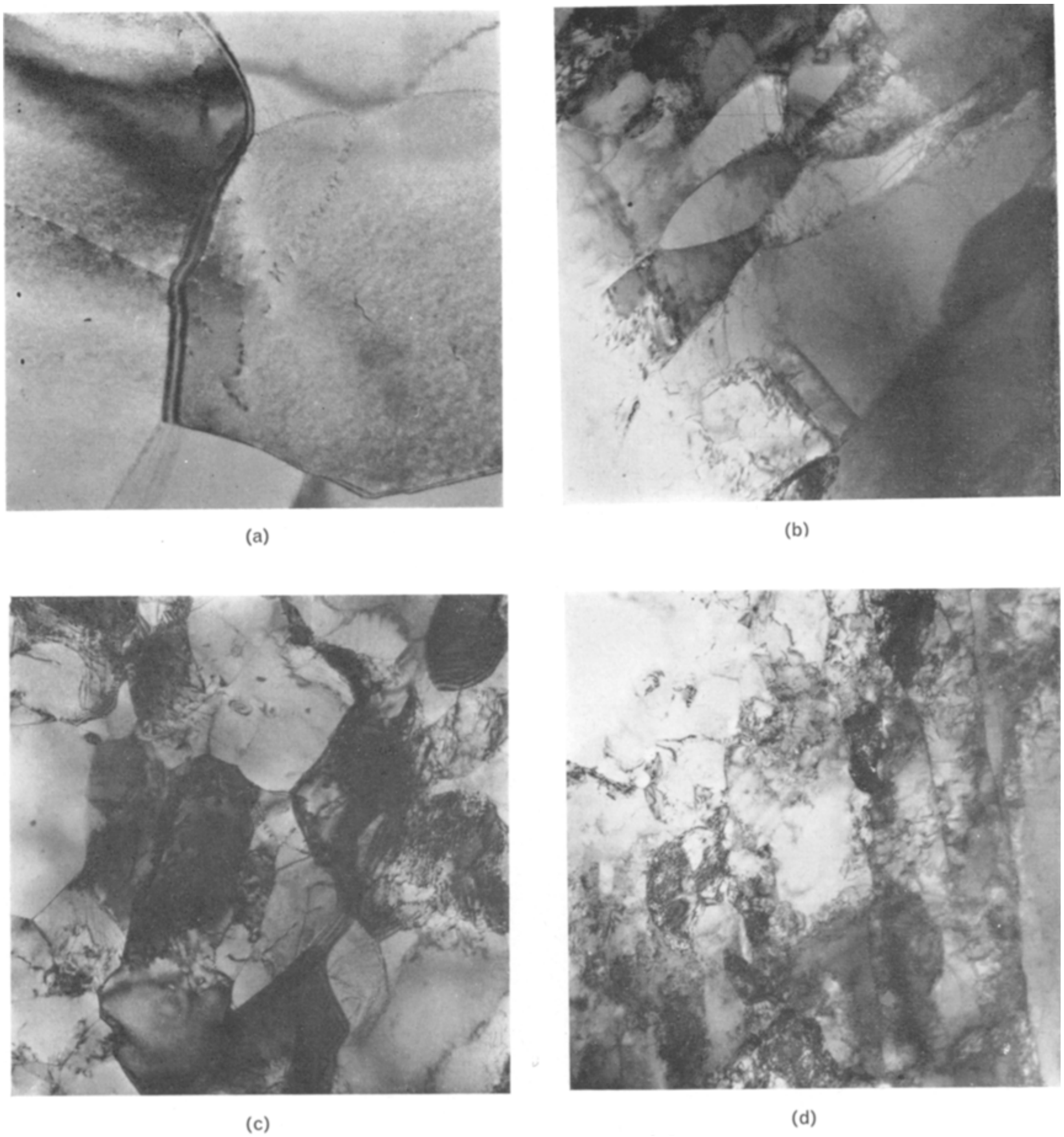


Figure 6 Electron micrographs of (a) forged alloy, worked + 1 h at 600° C, structure in the rolled surface (× 60 500). (b) as (a) but structure observed looking along the rolling direction (× 30 250). (c) Nitrogen-doped alloy, worked + 1 h at 600° C, structure in the rolled surface (×27 500). (d) as (c) but structure observed looking along the rolling direction (× 30 250).

3.3.2. Wire Specimens

The change in J_c with field for the 99.93% deformed, forged and nitrogen-doped alloy wires as-worked and aged for 1 h in the range 300 to 600° C are shown in figs. 8 and 9. In the as-worked condition both materials had a peak in J_c occurring around 80 kG, and this may be associated with the upper critical field, H_{c2}, of this alloy $\sim$120 kG. These H_{c2} peaks have been observed previously [9, 10]. The peak in J_c is more pronounced in the nitrogen-doped alloy wires. The peaks disappeared on ageing and J_c increased, reaching a maximum after 1 h at 300 to 400° C in the forged alloy wire and 400 to 500° C in the nitrogen-doped alloy wire.

The results are more easily interpreted if they

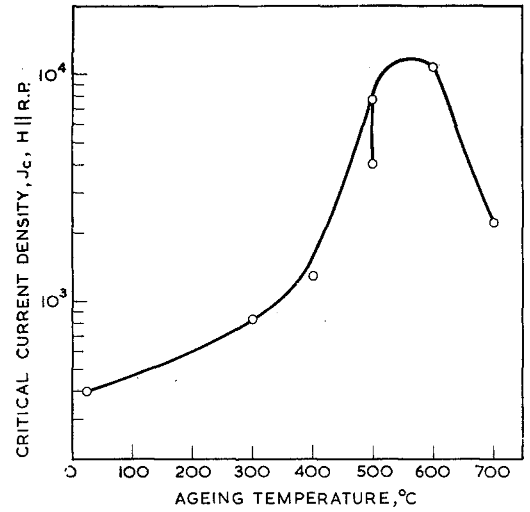


Figure 7 Critical current density, J_c, at 30 kG against ageing temperature, 1 h ageing time, for the worked, forged alloy re-water-quenched from 1100° C.

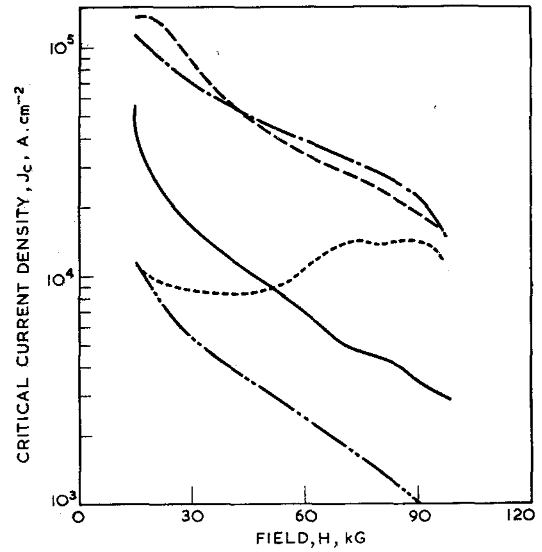


Figure 8 J_c against field plots for the forged alloy wire-drawn 99.93%, as-worked and aged 1 h at a number of temperatures: - - - as-worked; — — — as-worked + 1 h at 300° C; ——— as-worked + 1 h at 400° C; ——— as-worked + 1 h at 500° C; — - — - as-worked + 1 h at 600° C.

are replotted as the variation in J_c with ageing temperature at constant field, e.g. at 30, 45, 60 and 90 kG, (figs. 10 and 11). The points to notice are:

(i) The peak effect disappears on ageing.

(ii) In the as-worked condition J_c of the forged alloy wire is at least twice that of the nitrogen-

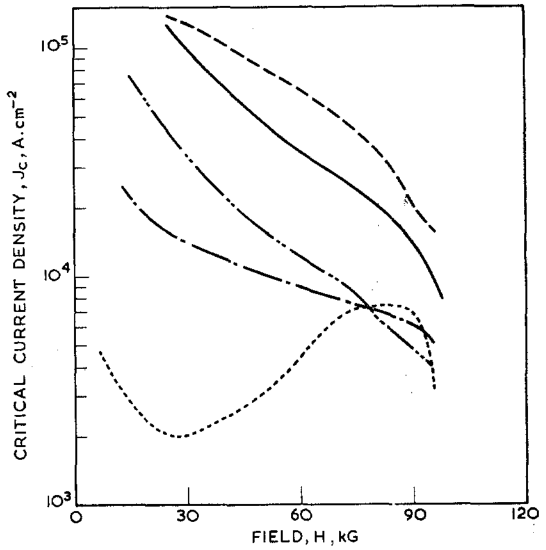


Figure 9 J_c against field plots for the nitrogen-doped alloy wire-drawn 99.93%, as-worked and aged 1 h at a number of temperatures: - - - as-worked; — — — as-worked + 1 h at 300° C; — — — as-worked + 1 h at 400° C; ——— as-worked + 1 h at 500° C; — - — - as-worked + 1 h at 600° C.

doped alloy wire over the whole field range.

(iii) The forged alloy wire has a maximum in J_c for an ageing temperature of ~ 350° C at all fields and a rapid decrease in J_c for temperatures in excess of 400° C. The nitrogen-doped alloy has a maximum in J_c for an ageing temperature of ~ 450° C at all fields and a rapid decrease in J_c for temperatures in excess of 500° C.

(iv) Whereas the maximum J_c obtained on ageing the nitrogen-doped alloy wire is relatively independent of field, in the forged alloy wire the maximum J_c appears to shift to lower ageing temperatures at higher fields.

(v) J_c of the nitrogen-doped alloy wire is roughly equivalent to that of the forged alloy at a 100° C lower ageing temperature.

3.3.3. Worked Sheet

J_c plots for the worked sheet with the field both perpendicular and parallel to the rolled plane are shown in fig. 12 for both the forged and nitrogen-doped alloy sheets. The field is 30 kG and the ageing time 1 h. The following points are to be noted:

(i) In the as-worked condition J_c is higher in the forged alloy sheet with the field both parallel and perpendicular to the rolled plane, than in nitrogen-doped alloy sheet.

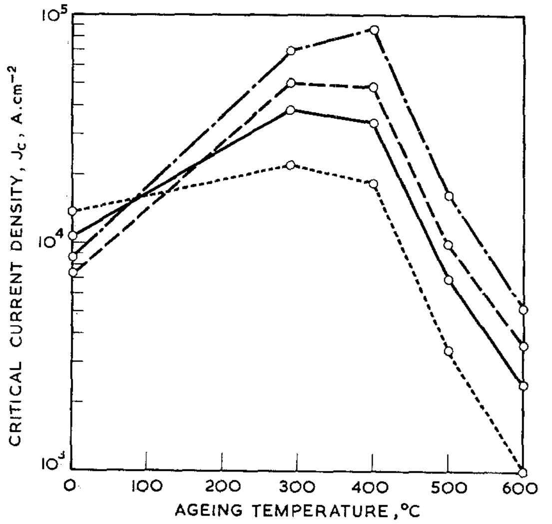


Figure 10 J_c against ageing temperature, 1 h ageing time, at a number of fields for the 99.93% deformed, forged wire. Field: - - - 30 kG; — — — 45 kG; ——— 60 kG; - - - - 90 kG.

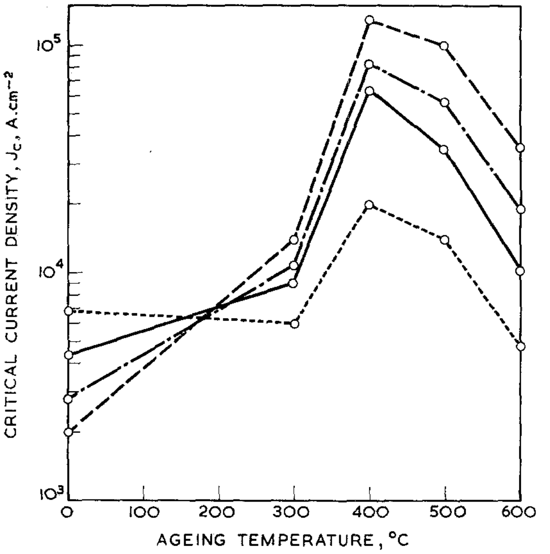


Figure 11 J_c against ageing temperature, 1 h ageing, at a number of fields for the 99.93% deformed, nitrogen-doped wire. Field: — — — 30 kG; — — — 45 kG; ——— 60 kG; - - - - 90 kG.

(ii) The peak in J_c on ageing occurs at a temperature 100° C higher in the nitrogen-doped alloy sheet than in the forged alloy sheet.

(iii) The anisotropy in J_c in the forged alloy sheet is only reduced when J_c (H || RP) is also falling rapidly, whereas in the nitrogen-doped alloy sheet the anisotropy in J_c is reduced even before J_c (H || RP) reaches its maximum value.

(iv) Although the maximum values of J_c (H || RP) were comparable in the forged and nitrogen-doped alloy sheet, the maximum value of J_c (H $\perp$ RP) in the nitrogen-doped alloy sheet is many times that in the forged alloy sheet.

4. Discussion

In an extensive investigation on superconductivity in deformed niobium alloys Narlikar and Dew-Hughes [1] showed that the maximum critical current densities were obtained with a non-uniform dislocation distribution. The results of the present investigation are broadly in agreement with this conclusion although the dislocation structure for maximum pinning is shown to be difficult to characterise.

For the rolled sheet the low J_c's (H $\perp$ RP) are explainable in that the dislocation structure is fairly uniform. In the nitrogen-doped alloy, where there is no cell structure visible in the rolled plane, J_c is even lower than the forged alloy in which indistinct cells are visible. The high J_c's (H || RP) are also understandable in that the dislocation structure observed in the electron microscope looking along the rolling direction consists of elongated cells ~ 1000 Å wide. These cells have heavily dislocated boundaries, as is observed from the varying contrast of adjacent cells, with a high dislocation density inside the cells. The effect of quenching is to produce a supersaturated solid solution of interstitials and little precipitation, and hence the elongated cell structure is only observed near the undissolved titanium nitride crystals and J_c (H || RP) is low. Embury, Keh and Fisher [3] have also found that the cell structure is considerably influenced by a dispersion of a second phase precipitate.

Similar results are obtained for the wire-drawn specimens; the forged alloy specimens having a smaller cell size, a more non-uniform dislocation distribution inside the cells, and a higher J_c than the nitrogen-doped alloy specimens. Both as-worked wire specimens displayed a peak in J_c near H_{c2}. The peak in the nitrogen-doped alloy wire was sharper and more pronounced, indicating perhaps that interstitials are a contributory factor. This peak near H_{c2} has been observed in many systems [9, 10] and many explanations have been put forward [11, 12]. The most likely is that of Anderson and Kim [11] who attribute the peak to the flux line lattice becoming rigid near

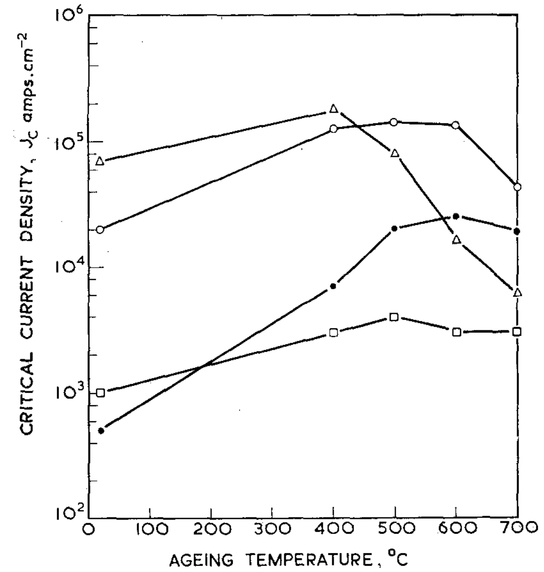


Figure 12 J_c at 30 kG against ageing temperature, 1 h ageing for both the forged and nitrogen-doped alloy sheets with the field both parallel and perpendicular to the rolled surface. △ forged (H $\parallel$ RP); O nitrogen-doped (H $\parallel$ RP); □ forged (H $\perp$ RP); ● nitrogen-doped (H $\perp$ RP).

H_{c2} so that the flux lines cannot move independently of each other. A dispersion of many relatively weakly pinning defects has been shown to produce a peak [10].

On ageing in the range 300 to 600° C, J_c increases to a maximum value and then decreases and the peak in J_c near H_{c2} is not observed. Metallographically the overall dislocation density decreases, the cell or fibre size increases and the dislocation structure inside the cells becomes less uniform. J_c decreases when recrystallisation commences and the cells become clear of dislocations. In fig. 13 a plot is shown of the critical current density as a function of fibre diameter in a field of 45 kG for both the forged and nitrogen-doped wires. From this figure it can be seen that the data on the wire samples divides into two groups. One group consists of both the as-worked alloy wires, and the 300° C aged nitrogen-doped alloy wires which have fibre sizes in the range 500 to 800 Å and a relatively low J_c. The fibres in the wires of this group contain a high, uniform density of dislocations. The other group which contains the rest of the data for both alloy wires fits an inverse dependence of J_c on fibre diameter and for fibre diameters of approximately 800 Å the J_c is approximately an order of magnitude greater than that of the first group.

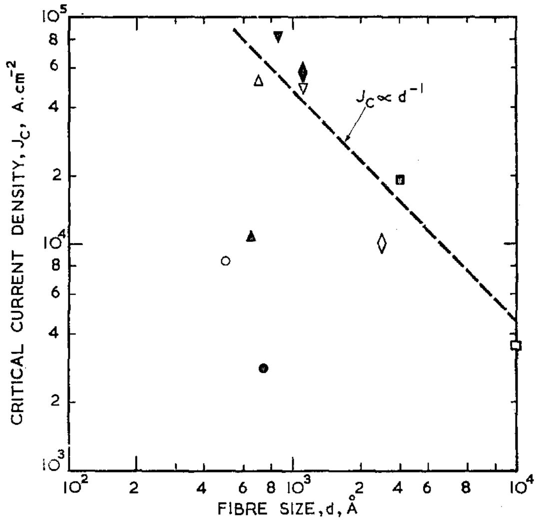


Figure 13 Effect of fibre size on the critical current density at a field of 45 kG; temperature = 4.2° K; solid symbols nitrogen-doped alloy wire; open symbols forged Nb–Ti alloy wire.

Very similar behaviour is found for all field values in the range 30 to 90 kG. The change from the first to the second group can be roughly characterised by the ageing temperatures at which individual dislocations and networks become resolvable inside the fibres. This inverse dependence on fibre diameter implies that the critical current density is a linear function of the number of fibre walls/unit length. Thus to maximise pinning there must be clearly defined fibre or cell walls with the fibre diameter as small as possible. Similar dependence of J_c on the number of boundaries/unit length have been observed; for example in Nb_3Sn [13] the appropriate boundaries are grain-boundaries, and in Pb–Bi alloy [14] the appropriate boundary is that between a normal bismuth precipitate and the matrix.

The main effect of the interstitials in solution is to impair the dislocation mobility on ageing because the dislocation core will be anchored by the interstitial cloud [8]. Thus higher temperatures will be required to produce dislocation motion and rearrangements in the nitrogen-doped alloy, and this is reflected in the critical current measurements and electron metallographic observations. Although interstitial precipitates could be observed on ageing the quenched alloy in the absence of cold-work, when this alloy was deformed no uniform and consistent precipitation was found. However, this is not surprising considering the high dislocation densities and low interstitial contents of the worked alloys. The interstitial precipitates are some complex of (titanium niobium); (oxide, nitride or carbide).

The J_c results for the sheet specimens are more difficult to explain. The maximum J_c (H || RP) occurs in the forged alloy sheet at variance with the wire results. The percentage deformation given to the sheet, is, however, very small and the nitrogen-doped alloy sheet shows no cell structure, looking along the rolling direction, away from the undissolved titanium nitride particles. The low anisotropy factors in the nitrogen-doped alloy sheet must be related to the uniform three-dimensional dislocation distribution in the worked state, and the high anisotropy of the forged alloy sheet follows from the two-dimensional nature of the dislocation distribution.

Other authors [15, 16] have studied the effects of interstitial addition on the critical currents of wires and find that with suitable heat-treatment the interstitially doped alloys can yield higher J_c's on ageing.

5. Conclusions

This investigation has shown that varying the heat-treatment both before and after working, changes the critical current density of a superconductor. However, to define the optimum course of treatment, it is necessary to know the field range and form of material to be used and the degree of deformation.

The results can be summarised as follows:

(i) Interstitials out of solution as preciptates are necessary to generate a uniform fine cell structure on deformation.

(ii) As-worked, the alloy with the interstitials in solution has a less well-defined fibre structure and a lower J_c than the alloy with the interstitials precipitated.

(iii) J_c is always improved by low temperature ageing after deformation.

(iv) When the interstitials are in solution the dislocation mobility is impaired and ageing temperatures 100° C higher than in the forged alloy are needed to give equivalent J_c values and dislocation distributions.

(v) For the wire-drawn alloys, in the optimum aged condition, the higher J_c is obtained with the nitrogen-doped alloy up to 75 kG and in the forged alloy above 75 kG.

(vi) For the cold-rolled alloys, the forged alloy exhibits far greater anisotropy and, in the optimum aged condition, a slightly higher J_c (H || RP) than the nitrogen-doped alloy.

(vii) The maximum J_c values occur when the cell or fibre wall is clearly differentiated from the fibre interior, in this condition the critical current density is found to be inversely proportional to fibre diameter. Thus the maximum J_c is obtained at the ageing temperature at which the dislocations inside the fibres or cells begin to move.

(viii) The results suggest that the highest J_c values should be obtained by a heat-treatment which produces a fine dispersion of precipitates and a high percentage of interstitials in supersaturated solid solution. For the particular alloy studied, 1000 ppm carbon and 1000 ppm nitrogen and quenching from 1100° C would seem to be suitable. This would leave some of the carbon as a carbide precipitate [17] and the nitrogen in solution. Working should lead to a well-developed cell structure and the nitrogen in solution should give beneficial increases in J_c on ageing.

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