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The relation between crossover of the intergrain loss-peak temperature-field characteristics of the Ag-$Bi_2Sr_2CaCu_2O_x$ screen-printed tapes and their J_C values

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Abstract

A study of the influence of the processing conditions of Ag-$Bi_2Sr_2CaCu_2O_x$ screen-printed tapes on the temperature, field and frequency dependence of their a.c. susceptibility has been conducted. Samples have been prepared by melt-solidification and subsequent sintering on silver substrates under the same conditions but with different cooling procedures and reannealing. These procedures lead to different T_C values and field dependency of the loss peak temperature T_M , which cause the crossover in the T_M versus applied field characteristics. It was established that the above crossover phenomenon is correlated to the crossover in the J_C versus temperature characteristics.

1. Introduction

The measurement of a.c. susceptibility, $\chi = \chi' - i\chi''$, has been widely used to characterize the high-T_C superconductors (HTSC). The imaginary part, χ'', of the susceptibility, which represents the hysteresis loss in a sample, becomes maximum (or peak) at characteristic temperature T_M, when the intergranular vortices reach to the centre of the sample volume [1]. The field dependence of T_M which gives qualitative information about intergranular coupling in the sample, is strongly influenced by the parameters of sample fabrication, and thermal and chemical treatments [2,3]. Therefore, the investigation of this dependency is crucial not only for a physical understanding of the flux dynamics in the HTSC, but also for a practical reason to obtain the optimum conditions for sample preparation.

In this paper we report a dependence of the intergrain loss-peak temperature T_M on a.c. magnetic field and driving frequency for Ag-$Bi_2Sr_2CaCu_2O_x$ screen-printed tapes, and their critical transport current density, J_C, values. Samples have been prepared by melt-solidification and subsequent sintering [4] on silver substrates under the same conditions but with different cooling procedures and reannealing. These treatments affect both their superconducting critical temperature, T_C, and field dependency of T_M, which gives the crossover in the T_M versus applied field, $H_{a.c.}$, characteristics [5]. It is established that the above crossover phenomenon is correlated to the crossover of the J_C versus temperature, T, characteristics. However in the case of slow cooled sample, the correlation between the crossover in (T_M vs $H_{a.c.}$) and (J_C vs T) varnished (probably due to the increase of thermally activated flux creep).

2. Experimental

Bi-based "2212" tapes were prepared by the screen-printing method using Dowa Mining Co. Ltd."2212" paste. The paste was usually printed on one side of an Ag substrate, while some tapes were prepared by printing paste on both sides. After paste deposition the tape was pre-sintered at 500 °C for 2 h in air to remove the organic binders. Then it was subjected to the melt-

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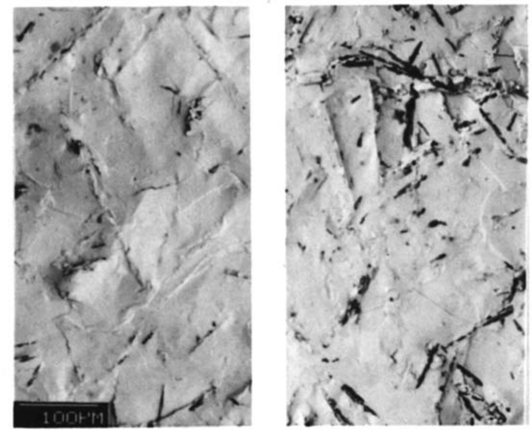
solidification process which was carried out at 890 °C and subsequently cooled to 850 °C at a rate of 10 °C/h in air. After sintering at 850 °C for 10 h, the samples were cooled to room temperature according to three different cooling procedures; (a) cooled slowly in the furnace (labelled sample SC), (b) cooled rapidly by removing the sample from furnace and simultaneously blowing nitrogen gas onto the sample (RC), (c) dropped into liquid nitrogen a vertical furnace (Q). Some of the slowly cooled samples were annealed at 500 °C for 15 h in N_2 (AN).

The microstructure of the sample was investigated by X-ray diffraction analysis (XRD) and scanning electron microscopy (SEM). The composition of the oxide was determined by energy dispersive X-ray analysis (EDX). The grain boundaries in the sample were investigated by high-resolution transmission electron microscopy (HRTEM). Critical current, I_C, was measured by a d.c. four probe method using an electric field criterion E=1 μV/cm. The a.c. susceptibility was measured as a function of temperature in various a.c. magnetic fields ranged from 0.1 Gauss to 9 Gauss using a driving frequency ranged from 33.3 Hz to 1000 Hz. The direction of applied field was always perpendicular to the surface of disk-formed samples.

3. Results and discussion

Figure 1 shows SEM micrographs of the tape surface for the samples (a) Q and (b) SC. Highly textured microstructure of the "2212" phase, shown by grey area in the Figure 1, are confirmed by the XRD results for the tape surface, which shows the strong (00l) reflection of the "2212" phase, and SEM observation of the cross section of the sample. An impurity phase, shown by black crystals in the form of whiskers, dispersed on the tape surface, has been identified as $(Sr,Ca)_2CuO_x$ by EDX analysis. These whiskers increase in number and length with decrease in the cooling rate. The increase of whiskers is enhanced remarkably by additional annealing in N_2 (sample AN), compared with sample SC. The observation of grain boundaries in the samples was conducted by the HRTEM. In sample Q, there are wide grain-boundaries (average width of 30 nm) mainly containing amorphous phases. On the other hand, narrower grain-boundaries (approximately 20 nm width)



(a) (b)

Figure 1. SEM micrographs of the surface of the samples: (a) quenched (Q), (b) slowly cooled (SC)

containing mainly crystalline phases, exist in sample SC. The dependence of width of the grain boundaries on the cooling procedure is related to the morphology of the sample surface. During slow cooling, the recrystallization of impurities from the grain-boundary region takes place and results in the appearance of whisker-form crystals on the surface of the sample. However, the remains of the wide grain-boundaries in sample Q prevents a fine morphology of the surface of the sample.

The difference in thermal contraction between the oxide and Ag substrate causes bending of the sample during cooling. SEM observation of the fracture surface of the samples indicated that sample Q contains a lot of cracks parallel to the surface of the tape induced by delamination of the Ag-"2212" composite. To avoid the above degradation, sample Q was prepared by printing on both sides of the Ag substrate, and in this case the bending of Q is eliminated. (The samples Q printed on the one side and both sides of the Ag substrates are labelled Q1 and Q2, respectively.)

A.C. susceptibility as a function of temperature was investigated for all samples with different applied fields. From the real part of the susceptibility, χ', versus temperature, the T_C value was defined for all samples as shown in Table 1, which also presents J_C values at 77.4 K and 0 T. The T_C value of the sample decreases with decrease of cooling rate. However, additional annealing of sample SC in N_2 increases the T_C value of this sample. This result confirms that the amount of oxygen content, i.e. the hole concentration in the "2212"

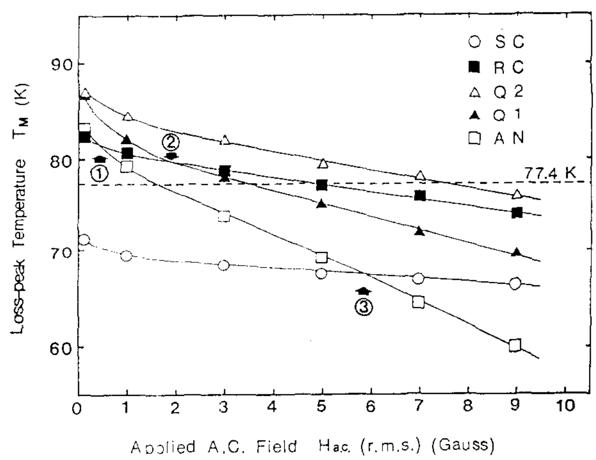


Figure 2. Field dependence of the loss peak temperature

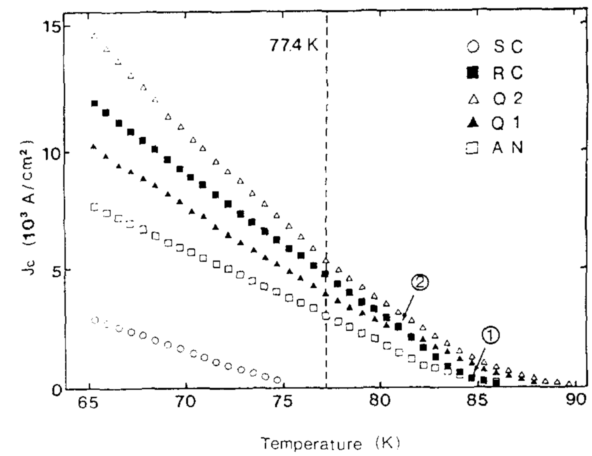


Figure 3 Temperature dependence of transport current density

phase can be changed by the cooling procedures and the reannealing [6].

From the imaginary part of the susceptibility, χ'', as function of temperature, the intergranular loss-peak temperature T_M is obtained with various a.c. applied fields. Figure 2 shows T_M versus applied field characteristics for all samples. As can be seen, each sample has a characteristic field dependence of T_M value. The sample that has a higher intergrain flux pinning force density, i.e. stronger intergrain coupling, has a smaller reduction of T_M with increase of applied field [7]. According to that, the order of the sample that has a strong intergrain coupling deduced from Figure 2 would be sample SC, RC, Q2, Q1 and AN (see Table 1). These different properties cause the crossover in T_M vs applied field characteristics.

Table 1. T_c and J_c values of the samples

Samples	T_c(onset) (K)	J_c(77.4K, 0T) (A/cm^2)
slow cooled/SC	76	0
repid cooled/RC	88	4.8×10^3
double-printed quenched/Q2	94	5.1×10^3
single-printed quenched/Q1	92	3.7×10^3
SC- annealed in N_2/AN	88	3.0×10^3

As can be seen in Figure 2, there are three crossover-points indicated by arrows in this figure; (1) between sample AN and RC at 81.5 K and 0.3 Gauss, (2) between sample Q1 and RC at 79.3 K and 2.5 Gauss and (3) between sample AN and SC at 67.0 K and 6.0 Gauss. The phenomenon of the crossover indicates that the intergrain-coupling strength may alter as a function of temperature. That is, above the crossover temperature a sample characterized by a higher T_C value has a better intergrain-coupling than another sample characterized by a lower T_C value, while below the crossover temperature this priority is reversed.

According to the Bean critical-state model, the relation between J_C value and a.c. applied field, $H_{a.c.}$, at T_M can be explained as follows [1],

$$J_C(T) \propto H_{a.c.}(T_M). \qquad (1)$$

Therefore, the crossover phenomenon should be related to the J_C versus temperature characteristics of the samples. Actually, the sample that has a higher J_C value at 77.4 K and 0 T (shown in Table 1) has a higher $H_{a.c.}$ value at 77.4 K in Figure 2 (the broken line indicates 77.4 K-level in Figure 2.). Figure 3 shows J_C versus temperature characteristics for all samples. As can be seen, the crossover points are also observed in this case; (1) between sample AN and RC at 84.7 K and 0.5 x 10^3 A/cm^2 and (2) between sample Q1 and RC at 81.3 K and 2.3 x 10^3 A/cm^2. This result proves that the alternation of the intergrain-coupling strength at crossover temperature is related to the transport properties of this group of samples. However, not only does an expected crossover between sample AN and SC (in Figure 3) not exist, but also the J_C value of sample SC is significantly below the J_C value of sample AN at lower temperature. Such inconsistency between (J_C vs T) and (T_M vs $H_{a.c.}$) for sample SC can be caused by a stronger flux creep effect in this sample, which leads to a strong frequency-dependence of T_M.

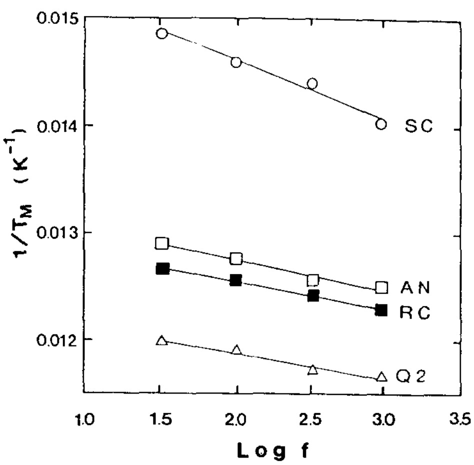


Figure 4. Logarithmic frequency dependence of the inverse loss-peak temperature

Figure 4 shows T_M versus frequency, f, characteristics at 1 Gauss, which are plotted T_M^{-1} as function of log (f). The behaviour can be interpreted by the following relation

$$f \propto \exp(-E_a/kT) \qquad (2)$$

where E_a is an energy that characterises the effect of flux creep on the a.c. susceptibility [8]. According to this relation, E_a values (at 1 Gauss) were estimated to be: 0.85 eV for sample Q2, 0.78 eV for RC, 0.71 eV for AN and 0.37 eV for SC. Remarkably, the SC has almost half the E_a value of other samples. This suggests that the a.c. susceptibility of sample SC is affected strongly by flux creep. In such a case, the flux penetration into the sample SC can differ from the rest of the samples, which causes difficulties in deduction of the transport property of sample SC from the field dependence of T_M.

4. Conclusion

The study of the influence of the processing conditions of the screen-printed "2212" tapes on the temperature, field and frequency dependence of their a.c. susceptibility has been conducted. The crossover phenomenon appears in the T_M versus applied field characteristics, which is correlated to the crossover of the J_C versus temperature characteristics. However, the sample SC shows a strong effect of flux creep on the measurement of a.c. susceptibility, which causes difficulties in deduction of the transport property of this sample from the field dependence of T_M. The reason that the SC has a smaller energy of E_a compared with another samples requires still further investigation.

Acknowledgement

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