PHYSICAL CHEMISTRY 2006

Proceedings

of the 8th International Conference on Fundamental and Applied Aspects of Physical Chemistry

September 26-29,
Belgrade, Serbia

ISBN	86-82139-26-X
Title:	Physical Chemistry 2006. (Proceedings)
Editors	Prof. dr A. Antić-Jovanović
Published by:	The Society of Physical Chemists of Serbia, Studentski trg 12-16, P.O.Box 137, 11001 Belgrade, Serbia
Publisher:	Society of Physical Chemists of Serbia
For publisher:	Prof. dr S. Anić, president of the Society of Physical Chemists of Serbia
Printed by:	"Jovan" Printing and Published Comp; 250 Copies; Number of Pages: x + 442; Format B5; Printing finished in September 2006.
Text and Layout:	Aleksandar Nikolić

250 – copy printing

SYNTHESIS OF FIBROUS SiC FROM NATURAL PRECURSOR

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Abstract

Fibrous magnesio-silicate (mountain leather asbestos) of Serbian origin was used as Si precursor for the synthesis of SiC by carbothermal-reduction process [1, 2]. As a reducing agent, sugar (saccharose) was used. Formation of SiC was confirmed by XRD analysis and optical microscopy images. Results showed that obtained SiC possess fibrous morphology. Due to the experimental procedure MgF_2 is also formed, which is known as an excellent additive for sintering of non-oxide ceramics.

Experimental

Mountain leather asbestos (MLA) sheets were cuted in 20mm x 10mm pieces, soaked into the sugar aqueous solution, dried and then heat treated at 1673K for 1 hour in continuous Ar flow (MLAC). Samples are then oxidized in air at 873K (MLAO), leached in diluted HF (1 part of HF + 3 parts of H_2O) for 5, 10 and 20 minutes, washed with H_2O and dried (MLAO5, MLAO10 and MLAO20).

Structural analysis was carried out by a Siemens D-500 powder diffractometer. CuKα radiation was used in conjuction with a CuKβ nickel filter.

Morphology was examined by the use of optical microscop »Zeiss«, AXIOVERT 25 (50-1000x magnif.), Germany.

Results and Discussion

XRD patterns of starting MLA and samples treated according to various steps of experimental procedure are presented in Fig. 1. In XRD pattern of MLAC sample (Fig. 1b), the reflections of original MLA disappeared and formation of SiC (JCPDS number 29-1129) is observed as well as the peaks of forsterite (Mg_2SiO_4, JCPDS number 34-0189). Broad reflection about $2\Theta = 26$ degree that belong to amorphous carbon, disappeares after exposure to air at 873K (not showed). During leaching the samples in diluted HF, intensities of forsterite reflections decrease with prolonging leaching time (Fig. 1c-1e) whilst the forsterite reflections for MLAO20 sample are completly disappeared. FWHM of strongest SiC reflection ($2\Theta \approx 35.5^0$) also increases with increasing HF leaching time, thus indicating that SiC grains are reduced in size, probably due to high stress exposition of SiC in presence of HF. Together with disappereance of forsterite reflections, new broad reflections arised in XRD paterns of HF leached samples ($2\Theta \approx 27^0$, 40^0 and 43^0), indicating formation of new amorphous phase. To clarify the nature of this phase, another MLAO sample is lefted overnight in HF solution (MLAO∞ - Fig. 1f). After this tretment mentioned reflections become more pronounced and they can be attributed to MgF_2 (JCPDS file number 06-0290), which is obviously formed during reaction of forsterite with HF. The strongest SiC

reflection is also broaden and further decreasing in SiC crystallite size. This behaviour confirmes that the SiC is unstable in presence of HF.

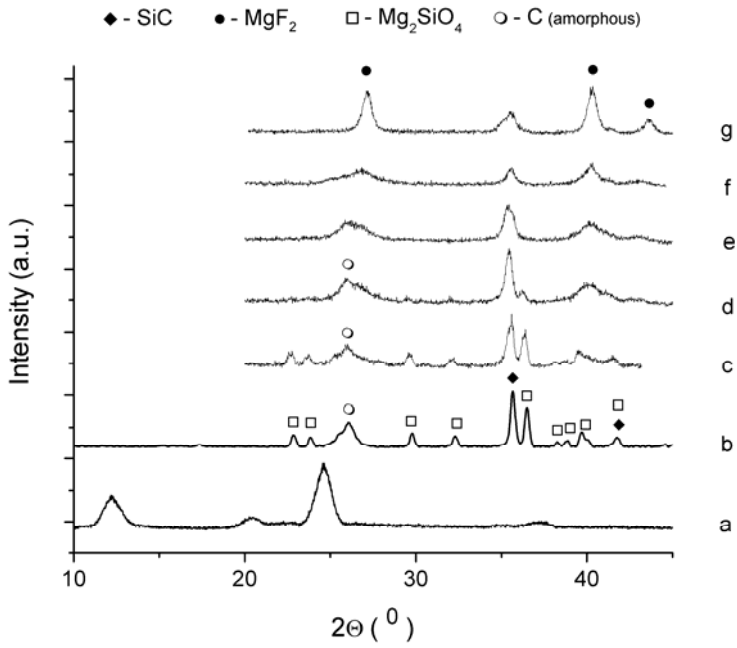

Fig. 1. XRD patterns: a) MLA; b) MLAC; c) MLAO5; d) MLAO10; e) MLAO20; f) MLAO∞; g) MLAO∞ exposed to air at 873K;

Relatively sharp peak at ≈ 26 degree in MLAO5, MLAO10 MLAO20 and MLAO∞ XRD patterns, belong to carbon which is formed during SiC decomposition. This is proved by exposing MLAO∞ again to air at 873K: peak at 26 degree disappeared (Fig. 1g), lefting only a peak centered at 27 degree which belongs to MgF_2. It should be also noticed that all MgF_2 reflections became sharper after this 873K treatement i.e. MgF_2 phase is now rather crystalline than amorphous. Presence of MgF_2 is quite appropriate, because it is known that fluorides of earth-alkali metals are very good additives for sintering of non-oxide ceramics [3].

Optical micrographs of samples are presented in Fig.2. MLAC sample retain its original shape after 1673K treatement (≈ 20mm x 10mm pieces). However, when MLAC samples are dipped into the HF solution, they divide into longish plate-like particles, ranging in size from several milimeters to less than one milimeter. Image of one such particle is given in Fig. 2a. Images of single SiC fibres falled off from big particles are presented in Fig. 2b-2d.

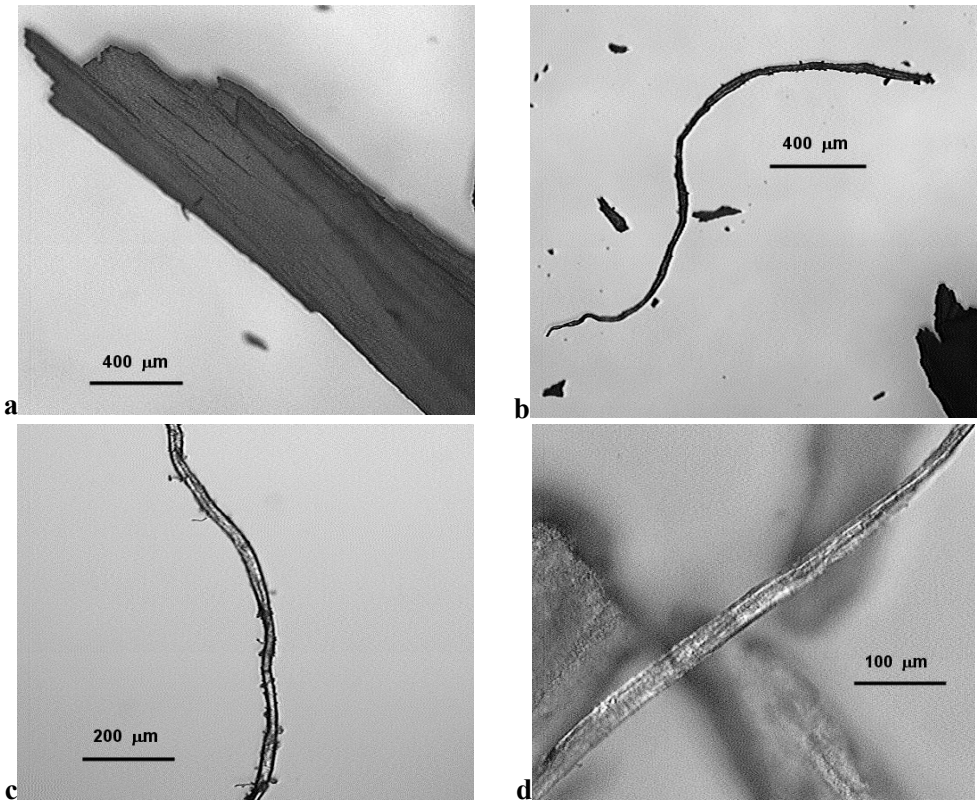

Fig. 2. Optical micrographs of samples, showing SiC fibrous morphology

Acknowledgment

This project was financially supported by the Ministry of Science and Environmental Protection of Serbia (project number: 142016).

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