분무열분해법에 의한 나노 MgB_2 분말 제조 연구

Nano MgB_2 Powder Synthesis by Spray Pyrolysis Method

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Abstract: Nano-sized spherical MgB_2 powders were synthesized by spray pyrolysis method. The influence of solution concentration and furnace reaction temperature on morphology and average particle size were investigated. For adequate preparation conditions, it has mostly spherical, solid and narrow particle size distribution. Average particle size(X_{50}) distribution was below 100 nm. The critical temperature for the synthesized MgB_2 was around 36K.

Key Words: MgB_2 nano powder, spray pyrolysis, flux pinning.

1. Introduction

In spite of the significant advancement made on critical current density of high temperature superconductors, the transport properties of BSCCO 2223/Ag tapes are still limited by the grain connectivity. Many magnetization and transport measurements show that MgB_2 does not exhibit weak-link electromagnetic behavior at grain boundaries[1] or fast flux creep phenomena[2], which limit the performances of high-T_c superconducting cuprates.

As already reported, high critical current densities have been observed in MgB_2 bulk samples, regardless of the degree of grain alignment[3,4]. Eom *et al.*[5] reported grain boundaries may act as pinning centers in MgB_2 as in Nb_3Sn. These would be advantages for making wires or tapes with no degradation of *Jc* due to grain boundary, which will induce weak-links and is a common and serious problem in cuprate high temperature superconductors.

The enhancement in *Jc* with metal powder addition or doping is reported [6-8]. In our previous work, J_c(B) of MgB_2 tapes was enhanced with Cu addition[9]. Another approach to enhance flux pinning property is increasing in the number of grain boundary that may act as pinning centers. In order to this, nano size MgB_2 is required. The easiest way to make nano MgB_2 powder is spray pyrolysis method. Spray pyrolysis[10] is a relatively low cost, simple to manipulate, applicable to large-scale production; which enables one to prepare homogeneous, spherical and narrowly size distributed ceramic powders.

In this work, MgB_2 powders were synthesized by spray pyrolysis method with an attempt to have a homogeneous nano-sized spherical shape. The influence of solution concentration and pyrolysis temperatures on the particle size of MgB_2 powders is studied and the superconducting property of the powder is evaluated by magnetic measurement.

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2. Experimental

The experimental set-up employed to study MgB_2 synthesis is shown in Fig. 1. It mainly consists of three parts. The first is the ultrasonic spray zone which consists of the mist-generating system of liquid source with an ultrasonic nebulizer (ultrasonic power of mist generator is about 100 W, 2.56 MHz) and the mist-droplet-carrying system with H_2/Ar mixed carrier gas. The second is the heating zone in which the misted droplet is pyrolyzed in a pre-heated chamber. And the last is

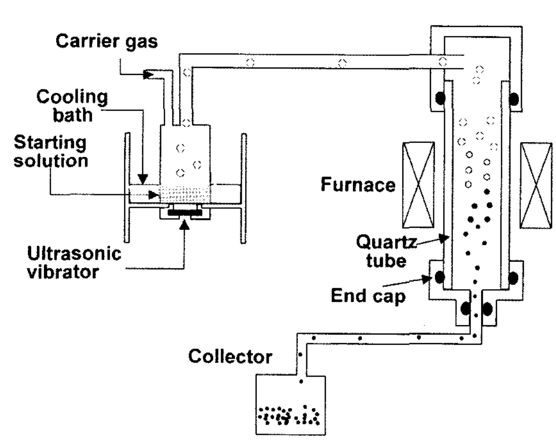


Fig. 1. Schematic diagram of the spray pyrolysis system utilized to prepare MgB_2 based nano-powders.

the pumping zone which consists of pumping evaporated/decomposed gases and trapping the produced powders with gab filters.

MgB_2 powders were prepared from a solution of an aqueous solution which contains Mg and B ions and absolute ethanol. The total precursor concentration of 0.05, 0.1, 0.2 and 0.5 mol/l were tested. The reaction temperature was between 600 and 900°C.

The microstructure, chemical and phase analyses of powders were characterized by SEM, EDS and XRD. The superconducting property of the sample was characterized by zero-field cooled susceptibility measurement.

3. Results and discussion

Fig. 2 and 3 shows SEM micrographs of the MgB_2 powder with different pyrolysis temperatures and solution concentration. It follows, from the images, that the MgB_2 particle size was decreased with dilution of solution concentration. Other important characteristics of the powders obtained by this method were that the aggregation-free particles had a narrow size distribution around 100 nm as shown in Fig. 4.

The MgB_2 phase fraction calculated from the XRD patterns is given in Fig. 5. It was increased with spray pyrolysis temperature.

Fig. 6 shows XRD patterns of the MgB_2 powders, which were prepared at 900°C. Most of the peaks are assigned to the MgB_2 phase. Some secondary phase was identified as MgO.

As shown in Fig. 7, the diamagnetic moment M for the MgB_2 powder sample synthesized from a solution of 0.1 mol/ℓ was measured under zero-field-cooled(ZFC) conditions in an applied field H=20G. The onset of superconducting transition temperature for the sample with spray pyrolysis temperature of 900°C was around 36K. It seems likely that superconductive volume fraction in the sample synthesized is not high. The process condition will be optimized for higher superconductive volume fraction.

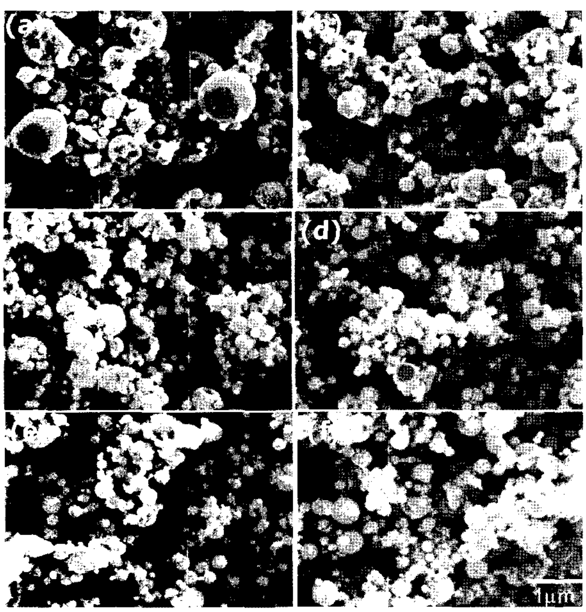


Fig. 2. SEM images of spray pyrolysis powders for various pyrolysis temperature, (a) 600°C, (b) 700°C, (c) 800°C, (d) 830°C, (e) 850°C and (f) 900°C.

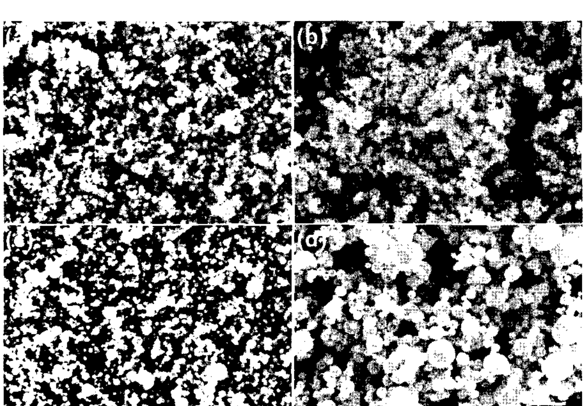
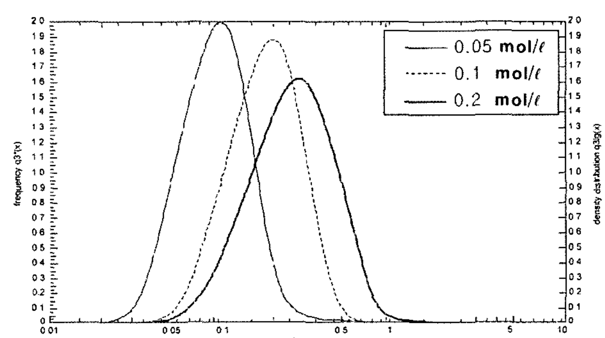


Fig. 3. SEM images of spray pyrolysis powders for various solution concentration, (Pyrolysis temperature: 850°C). (a) 0.05 mol/ℓ, (b) 0.1 mol/ℓ, (c) 0.2 mol/ℓ (d) 0.5 mol/ℓ.



	X_{50}	X_{90}	X_{99}
0.05 mol/ℓ	96nm	180nm	260nm
0.1 mol/ℓ	200nm	420nm	500nm
0.2 mol/ℓ	280nm	580nm	800nm

Fig. 4. Particle size distribution data for spray pyrolysis powders.

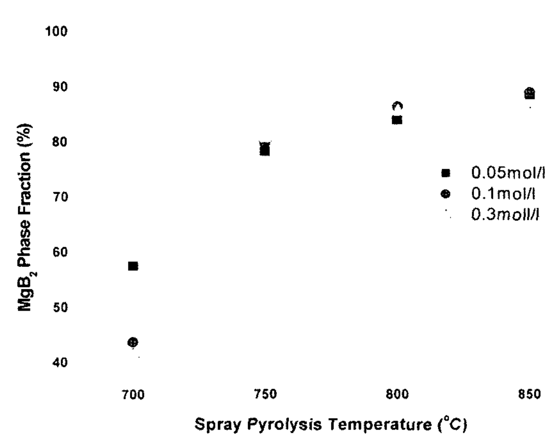


Fig. 5. MgB_2 phase fraction with pyrolysis temperature and solution concentration.

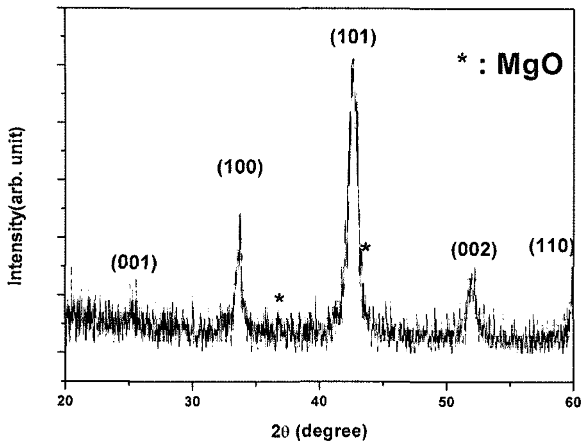


Fig. 6. XRD patterns for MgB_2 powder obtained from a 0.05 mol/ℓ solution, at 900°C.

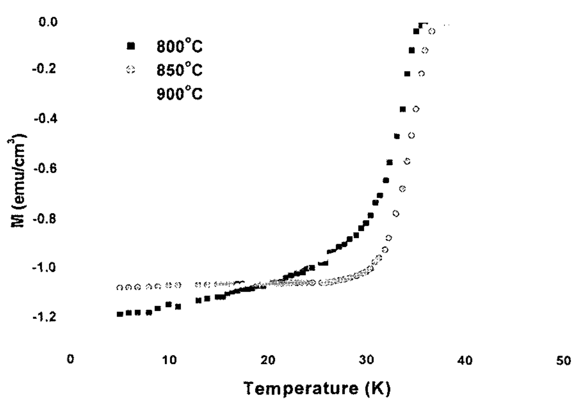


Fig. 7. Diamagnetic transition of MgB_2 superconductor produced by this experiment in zero-field cooled states at 20G for all the samples. (Reaction temp. : 850°C, Solution conc. : 0.1 mol/ℓ).

4. Conclusions

Nano-sized MgB_2 powders were synthesized by spray pyrolysis method. It has been demonstrated the feasibility to produce MgB_2 nano-powders without post-heat treatment with spray pyrolysis technique. The onset of superconducting transition temperature was 36 K. For adequate preparation conditions, powders were mostly spherical, solid and narrowly size distributed. Average particle size(X_{50}) was between 100 and 400 nm.

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