Periodic pin array at the fluxon lattice scale in a high-field superconducting wire

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A periodic pin array has been made at dimensions approaching the diameter and spacing of the fluxon cores in an "island"-type of artificial pinning center superconducting composite wire. Gun-barrel drilling was used to precisely incorporate a hexagonal array of Nb 1.3 wt. % Ti pinning centers into an ingot of Nb 46.5 wt. % Ti, before extrusion and wire drawing. Transmission electron microscopy images showed little evidence of pin geometry alteration down to 100 nm pin diameter. At ~30 nm effective pin diameter, the aspect ratio of the pins was 3–5, and the pin array retained its hexagonal geometry. Bulk pinning force curves had temperature-independent structure down to 15 nm effective pin diameter, suggestive of matching between fluxons and pins. Because of the uniform pin geometry, the maximum bulk pinning force exceeded that of the best conventional composites, even though the upper critical field and the volume fraction of pins were significantly lower.

Artificial pinning center (APC) superconducting composites offer exciting new opportunities for high-field magnet applications.[1,2] They permit volume fractions and types of pins which are not constrained by the thermodynamics and kinetics of the particular high field superconducting matrix, and therefore offer great potential to increase the critical current density (J_c).[3] In Nb-Ti based APC composites, a volume fraction of pins as high as 50% has been reported,[4] as compared to the 17%–22% of α-Ti precipitates possible in conventional Nb 46.5 wt. % Ti wires.[5,6] A bulk pinning force ($F_p = J_c B$) exceeding 28 GN/m^3 has been achieved at 3 T, 4.2 K in an APC composite with 34 vol % of pins,[7] far exceeding the best pinning force in a conventional round Nb-Ti wire, 18–19 GN/m^3 at 5 T, 4.2 K.[8]

In principle, APC construction is simple, since the pin geometry is determined by billet stacking at a scale of a few cm. Of course, optimum flux pinning requires control of the microstructure on the scale of the fluxon diameter, 1–10 nm for useful high-field superconductors, and the fabrication of a nanometer-scale microstructure thus requires unusually large mechanical strains. In this letter, we report recent success in achieving regular nanometer-scale pin arrays in 3721 and 26 047 filament APC Nb 46.5 wt. % Ti composites, and we show that such arrays can have unusually good superconducting properties.

The APC composite was made by gun-barrel drilling a hexagonal array of 61 holes into a 146-mm-diam ingot of Nb 46.5 wt. % Ti, and filling the holes with 61 rods of Nb 1.3 wt. % of Ti, the pinning center. The edge-to-edge separation of the holes nominally equaled the 7.3 mm diameter of the holes; the ratio of the pin diameter to the ingot diameter was thus 1:20. The overall volume fraction of the pinning centers was 15.3%. Nb 1.3 wt. % Ti was chosen for the pins because its mechanical properties were presumed to be similar to those of Nb 46.5 wt. % Ti, because of its availability, and because its upper critical field (B_{c_2}) of about 0.8 T at 4.2 K is much less than the ~11 T B_{c_2} of Nb 46.5 wt. % Ti.

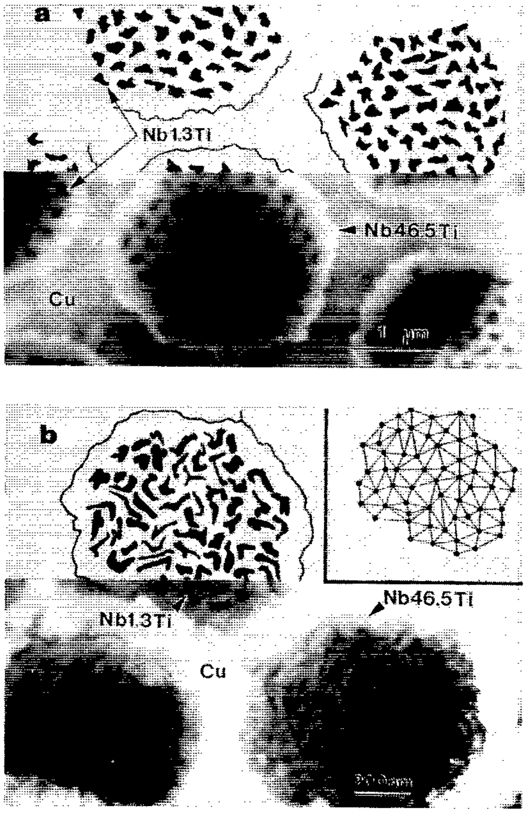

FIG. 1. APC composite cross sections, as observed by transmission electron microscopy. (a) Filament cross sections at $d_p = 92$ nm, shown in a photograph (lower half) and a sketch (upper half). The components are labeled. (b) Filament cross sections at $d_p = 29$ nm. Inset: Centroids of the pins, represented by dots, for the top left filament. The lines that connect the dots are described.

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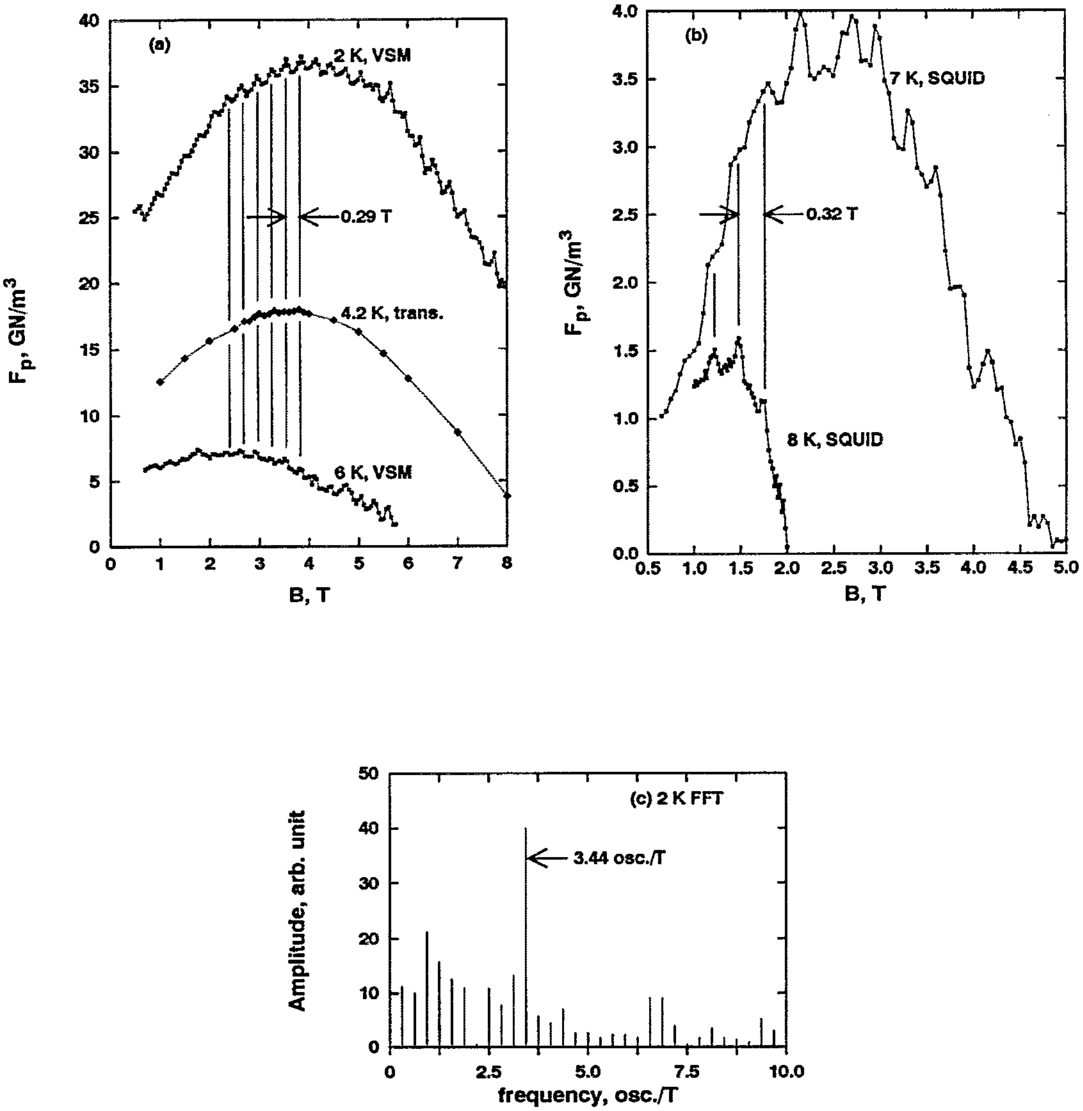

FIG. 2. (a) The bulk flux pinning force at 2–6 K for a 15 nm effective pin diameter. The 2 and 6 K data are derived from magnetic measurements using a vibrating sample magnetometer (VSM), and the 4.2 K data are obtained from a measurement of the transport critical current density (trans.). (b) The bulk pinning force at 7 and 8 K, derived from magnetic measurements using a superconducting quantum interference device (SQUID). The vertical lines in (a) and (b) are drawn as a guide to the eye. (c) A fast Fourier transform (FFT) of the 2 K data shown in (a), after a least-squares fit was subtracted, for the 128 data points between 0.5 and 6.9 T. The data points are spaced by 0.05 T.

The ingot was extruded at ~676 °C to ~ 41 mm diameter. All subsequent processing was by wire drawing at room temperature; no precipitation heat treatment was given. After a reduction to ~6.9 mm diameter, the Nb-Ti rods were clad in Cu. The wire drawing of the Nb-Ti/Cu composite used a series of stacking stages: Two stacks of 61 elements gave effective pin diameters down to 29 nm. An additional stack of 7 elements gave effective pin diameters down to 7 nm. The final products thus contained a total of 3721 and 26 047 filaments. The effective pin diameter (d_p) used to characterize the pins is calculated from the number of filaments in the composite (N), the area (volume) ratio of copper to superconductor (R), and the wire diameter (d_w) using the following relationship:

$$d_p = d_w/20\sqrt{N(1+R)}. \qquad (1)$$

The regularity of the microstructure at different composite sizes can be assessed from Fig. 1. Figure 1(a) shows that the pins are extremely regular in position and shape for $d_p = 92$ nm. At $d_p = 29$ nm, Fig. 1(b), the pins' cross sections have become more planar, their aspect ratio being about 3–5:1. Below this size, the pins' aspect ratio increased, and they became hard to distinguish in bright-field transmission micrographs; evidently, the microstructure became extremely irregular below $d_p \sim 30$ nm.

The image in Fig. 1(b) describes the microstructure with the strongest F_p. It is interesting that the aspect ratio of the pins is much less than is observed for α-Ti precipitates at the optimum composite size in conventionally processed Nb 47.8 wt. % Ti, 30–50:1.[9] Also, the shape and the orientation of the individual pins do not appear correlated across the filament cross section. Viewed more collectively, however, much of the hexagonal coordination of the original pin geometry is maintained. The pins are easily counted; they tend to line up in rows. The positions of the centroids of the pins, shown in the inset of Fig. 1(b) for the top left filament, retain their original sixfold coordination. In the inset, the dots represent the positions of the centroids. The centroids of the initial pin array are connected by 156 lines; here the same number of lines is drawn, such that the total line length is minimized. Of the internal 37 centroids, many (19) have six lines connected to them, and an equal number of centroids (9) have either fewer lines or more lines. Three average orientations of the rows of centroids emerge, being ~60° from each other.

A periodic flux pinning potential should result from the periodic pin array. Electromagnetic results support this hypothesis: Matching of the flux lattice to the pin geometry is observed from large sizes down to 15 nm effective pin diameter. The data for $d_p=15$ nm is shown in Fig. 2. In plots a and b, periodic structure appears on the F_p curves at 2, 4.2, 6, 7, and 8 K. The periodicity is not dependent on the temperature, being about 0.3 T. The absolute positions of the peaks also appear to be temperature independent. The periodicity appears for the three independent measurement techniques, and thus cannot be attributed to noise or artifacts of the apparatus. Furthermore, a Fourier transform of the 2 K data measured by vibrating sample magnetometer covering the 128 data points between 0.5 and 6.4 T was made after subtracting a smooth curve generated by a least-squares fit, as shown in Fig. 2(c). The subtraction removed the fundamental signal of the sine-wave-like bulk pinning force curve. A peak occurs at a frequency of 3.44 oscillations per tesla, and an overtone at a frequency of 1.72 oscillations per tesla, giving a periodicity of 0.29 T. We believe that the structure is due to a slight enhancement of the pinning force when the fluxon spacing matches that of the pin array, as discussed more fully in a forthcoming article.[10] Since any matching effect must be the result of a collective fluxon interaction, the underlying pin geometry must be uniform over several fluxon spacings.

Figure 2 also demonstrates that the bulk pinning force of this APC composite is very high. The maximum value of F_p at 4.2 K is about 18 GN/m^3 in Fig. 2(a), and the highest value of F_p obtained for this APC composite exceeded 22 GN/m^3 at 4.2 K at an effective pin diameter of 25 nm.[11] This pinning force exceeds the highest pinning force that can be attained in a conventionally optimized Nb-Ti composite, 18–19 GN/m^3, even though in this APC composite the upper critical field is ~2 T lower (~9 T vs 11 T) and the volume fraction of pins is about two-thirds (15.3% vs 17%–22%). We believe strong flux pinning in the APC composite directly results from its uniform microstructure, which maximizes the efficiency of the pins.

The $d_p=25$ nm sample achieved the strongest bulk pinning force because the elementary core pinning force was maximum. The core pinning force should depend upon how much of the fluxon core can be placed inside a pin; it should be maximum because the thickness of the $d_p=29$ nm pins in Fig. 1(b), ~12 nm, is about the same as the diameter of the fluxon core at 4.2 K. In contrast, strong flux pinning is obtained in optimized conventional Nb-Ti composites because the high number density of the α-Ti precipitates offsets their relatively weak elementary pinning force.[12] The average thickness of the precipitates is ~0.2 times the diameter of the fluxon core, and their elementary pinning force is correspondingly ~20% of the maximum elementary pinning force. Clearly, the APC composite is more efficient.

In conclusion, we have shown for the first time that a highly uniform, periodic array of flux pinning centers can be made on a scale below 30 nm in a Nb-Ti APC composite wire. These results are the first to be obtained near to the optimum flux-pinning size for an "island"-type of APC composite, and are in contrast to the very inhomogeneous microstructure seen for some "sheath"-type APC composites at optimum size.[13]

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