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In niobium bicrystals of various preassigned misorientations, the superconducting critical current, I_C , has been measured as a function of the applied magnetic field, H , and the azimuthal angle, ϕ , between the grain boundary and H . Frequently, peaks in $I_C(\phi)$ with half width $\Delta\phi_{1/2} \sim 30^\circ$, as well as high pinning force, f_p , up to 10^{-4} Nm⁻¹ at 0.2T have been found for H both parallel and perpendicular to the grain boundary plane. Using f_p and $\Delta\phi_{1/2}$, obtained from bicrystals, the critical current density, J_C , has been calculated for polycrystalline samples. The results are in good agreement with experimental measurements both in magnitude and grain size dependence.

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INTRODUCTION

It is now well recognized that in a wide variety of both commercial superconductors, such as those based on the A-15 compounds, and other model type II superconducting materials, grain boundaries can be very effective in flux pinning. However, in the past, the underlying flux pinning mechanism has remained uncertain. A number of conflicting mechanisms such as interaction through the anisotropy [1] of Hc₂, or stress interaction between grain boundary dislocations and fluxoids [2] have been proposed. More recently, some definitive experiments on niobium bicrystals [3] and other work, both theoretical [4,5,6] and experimental [7,8], have pointed to an electron-scattering mechanism at grain boundaries. The use of bicrystals has been of great value in studies of flux pinning by grain boundaries. The advantages in using bicrystal samples are that the complications and uncertainties associated with the statistical summation of f_p are eliminated, and the pinning properties can be isolated for individual grain boundaries. The purpose of the present work has been twofold: 1. to extend flux pinning investigations to bicrystal specimens of various misorientations, which have thus far not been possible to grow, and 2. to calculate the critical current density in polycrystalline specimens, using the pinning parameters for individual grain boundaries from the bicrystal experiments. It will be shown that the critical current densities thus simulated for polycrystals not only agree well with the experimental data in magnitude, but also give the correct grain size dependence.

EXPERIMENTAL PROCEDURE

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Niobium bicrystals grown in a vertical floating-zone electron beam furnace with various preassigned misorientations were cylindrical in shape and approximately 0.6 cm in diameter. The details of the bicrystal growth procedure can be found elsewhere [9]. Cylindrical bicrystals, thus obtained and with grain boundaries at the center run-

ning parallel to the axis, were thinned to dumbbell shape for resistive measurements of the critical current. The procedure for sample preparations and its geometry have been published previously [3]. As in [3], the specimens were oxidized in air for 5 mins. at 400°C prior to the superconducting measurements to eliminate the surface contribution to flux pinning. Fig. 1 shows the experimental arrangement for the resistive measurement of I_c , which was measured as a function of H , and ϕ . All measurements were made at 4.2K.

RESULTS AND DISCUSSION

In contrast to the prior studies in niobium bicrystals [3], where some specific symmetric tilt boundaries were investigated and for which typical results are shown in Fig. 2, the present studies have generally revealed wide peaks in $I_c(\phi)$ with $\Delta\phi_{\frac{1}{2}} \sim 30^\circ$ and high f_p up to 10^{-4} Nm $^{-1}$ at $H = 0.2T$. Some typical results from the present studies are shown in Fig. 3. Similar observations have been made in a wide variety of niobium bicrystals with different misorientations. As outlined in [3], one can calculate f_p by equating the Lorentz force with the pinning force at a peak in $I_c(\phi)$. Thus,

$$f_p = \frac{I_c B a_0}{d} \quad (1)$$

where B is the magnetic induction, a_0 the flux line lattice parameter and d the diameter of the specimen. Using typical values from our present measurements, we obtain $f_p = 7 \times 10^{-5}$ Nm $^{-1}$ at $H = 0.2T$. In the present measurements, in addition to the peaks in $I_c(\phi)$ at $\phi=0$, i.e., for H parallel to the grain boundary, peaks were also found at $\phi = 90^\circ$, i.e., for H perpendicular to the grain boundary. The origin of the latter peaks in $I_c(\phi)$, as well as the reasons for the large difference in $\Delta\phi_{\frac{1}{2}}$ between different grain boundaries, are not understood at the present time.

CALCULATION OF J_c FOR POLYCRYSTALLINE NIOBIUM

Using average f_p and $\Delta\phi_{\frac{1}{2}}$ from our experiments on approximately ten bicrystal samples, it is possible to simulate J_c for polycrystalline niobium. The present section outlines the procedure. Consider a cylinder of polycrystalline niobium of length l , and average grain size $\langle D \rangle$. Then, the number of grains/cm 2 of cross section = $\langle D \rangle^{-2}$ and the total grain boundary length/cm 2 of cross section = $K_1 \langle D \rangle$, where K_1 = average ratio of the perimeter of a grain to its size. $K_1 \approx 3$. Of the total length of grain boundary per cm 2 in the cross section, a fraction $K_2 = 2\Delta\phi_{\frac{1}{2}}/\pi$ will lie within a favorable angle from H to enter into grain boundary pinning interaction. The factor 2 enters into the numerator of the expression for K_2 because of the two peaks in $I_c(\phi)$ at $\phi = 0^\circ$ and $\phi = 90^\circ$, which are of approximately equal height. Using force balance between the Lorentz force and the pinning force under the critical current condition, we obtain

$$(lA) J_c B = K_2 \frac{K_1}{\langle D \rangle} \frac{l}{a_0} f_p \quad (2)$$

Thus

$$J_c = \frac{1}{(\phi_0 B)^{\frac{1}{2}}} \frac{K_1}{\langle D \rangle} \left(\frac{2\Delta\phi_{\frac{1}{2}}}{\pi} \right) f_p \quad (3)$$

where ϕ_0 = flux quantum and $a_0 \approx (\phi_0 J_c B)^{1/2}$. We note that in deriving Eq. (3) a direct summation of f_p has been made to obtain the bulk pinning force. The direct summation is justified in view of the relatively long distance between the pinning segments (\sim grain size) of grain boundaries. In the presence of background pinning through pinning centers other than grain boundaries, we expect

$$\begin{aligned} (J_c)_{\text{total}} &= (J_c)_{\text{background}} + (J_c)_{\text{grain boundary}} \\ &= (J_c)_{\text{background}} + K \langle D \rangle^{-1} \end{aligned} \quad (4)$$

$$\text{where } K = \frac{K_1}{(\phi_0 B)^{1/2}} \left(\frac{2\Delta\phi_{1/2}}{\pi} \right) f_p.$$

Equation (4) can be looked upon as the analogue in superconductivity of the Hall-Petch relationship in metallurgy [10], which relates the yield strength to the grain size in a polycrystalline material. Using Eq. (3), $J_c(\langle D \rangle)$ could be calculated from the typical experimental values of f_p and $\Delta\phi_{1/2}$ in our experiments. These results are plotted in Fig. 4, and compared with published experimental results on polycrystalline niobium [11,12]. The agreement in terms of both the magnitude of J_c and its dependence on $\langle D \rangle$ seems to be good in view of the uncertainties in the fit of the experimental data over several decades and the gross assumptions made in the calculation of J_c . The polycrystalline experimental results plotted in Fig. 4 show a somewhat steeper slope than our calculated results; although, generally, in other type II superconductors [13,14], J_c is found to be proportional to $\langle D \rangle^{-1}$.

CONCLUSION

Use of niobium bicrystals has proven to be of great value in studying the flux pinning characteristics of individual grain boundaries. In contrast to previous work, we have found broad $I_c(\phi)$ peaks in some bicrystal samples with peaks for H both parallel and perpendicular to the grain boundary. Using the pinning properties of single grain boundaries, f_p and $\Delta\phi_{1/2}$, it has been possible to synthesize and thus simulate J_c in niobium polycrystals, which is in good agreement with published data both in magnitude and its grain size dependence.

LIST OF SYMBOLS

- a_0 = lattice parameter of the flux line lattice
- A = cross sectional area of niobium sample
- B = magnetic induction
- d = diameter of sample
- $\langle D \rangle$ = average grain size
- H = applied magnetic field
- I_c = critical current
- J_c = critical current density
- K_1 = average ratio of the perimeter of a grain to its size in the cross section
- K_2 = fractional part of grain boundary in the cross section, which can enter into flux pinning
- l = length of niobium sample

- ϕ_0 = flux quantum
 ϕ = azimuthal angle between I_c and H
 $\Delta\phi_{\frac{1}{2}}$ = half-intensity width of a $I_c(\phi)$ peak

ACKNOWLEDGMENTS

This work was co-sponsored by the National Science Foundation (Grant No. DMR-79 15799), and the Division of Materials Science, ^{U.S.} Department of Energy under contract W-7405-eng-26 with the Union Carbide Corporation.

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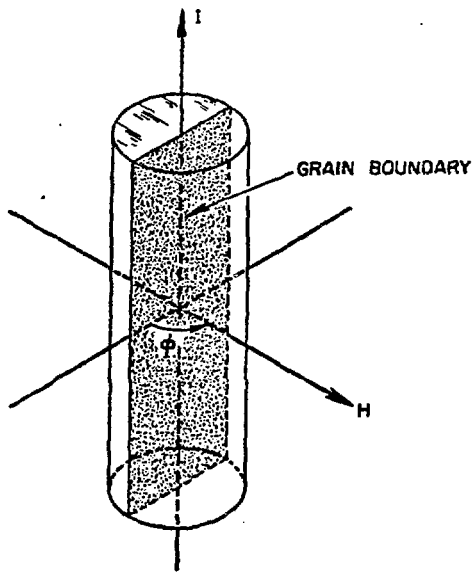


Fig. 1 Schematic view of experimental arrangement in the measurement of I_c

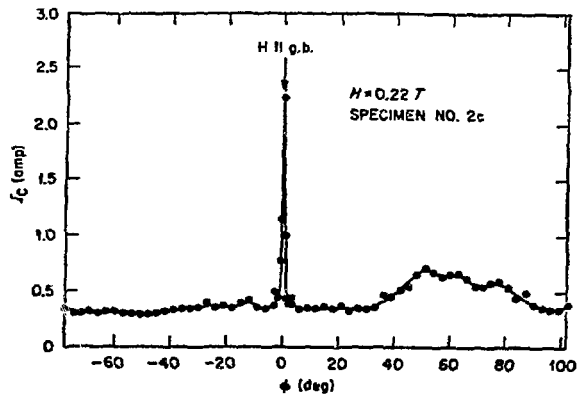


Fig. 2 Typical results of $I_c(\phi)$ from [3]. The bicrystal has a symmetric tilt boundary of 16° misorientation angle between $[011]$ vectors. The tilt axis is $[11\bar{1}]$

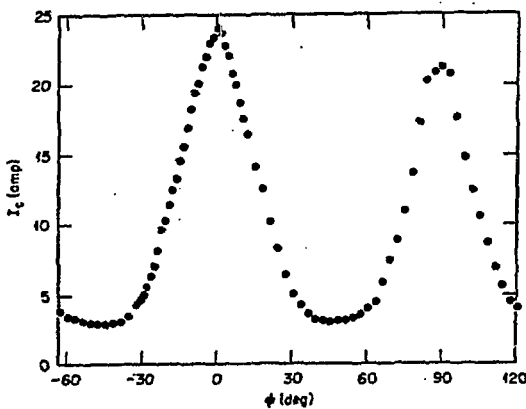


Fig. 3 Typical results of $I_c(\phi)$ in the present studies. $H = 0.248T$. The bicrystal has a symmetric tilt boundary of 5° between $[011]$ vectors. The tilt axis is $[01\bar{1}]$

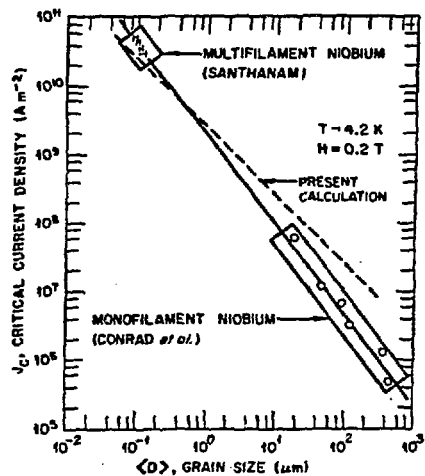


Fig. 4 Comparison of calculated $J_c(\langle D \rangle)$ with published data