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IRREVERSIBILITY BEHAVIOUR IN Ag-SHEATHED Bi-BASED SUPERCONDUCTING WIRES

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ABSTRACT

Irreversibility lines for Ag/$(Bi,Pb)_2Sr_2Ca_2Cu_3O_y$(2223) wires prepared through a phase formation–decomposition–recovery(PFDR) process and normal annealing process were determined using both AC susceptibility measurements under DC fields and magnetisation measurements. It was found that flux pinning was enhanced in the PFDR processed samples over the normal processed samples, in particular at temperature above 77 K. The PFDR process results in high mass density, grain alignment, uniform distribution of impurity precipitates and high density of defects. The irreversibility temperatures scaled with the applied field according to $H^{1/3}$, which is in contrast to $H^{2/3}$ law for $YBa_2Cu_3O_{7-x}$ and conventional superconductors. The irreversibility lines for PFDR processed tapes showed a crossover with those for normal processed tapes at temperature below T_c of the $(Bi,Pb)_2Sr_2CaCu_2O_{8+x}$ (2212), suggesting that at temperature above T_c of the 2212 phase, the 2212 as nonsuperconducting region, may serve as effective pinning sites for fluxoids.

INTRODUCTION

A number of factors influencing the J_c–H behaviour in Ag-clad $(Bi,Pb)_2Sr_2Ca_2Cu_3O_{10+x}$ wires [1–4] have been discussed previously, which includes grain alignment, temperature, duration and atmosphere of heat treatment, doping and substitutions, and phase assemblage. It is clear that densification and grain alignment, derived from repeated rolling, pressing, and sintering, are critical for raising the J_c. Other factors, such as Ag additions [5], Ca_2CuO_3 excess [6], uniform distribution of impurities, elimination of carbon [7], and partial melting, are also beneficial to the J_c–H characteristics. However, some factors are still not well understood. For example, the low-T_c phase $(Bi,Pb)_2Sr_2CaCu_2O_{8+x}$ (2212) and various defects have been observed in Ag-clad tapes but the effect of these phases on the J_c is not clear.

Recently, a high-T_c phase formation–decomposition–recovery process(PFDR) through the use of a short period of melting has been developed for fabrication of Ag-clad Bi–Pb–Sr–Ca–Cu–O (BPSCCO) wires[8]. The PFDR processed tapes exhibit 3 to 10-fold increase in the J_c at 77 K and 1 Tesla over the normally processed tapes. A J_c of 40000 A/cm^2 at 77 K and 0 T and 9000 A/cm^2 at 77 K and 1 T have been achieved. The weak links and flux pinning have been significantly improved and an extended plateau regime in the J_c–H curve has been observed. The improved J_c – H characteristics is attributed to the desirable microstructures consisting of dense, well aligned $(Bi,Pb)_2Sr_2Ca_2Cu_3O_{10+y}$ (2223) grains, fraction of $(Bi,Pb)_2Sr_2CaCu_2O_{8+y}$(2212)

and high density of dislocations produced through PFDR process.

In the present work, we report results on the irreversibility behaviour for the PFDR processed Ag-clad $(Bi,Pb)_2Sr_2Ca_2Cu_3O_{10+y}$ wires in comparison with the normal processed tapes.

EXPERIMENTAL PROCEDURE

Powders were prepared both by freeze drying and thermal decomposition of metal nitrate solutions having the ratio Bi:Pb:Sr:Ca:Cu = 1.85:0.35:1.90:2.05:3.05. The powders were calcined at 830°C for 10 h, pressed into pellets, and sintered in a muffle furnace at 845°C for 20 h. X-ray diffraction patterns obtained using a SIEMENS D5000 diffractometer showed that the major phase in the samples was $(Bi,Pb)_2Sr_2CaCu_2O_{8+x}$ (2212); the high-T_c phase $(Bi,Pb)_2Sr_2Ca_2Cu_3O_{10+x}$ (2223) had not been formed at this stage. The pellets were crushed and ground with a mortar and pestle for ~1/2 h or ball milled for 12 h with hexane as medium in order to compare the effect of particle size on the properties of the final products. The powders were then pressed into round bars of dimensions 50 mm length and 8 mm diameter. The bars were loaded into silver tubes of 10 mm outer and 8 mm inner diameters, and the compositions were then drawn to a final diameter of 0.7–1.0 mm. The wires were rolled into tapes of overall thickness ~0.1 mm and width 2–3 mm. The resultant tapes were heat treated through three annealing cycles as described previously[3] which is referred as a normal process in this paper. Alternatively, the tapes were treated with the PFDR process[8].

The transport critical current density was determined from the current/voltage curve under magnetic fields (H) varying from nil to 1.2 T using a 1 μV/cm criterion. Measurements of the AC magnetic susceptibility were obtained using a mutual inductance bridge. In order to measure the real (χ') and the imaginary (χ'') components of the susceptibility, the phase angle of the lock-in amplifier was adjusted to null χ'' output at 120 K. A DC magnetic field was superimposed on an AC field in order to compare the behaviour of the samples treated differently. The magnetic hysteresis curves were obtained with a commercial superconducting quantum interference device (SQLIID) at various temperatures and magnetic fields up to 5 T. The irreversibility lines were determined from both magnetisation and AC susceptibility measurements. The irreversibility temperatures were determined at the lowest temperature where the magnetisation measured both in zero-field cooling (ZFC) and field cooling (FC) became reversible.

RESULTS AND DISCUSSION

As shown previously[8], the PFDR processed BPSCCO tapes exhibited a significant enhancement of the J_c in magnetic field. In order to confirm the effect of the phase decomposition on the flux pinning the irreversibility lines (IL) for PFDR processed and normal processed samples were determined through the magnetisation measurement in a wide range of temperatures between 4.2 K and T_c and AC susceptibility measurements at temperatures near T_c. Figure 1 shows the irreversibility lines for a PFDR processed tape A (J_c = 19,000 A/cm^2 at 77 K and 0 T) and a normal processed tape B (J_c = 9,900 A/cm^2 at 77 K and 0 T). It is evident that the PFDR sample A shows significantly stronger flux pinning than the normal sample B at high temperatures.

It should be noted that the two irreversibility lines showed a crossover at low temperature. The magnetisation hysteresis loops for samples A and B at 40 K and 4.2 K are shown in Figure 2. The magnetisation hysteresis deference (ΔM) is a measure of flux pinning strength. As seen from Figure 2 the ΔM for sample A is larger than that for sample B at fields greater than 1.0 T at 40 K, whereas the ΔM for sample A is smaller than that for sample A at 4.2 K i.e. the PFDR processed tape A has stronger pinning at high temperatures, while the normal processed tape B has stronger pinning at low temperature.

The irreversibility lines for the PFDR processed tapes A and C, and normal processed tape D (without melt) determined from AC susceptibility measurements under DC fields are presented in Figure 3. The irreversibility temperature at a given DC field is determined from the position of the loss peak in the imaginary part (χ'') of the AC susceptibility with a fixed AC field of 0.3 Oe and a frequency of 1000 Hz. As seen from Figure 3 the IL for the PFDR processed tapes A (J_c = 19,000 A/cm^2) and C (J_c = 14,000 A/cm^2) are positioned at higher temperature than that for the normal processed sample D. This confirms that the PFDR processed tapes have greater pinning strength than the normal sintered sample D.

As has been observed previously [8], the 2223 phase decomposed into 2212 phase and some impurity phases Ca_2CuO_3 and $SrCaCu_2O_4$ during a short

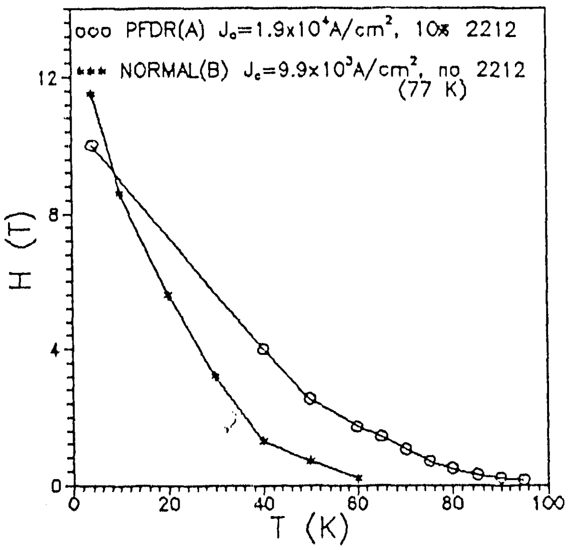

Fig. 1. Irreversibility lines for the PFDR processed tape A and normal processed tape B

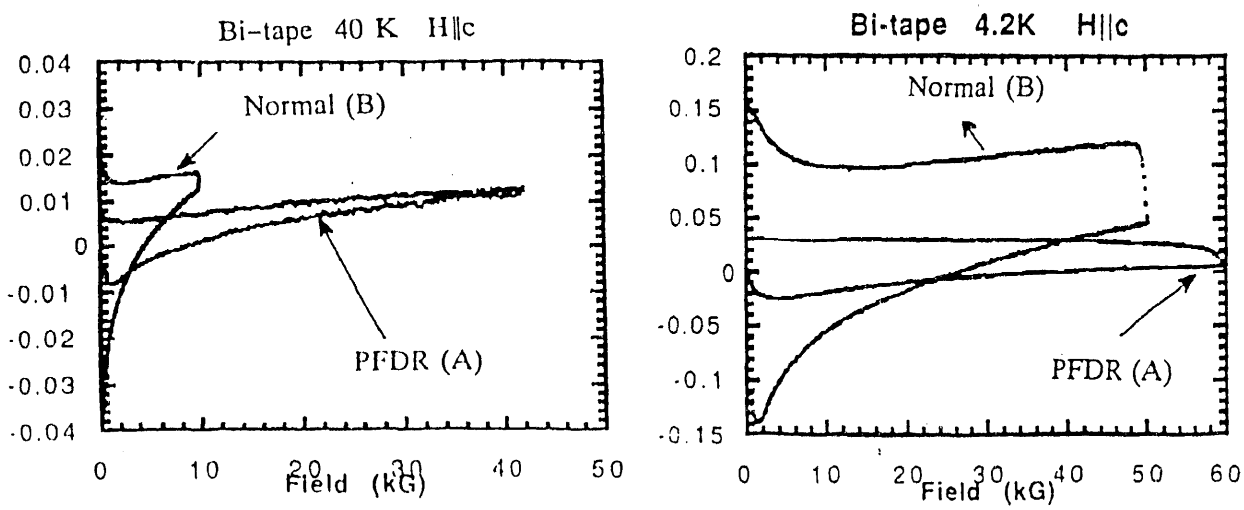

Fig. 2. Comparison of magnetic hysterisis curves for the Ag/2223 tapes A and B at (a) 40 K and (b) 4.2 K

period of melt processing at 860°C. The 2223 phase was then recovered through a post annealing. However, the degree of the 2223 phase recovery was variable depending upon the length of the melt period and the post annealing time. The fraction of 2223 phase decreased with increasing duration of melt. X-ray diffraction studies reveal that the typical PFDR processed tapes all contain a fraction of 2212, while the 2212 content in the normal processed tapes is usually very low. The amount of 2212 is estimated from the ratio of the corresponding 2223 peaks to be 10% for tape A and 6% for for tape C. The electron micrograph(Figure 4) shows that the microstructure of the PFDR processed tape A has well aligned and elongated grains, with a high apparent density and no large secondary phase particles. In addition, elongated 2212 phase (white), with a thickness smaller than 1 μm and the impurities in the PFDR processed tape A are uniformly distributed within the textured 2223 matrix [9]. It is argued that at temperature above T_c of 2212 and below that for 2223 the highly dispersive 2212 phase and Ca-Cu-O and Sr-Ca-Cu-O precipitates, as nonsuperconducting regions, serve as desirable pinning sites, contriduting to the enhancement in the flux pinning strength in the PFDR processed samples.

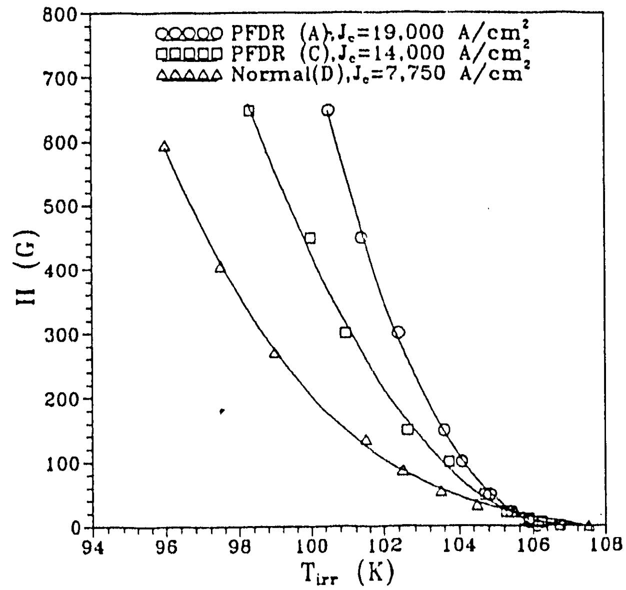

Fig. 3. Irreversibility lines for the PFDR processed Ag/Bi-2223 tapes A, C and normal processed tape D

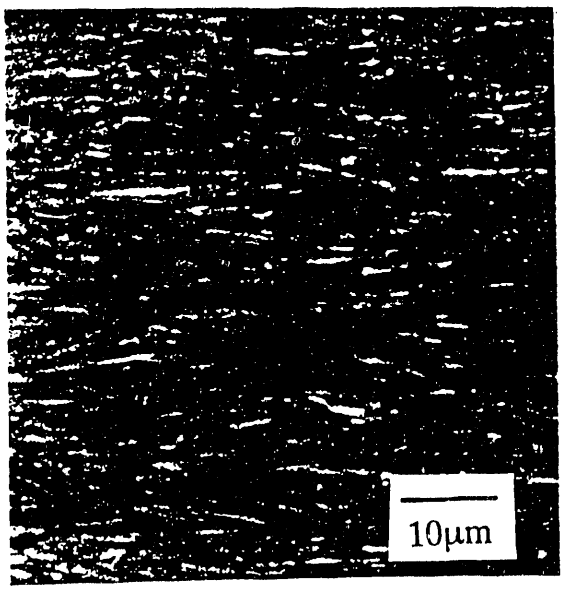

Fig. 4. SEM images of the PFDR processed Ag-clad 2223 tape A (J_c=19000 A/cm^2)

To study the scaling behaviour of the irreversibility temperature with magnetic field, the following relationship was obtained from the AC susceptibility data:

$$(1 - T_p / T_c) = \alpha H^{1/3} \qquad (1)$$

where α is a numerical constant and T_{irr} / T_c is a reduced irreversibility temperature. Figure 5 shows the linear relationship between $(1 - T_{irr} / T_c)$ and $H^{1/3}$ for tapes A, C and D, from which α can be calculated. Magnetisation measurements under field cooling as well as zero field cooling have been used to determine the IL for tapes A and B, which shows a close linear relationship for $(1 - T_{irr} / T_c)$ versus $H^{1/3}$ in the range from 600 Oe to 4 T. This is consistent with the scaling law in the range from 100 Oe to 594 Oe, as determined by AC susceptibility measurements. The scaling behaviour of $H^{1/3}$ is consistent with that for Bi-based and Tl-based bulk materials, as determined by microwave absorption [10], but is in contrast to the well known $H^{2/3}$ behaviour of $YBa_2Cu_3O_{7-x}$ [11] and conventional low-T_c superconductors [12]. The different dependence of $(1 - T_{irr} / T_c)$ on H for Bi-based materials and $YBa_2Cu_3O_{7-x}$ has been attributed to the difference in the anisotropy of these materials. Both Bi-based superconductors and $YBa_2Cu_3O_{7-x}$ have layered structures, which lead to strong anisotropy of their properties. The anisotropy parameter, γ, is $(m_c / m_a)^{1/2}$, where m_c and m_a are the effective superconducting masses for pair motion along the *c* direction and the *a*–*b* plane, respectively. γ is reported to be in the range 25–50 for Bi-based materials [13], while it is estimated to be 5 for $YBa_2Cu_3O_{7-x}$ [14]. This difference leads Bi-based materials to be considered two dimensional, whereas $YBa_2Cu_3O_{7-x}$ is considered three dimensional. Based on the two dimensional nature of the Bi-based materials, pinning potential, U_o, can be expressed in terms of the Anderson – Kim flux creep model[15] as follows:

$$U_o = \frac{KT_c(Ln\ (f_o\ /f))(1-t)^{3/2}}{\alpha^{3/n}\ B^{1/2}} \qquad (2)$$

where f_o and f are the flux creep velocities in the presence and absence of pinning, respectively, t is T / T_c, and k is the Boltzmann constant. By using T_c values and $\ln(f_o / f) = 21$ [16], the pinning potential, U_o, at 77 k and 1 T for is calculated to be 20 eV, 4 eV and 0.4 eV for tapes A, C and D respectively. It is noted that the pinning potential for the PFDR processed tapes increases 10 to 50-fold over the the normal processed tape.

The results presented above indicate that the high mass density, grain alignment and uniform distribution of fine impurity precipitates are responsible for the enhancement of flux pinning in the high T_c phase formation-decomposition-recovery processed tapes. A small proportion of highly divided 2212 and remaining impurity precipitates produced from the PFDR process may also serve to enhance flux pinning in the Ag-sheathed Bi-

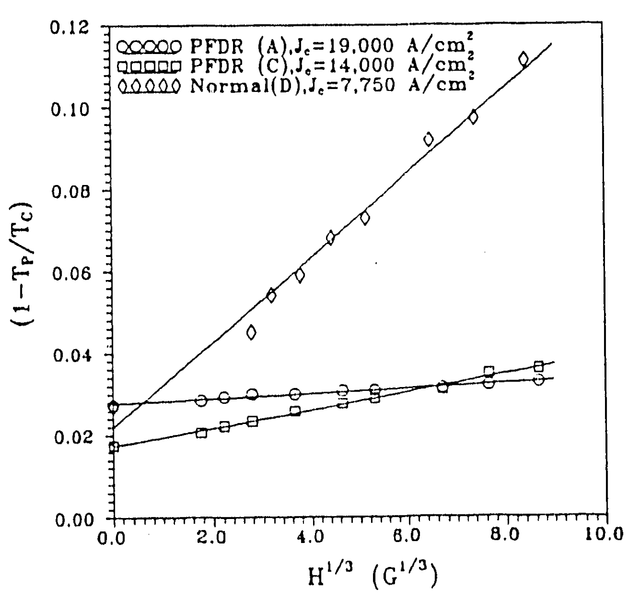

Fig. 5. Linear relation between $(1 - T_{irr} / T_c)$ and $H^{1/3}$ for Ag/Bi-2223 tape A, C and D

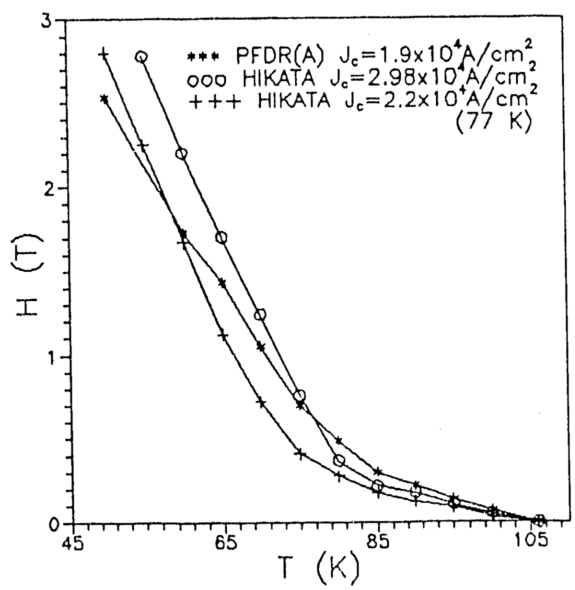

Fig. 6. Comparison of the irreversibility lines of the PFDR processed tape A with Hikata's samples

based superconducting wires. To confirm this, the irreversibility line of sample A is compared with those reported by Hikata et al [17] as shown in Figure 6. As these authors described, their samples were nearly single 2223 phase, but with varying amount of nonsuperconducting phases. It is interesting to note that the irreversibility line for sample A showed a crossover with that of Hikata's samples at 60 to 77 K. This indicates that the sample A (10% 2212) has stronger pinning at higher temperatures, while Hikata's samples with a proportion of impurities(no 2212) have stronger pinning at lower temperature, suggesting that two different pinning mechanisms operate in the PFDR processed tapes and Hikata's tapes. In the former, the 2212 together with impurity phases serves as pinning sites at temperature above T_c of 2212, while the impurity phases are main pinning sites in the latter. The role of 2212 phase in the Ag clad 2223 tapes deserves a further attention.

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