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Magnetic and microstructural properties of $YBa_2Cu_3O_{7-x}$-Ag composite

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Abstract. The magnetic response of $YBa_2Cu_3O_{7-x}$–5 mol% Ag composite to low-frequency magnetic field and its microstructure have been studied. Microstructural analysis shows evidence of platelet-type grain growth and silver fills the intergranular regions. The granular nature of the sample is revealed from the strong decrease in a.c. response in the presence of d.c. magnetic field. The intergranular shielding current estimated from the complex response and using the Bean's model sharply increases with temperature below transition temperature.

Keywords. Superconductors; YBaCuO-Ag composite; a.c. susceptibility; intergrain shielding current.

1. Introduction

The complex magnetic response near and below T_c provides important information about the macroscopic state of ceramic superconductors (Hanic *et al* 1989; Flippen and Askew 1989; Murphy *et al* 1988). The magnetic response of the material is profoundly affected if the material is an aggregate of superconducting grains which are coupled by weak links. The effect of these links on the superconducting properties can be assessed by studying the magnetic behaviour of the material under the influence of a.c. and d.c. magnetic fields (Flippen and Askew 1989). We present the results of such an investigation on $YBa_2Cu_3O_{7-x}$-Ag composite and also the microstructural analysis which indicates that the intergranular space is occupied by silver. Earlier results on such a composite suggest the possibility of improving the critical current of these materials (Pavuna *et al* 1988).

2. Experimental method

The ceramic composite was prepared by calcining a mixture containing appropriate amounts of yttrium oxide and nitrates of barium, copper and silver at 920°C in air for 40 h. The calcined mass was ground, pelletized and sintered at 950°C under flowing oxygen for 24 h. The sample was cooled at 1°C/min, under oxygen, to room temperature. For magnetic measurement, copper wire (300 turns) was tightly wound at the centre of the cylindrical sample (radius = 0.18 cm). Magnetization was measured by measuring the integrated pick-up voltage appearing in the presence of low frequency (214 Hz) a.c. magnetic field. The sample was cooled under zero d.c. field and measured in heating cycle in the presence of d.c. magnetic field.

Figure 1. S.E.M. of $YBa_2Cu_3O_{7-x}$-Ag (5 mol%) pellet sintered at 950°C—fractured surface; chemical map of silver.

3. Results and discussion

The sintered samples were essentially single-phasic as determined by X-ray diffraction analysis. Scanning electron micrograph (figure 1) shows evidence of extensive grain growth and SEM-EDAX analysis reveals that silver is mostly clustered at grain boundaries. ESCA analysis of sputter-cleaned surface of the fractured faces of the samples shows that silver is mostly in elemental form. The onset transition temperature as measured by flux expulsion is 93 K. Unlike the 1–2–3 compound this sample was found to be stable against environmental degradation in humid atmosphere for over 1 year. The susceptibility of YBCO-Ag composite measured with an a.c. field of $H_a =$ 130 mOe amplitude decreases slowly initially near T_c and then falls rapidly to reach a constant value near 82 K (figure 2) in the absence of d.c. magnetic field. The fraction of flux expelled from the sample at this field was estimated to be about 89%. In the presence of a small co-linear d.c. field (5 Oe) no appreciable change in flux exclusion was observed. As the d.c. field is increased the absolute value of susceptibility drops and the transition is broadened over a few degrees (figure 2). The rate of decrease of screening just below the onset temperature is very sharp at low d.c. field and becomes constant as field continues to increase (figure 3a). But at lower temperatures the screening decreases slowly and continues to decrease up to 3 kOe field (figure 3b). These results can be adequately described assuming that the sample consists of aggregates of superconducting grains coupled by Josephson junctions. The superconducting screening current induced due to small a.c. field can travel from grain to grain through the Josephson junctions and thus generate macrocurrents within the sample. The signal measured in this case is proportional to volume enclosed by the current rather than the individual grains. The intergranular couplings tend to break up as the d.c. magnetic field, applied co-axially to the a.c. field, increases from zero. Macroshielding current is thus

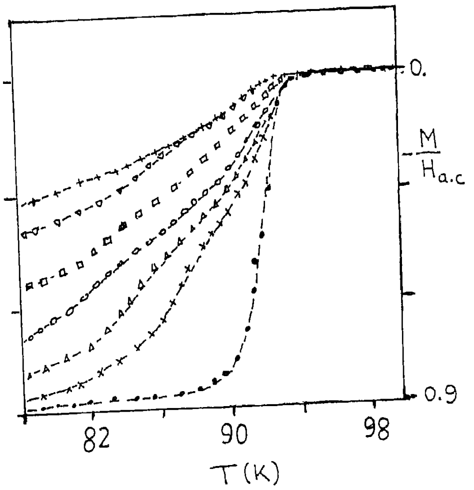

Figure 2. The variation of $-M/H_{ac}$ with temperature T of YBCO-Ag pellet in the presence of d.c. magnetic field; $\cdot$ – 0 Oe, × – 30 Oe, △ – 50 Oe, ○ – 100 Oe, □ – 300 Oe, ▽ – 1 kOe, and + – 2 kOe. The amplitude of a.c. field $H_{ac} = 130$ mOe.

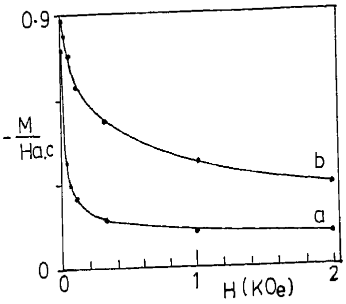

Figure 3. Variation of ZFC susceptibility ($-M/H_{ac}$) with d.c. magnetic field at (a) 90 K and (b) 80 K of the same sample.

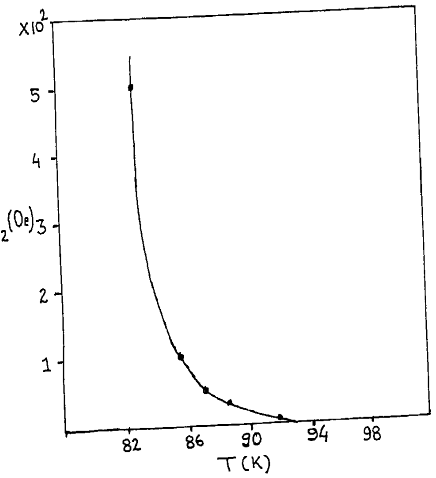

Figure 4. Temperature dependence of intergranular upper critical field of YBCO-Ag composite.

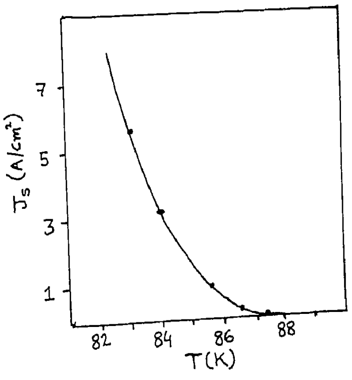

Figure 5. Temperature dependence of intergranular shielding current of YBCO-Ag composite in the absence of d.c. magnetic field.

interrupted and the signal drops to a value determined from the flux exclusion from the individual grain. The strength of Josephson or other weak-link coupling increases as the sample temperature is reduced. So when the temperature is close to the transition temperature the coupling is weak and the links are destroyed at low field resulting in sharp decrease of signal. At low temperature, as the coupling gets stronger and stronger, slower decrease of the signal is expected as observed here (figure 3). An approximate value of upper critical field, H_{c2}, where superconductivity within intergranular space vanished can be estimated. Taking H_{c2} as the field where the signal is reduced to half of its full value at low T and zero field, H_{c2} is estimated from the data (figure 2) and is plotted in figure 4. Near T_c the value of this field is low but increases steeply as the temperature is reduced. This clearly indicates that the strength of intergranular Josephson coupling is a strong function of temperature.

The imaginary part $\chi''(T)$ of the a.c. susceptibility shows a maximum below T_c. The position of the peak shifts towards lower temperature as the a.c. field amplitude increases. This result is usually interpreted on the basis of the critical state model (Bean 1964; Gomory and Lobotka 1988) of type-II superconductors. Using this model, the macroscopic shielding current density $J_s = H/r$, where H is the magnetic field at the surface and r is the radius of the sample. The temperature dependence of J_s is shown in figure 5. This value is comparable to that observed in 1–2–3 compound (Murphy *et al* 1988; Gomory and Lobotka 1988).

4. Conclusion

Silver in YBCO-Ag composite segregates in the intergranular space in elemental form and stabilizes the material against environmental degradation. The magnetic field dependence of a.c. response can be used to study the nature of intergranular links. Addition of small amounts of silver to 1–2–3 does not however change the shielding current.

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