In Situ Synchrotron X-ray Diffraction Study of Reaction Kinetics for Reactively Processed Mg/MgB₂ Metal Matrix Composites

J.D. DeFouw,¹ J.P. Quintana,^{1,2} D.C. Dunand¹

¹Department of Materials Science and Engineering,

Northwestern University, Evanston, IL, U.S.A.

²Advanced Photon Source (APS), Argonne National Laboratory, Argonne, IL, U.S.A.

Introduction

Magnesium diboride is a recently discovered superconductor with a critical temperature of 39K [1] and other superconducting properties comparable to commercially available Nb₃Sn and high-temperature cuprate superconductors [2]. Mechanically, MgB₂ is a brittle ceramic and requires a ductile sheath or dispersion within a metal matrix for commercial applications. We have produced superconducting MgB₂ fibers in situ within a magnesium matrix by reacting commercially available boron fibers and magnesium liquid to form MgB_2 and subsequently solidifying the magnesium [3]. To gain a full understanding of this reactive process, reaction kinetics are studied in situ by using synchrotron x-rays.

Methods and Materials

Boron filaments (Goodfellow, UK) 100 µm in diameter were aligned within a titanium crucible (8-mm inner diameter, 8.5-mm outer diameter) and gas-pressureinfiltrated with magnesium at 800°C [3, 4]. The initial volume fraction of the filaments was 20%. The sample was enclosed within an evacuated quartz tube backfilled with 1/3 atm of argon. A custom infrared lamp furnace heated the sample to 950°C from the side to melt the magnesium and react it with the boron filaments within the titanium crucible. The reaction was monitored by exposing the sample to 65-keV x-rays with 30-second exposure times captured on a charge coupled device (CCD) camera (Photonic Science Limited, UK). Experiments were performed at DND-CAT beamline station 5-BM-D at the APS. Diffraction ring images were converted to 1-D intensity versus 20 plots by using FIT2D software [5].

Results

The formation of MgB_2 was observed through the appearance of a diffraction ring (peak) (Fig. 1) after reaction at 950°C. The intensity of the peak increased with time corresponding to a higher degree of reaction in the fibers (Fig. 2).

Discussion

These preliminary results show that the reaction of $Mg + 2B = MgB_2$ can be studied with synchrotron x-rays and CCD on magnesium-infiltrated boron fibers in a titanium crucible. Future experiments will monitor the reaction of boron fibers and powders having different diameters with magnesium liquid and vapor at temperatures between 700 and 1100°C.

Acknowledgments

This work was supported by the National Science Foundation (NSF) through Grant No. DMR-0319051. Also, the authors thank the DND-CAT staff for help in performing these experiments. DND-CAT is supported by E.I. DuPont de Nemours & Co., the Dow Chemical Company, NSF through Grant No. DMR-9304725, and the State of Illinois through the U.S. Department of Commerce and Illinois Board of Higher Education, Higher Education Cooperation Act Grant IBHE HECA NWU 96. Use of the APS was supported by the DOE Office of Science, Office of Basic Energy Sciences, under Contract No. W-31-109-ENG-38.

References

[1] J. Nagamatsu, N. Nakagawa, T. Muranaka, Y. Zenitani, and J. Akimitsu, "Superconductivity at 39K in magnesium diboride," Nature **410**, 63-64 (2001).

[2] D. Larbalestier, A. Gurevich, D.M. Feldmann, and A. Polyanskii, "High-T-c superconducting materials for electric power applications," Nature **414**, 368-377 (2001).

[3] J.D. DeFouw, and D.C. Dunand, "In situ synthesis of superconducting MgB_2 fibers within a magnesium matrix," Appl. Phys. Lett. **83**, 120-122 (2003).

[4] D.C. Dunand, "Synthesis of superconducting Mg/MgB_2 composites," Appl. Phys. Lett. **79**, 4186-4188 (2001).

[5] A.P. Hammersley, S.O. Svensson, M. Hanfland, A.N. Fitch, and D. Häusermann, "Two-dimensional detector software: From real detector to idealised image or two-theta scan," High Pressure Res., **14**, 235-248 (1996).

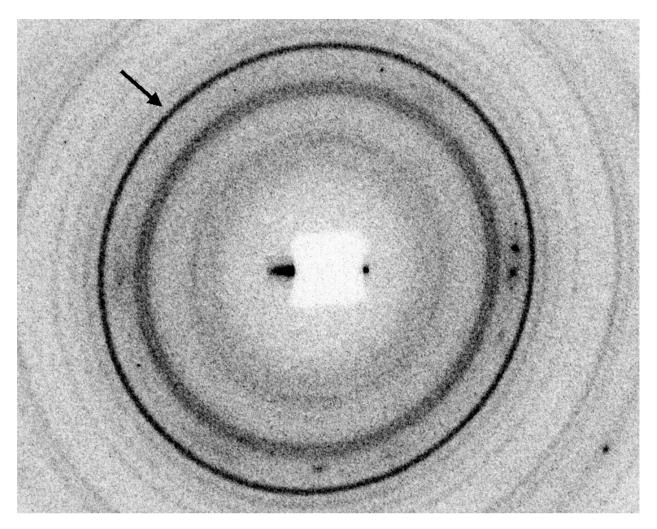


FIG. 1. CCD diffraction image showing appearance of MgB_2 ring (arrow) after reaction of boron fibers with magnesium liquid at 950°C for 2 hours.

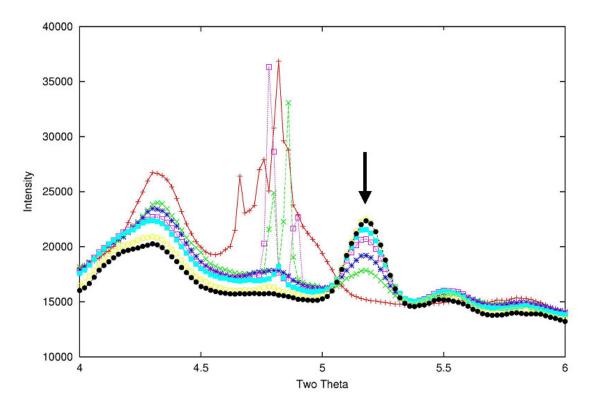


FIG. 2. Intensity versus 2θ plot showing growth of MgB_2 peak (arrow) with time during reaction.