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Evidence for magnetic organization of high T_c superconductor YBa₂ Cu₈O₇ observed by neutron diffraction

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Résumé.- Des expériences de diffraction de neutrons sur des poudres de YBa₂Cu₃O₇ montrent l'apparition d'intensité diffuse sous champ magnétique au dessous de la température critique. Cette diffraction a été observée sur l'anneau de poudre correspondant à la réflexion 001 de la maille orthorhombique (d = 11,65 Å). Un pic d'intensité double de celle de la diffraction nucléaire apparaît dans le plan perpendiculaire à la direction du champ. Le phénomène est réversible et disparaît pour $T > T_c$. L'intensité de l'anneau de diffraction ne montre aucune trace de texture même en présence de la diffraction supplémentaire observée sous champ.

Abstract.- Neutron diffraction experiments on powder in a magnetic field on the high T_c superconductor YBa₂Cu₃O₇ reveal the existence of diffuse intensity below the critical temperature. This diffraction is observed on the ring corresponding to the 001 reflection (d = 11.65Å) of the orthorhombic lattice. Two symmetric spots, with intensity twice that of the nuclear diffraction appear in the plane normal to the magnetic field. This phenomenon is reversible and vanishes for $T \succ T_c$. The nuclear diffraction ring does not exhibit any texture even in presence of the additional diffraction observed with the magnetic field.

1. Introduction.

In an external magnetic field between H_{c_1} and H_{c_2} a superconductive material exhibits a regular organization of parallel flux lines which each one carry a quantum of flux. The value of the magnetic field in the centre of the flux line is equal to H_{c_2} . This result, first theoretically established by Abrikosov[1], has been confirmed by neutron diffraction which indicates that the flux lines are ordered in a triangular lattice [2,3]. In order to understand the properties and superconductivity mechanism of the new high T_c superconductors, we have undertaken a systematic investigation of the magnetic flux organization in these systems.

Preliminary experiments revealed that there is no observable magnetic diffraction on powders in the range $2.5 \times 10^{-3} \text{ Å}^{-1} < q < 0.5 \text{ Å}^{-1}$ in which the regular superconductors (A 15 type for example) exhibit a characteristic coherent diffraction due to the regular lattice of the flux lines. But an examination of the diffraction pattern reveals the apparition of intense spots superimposed to the nuclear diffraction, in a magnetic field and below T_c , for powders of YBaa₂Cu₃O₇. We report here this first result.

2. Experimental.

a) Preparation of the samples. The powders of YBa₂Cu₃O₇ ($T_c = 95K$) are prepared starting from a mixture of Y₂O₃, BaCO₃ and CuO reacted around 900°C in air. After several grinding and annealing the powder is treated in an oxygen flux at 900°C and slowly cooled down to room temperature. X-ray powder diffraction (CuK_{α}) on Guinier-Lainé camera reveals that under these conditions the pure triple *perovskite-like* phase is obtained. The superconductivity is verified by magnetic field exclusion in liquid nitrogen.

b) Neutron diffraction experiment was done with the small angle scattering spectrometer PAXY of the Laboratoire Léon Brillouin, located at the exit of a cold neutron guide of the Orphée reactor. This spectrometer is equiped with a two dimensional area detector with 128 * 128 cells of 0.5*0.5 square cen-

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timeters [4]. The incoming beam wavelength, defined by a mechanical selector, was 3.23 ± 0.22 Å and the sample-detector distance was fixed at 0.89 meter. The direct beam impinges the detector in the middle, so that the whole first diffraction ring ((001) of the orthorhombic cell) can be observed in a single experiment. The size of the incoming beam was 8 millimeters in diameter.

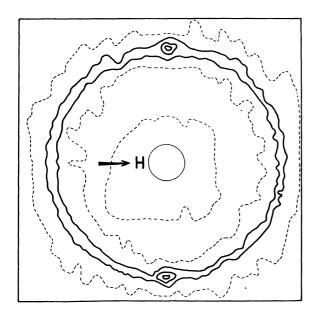
The powder sample was hold in an aluminium can of 12 millimeters in diameter and 12 millimeters high set at the bottom of a helium cryogenerator. The sample temperature, measured by a platinum resistance, was recorded during experiment and the temperature regulation was measured by another platinum resistance. Great care was taken in the temperature variation in order to obtain an homogeneous sample temperature. The temperature control was done by a standard electronic device, with an accuracy of 0.01 K. The sample was hold vertically in an horizontal magnetic field given by an electromagnet with a maximum flux of 1.6 Teslas. Data acquisition time was typically one hour.

3. Results.

Measurements were done for two temperatures, 130 K and 80 K, and with two magnetic field values, zero and 1.6 Teslas. No differences were observed for data recorded at high temperature for the two magnetic field values and at low temperature in zero field, the intensity of the diffraction ring $(d = 11.65\text{\AA})$ remaining constant. But at 80 K with 1.6 Teslas field, two strong peaks appear in a plane perpendicular to the magnetic field direction superimposed to the nuclear diffraction ring (Fig.1). Figure 2 shows the relative intensity with and without magnetic field in the direction normal to the applied field. Whereas there is no appreciable modification of the pattern at small angle, the intensity is multiplied by more than twice at the nuclear diffraction position. For the other parts of the ring there is no change in intensity when the magnetic field is applied. This observation is the first evidence of a magnetic structure in the supraconductor state.

This effect is reversible : the peaks disappear when the field is switched off, and appear again with the field switched on. Experiments done with a vertical field leads to the same result : two symmetric peaks appear at the position of the first diffraction ring, in a plane normal to the field direction.

The diffraction pattern observed in the supraconductor state with applied magnetic field is the evidence of a magnetic structure. Further experiments are on progress in this particular reflection and others, in order to determine the nature of this structure and the influence of the magnetic field and of the temperature.



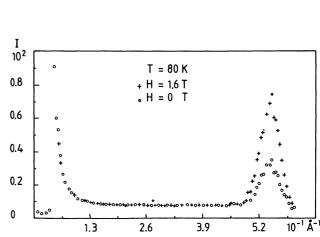


Fig.1.-Iso-intensity curves of the diffraction pattern ($\lambda = 3.23$ Å) of a YBa₂Cu₃O₇ powder sample at 80K with external horizontal magnetic field H = 1.6T, recorded with the XY detector. Two spots are clearly observed superimposed to the nuclear diffraction ring (001) (the full line) in the plane normal to the direction of the magnetic field.

Fig.2.-Intensity distribution along a direction normal to the magnetic field at 80 K. Crosses and open circles correspond respectively to data recorded with magnetic field (1.6 T) and without magnetic field (Data acquisition time 20 minutes).

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