Observation of an anomalous transmission in x-ray scattering by a superconducting $Nd_{1.85}Ce_{0.15}CuO_{4-\delta}$ crystal

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An anomalous-transmission effect in x-ray scattering, characteristic of perfect crystals, has been observed in a superconducting $Nd_{1.85}Ce_{0.15}CuO_{4-\delta}$ crystal. © *1994 American Institute of Physics.*

The observation and study of perfect high T_c superconducting crystals would be important for clarifying the nature of high T_c superconductivity, by analogy with the case of semiconductors. Such research would make it possible to avoid the masking effects of defects on the results of measurements. It would also make it possible to study the structure and properties of superconductors by means of the highly precise diffraction methods which have been developed for studying perfect Si and Ge crystals, and which are based on the observation of dynamic diffraction effects.

Dynamic effects (an anomalous transmission or Borrman-Pendellösung effect: narrow reflection curves with a width close to the theoretical width for a perfect crystal) were observed in neutron and x-ray diffraction in Refs. 1 and 2 in nonsuperconducting Nd_2CuO_4 and $Nd_{1.85}Ce_{0.15}CuO_4$ crystals grown by shattering a single-crystal seed on a crystal holder in a molten solution.[3] That such effects would occur in superconducting crystals is not obvious, since imparting superconducting properties to a crystal requires creating a large number of oxygen vacancies by high-temperature annealing. The nonuniform vacancy concentration which is particularly characteristic of thick crystals ($t > 300\ \mu m$) grown by the method of Ref. 3 might lead to a significant stress, which would suppress dynamic effects.

In this letter we are reporting observation of an anomalous transmission in the scattering of x rays by a thin superconducting $Nd_{1.85}Ce_{0.15}CuO_{4-\delta}$ crystal grown by spontaneous crystallization from molten solution.[4]

The crystal was a platelet with a thickness $t \simeq 80\ \mu m$ and an area $S \simeq 2\ mm^2$. It had a uniform black color and a "mirror" finish. The plane of the platelet coincided with the

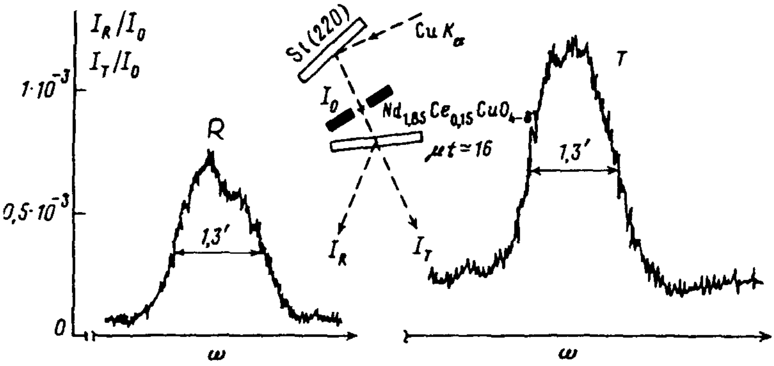

FIG. 1. Curves of the reflection R and transmission T of the crystal, along with the experimental layout.

(001) crystallographic plane. The superconducting transition temperature was 21 K according to measurements of the magnetic susceptibility.

The experiments were carried out on a two-crystal diffractometer, in which a Si crystal served as monochromator. We used CuKα radiation. Curves of the x-ray reflection $R(\omega)$ and transmission $T(\omega)$ were measured for the (200) Laue reflection. A method involving x-ray topography and selective etching was used to visualize the defects of the crystal. Topograms were recorded by scanning the crystal across the R beam, without the help of the monochromator. The etching was carried out by the method of Ref. 5.

Peaks were found on the $R(\omega)$ and $T(\omega)$ curves. The intensities of the R and T beams at the peaks are $\simeq 1\times10^{-3}$ of the intensity of the beam incident on the crystal (Fig. 1). Since the calculated product of the linear absorption coefficient μ and the crystal thickness is $\mu t \simeq 16$, the presence of these peaks is evidence of an anomalous x-ray transmission in the crystal.[6] Working from the experimental value of the integral reflection coefficient, $\rho \simeq 2.8\times10^{-7}$, we calculated the ratio of the effective (interference) absorption coefficient μ_i to the "ordinary" absorption coefficient μ, by analogy with Ref. 1. The result, $\mu_i/\mu \simeq 5.3$, is lower than for either $Nd_{1.85}Ce_{0.15}CuO_{4-\delta}$ or Nd_2CuO_4 crystals which have not been annealed [$\mu_i/\mu \simeq 10$ (Ref. 2) and $\mu_i/\mu \simeq 14$ (Ref. 1), respectively]. The widths of the peaks on the R and T curves are 1.3′, in comparison with the theoretical 17″ for a perfect crystal.

To determine the reason for the broadening of the he peaks, we studied defects in the crystal. It was found by x-ray topography that there are no small-angle boundaries in the crystal, but defects of a nondislocation nature are observed. On the topograms they are seen as an alternation of bright and dark bands along the plane of the crystal platelet (the period is $\simeq$0.1 mm). We did not see defects of this type in previous studies of crystals which were not annealed.[1,2] These bands can be attributed to the presence of a periodic stress field in the crystal, apparently associated with a nonuniform distribution of oxygen vacancies. A final resolution of the nature of this stress, however, will require a comparison of topograms recorded before and after a sample is annealed. The stress makes it difficult to see individual dislocations on the topograms (Fig. 2).The dislocation density was accordingly determined by observing etching pits, which (as was shown in Ref. 5)

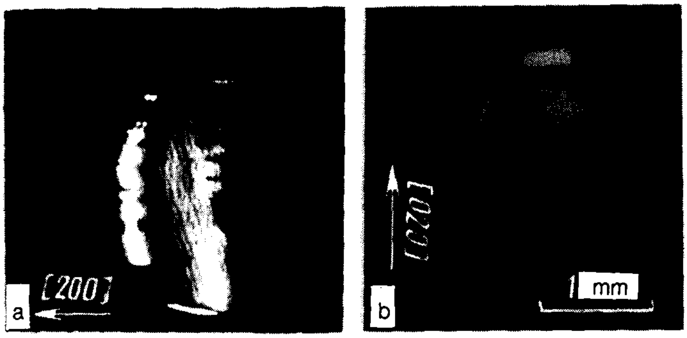

FIG. 2. Some x-ray patterns recorded in the *R* beam for the [200] and [020] reflections (a and b, respectively).

correspond unambiguously to dislocations in $Nd_{1.85}Ce_{0.15}CuO_{4-\delta}$ crystals. The dislocation density was $\simeq 2\times 10^4$ cm^{-2}, or an order of magnitude higher than in $Nd_{1.85}Ce_{0.15}CuO_{4-\delta}$ and Nd_2CuO_4 crystals (grown by the method of Ref. 3) which have not been annealed.[1,2]

Despite the defects in the crystal, the occurrence of the Borrman effect shows that it is possible to achieve a quality high enough for the existence of dynamic effects in x-ray scattering, at least in thin superconducting crystals. Crystals of this quality might be used (for example) in x-ray-acoustic experiments near the superconducting transition, like some experiments which have been carried out on nonsuperconducting crystals at room temperature.[7]

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