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Low Temperature Formation of MgB₂ from Mg/B Composite Particles[†]

ABE Hiroya*, NAITO Makio**, NOGI Kiyoshi** and FUKUI Takehisa***

Abstract

The formation of superconducting MgB₂ phase from Mg/B composite particles was investigated. Mg/B composite particles were fabricated by an advanced mechanical method from the corresponding element particles, Mg and B. The mechanically treated particles were annealed under atmospheric pressure of argon. MgB₂ phase clearly appeared after relatively low temperature annealing. The structural analyses revealed that the superconducting phase was formed at the Mg and B composite region of the processed particles. These results indicate that the mechanical treatment of the powder elements can enhance the reactivity for the formation of MgB₂.

KEY WORDS: (MgB₂), (Superconductor), (Advanced mechanical method), (Composite particle)

1. Introduction

After the discovery of superconductivity in MgB₂¹⁾, attempts have been made to synthesize this material with the aim of improving superconducting properties. Although systematic studies relating to the effect of synthesis parameters on the superconducting properties have been reported, some of the practical aspects for the formation of MgB₂ are not addressed, such as the influence of mechanical treatment. Mechanical treatment has been known to be an effective technique for mixing as well as for driving a wide range of chemical reactions²⁾. In the latter case, it utilizes mechanical energy instead of thermal energy to provide the activation energy for solid-state reaction and it can be designed as an intermediate step to promote reactions that can be completed only at high temperature³⁾. In order to prove the possibility of MgB₂ for various practical applications, the accumulation of experimental data on various physical properties related to the superconductivity is urgently needed.

In this short note, we show the influence of the mechanical treatment of Mg and B powders on the formation of superconducting MgB₂ phase.

2. Experimental

The raw materials used in this study were 99.5% pure Mg powder with a sieve size of -330 mesh (Wako Pure Chemical Industries, Japan) and 97% pure amorphous B powder with an average particle size of 0.8

μm (Rare Metallic, Japan). Powder mixtures consisting of an Mg:B molar ratio of 1:2 were processed using an advanced mechanical method (Mechanofusion System[4], Hosokawa Micron Corp., Japan) with the rotation speed of the milling vessel at 2000 rpm for 5 min and 60 min under atmospheric pressure of argon (flow rate 1 l/min). The processed powder mixture was removed from device for analysis and for subsequent annealing, and the superconducting and structural characterization were performed.

3. Results and Discussion

Figure 1 shows the temperature dependence of field cooled (FC) magnetization for the powder mixtures annealed at 500°C. In this measurement, the sample temperature was reduced to 4 K through the critical temperature T_C under a fixed field of 10 G. The diamagnetism magnitude for the powder mixture processed for 60 min is about 50 times larger than that for 5 min. Clearly, a larger superconducting phase was noted for the longer mechanical treatment powder mixture. When annealing at 630°C, the magnetization curves for both powder mixtures were almost the same, showing T_C ~ 38K.

Figure 2 shows Mg element mapping on the cross sectional plane of a particle obtained by processing for 60 min. As can be seen, Mg concentration is less in the region of about 1 μm width from the particle, which

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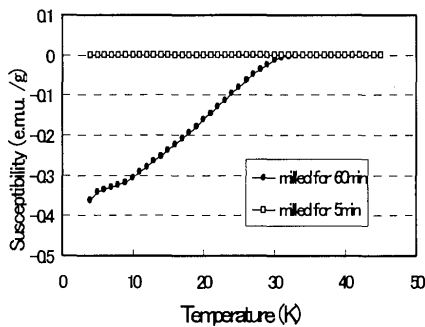


Fig.1 Temperature dependence of field cooled magnetization under 10 G for 500°C annealed powder mixture.

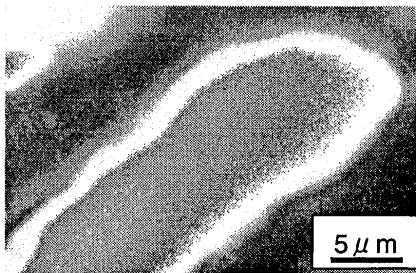


Fig.2 Cross-sectional EPMA image of the processed powder mixtures for 60min (Mg mapping)

indicates the derived particle was B embedded composite particles.

Figure 3 shows the surface structure of the B embedded composite particle annealed at 500°C. The crystallites of closed-packed hexagonal MgB₂ are clearly visible. Thus, the MgB₂ phase was formed by annealing at 500°C at the surface region of the composite particle.

It has been already shown that the solid state reaction was enhanced by mechanical processing^{2,3}. This study also shows that the longer mechanical milling of Mg and B powders raised the reactivity for the formation of MgB₂ compound and led to the low temperature synthesis of MgB₂.

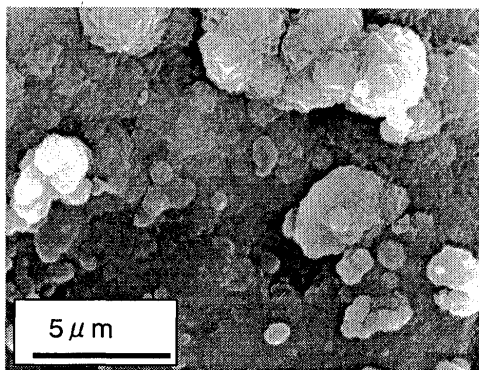


Fig.3 Surface morphology of the composite particle annealed at 500°C.

It was found that the enhanced reactivity observed was strongly related to the structure of the composite particle in which B particles were embedded into the surface of Mg. This embedded region may have a highly disordered crystal structure and microscopic mixing state, which would act as strong driving force for the formation of MgB₂.

4. Conclusion

Large Mg particles (-330 mesh) and submicron amorphous B particles (average particle size, 0.8 μm) were processed for 60min using an advanced mechanical device. Then, B/Mg composite particles were obtained, in which B particles were embedded into Mg particles at about 1 μm depth. MgB₂ phase was synthesized at this embedded region by annealing at relatively low temperatures.

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