SYNTHESIS OF CARBIDES COMPOUNDS FROM ELEMENTAL POWDER BY MECHANICAL MILLING



INSTITUT PENYELIDIKAN, PEMBANGUNAN DAN PENGKORMERSILAN UNIVERSITI TEKNOLOGI MARA 40450 SHAH ALAM, SELANGOR MALAYSIA

DISEDIAKAN OLEH:

AZNIFA MAHYAM ZAHARUDIN RIZAL MOHAMAED NOOR RASDI DERAMAN

JUNE 2005

Surat Kami : 600-UiTMCPP (UPP.5/2/1)

Tarikh : 25 Dis 2003

Aznifa Mahyam Zaharudin (Ketua) Rizal Mohamed Noor Rasdi Deraman Pensyarah Universiti Teknologi MARA Cawangan Pulau Pinang



CAWANGAN PULAU PINANG

Unit Penyelidikan, Pembangunan dan Pengkomersilan

Permatang Pauh 13500 Permatang Pauh Pulau Pinang

Tel : 04-3823441 (URDC), 3822888 (AM) Faks : 04-3823602 email: peridahb@ppinang.uitm.edu.my

Tuan/Puan/Prof.Madya,

KELULUSAN PERMOHONAN UNTUK MENJALANKAN PROJEK PENYELIDIKAN

Perkara di atas adalah dirujuk.

Sukacita dimaklumkan permohonan tuan/puan/Prof.Madya untuk menjalankan penyelidikan telah diluluskan dalam Mesyuarat Jawatankuasa Teknikal Unit Penyelidikan, Pembangunan dan Pengkomersilan Universiti Teknologi MARA (UiTM) Cawangan Pulau Pinang yang telah diadakan pada 16 Dis 2003.

Butiran permohonan yang diluluskan adalah seperti berikut:

Nama Ketua Projek	:	Aznifa Mahyam Zaharudin
Tajuk Penyelidikan	:	Synthesis Of Carbides Compounds From Elemental Powder By Mechanical Milling
Keputusan	:	Diluluskan dengan peruntukkan RM10,000
Tempoh	:	Jan 2004 – Jan 2005

Berikut disertakan dokumen-dokumen untuk panduan dan tindakan pihak tuan/puan/Prof.Madya selanjutnya.

- 1. Perjanjian bagi menjalankan Projek Penyelidikan, sila isi dan kembalikan kepada pihak kami untuk ditandatangani oleh pihak seterusnya.
- 2. Borang laporan kemajuan yang perlu dikemukakan kepada pihak kami setiap enam (6) bulan.

ABSTRACT

Silicon carbides (SiC), was prepared by mechanical milling using high planetary ball mill. Four parameters studied are the effect of milling time, effect of annealing temperature, effect of environment and effect of two stages of milling. Powders of silicon and graphite mixture were milled for 15 hours, 25 hours and 60 hours with ball to powder weight ratio (BPR) of 30:1 at 130 rpm. The as-milled samples were then annealed at 800°C, 1000°C and 1200°C. The phase formation and structural and morphology changes during mechanical milling were studied by X-ray diffraction (XRD), differential thermal analysis (DTA) and scanning electron microscopy (SEM). No formations of silicon carbide were found in powder milled for up to 60 hours. It was found that silicon carbide could be formed in powders milled for 15 hours, 25 hours or 60 hours after annealing in argon atmosphere at 1200°C. XRD peaks of silicon carbide in the annealed powder shows very low intensities and broad, indicating silicon carbide phase was in nano-phase. Using Scherrer equation, the average crystallite size calculated was 22 nm. In an effort to synthesized silicon carbide at lower milling time, two stages milling was performed. Results indicate that two stage milling improved production of silicon carbide as more silicon carbide formed. Extended second milling produced silicon carbide crystallite size of 9 nm.

CONTENT

Acknowledgement	i
Abstract	îį
List of Tables	vii
List of Figures	viii

CHAPTER 1 INTRODUCTION

1.1 Background	1
1.2 Significant of Project	3
1.3 Objectives	4

CHAPTER 2 LITERATURE REVIEW

.

2.1	Introduction to Mechanical Milling		
	2.1.1	Historical perspective	6
	2.1.2	Mechanical Milling	7
	2.1.3	Type of milling	8
2.2 The process of mechanical milling			
	2.2.1	Process parameters	12
	2.2.2	Atmosphere Control	12
	2.2.3	Selection of BPR	13
	2.2.4	Selection of PCA	13

í

CHAPTER 1

INTRODUCTION

1.1 Background

Silicon Carbide is the only chemical compound of carbon and silicon. It was originally produced by a high temperature electro-chemical reaction of sand and carbon. Silicon carbide is an excellent abrasive and has been produced and made into grinding wheels and other abrasive products for over one hundred years. Today the material has been developed into a high quality technical grade ceramic with very good mechanical properties. It is used in abrasives, refractories, ceramics, and numerous high-performance applications. The material can also be made an electrical conductor and has applications in resistance heating, flame igniters and electronic components. Structural and wear applications are constantly developing.

Silicon carbide, as a technical ceramic, is produced basically in two main ways. Reaction bonded SiC is made by infiltrating compacts made of mixtures of SiC and Carbon with liquid Silicon. The silicon reacts with the carbon forming SiC. The reaction product bonds the SiC particles. Sintered SiC is produced from pure SiC powder with non-oxide sintering aids. Conventional ceramic forming processes are used and the material is sintered in an inert atmosphere at temperature up to 2000°C or higher.

A number of techniques have been used to prepare silicon carbide. Preparation of silicon carbide is relatively easy, but to make them oxygen-free is difficult and subtle processes which many investigators have failed to appreciate. It has been the