Evidence for two superconducting components in oxygen-annealed single-phase Y–Ba–Cu–O*

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Received 8 June 1987

The complex susceptibility of a sintered Y–Ba–Cu–O superconductor is strongly dependent on a.c. field amplitude, h. Very small values of h must be used for the real part of susceptibility, χ', to reach a value corresponding to bulk diamagnetism just below the critical temperature, T_c. The imaginary part, χ'', represents hysteresis loss in the sample. Thus, χ'' *versus* temperature becomes positive when h exceeds the lower critical field, H_{cl}, of the superconductor.

Annealing the material in oxygen gives rise to two distinct components, a relatively high-T_c, high-H_{cl} superconductor (denoted as 'G' or 'good') and a relatively low-T_c, low-H_{cl} superconductor (denoted as 'B' or 'bad'). Curves of susceptibility *versus* increasing temperature reflect the dual nature of the annealed sample: χ' has an inflection point at T_c of the B component and approaches zero at T_c of the G component, while χ'' has a peak at each T_c. Both critical temperatures decrease linearly with increasing h, though at very different rates. H_{cl} of the G component is considerably greater than H_{cl} of the B component. The lower critical fields are linearly decreasing functions of temperature.

Two models might explain the susceptibility data. In the grain model, the G component consists of superconducting grains and the B component is either intergranular material, unfavourably orientated anisotropic grains, or oxygen-depleted grain boundaries. In the surface model, the G component is in the interior of the sample and the B component is at the sample's surface. This condition could arise if there was oxygen depletion at the surface subsequent to total enrichment during annealing.

Keywords: superconductors; Y–Ba–Cu–O superconductors; high critical temperature superconductors; a.c. losses; susceptibility; lower critical field

The measurement of a.c. susceptibility, χ, as a function of temperature, T, is often used to determine the critical temperature, T_c, of superconductors[1,2]. In general, the real part of susceptibility, χ', goes from -1 (SI units) below T_c, indicating perfect diamagnetism and flux shielding, to a small positive value above T_c, indicating the normal paramagnetic state. The imaginary (loss) part of susceptibility, χ'', goes from zero below T_c, through a peak near T_c, to zero in the normal state.

The derivative of the initial curve of magnetization *versus* magnetic field is volume susceptibility. For a large enough field amplitude, hysteresis loss will appear as an enclosed area in the magnetization-field plane. In such a state, the material is not perfectly diamagnetic, χ'' is greater than zero, and χ' is no longer equal to -1.

We examine χ' and χ'' as functions of T for different values of a.c. field amplitude, h, and frequency, f, for a high-T_c, sintered, X-ray single-phase, copper-oxide superconductor, $Y_1Ba_2Cu_3O_{8-\delta}$, where δ represents an unknown oxygen deficiency. The character of the χ curves changes significantly after annealing the sintered material in oxygen. The curves indicate a transformation of the original material into two distinct superconducting components. From the χ curves we deduce $T_c(h)$ and $H_{cl}(T)$ for each component, where H_{cl} is the lower critical field. One component, with relatively high T_c and H_{cl}, is denoted as 'G' or 'good'. The other, with relatively low T_c and H_{cl}, is denoted as 'B' or 'bad'.

Two physical models are consistent with the susceptibility data. In the grain model, the G component is identified as superconducting grains and the B component is either intergranular material, unfavourably orientated anisotropic grains, or oxygen-depleted grain boundaries. The two components are dispersed throughout the sample. In the surface model, the G component is

*Paper presented at CEC/ICMC, St Charles, Illinois, USA, 14–18 June 1987. Contribution of the National Bureau of Standards, not subject to copyright

0011-2275/87/090475-06 $03.00

located in the interior of the sample and the B component is the region at the sample's surface.

In conventional 'hard' type-II superconductors, the lower critical field, H_{c1}, corresponds to the field above which: 1, magnetic flux penetrates into the superconductor and is subjected to pinning forces; 2, hysteresis loss appears; and 3, the curve of magnetization *versus* field deviates from linearity*. Therefore, when the h used in a susceptibility measurement exceeds H_{c1}, positive χ'' is expected[4]. (If necessary, the value of h should be corrected for the sample demagnetization factor.) Thus, measurements of χ'' *versus* T for different values of h may be used to deduce $H_{c1}(T)$†. H_{c1} is sometimes difficult to obtain from curves of magnetization *versus* field because deviation from linearity is often not readily apparent.

In granular superconductors, superconducting grains may be coupled via Josephson currents through insulating barriers (tunneling) or normal-metal junctions (proximity effect)[6–8]. Since this description might apply to Y–Ba–Cu–O superconductors[9–13], one would distinguish between the lower critical field of the grains and the significantly smaller 'Josephson lower critical field'[14]. In this model, the definition of H_{c1} above would apply to the Josephson lower critical field rather than the superconducting grains. In variations of the grain model, the definition of H_{c1} would apply to either unfavourably orientated anisotropic grains[15–17] or to oxygen-depleted grain boundaries.

In the surface model, the region near the sample surface is simply a B superconductor, not Josephson material. H_{c1} in the traditional definition above would apply to the surface region, not the G interior. In any case, we use the symbols T_{cg} and H_{c1g} to refer to T_c and H_{c1} of the G superconductor. Similarly, we use T_{cb} and H_{c1b} to refer to T_c and H_{c1} of the B material.

Experimental details

The $Y_1Ba_2Cu_3O_{8-\delta}$ superconductor was fabricated as described by Panson *et al.*[18]. If δ were equal to zero, it would imply that all Cu was in the trivalent state. For δ greater than zero, some Cu is divalent. The pressed powder was sintered at 940°C and cooled in oxygen. The material is single-phase orthorhombic as determined by X-ray diffraction. Grain size is of the order of 10 μm. Prior to measurement, the two large sample surfaces were sanded to remove surface contamination and to ensure flat faces. After measuring χ for different h and f, the same sample was annealed in flowing oxygen at ambient pressure ($\approx$80 kPa) at 700°C for $\approx$40 h and slowly furnace cooled to room temperature. It took 2 h for the temperature to fall from 700 to 350°C. Samples so prepared are crystallographically single phase within the resolution of X-ray diffraction[18]. After annealing the sample, χ was again measured. Between measurements the sample was stored in a desiccator.

A proper combination of sample dimensions and maximum f, appropriate to the normal-state residual resistivity, eliminates normal-state eddy-current effects near T_c. Intergranular residual resistivity is 350–400 $\mu\Omega$ cm. The normal-state skin depth at 1000 Hz is $\approx$3 cm, compared to a sample thickness of $\approx$1 mm. Thus, in this sample, χ'' represents only hysteresis loss. However, if sample dimensions perpendicular to the applied field were large and normal-state resistivity were low enough, eddy currents could contribute to χ near T_c.

Volume susceptibility was measured with an a.c. susceptometer calibrated, for these measurements, with a Nb sphere at 4 K[19]. (Normal-state eddy-current effects would not be seen this far below T_c of $\approx$9.6 K.) The susceptibility values reported are thus accurate to the extent that the sample moment approximates a dipole. The sample used for the susceptibility measurements was a thin, flat piece with h applied in the plane. Sample dimensions were $\approx 12 \times 7.2 \times 0.9$ mm, with an estimated[20] effective demagnetization factor of 0.05. External susceptibilities, not corrected for this small demagnetization factor, are presented in the results. The overall sample volume, including voids, was used to compute χ. Sample density was 4.8 g cm^{-3}. Based on a comparison to the 5.3 g cm^{-3} pycnometric density of the powder, 10% of the sample volume would be voids. Based on an ideal 6.4 g cm^{-3} X-ray density, 25% of the sample volume would be voids.

A high-permeability shield around the Dewar reduced the Earth's field to less than 0.4 A m^{-1} (5 mOe) in the measurement axis. The sample was cooled to either 4 or 76 K in zero magnetic field. Measurements were made with T increasing at the rate of 0.2 K min^{-1} or less. As T increased, f was cycled between 10, 100 and 1000 Hz. Hence, each measurement run yielded χ curves for three frequencies at constant h. The phase angle of the lock-in amplifier used in the experiment was determined for each measurement frequency to null χ'' for a small h at 4 K.

Results and discussion

Unannealed sample

Both χ' and χ'' for the unannealed sample are plotted from 75 to 95 K in *Figure 1* for a range of h at 100 Hz. The curves are labelled with numbers equal to the r.m.s. measuring field in Oe. The transition from the normal to the superconducting state begins at 90 K, but the shapes of the χ curves are strongly dependent on h. In fact, very small values of h must be used for χ' to reach -1 just below T_c and for the apparent width of the transition to be small. For the smallest h, the transition width is 3 K, similar to that measured resistively. Most papers reporting susceptibility of high-T_c, copper-oxide superconductors either present χ values in arbitrary units or report measurements made in applied fields that are too large for perfect diamagnetism to be seen just below T_c. There are some exceptions[4,21–24].

As h increases, the character of the χ' and χ'' curves changes. The χ'' curve goes from having a peak to having a

*In an ideal type-II superconductor, assuming zero demagnetization factor and thus no intermediate state, the field of maximum (negative) magnetization also coincides with H_{c1}. However, in hard type-II superconductors, those characterized by flux pinning, it corresponds to the 'full-penetration' field at which flux lines first penetrate to the centre of the superconductor in the mixed state. For higher fields, the critical current density, and therefore the magnetization, decrease in magnitude[3]

†An alternate, but conceptually equivalent, method to obtain H_{c1} from χ'' is to measure low-field a.c. χ in a variable d.c. bias field[5]. In such an experiment we observe features in the χ curves that are the same as those we report here. There is not, however, a perfect correlation between the magnitudes of the d.c. bias field and h

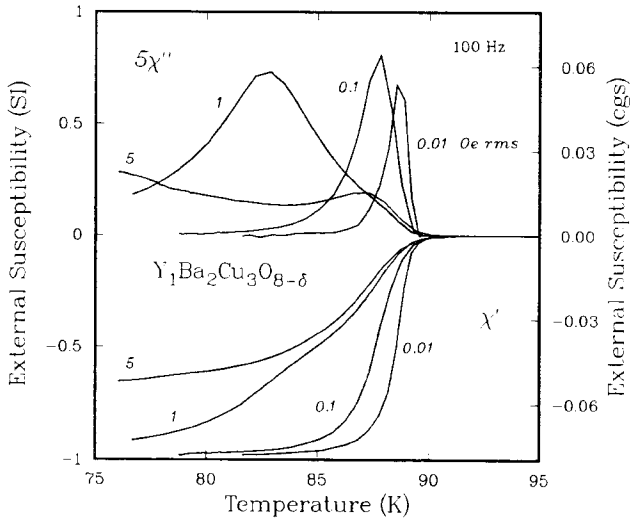

Figure 1 Real (χ') and imaginary (χ'') susceptibility for sintered but unannealed $Y_1Ba_2Cu_3O_{8-\delta}$. The numbers labelling the curves are the r.m.s. values of the measuring fields in Oe. Measurements were made for field amplitudes, h, of 1.1 A m^{-1} (14 mOe) [curves 0.01], 11 A m^{-1} (0.14 Oe) [curves 0.1], 113 A m^{-1} (1.4 Oe) [curves 1] and 563 A m^{-1} (7 Oe) [curves 5]. Each curve is composed of straight-line segments connecting about 30 data points. For purposes of clarity, χ'' is multiplied by 5 and only data for 100 Hz are shown. Curves for f of 10 and 1000 Hz are virtually identical. (After Reference 4)

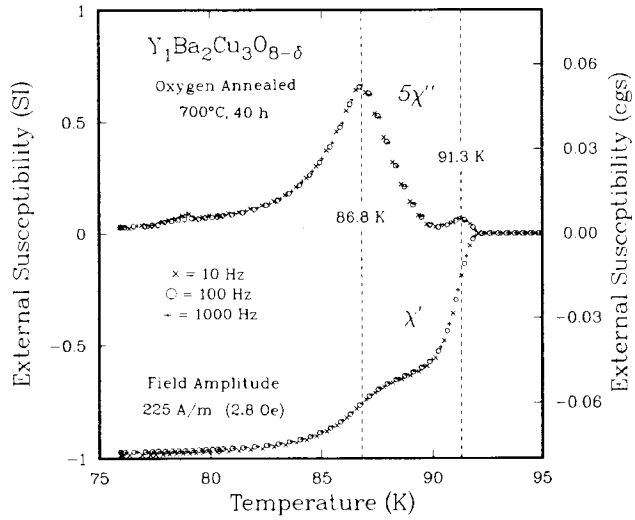

Figure 2 Real (χ') and imaginary (χ'') susceptibility illustrating the two-component nature of $Y_1Ba_2Cu_3O_{8-\delta}$ annealed in oxygen at ambient pressure at 700°C for 40 h. Measurements are shown for a field amplitude, h, of 225 A m^{-1} (2.8 Oe) at frequencies, f, of 10 Hz (x), 100 Hz (○) and 1000 Hz (+). Susceptibility is virtually independent of f. For purposes of clarity, χ'' is multiplied by 5. The peaks in χ'' coincide with approximately the temperatures of the midpoints of the transitions in χ' for each component

broad maximum. For the highest field, the maximum of χ'' is below 75 K and a secondary peak appears near 87 K. When h exceeds the lower critical field, H_{c1}, of the superconductor at a given temperature below T_c, χ'' becomes positive and χ' is no longer equal to -1. Thus, measurements of χ as a function of T allow $H_{c1}(T)$ to be determined, as shown in more detail for the annealed sample below. For a field amplitude, h, equal to 1.1 A m^{-1} (14 mOe) [curves 0.01], χ'' first becomes positive upon warming at 87 K, indicating $H_{c1}(87) = 1.1$ A m^{-1}. By the same logic, $H_{c1}(85) = 11$ A m^{-1} (0.14 Oe) [curves 0.1]. Because H_{c1} is extremely small for the unannealed material, even moderate values of h prevent χ' from reaching -1 below T_c.

Annealed sample

Susceptibility data for the sample after annealing at 700°C for ≈40 h are plotted in *Figure 2* for field amplitude, h, equal to 225 A m^{-1} (2.8 Oe) at f of 10, 100 and 1000 Hz. The data illustrate that χ is independent of f. This value of h was selected because there are very distinguishable features in χ' and χ'' which we interpret as corresponding to two superconducting components in the material. Peaks in χ'' and inflection points in χ' denote their critical temperatures. The critical temperature of the B material, T_{cb}, is ≈86.8 K and the critical temperature of the G material, T_{cg}, is ≈91.3 K. These temperatures correspond to approximately the midpoints of the transitions.

Data for a full range of h at 100 Hz are plotted in *Figure 3*. The curves are labelled with numbers equal to the r.m.s. measuring field in Oe. For a very small field amplitude, h, of 1.1 A m^{-1} (14 mOe) [curves 0.01], a single sharp transition is seen at 91 K, with a transition width of ≈2 K. For a larger h of 11 A m^{-1} (0.14 Oe) [curves 0.1], T_c moves to lower temperature and the transition width broadens. For h equal to or greater than 113 A m^{-1} (1.4 Oe) [curves 1–10], the presence of the two superconducting components is apparent. The χ'' curves have double peaks and the χ' curves have two fall-offs.

The critical temperatures, T_{cg} and T_{cb}, of the two components are shown in *Figure 4* as functions of h. With increasing h, the critical temperatures decrease linearly but at unequal rates. The slope dT_{cb}/dh is 50 times dT_{cg}/dh. The plotted values were determined from the peak

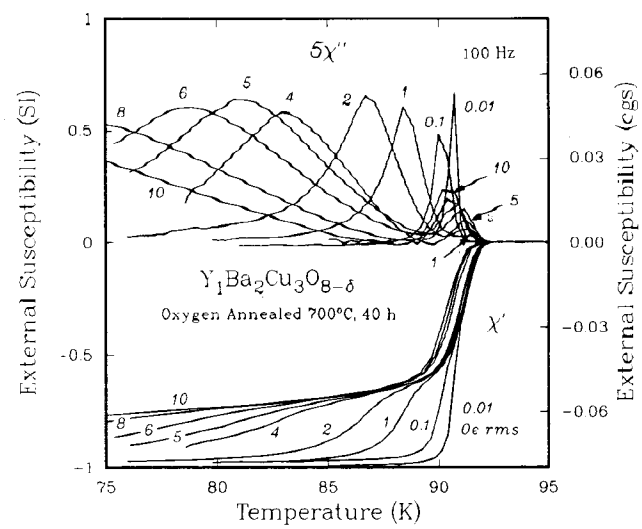

Figure 3 Real (χ') and imaginary (χ'') susceptibility for oxygen-annealed $Y_1Ba_2Cu_3O_{8-\delta}$. The numbers labelling the curves are the r.m.s. values of the measuring fields in Oe. Measurements were made for field amplitudes, h, of 1.1. A m^{-1} (14 mOe) [curves 0.01], 11 A m^{-1} (0.14 Oe) [curves 0.1], 113 A m^{-1} (1.4 Oe) [curves 1], 225 A m^{-1} (2.8 Oe) [curves 2, seen also in *Figure 2*], 450 A m^{-1} (5.7 Oe) [curves 4], 563 A m^{-1} (7.1 Oe) [curves 5], 675 A m^{-1} (8.5 Oe) [curves 6], 900 A m^{-1} (11.3 Oe) [curves 8] and 1125 A m^{-1} (14.1 Oe) [curves 10]. χ'' curves [1]–[10] have double peaks. Each curve is composed of straight-line segments connecting about 40 data points. For purposes of clarity, χ'' is multiplied by 5 and only data for 100 Hz are shown. Curves for frequencies of 10 and 1000 Hz are virtually identical

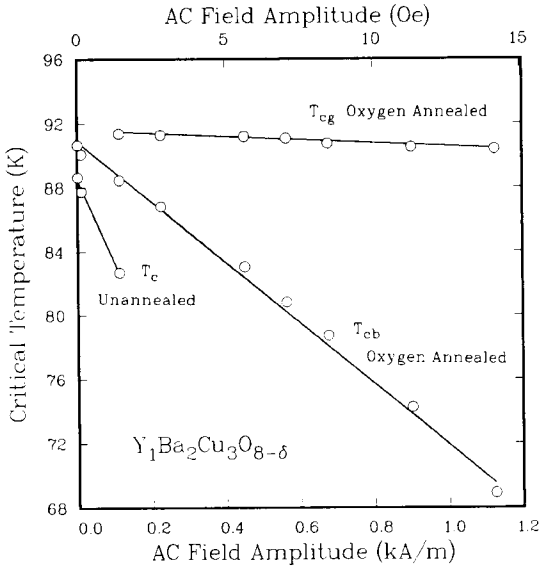

Figure 4 Critical temperatures, T_{cg} and T_{cb}, of the two superconducting components in $Y_1Ba_2Cu_3O_{8-\delta}$ (oxygen-annealed at 700°C for 40 h) as functions of a.c. field amplitude, h. For comparison, T_c for the unannealed sample is also shown. The critical temperatures were determined by the peaks in χ'', which correspond to approximately the midpoints of the transitions in χ', for each component

temperatures in χ'', interpolated by fitting a parabola to the three highest-valued χ'' data points. For the two smallest h values, T_{cg} cannot be found from χ'' because there is no second peak. In this limit of small h, T_{cg} and T_{cb} converge. Also plotted in *Figure 4*, for comparison, are T_c values for the unannealed sample. The decrease in T_{cb} with increasing h is consistent with the rapid decrease in transport critical current observed in applied fields in the range 80–8000 A m^{-1} (1 to 100 Oe) at liquid nitrogen temperature[10].

Lower critical fields

Because H_{clg} is significantly greater than H_{clb}, larger h is required to bring out the features in χ corresponding to flux penetration into the G component. Above T_{cb}, only the G superconductor contributes to χ. That is why, for h less than H_{clg}, all χ' curves merge near T_{cg} in *Figure 3*.

In general, H_{cl} is expected to be a decreasing function of T. Ginzburg–Landau theory, for example, shows that H_{cl} is proportional to $1 - T/T_c$ near T_c*. Consequently, there will be some value of T upon warming at which H_{cl} falls below h. At this T, χ'' will begin to rise from zero. As T increases, H_{cl} continues to decrease and χ'' continues to increase. As T_c is approached, the magnetization curve collapses and both χ' and χ'' approach zero.

The sequence of measurements using different h in *Figure 3* may be used to deduce $H_{cl}(T)$ for the two components. H_{clg} and H_{clb} are plotted *versus* T in *Figure 5*. The functions are linear, although there is some uncertainty in H_{clb} at the lower temperatures. H_{clg} is considerably greater than H_{clb} and decreases steeply with increasing T near T_{cg} with a slope, dH_{clg}/dT, of -289 A m^{-1} K^{-1} (-3.6 Oe K^{-1}).

Indentification of components

Because the annealing and slow cooling procedure is believed to yield material that is crystallographically single phase, we refer to the G and B superconductors as 'components', not 'phases'. The possibility that the G component is at the sample surface should be considered. This condition could exist if there was inadequate oxygen diffusion into the sample during annealing. If the G material were at the surface, however, it would effectively shield the B interior material. The latter would not be evident in the χ curves for any T or h.

We examine two other models. In the grain model, the two components are distributed throughout the sample as grains plus either intergranular material, anisotropic grains misaligned with the field, or oxygen-depleted grain boundaries. The intergranular material could be Josephson weak links or simply B superconductor. The misaligned grains might exhibit weak superconductivity due to crystalline anisotropy such as anisotropy in the superconductive energy gap. When h is small, χ' is characteristic of a bulk superconductor. For medium h, greater than H_{clb}, the grains decouple and χ' decreases in magnitude to a value corresponding to a collection of decoupled grains[14]. For large h, greater than H_{clg}, the grains are penetrated by magnetic flux and χ' is reduced in magnitude. These effects may be seen in *Figure 3* at appropriate temperatures, since H_{clb} and H_{clg} are functions of T.

In the surface model, the G material is in the interior of the sample and B superconductor, not Josephson material, is a surface sheath. The G superconductor would contain grains and voids, but the material would be contiguous. This condition could arise from oxygen depletion at the surface subsequent to total oxygen enrichment during annealing. The depletion could be a

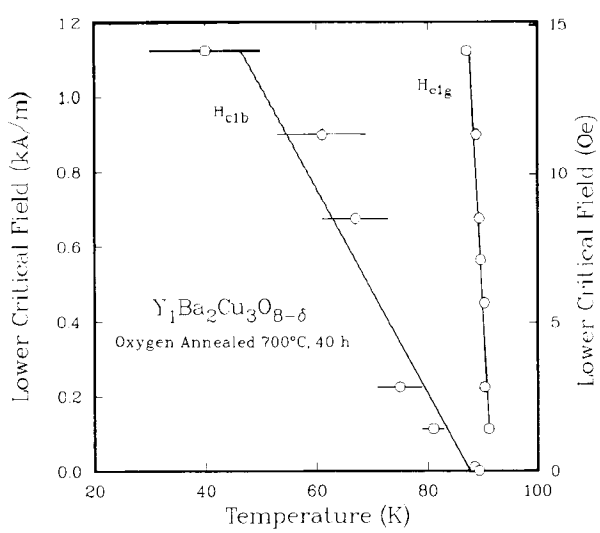

Figure 5 Lower critical fields, H_{clg} and H_{clb}, of the two superconducting components in oxygen-annealed $Y_1Ba_2Cu_3O_{8-\delta}$ as functions of temperature. The data were determined by the temperatures at which curves of χ'', most shown in *Figure 3*, rise from zero upon warming. The gradual rise in χ'' at lower temperatures gives some uncertainty in H_{clb}, indicated by error bars. Each line is a least-squares fit with H_{cl} as the independent variable

*This may be deduced from the theoretical expression for H_{cl} in terms of the penetration depth and the coherence length[25], both functions of temperature[26]

consequence of reaction with water or carbon dioxide. When h is small, χ' is once again characteristic of a bulk superconductor. When h exceeds H_{clb}, flux penetrates through the surface region until it reaches the G superconductor in the interior. When h exceeds H_{clg}, flux penetrates into the G material and χ' is reduced in magnitude.

We have seen susceptibility evidence of two superconducting components in other samples of Y–Ba–Cu–O and La–Sr–Cu–O made at different laboratories. Others have reported similar evidence[23,24]. Thus the two-component phenomenon appears to occur generally, as a consequence of current methods of materials preparation and oxygen annealing. Different methods might minimize the fraction of B material.

The relative volumes of the two components may be estimated from the value of χ' above T_{cb}. For example, in *Figure 2*, we assume that the G component is perfectly diamagnetic at 89 K and that the B material does not contribute to χ' for this T and h. Then χ' is entirely due to the G material. If χ' is ≈ 0.65 (SI), then 65% of the volume is occupied by G material. In the grain model, if the grains are large enough to avoid penetration-depth effects[14], the remainder of the volume is composed of B material and voids. In the surface model, the remainder of the volume is B material and the voids are distributed uniformly throughout the sample.

Conclusions

The critical temperature, T_c, of unannealed, X-ray single phase, sintered $Y_1Ba_2Cu_3O_{8-\delta}$ is relatively low and decreases rapidly with increasing a.c. field amplitude, h. The lower critical field, H_{cl}, is quite small.

Annealing the sample in oxygen and slowly cooling preserves the single crystallographic phase, but gives rise to two superconducting components. The components are identified as 'good' ('G') and 'bad' ('B') superconducting material, with critical temperatures T_{cg} and T_{cb}. The dual nature of the annealed sample is clearly evident in curves of complex susceptibility, χ, *versus* increasing temperature, T: χ' has an inflection point at T_{cb} and approaches zero at T_{cg}. χ'' has a peak corresponding to each T_c. Both critical temperatures decrease linearly with h, but at very different rates. For all h, T_{cg} is greater than T_{cb} which, in turn, is greater than T_c of the unannealed superconductor.

The lower critical fields of the two components, H_{clg} and H_{clb}, are linearly decreasing functions of T. H_{clg} is much greater than H_{clb} and H_{cl} of the unannealed superconductor. The G component appears to be much better superconductor than the B component based on the data for $T_c(h)$ and $H_{cl}(T)$. At temperatures greater than $T_{cb}(h)$, there is a reduction in the effective volume of superconductor and in the magnitude of χ'.

Added in proof

In order to possibly increase the oxygen content of the sample and, in particular, that of the B component, we reannealed the sample in flowing oxygen according to the following schedule: 650°C/2 h, 450°C/24 h, 400°C/24 h, 350°C/16 h, slow cool to room temperature. Susceptibility *versus* temperature was measured with h equal to 225 A m^{-1} (2.8 Oe) at 100 Hz for comparison with *Figure 2*. The low-temperature χ'' peak associated with T_{cb} shifted slightly upward to 87.3 K. The upper-temperature χ'' peak and the fall-off in χ' corresponding to T_{cg} both shifted slightly downward to 91.0 K. The magnitudes of the peaks remained about the same. Based on these results, oxygen deficiency might not be a factor for the B component.

Acknowledgements

We had helpful discussions with J.W. Ekin, L.F. Goodrich and J. Moreland. The a.c. susceptometer was constructed by C.A. Thompson. Y. Melchizedek and D.L. Ried assisted with computer programming and data reduction. Comparison samples of Y–Ba–Cu–O were generously provided by M. Hong, AT&T Bell Laboratories, and by J. E. Blendell, National Bureau of Standards. Comparison samples of La–Sr–Cu–O were generously provided by H.C. Ku and R.N. Shelton, Iowa State University. Work at NBS was supported by the National Engineering Laboratory, National Bureau of Standards. Work at Westinghouse was supported in part by the Air Force Office of Scientific Research, USA, contract No. F49620-85-C-0043.

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