View Article Online / Journal Homepage / Table of Contents for this issue

J. CHEM. SOC. FARADAY TRANS., 1990, 86(22), 3829–3830

Low-field Microwave Absorption in Mechanical Mixes of Superconducting $YBa_2Cu_3O_{7-x}$ with Al_2O_3 and $BaPbO_3$

Robert Janes, Kaushal K. Singh, Jane Blunt and Peter P. Edwards
IRC in Superconductivity, University of Cambridge, Madingley Road, Cambridge CB1 0HE, UK

Published on 01 January 1990. Downloaded by Michigan State University on 22/01/2016 11:26:56.

Magnetic-field-dependent (X-band) microwave absorption in superconducting $YBa_2Cu_3O_{7-x}$, diluted into insulating Al_2O_3 and metallic $BaPbO_3$ hosts, is reported here. X-Ray diffraction and a.c. susceptibility (mutual inductance) measurements were also carried out on the same samples to compare the sensitivity limits for detecting the presence of superconducting material. The intensity of the low-field microwave absorption and the mutual inductance signal increased linearly with increasing fraction of superconductor.

Studies on the microwave properties of high-T_c mixed copper oxide superconducting ceramics, using conventional electron paramagnetic resonance (EPR) instrumentation, have established that the transition to the superconducting state is always accompanied by an intense absorption at low magnetic fields.[1] EPR has now become a useful diagnostic tool for detecting superconductivity complementary to other more conventional methodology. Indeed we have recently demonstrated the close link between the detection of the superconducting transition temperature, as gauged by the field-dependent microwave absorption, and measurements of electrical resistivity and a.c. inductance.[2] In the present work, we further exploited this response, and investigate the magnitude of the low-field signal in 'two-phase' samples containing known amounts of superconducting and non-superconducting materials. To achieve this we have diluted a known superconductor ($YBa_2Cu_3O_{7-x}$, $T_c = 92$ K) into two nominally inert matrices, one of metallic character ($BaPbO_3$) the other an insulator (Al_2O_3). Synthesis of new superconducting materials is often hampered by the formation of mixed-phase samples, and the study of the microwave response in specifically produced inhomogeneous systems will provide a guide as to the general applicability of the low-field EPR technique.

$YBa_2Cu_3O_{7-x}$ and $BaPbO_3$ were prepared by standard solid-state reaction from the metal carbonates or oxides, and characterised by Cu Kα X-ray powder diffraction (Spectrolab CPS-120 diffractometer). By virtue of the synthetic technique employed, these materials will typically consist of grains of variable size, shape and homogeneity; however, as the $YBa_2Cu_3O_{7-x}$ used was from the same batch of material throughout, inconsistencies arising from the influence of ceramic microstructure on the low-field microwave signal have been avoided. Carefully weighed samples of the superconductor were thoroughly mixed into Al_2O_3(Johnson-Matthey Specpure) and $BaPbO_3$ in an agate mortar and pestle using cyclohexane as dispersant, to produce two series of samples having known dilutions by weight of $YBa_2Cu_3O_{7-x}$; these were as follows, $BaPbO_3$: 0.1, 0.25, 0.5, 0.75, 1.0, 2.5, 5.0% and Al_2O_3: 0.1, 0.25, 0.5, 1.0%. High-resolution X-ray diffraction patterns consisted only of a main peak due to $BaPbO_3$ (29.5°) and an $YBa_2Cu_3O_{7-x}$ peak (32.6°) whose intensity varied with composition. The latter line disappeared below the instrumental detection limits at 1% $YBa_2Cu_3O_{7-x}$. Fig. 1 shows typical XRD traces from the 1 and 5% dilutions.

X-Band EPR measurements were taken at 77 K using a Bruker ER200D spectrometer. In order to standardise each measurement, Suprasil quartz tubes were filled with the same amount of sample, whose position in the cavity (within a liquid-nitrogen-containing quartz insert Dewar) was kept constant. The incident microwave power, modulation amplitude and modulation frequency were all kept constant, and the low-field signal intensity was measured from the peak maxima to the baseline. Repeat scans were carried out to ensure reproducibility, in an attempt to eliminate the effects of flux-trapping, on sweeping back to zero-field. In Fig. 2 we show the low-field absorption from samples of $YBa_2Cu_3O_{7-x}$ diluted into $BaPbO_3$ at (*a*) 5% and (*b*) 1% levels, as well as a 0.1% mix in Al_2O_3 (*c*). The observation of a relatively strong absorption from such a low concentration

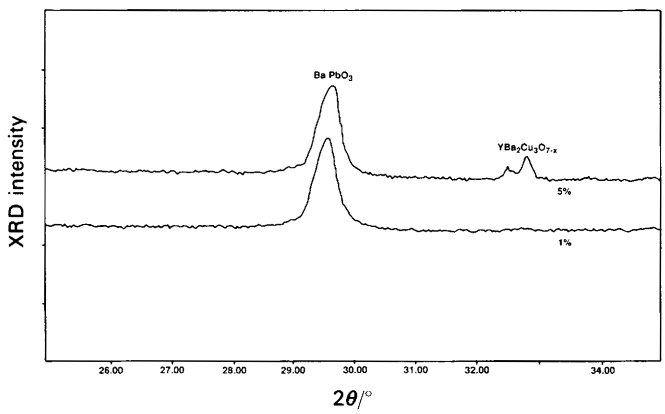

Fig. 1 X-ray diffraction pattern for $YBa_2Cu_3O_{7-x}$ diluted into $BaPbO_3$ at 1 and 5% levels

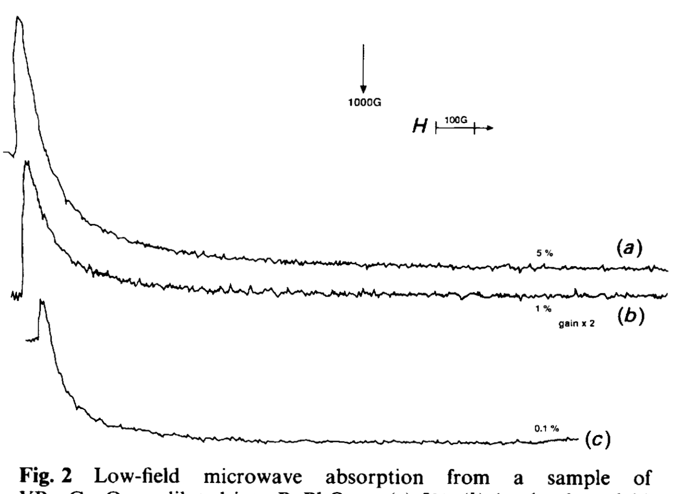

Fig. 2 Low-field microwave absorption from a sample of $YBa_2Cu_3O_{7-x}$ diluted into $BaPbO_3$ at (*a*) 5%, (*b*) 1% levels and (*c*) 0.1% mix in Al_2O_3 ($\nu = 9.1$ GHz, $T = 77$ K)

View Article Online

Published on 01 January 1990. Downloaded by Michigan State University on 22/01/2016 11:26:56.

of superconductor (0.1%) highlights the intrinsic sensitivity of the EPR technique. The intensity of the low-field signal for both compositional series is given in Fig. 3. Barium plumbate, being a metallic host, appreciably perturbs the field patterns in the microwave resonator, and would be expected to degrade the Q-factor, thus reducing the overall spectrometer sensitivity. This situation is commonly found when studying these ceramic superconductors, which tend to be poor metals in the normal state. In all cases, the signal was found to saturate at high microwave powers, a response common to all cuprate superconductors.[3] This effect has also been observed in microwave resistance measurements on $YBa_2Cu_3O_{7-x}$ where a resistance increase was observed with increasing microwave power at temperatures below T_c.[4]

Significantly, for both series the intensity of the low-field microwave absorption clearly scales with the superconducting fraction (Fig. 3), the amplitude increasing approximately linearly with the volume fraction of $YBa_2Cu_3O_{7-x}$, and deviating from linearity at higher concentrations of superconductor. The latter is probably a reflection of instrumental sensitivity changes, brought about by a change in the quality factor of the microwave cavity as the sample dielectric properties change with composition. We do not believe these deviations to be due to imperfect mixing as this potential problem would likely to be most obvious at the lower end of the concentration scale. The width of the signal was the same throughout the concentration range. Conjoint a.c. susceptibility measurements were carried out on these materials,

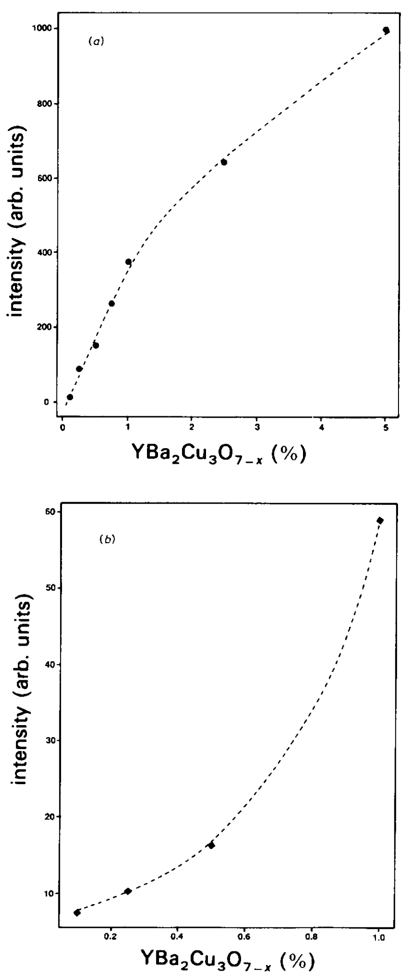

Fig. 3 (*a*) Intensity of the low field signal for a series of $YBa_2Cu_3O_{7-x}$–$BaPbO_3$ mechanical mixes, as a function of composition, (*b*) $YBa_2Cu_3O_{7-x}$–Al_2O_3

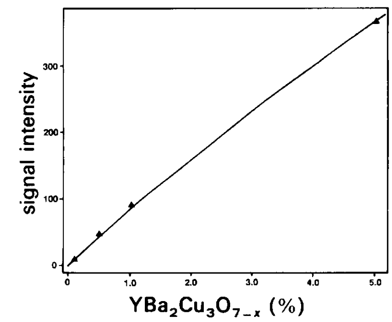

Fig. 4 Intensity of the mutual inductance signal, measured on an a.c. susceptometer, for four compositions in the $YBa_2Cu_3O_{7-x}$–$BaPbO_3$ series

using a mutual inductive technique operating at a frequency of 666 Hz (Lakeshore Cryotronics Inc., model 7000) which measures the dispersive component of the magnetic susceptibility. Fig. 4 shows the compositional dependence of the amplitude of the mutual inductance signal measured at 60 K, which again shows an approximate linear dependence on composition.

In summary, by studying a series of deliberately prepared mixed-phase materials, we have shown that the low-field EPR technique may easily be used to detect superconductivity down to extremely low levels of superconducting fraction, sensitivity being comparable with the more widely used a.c. susceptibility technique. A clear correlation between the fraction of superconducting material in the sample and the signal intensity was observed, which, with careful control, could be used to calibrate the amount of superconducting material in multiphase samples. It is generally accepted that microwave absorption at the lowest fields is attributed to weak intergranular Josephson links,[5] and certainly at the lowest concentrations prepared here we would expect individual grains to be well dispersed, and intergranular weak links to be greatly reduced. However, as pointed out by Deutscher and Muller[6] and Blazey *et al.*,[7] the microwave absorption at the lowest fields can be explained by invoking a 'superconducting glass' state, where superconducting loops having areas typically less than the grain size exist. In this case internal weak links, in addition to grain boundaries are considered. The former may occur at defects such as twin boundaries within grains, and form a network which divides the grains into weakly coupled superconducting regions.

We thank the SERC and BP for financial support.

References

1 See for example; M. D. Sastry, *Studies of High Temperature Superconductors*, Nova Science Publishers Inc., New York, 1989, ch. 10.
2 R. Jones, R. Janes, R. Armstrong, K. K. Singh, D. J. Keeble, M. R. Harrison and P. P. Edwards, *J. Chem. Soc., Faraday Trans.*, 1990, **86**, 683.
3 R. Janes, R. S. Liu, P. P. Edwards and J. L. Tallon, *Physica C*, 1990, **167**, 520; R. Janes, R. S. Liu, P. P. Edwards, A. D. Stevens and M. C. R. Symons, *Proc. Int. Conf. on Superconductivity*, Bangalore-India, 1990, in press.
4 Q. Li, K. W. Rigby and M. S. Rzchowski, *Phys. Rev.*, 1989, **39**, 6607.
5 Y. Maniwa, A. Grupp, F. Hentsch and M. Mehring, *Physica C*, 1988, **156**, 755.
6 G. Deutscher and K. A. Muller, *Phys. Rev. Lett.*, 1987, **59**, 1745.
7 K. W. Blazey, K. A. Muller, J. G. Bednorz, W. Berlinger, G. Amoretti, E. Buluggiu, A. Vera and F. C. Matacotta, *Phys. Rev. B*, 1987, **36**, 7241.

Communication 0/03336G; *Received* 24*th July*, 1990