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Effect of Boron powder purity on superconducting properties of bulk MgB$_2$

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Abstract

In order to study the influence of the amorphous Boron powder on the superconducting properties, MgB$_2$ bulk samples were prepared using 96% and 99% pure commercial Boron powder as well as 92% commercial Boron powder after purification process. The results showed that the original 96% and the purified 92% powders have larger particle size compared to the pure 99% Boron powder, which leads to reduce magnetic critical current densities. In order to get higher performance MgB$_2$, the purified low grade Boron powder need further control of their microstructure such as smaller particle size to enhance flux pinning from the grain boundaries which represent effective pinning centers.

Keywords: MgB$_2$; Particle size effect; Flux pinning; Critical current density

1. Introduction

Since the discovery of the superconductivity of MgB$_2$ [1], there has been a considerable amount of research undertaken to understand the enhancement of $J_c$ performance by means of dopants and nanoparticle additions such as SiC, C, hydrocarbon and Dy$_2$O$_3$ [2–8]. On the other hand, according to previous work [9], properties of the starting Boron powder, such as purity and particle size, may also play an important role in the superconducting properties. In this work, we focus the discussion on the particle size. It was found that MgB$_2$ prepared with 96% and 99% pure commercial Boron powders as well as 92% commercial Boron powder after purification process have different behaviors for the volume pinning force, which was calculated from critical current density curve and the respective values of the applied field.

It is suggested that the control of the microstructure, particularly the particle size, may enhance the flux pinning originated from the grain boundaries.

2. Experimental

MgB$_2$ samples were prepared by in situ solid state reaction of crystalline magnesium powder (99.8%, 325 mesh) and three different types of boron powders: commercial 99% (pure 99%), commercial 96% (original 96%) and commercial 92% after purification process (purified 92%). Mg and B were mixed in the atom ratio of 1:2, followed by hand milling (30 min). The pellets were sintered in Ar, at different temperatures, using heating rate of 10 °C/min and furnace cooling down. The X-ray diffraction patterns were recorded in a Philips PW1730 diffractometer using a Cu K$_\alpha$ radiation source. The magnetization hysteresis loops were performed with the magnetic field applied parallel to the longest dimension of samples with $2 \cdot 2 \cdot 3$ mm$^3$ (Quantum Design MPMS). The critical current density $J_c$ was determined from DC magnetization

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loops at 5 K and 20 K. Magnetic critical current density $J_c$ was estimated based on the Bean's Model \[10\].

3. Results and discussion

Table 1 shows the full-widths at half maximum (FWHM) determined by TRACES™ 6.3.0 software for three main indexed peaks observed in the studied samples. FWHM is related to the crystallite size by the Scherrer equation:

$$D_p = \frac{0.94k}{b_{1/2}} \cos h$$

where $D_p$ is the crystallite size ($\text{Å}$), $k$ is the radiation wave length ($\text{Å}$), $b_{1/2}$ is the determined peak FWHM ($^\circ$), and $2h$ is the considered peak position ($^\circ$). Consequently, from the values on the table, it can be concluded that the average crystallite domain sizes, which could be calculated for different orientations, are significantly smaller for pure 99% compared to the purified 92%. And the same is verified for purified 92% compared to the original 96% sample. Although this approach has been reported as to neglect the effect of the strain on the crystallite size \[11\].

In Fig. 1 the plots are showing the field dependence of the normalized volume pinning force ($F_p/F_{p,max}$), at (a) 5 K and (b) 20 K, for different samples. At 5 K, the highest $F_p$ curve was obtained for pure 99%, followed by the purified 92% and the original 96% sample. The Table along with these set of results indicate that the decrease of the particle size may enhance flux pinning originated from the grain boundaries improving the critical current density.

At 20 K, a two step behavior is observed. For applied fields below 1.3 T, the pure 99% sample presented the largest pinning forces, while the purified 92% and the original 96% samples showed nearly the same forces. For higher applied fields, the curve for the purified 92% sample turns out to be close to the pure sample, while the original 96% sample, for which was estimated to have the largest crystallite size, showed the lowest values for the pinning forces.

4. Conclusion

In summary, we have shown that the crystallite size and, consequently, the particle size of the starting boron powder are important parameters and should be controlled when a better $J_c(H)$ performance on MgB$_2$ is aimed. However, in order to verify the possibility of using purified 92% or even original 96% powders, instead of high cost pure powders, to make high $J_c$ performance MgB$_2$, further work need to be performed on obtaining smaller particle sizes for these impure powders.

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