Interface flux pinning in *in situ* formed superconducting composites

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Detailed flux flow studies of *in situ* formed wire and tape superconducting composites confirm our earlier findings that the filament-matrix interfaces are effective flux pinning centers. Angular dependence measurements on tape composites, where the ribbon-like filaments are parallel to the rolling plane, reveal a critical current anisotropy which depends on the magnetic field. The flux pinning force is also anisotropic; its complex field dependence, however, cannot be fully described using the existing flux pinning theories.

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INTRODUCTION

The interaction of plane boundaries with the flux line lattice in type II superconductors has long been of considerable interest. There is ample experimental evidence that in type II superconductors fluxoids interact strongly with both free surfaces and internal boundaries across which the superconducting properties change.[1] In this paper we report on flux motion in an inhomogeneous system containing a high density of plane superconductor-normal (S-N) boundaries. The samples used in the present study were prepared by an *in situ* technique and consist of 200–1000 Å thick discontinuous superconducting filaments embedded in a copper matrix.[2] An important feature of these composites is that the filaments are ribbon shaped due to the ⟨110⟩ texture formation and subsequent plane-strain deformation mode in niobium (or vanadium) during wire drawing.[3] This dense ribbon-like filament system provides the composite with an extremely high density of interfaces with which fluxoids can interact. In a wire composite, the filaments are neither uniformly stacked nor are their surfaces parallel to each other; the ribbon-like filaments are forced to fold, twist, and curl due to the constraints of the surrounding matrix. This greatly reduces the pinning capacity of most of the interfaces. When the wire composite is rolled into a tape, however, the filaments become flat and well aligned in the rolling plane of the tape (Fig. 1) leading to a maximum interaction of the flux line lattice with the internal boundaries.

The contribution of the filament-matrix interfaces to the bulk pinning force was studied by measuring the angular (θ) dependence of the critical depinning current and subsequent flux flow in a variety of composite tapes. A similar set of measurements was also performed on wire specimens identical with those used for rolling into tapes. Our results show that parallel internal boundaries in tapes are effective, and often dominant, pinning centers and contribute significantly to the current carrying capacity of *in situ* composites. Pinning force measurements show that pinning in the *in situ* composites is not fully explained by existing theories.

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EXPERIMENT

Our samples were prepared by first mixing copper and niobium, or copper and vanadium using rf melting followed by quick cooling in a water-cooled copper crucible. The two-phase as-cast alloys were then swaged and drawn, without intermediate annealing, to wires. Sections of these wires were then rolled to tapes. The Cu–Nb_3Sn composites were made by plating the wires or tapes with tin and then annealing them in vacuum at 560 °C for time spans of 200–300 h. The Cu–V_3Ga composites were made in a similar fashion but annealed at lower temperatures in the range 500–525 °C for

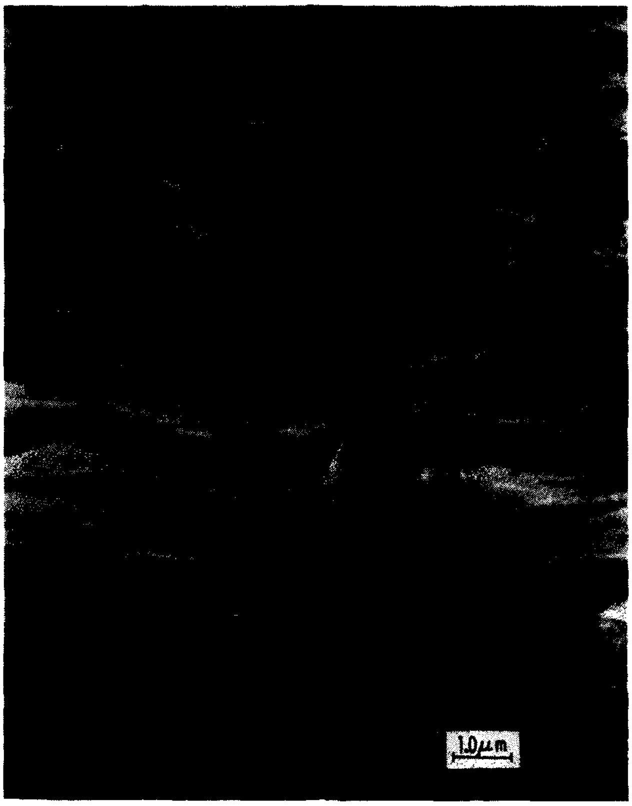

FIG. 1. Scanning electron micrograph of etched cross-section of an *in situ* Cu-Nb tape. Notice the dense and uniform distribution of the ribbon-shaped filaments.

350–450 h. More details on sample preparation are given in Refs. 4 and 5.

Critical currents J_c were obtained from the V-I curves at 4.2 K, using a 1 μV/cm criterion. The upper critical field H_{c2} was measured resistively by applying a small current density (about 5 A/cm^2) and sweeping the field. For the angular dependence study, the samples were mounted in a cryostat equipped with a variable-angle sample holder. The angle θ between the plane of the tape and the applied magnetic field could be varied during the test and was determined to within $\pm 1°$. For high H_{c2} samples, the applied magnetic field was produced by Bitter-type solenoid and kept perpendicular to both the axis of the tape or wire and the transport current. The experimental configuration is shown schematically in the inset of Fig. 2. Cu–Nb samples with low H_{c2} were studied using a conventional electromagnet and an otherwise similar setup.[6]

RESULTS AND DISCUSSION

A typical microgeometry of our *in situ* tapes is shown in Fig. 1 in which the Nb_3Sn filaments are exposed by etching away the copper matrix. The ribbon-like filaments are flat and fairly well aligned in the rolling plane of the tape. Along the direction normal to the rolling plane, however, the filaments are randomly stacked.

We find that critical currents of *in situ* wire composites are always lower than those measured in corresponding tape composites when the magnetic field is parallel with the surface of the tape. However, the critical currents of wires are higher than those of tapes when the magnetic field is perpendicular to the surface of the tape. This suggests that the filament-matrix interfaces play an important role in flux pinning. In fact, we find that by increasing the aspect ratio of the tape, the alignment of the filaments is improved and the flux pinning in the $\theta = 0$ direction is enhanced. Furthermore, we observe that the flux flow behavior of the tape composites is highly anisotropic. As the orientation of the flux lines deviates from being parallel to the surface of the tape to perpendicular, the critical depinning current decreases, and the flux flow characteristics gradually change.[7] The angular dependence of J_c obtained at $H = 3$ and 12 T in a Cu–Nb_3Sn tape composite is plotted in Fig. 2. The strongest pinning occurs when the flux lines are parallel to the filament-matrix interfaces and decreases as the flux lines intercept the interfaces. Similar behavior is also found in Cu–V_3Ga (Ref. 8) and CuNb samples.

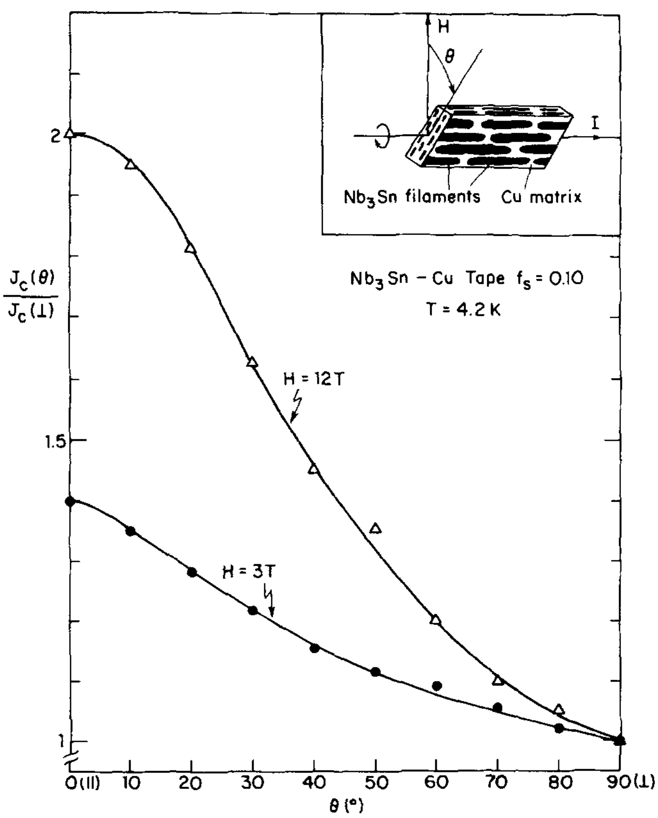

FIG. 2. Critical current anisotropy of a Cu-Nb_3Sn tape. $J_c(\theta)$ was obtained at constant magnetic fields $H = 12$ and 3 T. At a given magnetic field, interface pinning causes $J_c(\|)$ to be greater than $J_c(\perp)$.

The field dependence of the J_c anisotropy is expressed in terms of $\alpha(H) = [J_c(\|)/J_c(\perp)]_H$, where $\alpha(0) = 1$. For a typical Cu–Nb_3Sn sample, $\alpha(H)$ initially increases, passes through a plateau over the range $2 \lesssim H \lesssim 12$ Tesla and then rises steeply as H_{c2} is approached.[9] The increase of α at high field suggests that in the $\theta = 90°$ orientation, the normal state is reached at a field lower than that needed to drive the sample normal in the $\theta = 0°$ orientation. In fact, upper critical field measurements show anisotropy in H_{c2}.[6,7] Although this anisotropy in Cu-Nb_3Sn samples is not more than 2% and cannot completely account for the critical current anisotropy, it is incorporated in our present analysis by calculating $A(h) = [J_c(\|)/J_c(\perp)]_h$, where $h \equiv H/H_{c2}$. The variation of $A(h)$ with h is shown in Fig. 3 for two Cu–Nb_3Sn tapes of the same aspect ratio $\delta = 8$ but with different superconducting volume fractions $f_s = 0.10$ and 0.23. At zero field, $A(0) = 1$ and as h increases A increases and reaches a maximum value at h_m. For $h > h_m$, A decreases rapidly. We note that the magnitude of $A(h)$ increases with increasing f_s and the peak at h_m shifts to higher fields. Similar observations were also made in Cu–Nb tapes where f_s varied over a wider range.

The H_{c2} anisotropy in the Cu–Nb tapes[6] is more pronounced than in Cu–Nb_3Sn or Cu–V_3Ga tapes and analyzing the Cu–Nb data in terms of reduced fields is essential. For example, at intermediate fields, $\alpha(H)$ can be as high as 100 but $A(h)$ is about 2 (Fig. 4). We note that in Cu–Nb and Cu–Nb_3Sn tapes of similar aspect ratios $A(h)$ has not only a similar functional dependence on h but also similar values. This suggests a common origin of the critical current anisotropy and indicates that the origin is independent of the microstructure of the filaments.

The microgeometry of our *in situ* formed tapes allows for two distinct flux pinning centers: the microstructural features of the filaments, such as grain boundaries or dislocations, and the filament-matrix interfaces. For example, the Nb_3Sn filaments are wide enough to contain many grains across their width but too thin to have more than a few across their thickness. Therefore, flux motion is dominated by grain morphology and surface roughness[9] when the flux lines are perpendicular to the filaments. However, when the

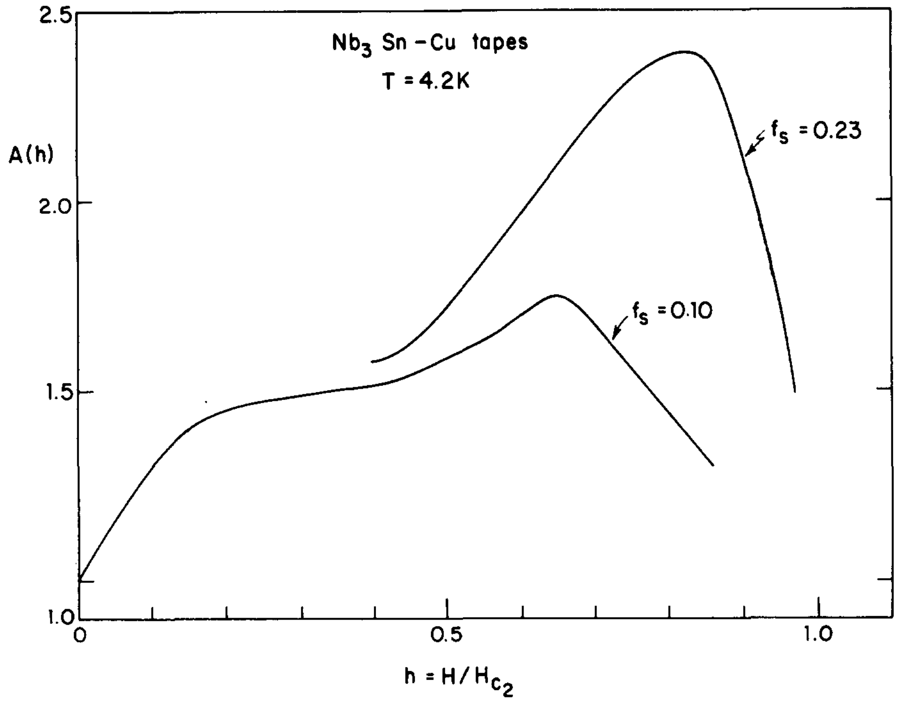

FIG. 3. Critical current anisotropy parameter $A(h)$ as a function of reduced magnetic field $h \equiv H/H_{c2}$. The magnitude of $A(h)$ decreases with decreasing superconducting volume fraction f_s and the peak shifts to lower h values.

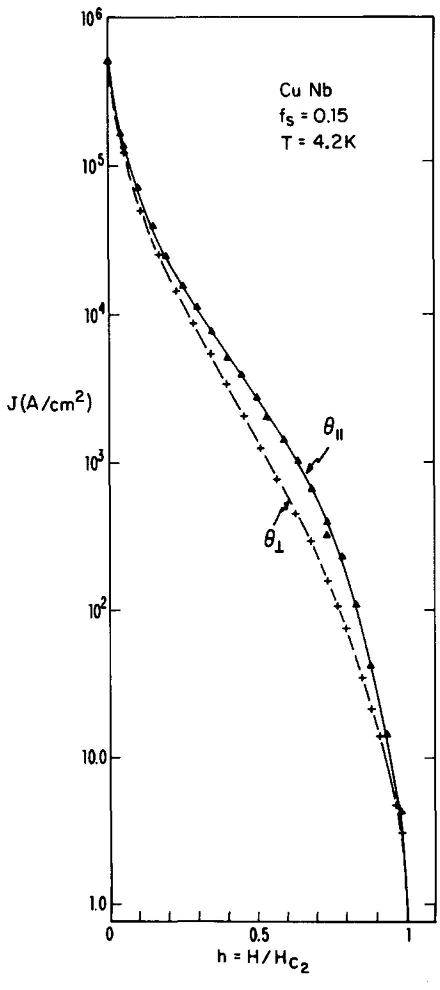

FIG. 4. Critical current $J_c(h)$ as a function of reduced magnetic field h, obtained on a Cu-Nb tape of aspect ratio 40 and area reduction of 4400. Notice the different field dependence of $J_c(\|)$ and $J_c(\perp)$.

flux lattice is parallel to the filaments, flux flow is hindered by the *S-N* interfaces. This may qualitatively account for the observed critical current anisotropy (Fig. 2).

The field dependence of the J_c anisotropy could be due to several factors. Although the *S-N* interfaces do not form a regular periodic array, one can expect that at higher fields (and smaller flux line lattice constant a_0) *S-N* interfaces with some degree of regularity will gain in pinning strength in comparison with grain boundaries since relatively more vortices will come into syncronization. This relative gain should be more pronounced in composites with higher interfacial density (i.e., higher f_s) and should occur at higher h, in agreement with data in Fig. 3.

Pinning force studies have been done on a number of Cu-Nb_3Sn, C-V_3Ga, and Cu-Nb samples. Qualitatively, $F_p(H)$ has similar behavior in all these samples, but the magnitude of F_p and the position of the peak are different. For example, as the composition of the V-Ga filaments in Cu-V_xGa_{1-x} samples approaches the stoichiometric composition ($x = 0.75$), F_p increases and the position of the peak, h_p shifts to lower fields[8]; the best samples had h_p close to 0.2. Another factor that affects the position of the peak is the coupling among filaments. In Cu-Nb_3Sn and also in Cu-Nb samples having the same aspect ratio but different volume fractions of Nb, we find that h_p shifts to lower h with decreasing f_s.

An important point can be drawn from our observations: all *in situ* samples have pinning forces that peak at very low fields and the position of the peak is not consistent with the predictions of theories or observations on bulk samples for the standard types of flux pinning sites. In materials with grain boundaries as pinning centers the pinning force peaks at h_p ranging from 0.2 to 0.5.[1] In our high critical current Cu-Nb_3Sn and Cu-V_3Ga samples, where the filaments consist of A-15 grains, h_p ranges between 0.09 and 0.2. Moreover, in Cu–Nb, where the filaments are essentially single crystals with a high density of dislocations, h_p is very low (in the range 0.05 to 0.1). F_p measurements on bulk samples with different dislocation density showed that h_p is about 0.2 to 0.3 and increases to 0.75 in samples with high dislocation density,[1] whereas theory predicts h_p of about 0.3.[1]

Over a limited range of h, F_p is found to be proportional in all samples to $h^{1/2}(1-h)^2$. This functional relationship has been often taken as evidence of the shearing of the flux line lattice at high fields.[11] Similar relationship has been derived also for surface pinning with a core interaction[10] and most recently for *N-S* boundary pinning.[12] Our results are invariably in poor agreement with at least some of the predictions of these theories. In particular, H_{c2} extrapolated from J_c data using Kramer's formula is much lower than resistively determined H_{c2}'s (Ref. 4 and Fig. 5) and peak positions of F_p in most samples fall short of the predicted values as discussed earlier. When applying Kramer's model, it is important to bear in mind its statistical nature, i.e., the assumption of a large number of pinning centers of strengths that have a normal distribution function. It is unlikely that such assumptions, which may be valid for bulk materials, are applicable in the *in situ* composites.

There are further qualitative discrepancies in the F_p behavior. As pointed out earlier, the field dependence of J_c, and therefore F_p, in *in situ* samples varies with the orientation of the magnetic field and F_p maxima occur at different h_p values. In Fig. 6, we show measurements of $F_p(H)$ for $\theta = 0$ and 90° obtained on a Cu–Nb sample. Notice that at a given field H, or h, $F_p(\|) > F_p(\perp)$ because of the additional interface pinning. However, $h_p(\|) > h_p(\perp)$ which is unexpected if Kramer-type flux shearing exists at high fields and unlike what is seen in bulk samples.[13] Moreover, theory predicts that $F_p(H)$ at high fields should not depend on the type of the pinning sites which, again, is contrary to our observations.

The source of these discrepancies may be in the fact that *in situ* samples are simply not very suitable to test theoretical predictions derived for less complex and more homogeneous systems. It is therefore difficult to argue on the basis of our results about relative merits of various pinning theories. It appears that flux pinning in *in situ* composites requires additional theoretical treatment which will take into account their characteristic microstructural features and, particularly at low fields, contribution from proximity-effect induced superconductivity. However, the conclusion that filament-matrix interfaces can be dominant pinning centers in composites with very small filaments remains valid and has been supported by additional experimental evidence.[14]

Additional information about flux flow can be obtained from the *V-I* characteristics. At a given field, the *V-I* curve, obtained for a bulk sample, has a nonlinear voltage dependence on current near I_c, followed by a linear dependence at higher currents. The origin of the nonlinear region is usually attributed to a range of local pinning strengths.[15,17] Therefore, a change in the characteristics is expected[18] and observed[17,19] near h_p. We examined *V-I* curves obtained mainly on Cu–Nb samples where the critical current is low enough so that heating is unimportant even for $I \gg I_c$. Figure 7 shows typical *V-I* curves obtained on a tape sample. We find that at high fields there is an extensive nonlinear region and as the magnetic field decreases the *V-I* curves become sharper. More quantitatively, we use the parameter I_0/I_c to measure changes in the *V-I* curves. I_c is here the critical current and is associated with the minimum pinning strength and I_0 is defined as $I_0 = I_f - I_c$ where I_f is the value of current above which uninterrupted flux flow occurs. The parameter I_0/I_c therefore represents the ratio of the spread in pinning strength to the weakest one and measures the variation of pinning strengths.

The *V-I* curves of CuNb wires and tapes were examined in terms of the I_0/I_c parameter. Uncommonly high values of I_0/I_c are obtained at high fields. For example, in a 45-μm-diam wire I_0/I_c is about 5 at 0.9 H_{c2} and decreases to about 1.5 at 0.2 H_{c2}. Below h_p ($\simeq$0.09 in this sample) we expect

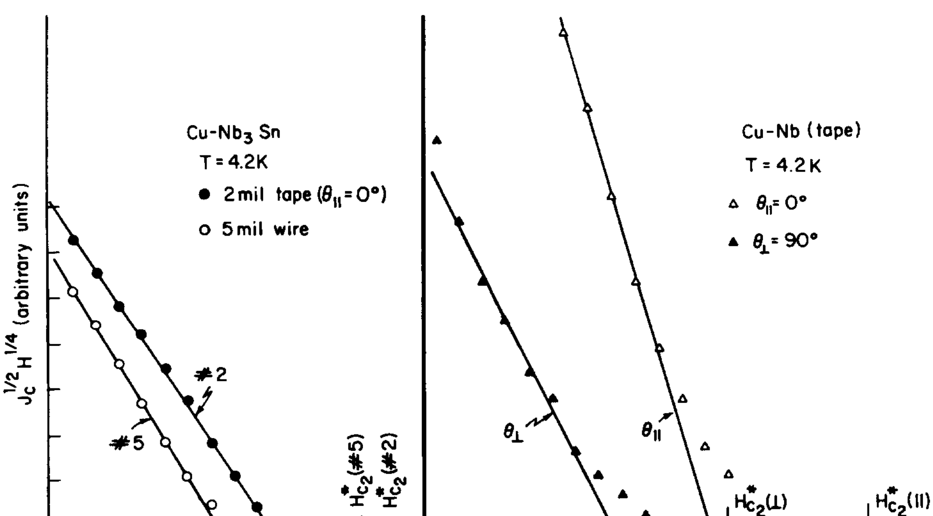

FIG. 5. Plot of Kramer shearing function, $J_c^{1/2}H^{1/4}$, as a function of applied magnetic field, (a) for a Cu-Nb$_3$Sn tape and wire; (b) for a Cu–Nb tape at different orientations.

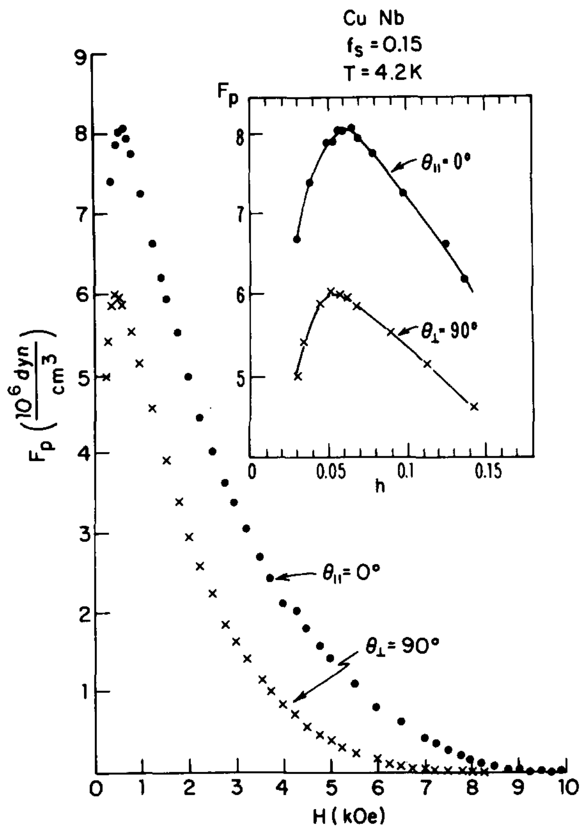

FIG. 6. Pinning force as a function of applied field, $F_p(h)$, for $\theta = 0°$ and $\theta = 90°$, obtained on a Cu-Nb tape made from a 127-μm wire and rolled to an aspect ratio of 40 and area reduction ratio of 4400. In the inset, $F_p(h)$ is plotted against h and shows the change of h_p with θ.

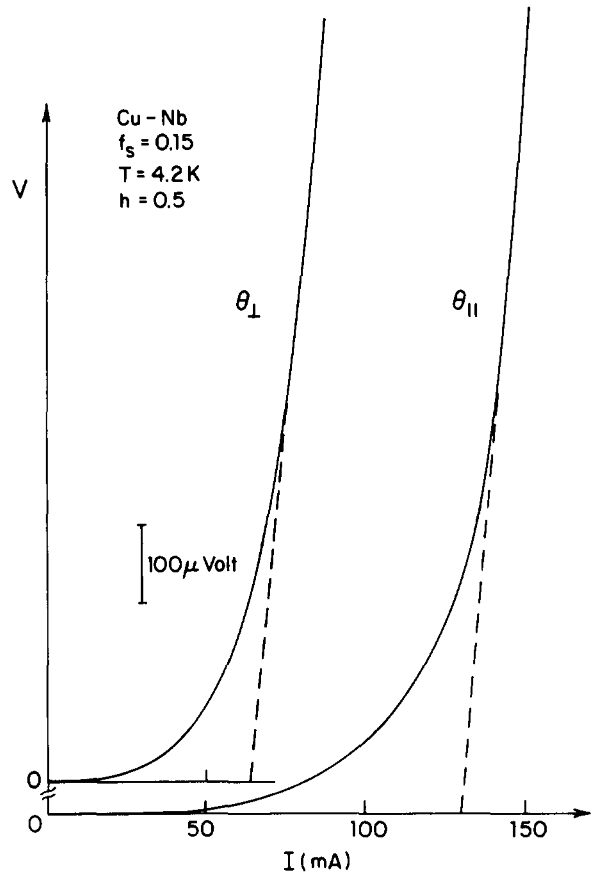

FIG. 7. V-I characteristics as a function of θ obtained at a constant reduced field $h = 0.5$ on a Cu-Nb tape of aspect ratio 40 and area reduction ratio 4400. The dashed line intersects the current axis at I_f.

sharp V-I curves corresponding to the pin breaking regime. Unfortunately, due to the high J_c at low fields we were not able to obtain V-I curves without excessive heating and hence it was not possible to follow the changes in I_0/I_c below h_p. Nevertheless, the high values above h_p indicate a wide distribution of pinning strengths which reflects the microstructure of these composites. Indeed, as the sample is drawn further and filaments become longer and closer spaced, I_0/I_c decreases. In a 127-μm-diam sample, I_0/I_c at $h = 0.5$ is about 3 and decreases to 1.5 when drawn to 45 μm. Moreover, in tape composites the relative spread in the $\theta = 0°$ direction is less than in the $\theta = 90°$ direction. In a CuNb tape of aspect ratio 40 (rolled from a 127-μm-diam wire), I_0/I_c values measured at $h = 0.5$ were found to be 2.3 and 3.3 for $\theta = 0°$ and $\theta = 90°$, respectively.

SUMMARY

We conclude that in the *in situ* formed superconducting composites, the internal S-N boundaries are effective pinning centers when the flux lines are parallel to the broad surface of the filaments. In those conductors where a high temperature diffusion anneal is necessary to convert the filaments to the A-15 structure this fact is of considerable practical importance. Whereas grain boundary pinning becomes less effective because of the rapid grain growth, the internal boundaries remain strong pinning centers as long as the filament integrity is preserved.

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