



Effect of ball milling time on the substitution of carbon in glucose doped MgB_2 superconductors: Dispersion behavior of glucose

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ABSTRACT

The effect of the ball milling time (BMT) on the substitution of the carbon in the glucose doped MgB_2 samples is investigated here. Using in situ solid state reaction, four different doped samples of $\text{Mg}(\text{B}_{0.98}\text{C}_{0.02})_2$ were prepared by mixing powders of Mg, boron and glucose for 2 h, 4 h, 8 h and 12 h using planetary ball milling. A reference sample of un-doped MgB_2 was also prepared under same conditions. The particle size distribution of the un-reacted samples show a decrease in the particle size as the BMT is increased. Both the average particle size as well as the standard deviation show a substantial decrease with the increase in the milling time up to 8 h. After 8 h, the size reduction is rather insignificant. From the XRD data, the crystallite size of the doped MgB_2 computed using the Scherrer formula was found to decrease with the increasing BMT, showing a saturation level after 8 h of the milling time. TEM images also confirm the crystallite size obtained from the XRD data. The substitution of the C in the MgB_2 lattice, measured from the change in the c/a ratio, increases with increasing BMT. The maximum carbon substitution is achieved at approximately 8 h of BMT. Moreover, a systematic enhancement of the residual resistivity and a decrease in T_C with an increasing BMT further confirms a progressive substitution of the carbon in the MgB_2 . These results suggest that a minimum ball milling time is necessary to disperse the glucose uniformly for a maximum substitution of nano C in the B plane of MgB_2 lattice. The optimum BMT is found to be 8 h. Thus, the decrease in the particle size due to the ball milling enhances the dispersion of the constituent materials thereby favoring a greater substitution of the dopant in the MgB_2 during the solid-state reaction.

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1. Introduction

The discovery of the superconductivity in the MgB_2 at 39 K [1] has led to extensive studies covering both its fundamental aspects as well as practical applications [2–4]. The two band nature of the MgB_2 imparts noteworthy features to its behavior in both the superconducting [5–7] as well as the normal states [8–10]. Moreover, the MgB_2 possesses simple crystal structure, relatively high transition temperature (T_C), large coherence length and transparency of grain boundaries to current flow [11]. The application of its pristine bulk sample is however limited due to the significant fall in the J_C in high magnetic fields due to weak pinning centers and low upper critical field ($H_{C2} \approx 14$ T at near-zero temperature) [2]. Recently, Eom et al. [12] and Braccini et al. [13] reported occurrence of very high H_{C2} and relatively large J_C in dirty thin films of

MgB_2 . This generated a great deal of renewed interest towards replicating similar improvement in the bulk MgB_2 samples [3]. Various kinds of techniques have been suggested in the literature. These include chemical doping [14–23], irradiation [24,25], and thermo-mechanical processing techniques [26–29]. Especially, the chemical doping technique looks promising in this context. The doping of nano particles has been reported to enhance the useable J_C - H behavior. Highest degree of improvement is achieved with the SiC [30,31] doping while the carbohydrate doping [32] has also yielded comparable enhancement. In both cases, it is the reactive nano-C that gets substituted in the B lattice. Moreover, the doping provides nano-sized impurities which act as enhanced flux pinning centers and also helps to remove the inter-grain resistive boundary [3]. It is in fact the uniform dispersion of the small amount of the dopant homogeneously throughout the MgB_2 matrix that poses an important challenge towards the exploitation of the full potential of the doping technique.

The planetary ball milling (BM) is extensively used in synthesizing the bulk MgB_2 samples owing to its flexibility of operation, the ease of usage and a faster mixing. The effect of various ball milling

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parameters on the properties of the bulk samples, tapes and wires has been reported in the literature [33–39]. Constituent materials can either be mixed in the liquid or the dry environment. Different gas environments can be chosen during the dry mixing. For example, a simple ball milling of the B powder in toluene is found to improve the J_c – H behavior of pure MgB_2 [40]. The effect of ball milling on the individual constituent powders of Mg and boron has also been studied. The milled B has been reported to improve the superconducting properties of pure MgB_2 [34]. In the present, the ball milling time is varied in the preparation of the bulk MgB_2 samples doped with the glucose. The objective here is to optimize the ball milling time for a maximum substitution of carbon. The glucose is chosen as the dopant here in view of the fact that it provides a reactive nano C source near the sintering temperature. The amount of the glucose was added in order to have a fixed 2% of C atom in the $\text{Mg}(\text{B}_{1-x}\text{C}_x)_2$. Four different samples of 2% glucose were prepared by varying the ball milling time from 2 to 12 h. A pure MgB_2 reference sample with 8 h of ball milling was also prepared for the comparison. The XRD measurements were carried out to identify the phases and quantify the percentage of C doping in the MgB_2 . The samples were also analyzed by TEM and SEM images. Resistivity measurements were carried out as a function of the temperature to confirm the variation of the substitution of the carbon in the MgB_2 .

2. Experimental details

In the present study, samples of 2% glucose doped MgB_2 superconductors were prepared by in situ solid state reaction method. Here, the 2% means the amount of glucose which substitutes 2% of B by C in MgB_2 lattice (i.e. $\text{MgB}_{1.96}\text{C}_{0.04}$). The constituent materials were 99.99% amorphous boron powder (Sigma Aldrich), 99.9% Mg powder (CERAC with average particle size 45 μm) and 99.9% glucose, $\text{C}_6\text{H}_{12}\text{O}_6$, (Fluke). These were mixed in stoichiometric ratio using planetary ball milling machine.

Four different sets of constituent materials were weighed and ball milled for 2, 4, 8, and 12 h, respectively in a 80-ml Zr_2O bowl using six 1-cm diameter ball of Zr_2O . These are henceforth termed as 2 h, 4 h, 8 h and 12 h samples. In all cases, the rpm was fixed at 100. Since the ball-milling time has a direct bearing on the size of the particles, three replicate runs were carried out to determine the particle size distribution of each sample using Microtrac 3500 Particle Size Analyzer.

After the ball milling, the powder was pelletized under 10 tons of the pressure. During pellet preparation equal amounts of the powder were used to have equal size of pellets. These were then wrapped in the tantalum foil and placed in a cylindrical crucible of soft iron. Crucibles were placed inside a long quartz tube closed at one end and valve fitted at the other end for evacuation. The quartz tube was evacuated to the order of 10^{-5} torr. It was placed inside the preheated furnace. All four samples were sintered at 650 $^\circ\text{C}$ for two hours under high vacuum under same conditions. After two hours of sintering, the furnace was switched off and samples were cooled under normal conditions. The fifth reference samples of pure MgB_2 of 8 h ball milling duration, henceforth termed pure sample, was prepared under the same condition as described above.

Samples were characterized by XRD for phase identification with the help of X-ray diffractometer (PANalytical X'Pert Pro MPD) using $\text{Cu K}\alpha$ radiation. Resistivity measurement from the room temperature (RT) to the 30 K were carried out using in-house standard four probe technique inside the APD closed cycle cryocooler. Susceptibility was measured using home-made ac susceptibility described elsewhere [41]. Grain morphology was studied using Scanning Electron microscope (JEOL ASEM 6360A).

3. Results and discussion

Since the most closely physical aspect related with the ball-milling time is the particle size distribution of the constituent elements of the sample, it is therefore first presented in Fig. 1. As mentioned earlier, three replicate runs were carried out in each case. A close agreement was observed in these runs. The average of all the three runs is shown in the figure. A substantial spread is seen in the particle size distribution varying from 0.5 μm to almost 250 μm . Note that peaks around 0.5 μm are of those of the B powder. On the other hand, main contribution to peaks around 45 μm is from the Mg powder used here. It is clear that the sample particle size distribution is mostly oriented towards large-size particles for small ball-milling times. In the case of 2 h sample, particle population with sizes greater than 100 μm exists that reaches up to 250 μm . As the ball-milling time is increased to 4 h, the size reduction of large-sized particle takes place. The particle population above 100 μm is now absent. Thus, as the ball-milling time is increased, there is shift in the particle size distribution towards the smaller size particles. Another noteworthy feature here is that

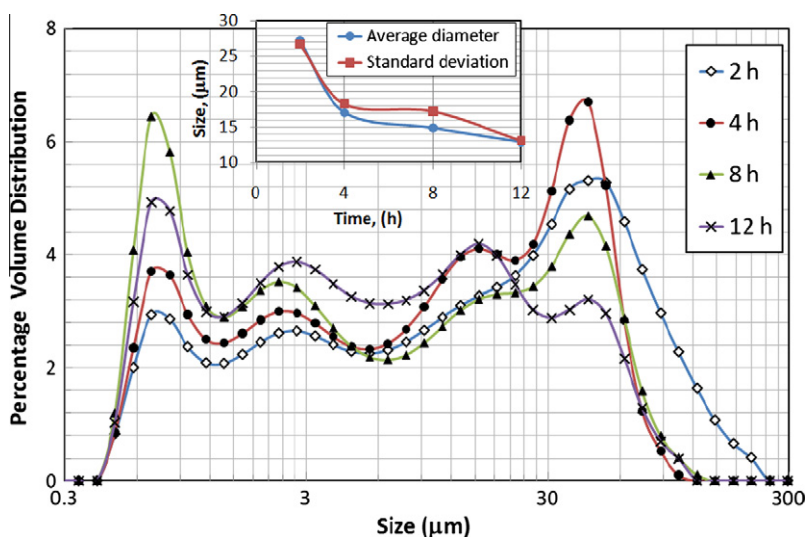


Fig. 1. Particle size distribution of 2% glucose doped MgB_2 samples at different ball-milling times.

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