Microstructure and pinning properties of hexagonal-disc shaped single crystalline MgB_2

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INTRODUCTION

The existence of impurities and structural imperfections on a microscopic scale can result in diverse transport and pinning properties in MgB_2, which was even observed for single crystalline samples made by several groups[1-3]. Here, we report the X-ray micro-diffraction measurements for, MgB_2 single crystals with hexagonal-disc shapes and shiny surfaces. The diagonal length and the thickness for the largest crystal was about 100 μm and 10 μm, respectively. The crystallinity was thoroughly identified by using the Laue pattern in the X-ray micro-diffraction measurement. Both the edge and the c-axis of the hexagonally shape disc were found to match the crystal symmetry.

EXPERIMENTAL

Two different procedures were used to grow the single crystals, and in both cases, excess Mg was critical for the growth of single crystals. The first involved a two-step method in which already synthesized pieces of MgB_2 bulk[4] were used as a seed material. They were heat treated in a Mg flux inside a Nb tube, which was sealed in an inert gas atmosphere. Then, the Nb tube was put inside a quartz tube, which was sealed in vacuum. The quartz tube was heated for one hour at 1050 °C, cooled very slowly to 700 °C for five to fifteen days, and then quenched to room temperature. The crystal images were observed using a polarizing optical microscope and a field-emission scanning electron microscope (SEM). We successfully separated single-crystalline MgB_2 from the Mg flux by using a thermo-mechanical spinning method.

For the X-ray micro-diffraction measurements, several crystals were fixed at the center of Cu crosshairs on the substrate, as shown in Fig. 1. The instrument used at the Advanced Light Source (ALS) for X-ray micro-diffraction is capable of producing a submicron-size X-ray microbeam and with submicron spatial resolution can probe the local texture in a single crystal[5]. The sample was positioned using the Cu fluorescence signal detected from the Cu crosshairs on the Si substrate by using a high-purity Ge ORTEC solid-state detector connected to a multichannel analyzer. The crystal orientation with respect to the substrate can be determined with an accuracy of 0.01 degree.

RESULTS

The crystal structure was identified by using white beam X-ray micro-diffraction measurements. After positioning these single crystals, a 100 μm × 100 μm region between the Cu crosshairs was scanned with a step size of 2 μm. At each step, the Laue pattern (together with the Cu *K*

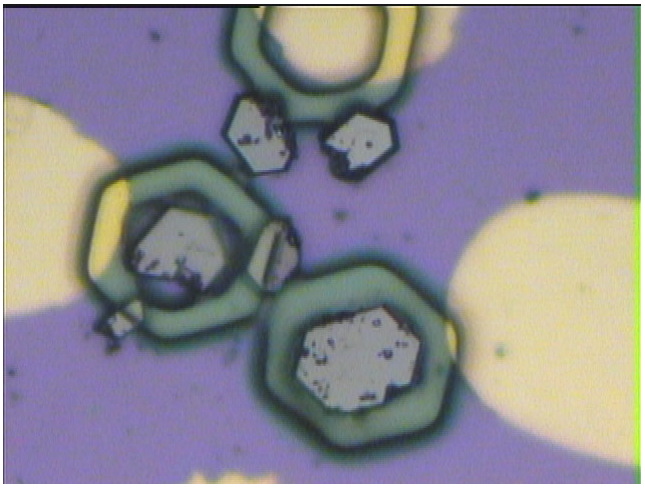

Figure 1. Polarizing optical microscope images of MgB_2 single crystals. An epoxy was used to fix six single crystals at the center of 100 μm-wide Cu crosshairs.

fluorescence signal) was collected with a BRUKER 6000 CCD camera which has an active area of 9 × 9 cm and was placed about 4 cm above the sample. [2500 images, 1024 pixel ×1024 pixel mode] The exposure time at each step, was 1 second. An example of a Laue pattern obtained from a MgB_2 single crystal is shown in Fig. 2(a). The Laue patterns are consistent with a hexagonal MgB_2 structure (a =0.3086 nm, c = 0.3524 nm, Space Group number =191, Ref. 6). The (0005) reflection in the center of the pattern in Fig. 2(a) corresponds to the direction of the normal to the crystal surface. This confirms that the surface plane normal is along the c-axis. Moreover, the hexagonal edges of the crystals were found to match the <1,0,-1,0> directions within a fraction of a degree resolution. Thus, the shapes of the crystals in the microscope image followed the MgB_2 crystal symmetry, which will be quite useful for any research of the direction dependencies of the physical properties in MgB_2.

Indexing the Laue patterns in Fig. 2(a) allowed us to calculate the complete orientation matrix of the X-ray illuminated volume. A finer step size of 1 μm was used for the white-beam scan. The orientation variations inside the single crystal shown in the right bottom corner of Fig. 1 are shown in Figs. 2(b) and 2(c). Figure 2(b) is the out-of-plane orientation variation calculated as the angle between the c-axis and the normal to the surface of the silicon substrate. The out-of-plane variation was about 0.2 degrees between the light orange and the red regions. The out-of-plane orientation shows a variation of about 0.2 degrees between the bottom and the top parts, indicating a slight bending of the crystal (which might be due to the photoresist used as an epoxy). Figure 2(c) shows the in-plane orientation variation calculated as the angle between the measured a-axis (or b-axis) and a reference directions. The variation was about 0.4 degrees between the light blue-green and the green regions. The in-plane orientation also showed some inhomogeneities of up to about 0.2 degrees.

These results demonstrate that the orientation of crystal axis of our hexagonal-disc-shaped single crystals was perfect, within 0.2 degrees[7]. A recent study showed that (0001) twist grain-boundaries, formed by rotations along the c-axis (typically by about 4 degrees), were the major grain boundaries in polycrystalline MgB_2 [8].

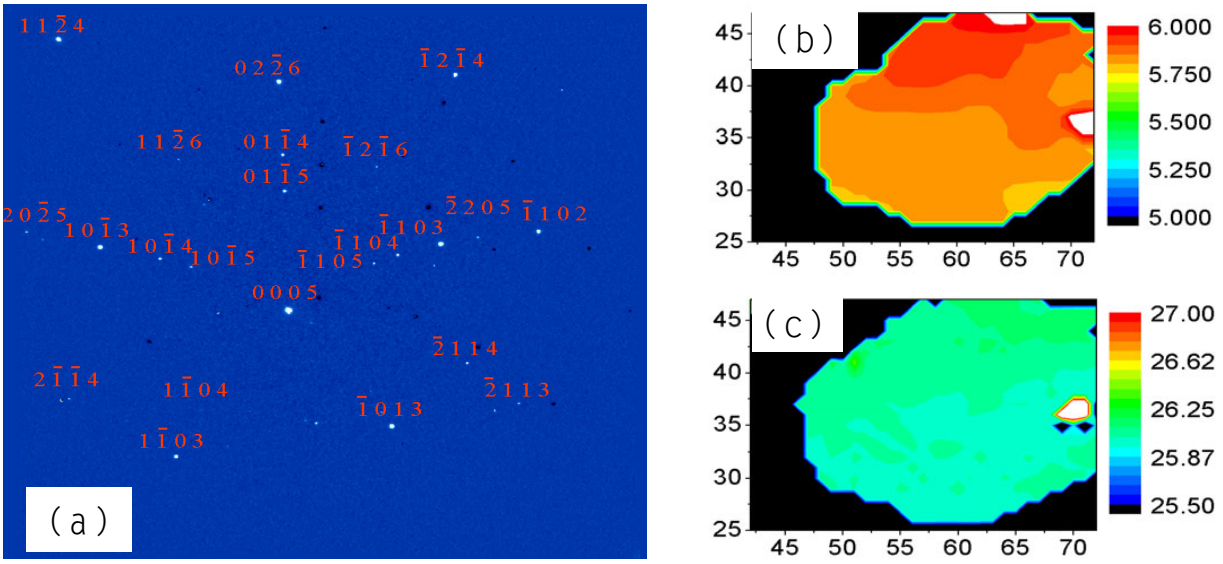

Figure 2. (a) A representative image of an indexed Laue pattern from X-ray micro-diffraction. (b) and (c) are, respectively, for the out-of-plane and the in-plane orientations inside a single crystal.

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