

# Microstructure and Superconductive Property of MgB<sub>2</sub>/Al Based Composite Materials<sup>\*)</sup>

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MgB<sub>2</sub> superconducting wires are interesting as the alternative conductors of Nb-based superconducting wires applied for the advanced nuclear fusion reactor because of the decay time of the induced radioactivity. However, MgB<sub>2</sub> is difficult for practical use because of its unworkable and lower critical current density ( $J_C$ ) in a high magnetic field than Nb-based superconductive materials. We have developed the original method of three-dimensional penetration casting (3DPC) to fabricate the MgB<sub>2</sub>/Al composite materials. In the composite materials we made, MgB<sub>2</sub> particles dispersed in the matrix uniformly. This makes these composite materials can be processed by machining, extrusion and rolling. And the  $T_C$  of the made composite materials was determined by electrical resistivity and magnetization to be 37~39 K. In this work, we made composite materials with ground MgB<sub>2</sub> particles with the purpose of extruding thinner wires of composite material. Furthermore, we successfully produced  $\phi$ 1 mm wires and also changed the matrix from pure Al to Al-In alloy.  $J_C$  of composite materials with the matrix of Al-In alloy was calculated with the width of the magnetic hysteresis based on the Extended Bean Model. The result was better than that of MgB<sub>2</sub>/Al composite material without Indium. Microstructures of these samples had been confirmed by SEM observation.

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## 1. Introduction

Superconductor MgB<sub>2</sub>, the Type II superconductor, was discovered in January, 2001 by Prof. Jun Akimitsu of Aoyama Gakuin University [1]. Its superconducting transition temperature ( $T_C$ ) is 39 K which is higher than that of Nb-based superconductor, however, the decay time of the induced radioactivity is shorter than the later one.

Therefore, MgB<sub>2</sub> superconducting wire was interested as an alternative conductor of Nb-based superconductor for the advanced fusion reactor application and so on [2]. And the studies on the MgB<sub>2</sub> superconductor have been focused on the application for superconducting magnets and Nb-based intermetallic compounds [3], as well as the projects for fabrication of wires and/or sheets are actively pursued [4]. In addition, in a high magnetic field, MgB<sub>2</sub> has low critical current density, which makes many researchers study how to improve its  $J_C$  behavior [5]. In our previous study, we fabricated composite materials with Al or age-hardenable Al alloys matrix reinforced by ce-

ramics particles such as Al<sub>2</sub>O<sub>3</sub>, SiC, and TiC, also investigated their hardening behaviors, microstructures, and aging properties [6]. Our special technique of 3DPC method for fabricating composite materials can disperse particles in the matrix homogeneously without any aggregation and control their volume fractions within the range of 4-50%, even when particle size is less than 1  $\mu$ m [6]. Thus, these composite materials can be processed by machining, extrusion and rolling. On the other hand, we also reported that MgB<sub>2</sub>/Al composite materials had superconductive behavior, and succeeded in extruding MgB<sub>2</sub>/Al composite billet to  $\phi$ 10 mm rod and  $\phi$ 3 mm wire [7]. In the present work, we made composite materials with ground MgB<sub>2</sub> particles with the purpose of extruding thinner wires of composite material. furthermore, we added Indium in the aluminum matrix to improve  $J_C$  of the MgB<sub>2</sub>/Al composite material in high magnetic fields, because it had been reported that Indium improved  $J_C$  of MgB<sub>2</sub> in high magnetic fields [8]. The microstructures of these samples were confirmed by SEM method. And the superconducting properties were evaluated using magnetization and electrical resistivity.

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## 2. Experimental Procedures

MgB<sub>2</sub> powders (Kojundo Chemical Laboratory Co., Ltd.) had a purity higher than 99%, and size smaller than 40 μm. These particles are called as “normal particles”, hereafter, the normal particles were ground with a mortar and pestle made by agate in the glove box with Ar-gas for the purpose of preventing particle oxidation. The particles with the size under 25 μm were obtained through the mesh with the sequence from 90, 75, 45, 32 to 25 μm (called as “fine particles”, hereafter). Initially, a preform was fabricated using compacted powders with 30 mm diameter and 42 mm length. This preform was set in the bottom of a steel mold. Molten Al at about 1173 K was poured into this steel mold and pressed into the preform by a pressing machine. This method was referred as the 3-dimensional penetration casting (3DPC) method. After cooling, the billet was removed from the steel mold by cutting. The volume fraction ( $V_f$ ) can be controlled to 10-50% by this method. The  $V_f$  of MgB<sub>2</sub> powders in the obtained billet was about 50% for the high  $V_f$  sample. The obtained billet was also extruded by a hot-extruding machine to a  $\phi$ 1 mm wire. On the other hand, it was added into the molten aluminum. And the indium-added sample was manufactured by 3DPC method with normal particle. The superconductivity of the composite materials with Al and Al-In matrix were measured by Physical Property Measurement System (PPMS, Quantum Design, Co., Ltd.). Samples for the measurement were cut from composite materials to 1 mm cubes. Electrical resistivity was measured by a DC 4-terminal method, with a direct current of 1.0 mA. magnetization was from room temperature to 4.2 K, and cooling rate was 0.003 K/s. Magnetization was measured by SQUID (Quantum Design, Co., Ltd.) using an applied magnetic field of 100 G. The range of the temperature employed for measurement of electrical resistivity, thermal conductivity, and The microstructures of composite materials were observed by a scanning electron microscope (SEM) of S-3500H (Hitachi, Co., Ltd.) operating at 20 kV. Samples for microstructure observation were simply cut from composite materials and polished using conventional polishing papers.

## 3. Results and Discussion

Figure 1 shows SEM images of (a) normal and (b) fine MgB<sub>2</sub> particle. There are no particles larger than 25 μm in (b) comparing to (a). Figure 2 shows a longitudinal cross section of the composite material billet made from the normal particles. No remarkable shrinkages, cracks, large aggregations of powders or any other defects are observed. Gray and bright contrasts in this figure correspond to the reinforced region and pure Al region without particles, respectively. The region of Al also exist at the bottom side of the steel mold, indicating that the molten Al sufficiently penetrated to the bottom side through the preform of MgB<sub>2</sub> and can be turned back to the preform by the applied pressure. The billet made with the fine particles show a similar

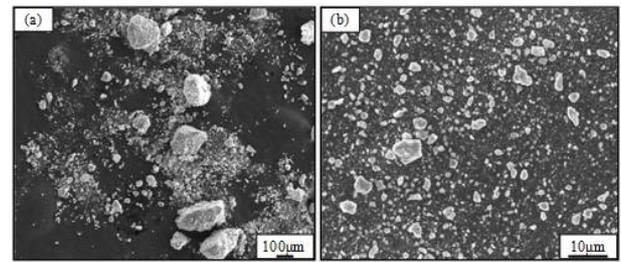


Fig. 1 SEM images of (a) normal and (b) fine particles of MgB<sub>2</sub>.



Fig. 2 Outlook of composite material billet.

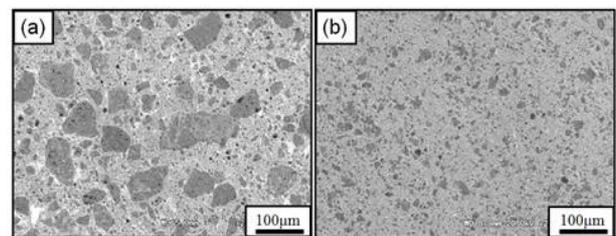


Fig. 3 SEM images for cross section of composite materials used (a) normal and (b) fine particles of MgB<sub>2</sub>.

result to Fig. 2, with is not shown here.

Figure 3 shows the longitudinal cross sections of the composite materials made with normal and fine particles. These images show a homogeneous distribution of the particles in the matrix, and no cracks between particles and the Al matrix at this magnification. Figure 4 shows a microstructure of the cross section of the extruded  $\phi$ 1 mm wire composite material, which indicates the homogeneous distribution, and no cracks between particles and the Al matrix at this magnification of particles.

Figure 5 shows the longitudinal cross sections of the composite materials made with Al and Al-In matrix. These images also show a homogeneous distribution of the particles in the matrix, and no cracks between particles and the Al as well as Al-In matrixes at this magnification.

Figure 6 shows the electrical resistivity of composite materials depending on the temperature. Superconducting onset temperature ( $T_C$ ) is determined about 37.7 K for the samples with Al matrix, and 38 K for those with Al-0.05wt.%In, Al-0.1wt.%In, Al-0.2wt.%In.

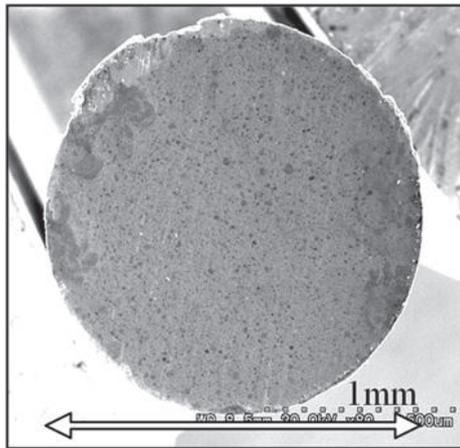


Fig. 4 SEM image of cross section of extruded  $\phi 1$  mm wire.

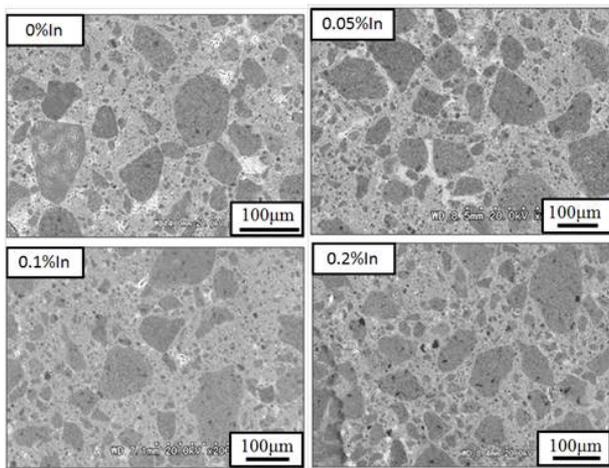


Fig. 5 SEM images of the longitudinal cross section of the composite materials made with Al and Al-In.

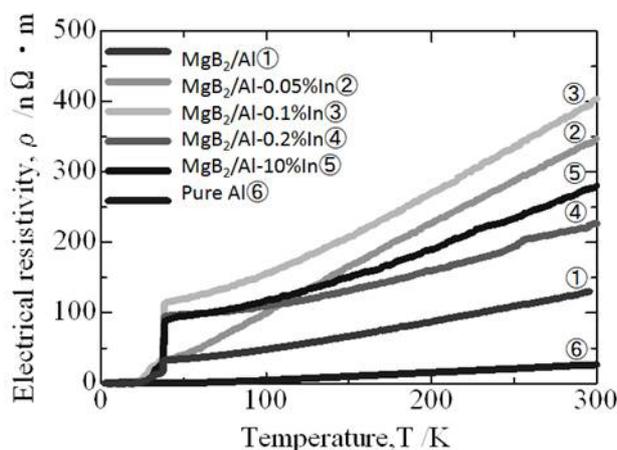


Fig. 6 Electrical resistivity depending on temperature.

Figure 7 shows the critical current density  $|J_C(H)|$  of composite materials. The critical current density  $|J_C(H)|$  of pristine and composites is estimated from the  $M(H)$

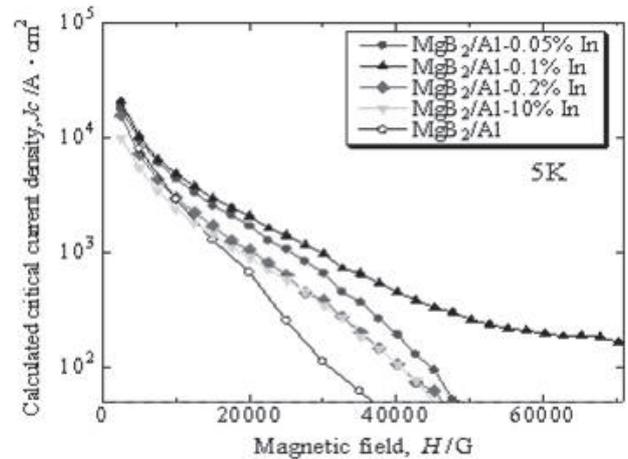


Fig. 7 Calculated critical current density of the composite materials.

loop by using the equation of an extended Bean critical state model [9] of  $|J_C(H)| = 20\Delta M/a(1-a/3c)$ , where  $\Delta M$  is the width of the  $M(H)$  loop in  $\text{emu}/\text{cm}^3$  at a given field and temperature. The  $\text{MgB}_2/\text{Al-In}$  composites causes the increasing tendency of  $J_C$  compared with  $\text{MgB}_2/\text{Al}$  composite, moreover, the  $J_C(H)$  of  $\text{MgB}_2/\text{Al-0.1wt.\%In}$  is the highest among  $\text{MgB}_2/\text{Al-In}$  composites.

#### 4. Conclusions

Billet and extruded rods of composite materials the  $\text{MgB}_2$  particles dispersed in aluminum matrix are fabricated, and confirmed to exhibit superconductivity. The composite materials with fine particles dispersed in aluminum matrix are successfully fabricated. No remarkable defects are observed in these composite materials. The composite billet is hot-extruded into a  $\phi 1$  mm wire, and no remarkable defects are observed. This extruded composite wire shows an onset  $T_C$  of magnetization about 38 K. Al or Al-In based  $\text{MgB}_2$  composite materials are also fabricated by the three-dimensional penetration casting method. The longitudinal cross sections of the  $\text{MgB}_2/\text{Al}$  and  $\text{MgB}_2/\text{Al-In}$  composite material billets show no remarkable shrinkages, cracks, large aggregations of powders or any other defects. All the composite materials are found to have similar  $T_C$  ( $\sim 38$  K) to that of  $\text{MgB}_2$  intermetallic compound. The critical current density  $|J_C(H)|$  of the pristine and composites are estimated from the  $M(H)$  loop by using equation of an Extended Bean Model. The  $\text{MgB}_2/\text{Al-In}$  composites cause the increasing tendency of  $J_C$  compared with  $\text{MgB}_2/\text{Al}$  composite, moreover, the  $J_C(H)$  of  $\text{MgB}_2/\text{Al-0.1wt.\%In}$  is the highest among  $\text{MgB}_2/\text{Al-In}$  composite materials.

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