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# Orientation of superconducting crystalline MgB<sub>2</sub> in magnetic field determined by x-ray diffraction

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# Introduction

It is well established experimentally and theoretically that MgB<sub>2</sub> exhibits unique and intriguing anisotropic properties. These are manifested in the ratios of the upper critical fields  $\gamma_H$ =  $H_{c2}^{\ ab}/H_{c2}^{\ c}$  and of the London penetration depth  $\gamma_{\lambda} = \lambda_c/\lambda_{ab}$  [1-8]. Experimental evidence shows that  $\gamma_H = \gamma_\lambda \approx 2$  near  $T_c$ , and at low temperatures  $\gamma_{\lambda} \approx 1$  and  $\gamma_{H} \approx 6$ . For a crystal with  $\gamma_{H} = \gamma_{\lambda}$  in a field along the *c*-axis, superconducting free single crystal grains experience a torque that tends to orient their ab plane parallel to the field [9,10]. However, for  $\gamma_{\lambda} \neq \gamma_{H}$ , recent theoretical predictions [11] suggest that a crystal will orient with its *c*-axis along the magnetic field. Thus, the temperature dependencies of  $\gamma_{\lambda}$  and  $\gamma_{H}$  suggest that near  $T_{c}$ , where  $\gamma_{H} = \gamma_{\lambda}$  and MgB<sub>2</sub> crystal will orient with its *ab*-axis parallel to magnetic field, and at low temperatures where  $\gamma_2 \neq \gamma_H$  it will orient with its c-axis along the field. Herein, we present synchrotron X-ray diffraction studies to determine the preferred orientation of crystalline powdered samples under applied magnetic fields at various temperatures.

## **Methods and Materials**

X-ray studies were conducted on the X-ray Operations and Research (XOR) beamline 4IDD, at the Advanced Photon Source, Argonne National Laboratory. A 4-T cryogen-free superconducting magnet mounted on a Huber diffractometer was used to produce magnetic field normal to the scattering plane. Two types of MgB<sub>2</sub> samples distinguished by the growth method and morphology (loosely sintered pellet, LSP [12], and thin filament fiber [13]) were used in this study. A portion of each sample was coarsely crushed to grains of  $50 - 100\mu$  in size and another portion was thoroughly ground to  $5 - 10 \mu$ . The four samples were loaded in thin walled cylindrical aluminum cans and sealed under helium. The intensity of each point in a 20 scan was integrated over 60 degree of sample rotation for a good powder averaging.

# **Results and Discussion**

Figure 1 (a,b) shows intensity versus scattering-angle 20 of the (0,0,1) Bragg reflections at T = 4.5 K demonstrating the effect of applied magnetic field on the coarse and fine powders. The coarse powders exhibit a 10 - 20% increase between zero and 4 Tesla, whereas the corresponding increase for the fine powders is 7 - 8 fold. The fiber growth fine powder shows even stronger effect under magnetic field. As grown MgB2 even when moderately ground consists of bound crystalline clusters and only thorough grinding yields isolated single crystals (5 -10  $\mu$  in size). By contrast to the (0,0,1) reflection, the intensities of (h,k,l) reflections  $h \neq 0$  or  $k \neq 0$  yielded significantly smaller changes in intensity under magnetic field. Figure 1(c) shows scans of the (0,0,1) reflection for LSP finepowder that was cooled under 4 Tesla from T = 50 K to 4.5 K in comparison with a similar scan of a ZFC sample. There is neither observable difference between FC and ZFC protocols on grain orientation nor observable difference between zero and 4 Tesla at 50 K, verifying the orientation occurs only in the superconducting state. The degree of crystalline orientation is both temperature and field dependent, as shown in Figure 1(d). At all temperatures the intensity of the (0,0,1) initially increases with field showing that the ground-state orientation does not change with temperature, in disagreement with theory[11]. The more detailed field dependent measurement, conducted at T= 32.5K, shows some features that correlate with  $H_{c2}^{min} \approx 0.5T$  and  $H_{c2}^{max} \approx 1.25T$ , as indicated by arrows. This yields  $\gamma_{\rm H} \approx 2.5$ , which is consistent with the value obtained in Ref. [2,8,12].



**Figure 1:** Intensity versus scattering-angle  $2\theta$  for (a) fiber grown samples at T=4.5K, (b) losely sintered pellet grown samples at T=4.5K and 50 K (c). The integrated intensity as function of magnetic fields at different temperatures for the LSP fine-powder sample is shown in (d).

The present study demonstrates the application of X-ray diffraction to characterize the anisotropic properties of MgB<sub>2</sub> in the superconducting state. Specifically, it shows the preferred orientation under magnetic field is for a crystal to align with its  $H_{\perp c}$  at low temperatures, where theory predicts that  $H_{\parallel c}$  orientation. Furthermore, this orientation persists at all temperatures with no evidence of orientation change up to  $T_c$ .

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