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Flux pinning, granularity and the irreversibility line of the high-T. superconductor HgBa₂CuO_{4+x}

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THE recent discoveries of superconductivity at 94 K in the single-CuO, layer compound HgBa₂CuO₄₊₂ (Hg-1201)¹ and at 133 K in its two- and three-copper-layer analogues* (Hg-1212 and Hg-1223) have renewed interest in the search for new hightransition-temperature (high- T_c) superconductors. But whatever the T_{in} high values of intergrain critical current density are required for large-scale applications; thus, the material must have good electromagnetic connectivity between grains (it must not be electromagnetically granular) and it should be able to maintain its current-carrying capacity in high magnetic fields (it should have a high irreversibility field, H*(T)). Putilin et al. surmised that the small CuO₂ layer spacing of Hg-1201 might yield a high $H^{*}(T)$. We report here that $H^{*}(T)$ is indeed significantly higher than for the two- and three-copper-layer Bi-Sr-Ca-Cu-O compounds (Bi-2212 and Bi-2223), but lower than for YBa₂Cu₂O₇ (Y-123). The low-temperature flux pinning is also strong. Like Y-123, however, Hg-1201 is electromagnetically granular. A high degree of grain alignment will probably be necessary to remove this granularity; by analogy with the bismuth compounds, this is likely to be easier if the two-mercurylayer counterparts of Bi-22XY (Hg-2201, -2212 or -2223) can be synthesized.

HgBa₂CuO₄₊, was prepared from stoichiometric mixtures of HgO and Ba₂CuO₃. Ba₂CuO₃ was prepared by reacting BaO₂ (<95%) and CuO (<99.9%) at 900°C in oxygen for 24 hours using one intermediate grinding step. Storage, grinding, and mixing of Ba₂CuO₃ with HgO was done in an inert atmosphere. Pellets of the mixture were placed in an alumina crucible, which in turn was placed inside a quartz tube. This was evacuated, scaled, then placed in a stainless steel tube filled with sand to protect against possible explosion (which did not occur). The samples studied here were heated to 800 °C at 5 °C min-1, then slowly cooled to room temperature over 10 hours. Samples heated at 845 °C exhibited sharper X-ray patterns for the 1201 phase but also showed free Hg, as also seen by Wagner et al. 3.

For this reason we worked at 800 °C. Subsequent transmission electron microscopy (TEM) on the 800 °C samples showed that they too had some free Hg (or HgO) within them, the size of these particles being 10-30 nm. Our X-ray, scanning electron microscopy (SEM), TEM, and magnetization measuremetrs showed that the proportion of 1201 phase was > 50%. After reaction, the samples were annealed at 200 °C for 2-6 hours in flowing oxygen. The grain size of all samples was ~ 5 am and the grain shape was nearly equiaxed. Mercury-rich compounds were observed at a number of grain boundaries, though others appeared clean, when examined by back scatter SEM and TEM.

The essence of our results is shown in Figs 1-4. Figure 1a shows the a.c. susceptibility transition of the as-reacted and subsequently oxygenated samples in a zero-to-peak field of 0.1 mT. Both transitions are rather more abrupt than in the original report¹. The treatment in oxygen markedly raised the T_s onset from ~ 80 K to ~ 91 K. Both samples appeared electromagnetically well-connected under this small a.c. field and the sharp transition indicates the basic good quality of the Hg-1201 phase. Raising the a.c. field however, from 0.1 to 4 mT, produced a marked broadening of the transition (shown in Fig. 1b) consistent with electromagnetic granularity caused by the field breaking the intergrain shielding current loops. The d.c. magnetization was measured in a vibrating sample magnetometer (VSM) at fields up to 12 T from 4 to 80 K. The critical current density (J_{cm}) was calculated from the modified Bean expression magnetization. hysteresis. $\Delta M = J_{con} n_{\beta} (1 -$ (a₂/3a₁))/10, where 2a₁, 2a₂ are the cross sectional dimensions and $a_1 > a_2$. The whole sample cross-section (1.5 mm \times 2 mm) was used to derive J_{cm} in the limit that all the current was flowing from grain to grain (intergrain). The grain size (- 5 µm) was

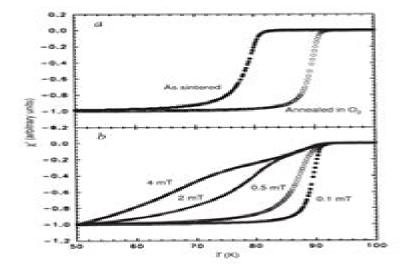


FIG. 1.a. Real component of the susceptibility (x') for the as-sintered and payeen annealed samples at M=0.1 mT. ϕ , $\chi^*(T)$ of the oxygen annealed sample for a.c. fields between 0.1 and 4 mT.

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Daniël Cornelis van der Laan

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