Electron backscatter diffraction of MgB$_2$

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Abstract. MgB$_2$ is one of important new functional materials, which can play significant role in emerging Hydrogen Economy. If adopted, Hydrogen Economy would be a low-CO$_2$ fossil-fuels-free answer to the current and future energy demands. To contribute to this economy, advanced methods of MgB$_2$ preparation and an insight into its properties are required. This paper reports electron backscatter diffraction (EBSD) of dense polycrystalline MgB$_2$ prepared by Hot Isostatic Pressing and Resistive Sintering. The EBSD study was performed in combination with polarised optical microscopy, scanning electron microscopy, transmission electron microscopy, atomic force microscopy and scanning conductivity probe microscopy. The superconducting properties were measured in order to select specimens with highest critical current density to associate this density with structural features of the samples. The investigation shows importance of twist grain boundaries in increasing critical current density in MgB$_2$.

1. Introduction
MgB$_2$ is a relatively new superconducting material promising to radically change the area of applied superconductivity. As the amount of available of fossil fuels steadily decreases while demand for energy continues to grow, superconductivity is posed to play an increasing role in the energy economy. This role is directly linked to the use of hydrogen as a clean fuel. Hydrogen could be derived in abundance from the renewable energy resources such as solar, wind, marine, hydro, geothermal, biomass and also produced in nuclear and thermonuclear power stations. The convenient way of hydrogen transport and storage is in its liquid form and the liquid hydrogen would enable wide use of superconductivity. There are many superconducting materials capable of working effectively at temperature of liquid hydrogen. MgB$_2$ has the far largest potential for these applications. The mechanical and superconducting properties of brittle and low-critical-field MgB$_2$ are still far from ideal for power applications. Significant efforts are required in order to improve its properties with the imaging playing crucial role in supporting these efforts. In this paper, several imaging techniques are applied to MgB$_2$ to clarify its structural properties on micro and nano scale in order to link them with superconducting properties. The electron backscatter diffraction (EBSD) appeared to be a central imaging technique. It allowed to obtain crucial information about grains, grain boundaries and crystal lattice of MgB$_2$. It gave access to a wealth of information not possible to reach with other imaging techniques and was guiding those techniques to obtain additional information.
2. Experimental
MgB$_2$ was prepared using hot isostatic pressing (HIP) and resistive sintering (RS). HIP is a standard technique successfully applied to MgB$_2$ [1]. RS is an original technique, its closest analogue being spark plasma sintering [2]. Details of HIP and RS in application to MgB$_2$ are described in [3,4].

Critical current density ($J_c$) of MgB$_2$ samples was derived from magnetisation measurements performed on a Quantum Design Magnetic Properties Measurement System. The samples were imaged by polarised optical microscopy (POM), scanning electron microscopy (SEM), transmission electron microscopy (TEM), electron backscatter diffraction (EBSD), atomic force microscopy (AFM) and scanning conductivity probe microscopy (SCPM). Moving the sample from one instrument to another, selected areas were visualised by all these techniques except for TEM, which requires special preparation of the sample. In this study, EBSD emerged as a central technique. It has been run on an electron microscope JEOL 7000. The crystallographic MgB$_2$ file for INCA software was generated by Oxford Instruments Ltd.

3. Results and discussion
POM, EBSD and AFM images of a selected area of MgB$_2$ are shown in figure 1. The grains in the material are clearly distinguished by the colour in POM and EBSD and by the topography in AFM. An important feature is that some grains, which are seen in POM as one, are split in EBSD in two or more grains by straight lines.

![Figure 1. EBSD, POM and AFM (left to right) images of an area of MgB$_2$. The grains 5-7, 10 and 12 seen in POM as one, are split in EBSD by straight lines in two or more grains. The AFM image is taken around the grain 8. Top left shows the crystal orientation of the parts of the grain 7, which are separated by a 15°-twist grain boundary.](image-url)
Analysis of the crystal lattice orientation in the grains separated by straight lines, like that shown for the grain 7 in the top left part of the plot, reveals that the lines are twist grain boundaries with rotations around $c$-axis of MgB$_2$. It is natural to expect the appearance of such twist boundaries in the pressed MgB$_2$ since it is a highly anisotropic layered material with weak forces between $ab$-planes. In HIP and RS, small grains are pressed into each other at an arbitrary angle creating large number of such boundaries on a micro and nano-scale. These boundaries coexist with other high-angle boundaries as shown in figure 2. In this figure, small square area is selected on a large EBSD map on the left. It is re-mapped in details by EBSD (not shown) and imaged by AFM (centre of the plot). The EBSD gives full description of the crystal orientation in the grains as is shown by the unit-cells around the AFM image, where green atoms are for magnesium and red are for boron. The grain boundary misorientation angles are also shown in the plot. The same square area was also mapped by conductivity scanning probe microscopy as described in [4].

Figure 2. EBSD map (left) and AFM image (centre) of a selected square area of MgB$_2$. Differently directed unit-cells around the AFM image show crystal lattice orientation in the grains. The grain boundary misorientation angles are also shown in the plot. In the AFM image, the grain boundary on the left is a low-energy coincidence site lattice boundary (CSLB). Most of the grain boundaries seen in AFM image in figure 2 are of high angle. The grain boundary on the left is low-energy coincidence site lattice boundary (CSLB) [3]. It is also a twist boundary with 28.4°-rotation around $c$-axis. CSLB boundaries are most frequent in MgB$_2$. Their high angles provide large density of dislocations that effectively pin magnetic flux and support high superconducting current in the sample [3]. The CSPM study [4] gives evidence that grain boundaries with relatively high angle are not detrimental for transport properties. This is further confirmed by $J_c$ measurements in a large number of samples with different grain size $D$ as shown in figure 3.
In this figure, open red circles are for the pure MgB$_2$ samples and black filled squares are for the samples with nanoparticles of external materials. The latter were added to further deform MgB$_2$ grains and increase the number of twist boundaries. The scattering of the points is lower for the samples with nanoparticles and $J_c$ is inversely proportional to the average grain size determined from the EBSD maps. The reciprocal $J_c(D)$ dependence is confirmed by the double-logarithmic presentation of the data in the inset. The experimental points fit well a model describing pinning on grain boundaries [3].

The clarification of the nature of pinning in MgB$_2$ became possible due to the use of EBSD. In addition to POM, EBSD detected twist boundaries, guided AFM, CSPM and TEM in selection of most important areas for imaging, allowed to determine grain size and visualised effect of external nanoparticles on micro and nano-structure of the samples.

![Figure 3](image-url)

**Figure 3.** Grain size dependence of critical current density in MgB$_2$ at 20 K and magnetic field of 2 T. Inset shows the data in double-logarithmic scale. Black filled squares are for the samples with nanoparticles of external phases and open red circles are for pure MgB$_2$. The red lines in the plot show reciprocal $J_c$ dependence on $D$.

4. Conclusions

Electron backscatter diffraction was successfully applied to MgB$_2$ produced by the advanced methods of resistive sintering and hot isostatic pressing. EBSD allowed to obtain important information about the micro and nano-structure of the samples and clarified nature of the pinning in the material. The EBSD findings can be useful in designing new processing techniques that would be able to increase critical current density in MgB$_2$. These techniques could be based on the treatment of pure MgB$_2$ or, alternatively, on the nano-additions of external phases.

References

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