

Microwave Synthesis of Fullerene-Doped MgB₂

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ABSTRACT: Unconventional in situ synthesis of MgB₂ undoped and doped with fullerene (C₆₀) was performed by using a single-mode microwave (MW) furnace. Structural properties suggested that C substitutes for B in the crystal lattice of MgB₂, while the J_c showed typical enhancement at high magnetic fields when C chemical doping occurred. This behavior was similar to published results for another field-activated unconventional method of processing, i.e., spark plasma sintering, and it was opposite to the conventional powder-in-tube method for which chemical substitution was not realized when using addition of C₆₀. Certain morphologically unique features such as the occurrence of needlelike grains were revealed in the MW samples. Our MW samples showed higher relative density ($\leq 83\%$) than previously reported data. Magnetic relaxation experiments suggested the presence of microscopic flux jumps in the MW samples, while macroscopic flux jumps were not observed.

1. INTRODUCTION

Magnesium diboride (MgB₂)¹ has several advantages over other low- T_c as well as high- T_c superconductors. MgB₂ is a light and simple compound that is composed of two elements arranged into a layered hexagonal structure, has low anisotropy, is nontoxic, is inexpensive, does not have the weak-link issue,² and has a relatively high T_c of 39 K. It has been found that chemical doping represents an effective method for the enhancement of $J_c(B)$ in MgB₂, and that carbon is among the most efficient doping.³ Literature indicates that carbon is substituting for B in the crystal lattice of MgB₂ and modifies the electronic structure with beneficial effects on the functional characteristics of MgB₂.

Microwave (MW) processing was less explored for the synthesis of MgB₂^{4–6} but represents an interesting and promising sintering technique at the same time. This is due to the fact that MW is fast, clean, and energy efficient and generally accelerates the synthesis and sintering processes.⁵ In the MW method, the heat in the system originates from the interaction of the electromagnetic field with induced or permanent molecular dipoles and/or ions. The adsorbed energy is distributed all over the material, while energy loss occurs only at the surface. Such a heating distribution produces an inversed thermal gradient in the sample (hotter inside, colder at the surface) that may significantly decrease the time necessary to reach the sintering temperature for the whole sample with respect to the traditional methods. The rapid synthesis may result in small and uniform grain size. In the case of MgB₂, a small particle size leads to an increased number of grain boundaries. A high density of grain boundaries is well-known to positively contribute to the enhancement of pinning and, hence, to the J_c increase in the MgB₂ superconductor.⁵ Recent research indicated that an electromagnetic field may generate certain “nonthermal effects” that produce enhanced activated sintering.^{7,8} Because of the presence of the electro-

magnetic field, nonequilibrium processes may occur with various consequences that are worth exploring. In the case of superconductors, for instance, the induced defects can be useful for pinning.

This work addresses the synthesis of MgB₂ and C₆₀-doped MgB₂ performed in a single-mode cavity MW furnace, in which only one mode of microwave propagation is permitted, and hence, the field pattern is well-defined so the material can be positioned accordingly. It is worth mentioning that previous work on conventional in situ powder-in-tube processing of Mg, B, and C₆₀ mixtures was not successful,⁹ so that the critical current density, J_c , and irreversibility field, H_{irr} , remained constant. On the other hand, unconventional ex situ spark plasma sintering (SPS) applied to mixtures of MgB₂ and C₆₀ powders produced excellent B-substituted samples with enhanced J_c and H_{irr} .¹⁰ In the first case, it was expected that fullerene (C₆₀) would provide a more efficient B substitution with carbon leading to the enhancement of properties because of its low decomposition temperature. This would allow the decrease in the processing temperature compared with those of samples doped with other efficient C-based compounds such as SiC or nanocarbon. The idea of low-temperature processing within the in situ approach and for the enhancement of J_c and H_{irr} is reviewed in ref 11. A reason for this situation might be the behavior of carbon when supplied from different source compounds. As noted by Dou et al.,¹² only certain “active” carbon can produce efficient B substitution effects in the MgB₂ lattice. For C₆₀, it is likely necessary to have an activated processing method. The influence of the electromagnetic field

Received: February 29, 2012

Revised: August 8, 2012

Accepted: August 9, 2012

Published: August 9, 2012