UNIVERSITI PUTRA MALAYSIA

OPTIMISATION OF THE SUPERCONDUCTING PROPERTIES OF MgB2 PREPARED BY THE REACTION OF (MgB4 + Mg) AND DOPING WITH SILICON CARBIDE

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OPTIMISATION OF THE SUPERCONDUCTING PROPERTIES OF MgB_2 PREPARED BY THE REACTION OF (MgB_4 + Mg) AND DOPING WITH SILICON CARBIDE

By

TAN KIM LEE

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in fulfillment of the Requirements for the Degree of Master of Science

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DEDICATIONS

To all my family members…

Especially to my beloved father, although you are not here to witness my achievements today but you'll always stand a special place in my heart. And to my beloved mother, for always being there for me through thick and thin…

In loving memory of,
Mr. Tan Thoo Yoong

"In order to reach the summit, you need to walk step by step"

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science.

OPTIMISATION OF THE SUPERCONDUCTING PROPERTIES OF MgB_2 PREPARED BY THE REACTION OF (MgB_4 + Mg) AND DOPING WITH SILICON CARBIDE

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Chair : Chen Soo Kien, PhD

Faculty : Science

In this work, MgB_2 was synthesized by reaction of (MgB_4 + Mg) and the superconducting properties of the samples were optimized by using the following routes: (i) variation of heat treatment, (ii) addition of excess Mg and (iii) addition of nano-SiC. The porosity of the samples was much reduced as compared to the sample prepared by reaction of (Mg + 2B). The samples were analyzed through XRD, SEM, physical density and superconducting properties measurements.

Firstly, MgB_4 powder was synthesized by direct reaction of Mg and B. Between HNO_3 and HCl, the use of the former for immersing time of 5 minutes was found to be effective in removing MgO phase in the MgB_4 sample sintered at 1050°C for 2 hours. Hence, the MgB_4 powder with reduced amount of MgO was used as the precursor powders for the following reaction.

The phase transformation from MgB_4 to MgB_2 was done by reacting MgB_4 and Mg powders at the temperature range from 650°C to 950°C for 4 hours and 8 hours, respectively. The estimated weight fraction of MgB_2 is the highest at 650°C and 750°C but it decreased above 850°C and upon increasing the sintering time, it changed slightly. With increasing sintering temperature, the transition temperature T_c decreased due to the lattice distortion of MgB_2. The sample sintered at 750°C for 4 hours showed the highest magnetic J_c (4.23×10^4 A/cm^2) at 5 K and 2 T which is consistent with the highest weight fraction of MgB_2.

Excess Mg powder was added into MgB_4 to synthesize nominal Mg_xB_2 (x = 1.2, 1.5 and 1.7) at 650°C and 750°C, respectively for 8 hours. The estimated weight fraction of MgB_2 decreased upon increasing x level and it increased with the sintering temperature. However, the effect of excess magnesium and sintering temperature on T_c was not pronounced. With increasing x, $Mg_{1.7}B_2$ sample sintered at 650°C showed the highest J_c (7.59×10^4 A/cm^2). Meanwhile at 750°C, $Mg_{1.5}B_2$ sample recorded the highest J_c (4.49×10^5 A/cm^2), both at 5 K, 2 T. The J_c was enhanced by higher estimated weight fraction of MgB_2 and the flux pinners (excess Mg).

In order to study the influence of nano-particle on J_c, 2 wt. %, 5 wt. % and 10 wt. % of nano-SiC were added into the samples with nominal composition of $Mg_{1.5}B_2$ and sintered at 750°C for 4 hours. The estimated weight fraction of MgB_2 decreased with SiC addition level. The *a*-axis decreased significantly indicating an increase in the C substitution at the B site upon increasing SiC addition level. Hence, T_c degraded with increasing SiC addition level due to the distortion of MgB_2 lattice. The highest J_c of 1.52 $\times 10^4$ A/cm^2 achieved by MgB_2 added with 2 wt. % of SiC were due to the C substitution that enhanced the scattering of the charge carrier and hence improved the field dependent J_c behavior.

This work demonstrated that inexpensive way of using excess Mg as compared to SiC, could result in samples with higher value of J_c (5 K, < 5 T) but SiC showed weaker field dependence of J_c.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan ijazah Master Sains

PENGOPTIMUMAN SIFAT SUPERKONDUKTOR MgB_2 YANG DISEDIAKAN DARIPADA TINDAK BALAS ANTARA (MgB_4 + Mg) DAN PENAMBAHAN SILICON CARBIDE

Oleh

TAN KIM LEE

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Pengerusi : Chen Soo Kien, PhD

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Dalam kerja ini, MgB_2 disintesis daripada tindak balas (MgB_4 + Mg). Sifat-sifat superkonduktor sampel telah dioptimumkan dengan menggunakan cara berikut: (i) perubahan rawatan haba, (ii) penambahan Mg berlebihan (iii) penambahan nano-SiC. Kelebihan kerja ini adalah keliangan sampel telah berkurangan berbanding dengan sampel yang disediakan melalui tindak balas (Mg + 2B). Sampel dianalisis melalui XRD, SEM, ketumpatan jisim dan pengukuran sifat superkonduktor.

Pertama, serbuk MgB_4 disintesis daripada tindak balas langsung antara Mg dan B. Antara HNO_3 dan HCl, perendaman serbuk MgB_4 dalam HNO_3 selama 5 minit didapati berkesan dalam menghapuskan fasa MgO di dalam sampel MgB_4 yang disinter pada suhu 1050°C selama 2 jam. Oleh itu, serbuk MgB_4 ini dengan fasa MgO yang telah dikurangkan digunakan sebagai serbuk permulaan bagi tindak balas yang berikutnya.

Transformasi fasa daripada MgB_4 ke MgB_2 dilakukan dengan tindak balas antara MgB_4 dan serbuk Mg pada julat suhu daripada 650°C kepada 950°C selama 4 jam dan 8 jam, masing-masing. Anggaran pecahan berat MgB_2 yang tertinggi dicapai pada 650°C dan 750°C tetapi ia berkurangan apabila suhu melebihi 850°C dan apabila masa pensinteran ditingkatkan, ia berubah sedikit sahaja. Dengan peningkatan suhu pensinteran, suhu genting T_c berkurangan disebabkan oleh pengherotan kekisi MgB_2. Sampel yang disinter pada suhu 750°C selama 4 jam menunjukkan J_c (4.23×10^4 A/cm^2) yang tertinggi pada 5 K dan 2 T yang konsisten dengan anggaran pecahan berat MgB_2 yang tertinggi.

Serbuk Mg berlebihan ditambahkan ke dalam MgB_4 untuk mensintesis sampel dengan komposisi nominal Mg_xB_2 (x = 1.2, 1.5 dan 1.7) pada 650°C dan 750°C, masing-masing selama 8 jam. Anggaran pecahan berat MgB_2 menurun apabila tahap x ditingkatkan dan ia meningkat dengan suhu pensinteran. Walau bagaimanapun, kesan magnesium berlebihan dan suhu pensinteran terhadap T_c adalah tidak ketara. Dengan tahap x yang semakin meningkat, sampel $Mg_{1.7}B_2$ yang disinter pada suhu 650°C menunjukkan J_c (7.59×10^4 A/cm^2) yang tertinggi manakala pada suhu 750°C, sampel $Mg_{1.5}B_2$ mencatatkan J_c (4.49×10^5 A/cm^2) yang tertinggi, kedua-duanya pada 5 K, 2 T. J_c telah dipertingkatkan oleh anggaran pecahan berat MgB_2 yang tertinggi dan pengepin fluks (Mg berlebihan).

Untuk mengkaji pengaruh nanopartikel pada J_c, 2 %, 5 % dan 10 % nano-SiC telah ditambahkan ke dalam sampel dengan komposisi nominal $Mg_{1.5}B_2$ dan disinter pada suhu 750°C selama 4 jam. Anggaran pecahan berat MgB_2 menurun dengan tahap penambahan SiC. Parameter kekisi *a* menurun dengan ketara dan ini menunjukkan peningkatan dalam penggantian C di tapak B apabila tahap penambahan SiC ditingkatkan. Oleh itu, T_c menurun dengan peningkatan tahap penambahan SiC yang ia disebabkan oleh pengherotan kekisi MgB_2. J_c yang tertinggi iaitu 1.52×10^4 A/cm^2 dicapai oleh MgB_2 yang ditambah dengan 2 % SiC di mana penggantian C meningkatkan penyebaran pengangkut cas dan secara tidak langsung ia memperbaiki ciri-ciri pergantungan medan J_c.

Penyelidikan ini menunjukkan cara yang lebih murah dengan pengggunaan Mg berlebihan berbanding dengan SiC, untuk menghasilkan sampel yang mempunyai nilai J_c yang lebih tinggi (5 K, < 5 T). Walaubagaimanapun, penambahan SiC menunjukkan pergantungan medan J_c yang lemah.

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I certify that a Thesis Examination Committee has met on 27 December 2012 to conduct the final examination of Tan Kim Lee on her thesis entitled “Optimisation of the Superconducting Properties of MgB_2 Prepared by Reaction of MgB_4 + Mg and Doping with SiC” in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

Members of the Thesis Examination Committee were as follows:

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Date: 2 March 2012

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

TAN KIM LEE

Date: 27 December 2011

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