

Preparation and Characterization of Iron incorporated Silicon Carbide Foam prepared via Polymer Precursor Route

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Abstract

In the present study, Iron incorporated Silicon Carbide foam (Fe-SiC) were prepared via polymer precursor route using optimized template method. X-ray diffraction confirms the formation of Fe-SiC with traces of Fe₃Si which may have induced the ferromagnetism in Fe-SiC. Microstructural study of Fe-SiC foam shows large variation in the range of pore size distribution. It is evident from SEM images that foam have open cell structures. Magnetic properties of Fe-SiC foam has also been investigated and compared. Addition of Fe induces the ferromagnetism in SiC which is very stable even at room temperature. Low coercivity in Fe-SiC and stability of ferromagnetism makes Fe-SiC suitable as soft Diluted magnetic Semiconductors (DMS) for device fabrication.

Keywords: *Ferromagnetism, Diluted Magnetic Semiconductors, Silicon Carbide Foam, Precursors*

1. Introduction

Recently, there is a surge in the research activities involving spin based microelectronics. Ferromagnetic semiconductors have attracted much interest due to potential new device applications in the spin based information processing technologies

[1]. Diluted Magnetic Semiconductors (DMSs) have been promising in view of possibility of integrating charge and spin degree of freedom in a single material [2], [3]. It is desirable in such applications that material possess wide bandgap so that heat and other external factors do not influence the performance of microelectronics.

Among such materials, Silicon Carbide (SiC) has been focus of research in the field of microelectronics. SiC has high breakdown electric field, high thermal conductivity and wide bandgap of 3.0eV for 6H-SiC [4], therefore SiC devices can operate at higher temperature and higher radiation levels than Si and GaAs-based devices [1]. Several transition metals (TMs) such as Cr, Mn, Ni, Fe etc have been incorporated into SiC to see the magnetic behavior of these materials. Magnetic behavior in Fe-SiC foam is discussed at length.

2. EXPERIMENTAL

2.1 Chemicals used

Commercially available LY-556 Epoxy resin was obtained from Huntsman (USA), Butanediol and 2 ethyl-2(hydroxyl methyl)-1,3-propanediol (TMP) were obtained from Merck India Ltd. and Lancaster (England) respectively. Hexamethyldiisocyanate (HMDI) 98% and

Methylenedianiline (MDA) 97% was procured from Acros chemicals were used as such. Iron (III) acetylacetonate [Fe(acac₃)] reagent grade 99% was purchased from Aldrich. The solvent used, xylene, was purified by distillation over sodium.

Polycarbosilane was prepared by the thermal backbone rearrangement of polydimethylsilane in strict inert atmosphere for 125 hours in the temperature range 250-450 °C as reported earlier [5], [6], [7], [8], [9], [10], [11], [12], [13], [14], [15].

2.2 Sample preparation

50 g of Polycarbosilane and epoxy resin (2.5 g) were added to sodium dried xylene (500mL) in a three necked round bottom flask fitted with a condenser and magnetic stirrer under strict inert atmosphere and refluxed for 5 hours with continuous stirring. To this solution 2.5 g Fe(acac)₃ was added and heated till reflux. Again, to this mixture 2.5 g of (TMP) mixed with butanediol (22.5 g) was added. The progress of the reaction was monitored using FTIR. Xylene was distilled off by vacuum distillation and the mixture was dried by heating it further for 3 hrs to ~300 °C. The solid obtained was crushed into a fine powder and then heated to ~140 °C in argon atmosphere. Above formed mixture was mixed with 0.675 gm MDA and 25 gm of HMDI, which on pyrolysis in the furnace upto ~1100 °C under argon atmosphere at the heating rate of 2 °C/min and then soaking at 1100 °C for 2 hrs. Afterwards, the furnace was allowed to cool until it attains room temperature under argon atmosphere. On cooling, the sample

thus obtained, was characterized using FT-IR, XRD, SEM and Physical property measurement.

2.3 Sample characterization

FTIR spectrophotometer of Perkin Elmer spectrum RX-1 model USA make was used for characterization of the material using KBr pallets in the range 4,000 – 400 cm⁻¹ and wave numbers reported in cm⁻¹. The X-ray diffraction experiments were performed on thin film samples by means of Rigaku X-ray diffractometer, operating at 40kV/30 mA and having Cu-K_α radiation selected by a graphite monochromator. The surface morphology, microstructure and scanning electron microscope (SEM) studies were carried out using Carl Zeiss Evo 50 SEM. Magnetic Properties were analysed using Physical Property measurement system (PPMS) 14T, USA.

3. Results and discussion

The polycarbosilane synthesized showed characteristic IR absorption peaks at ~1404, ~2462, ~2893, ~2951 cm⁻¹ (ν C-H), ~650, ~800 and ~1246 cm⁻¹ (ν Si-CH₃), ~1023 and ~1352 cm⁻¹ (ν Si-CH₂-Si-) and ~2100 cm⁻¹ (ν Si-H) [22,24]. With the progress of the reaction between polycarbosilane with epoxy, Si-H absorption peak at ~2100 cm⁻¹ starts diminishing, showing other peaks at ~1024 cm⁻¹ (ν Si-CH₂), ~1255 cm⁻¹ (ν SiCH₃) and ~3488 cm⁻¹ (ν OH). This on further reaction with iron acetylacetonate, polyols, di-isocyanates and diamine shows IR peaks at ~3324 cm⁻¹ (ν NH str), ~2941 cm⁻¹ (ν CH str), ~2107 cm⁻¹ (ν SiH), ~1691 cm⁻¹ (ν Si-CH), ~1540 cm⁻¹ (ν NH def),

~1261 cm^{-1} (ν SiCH₃), ~1044 cm^{-1} (ν Si-CH₂-Si). Moreover, as the reaction further proceeds, Si-H absorption peak at ~2100 cm^{-1} completely diminishes along with the disappearance of absorption peak at ~1580 cm^{-1} due to acetylacetonate group which may be attributed to the direct bonding of iron to Silicon linkage (fig. 1a, b, c).

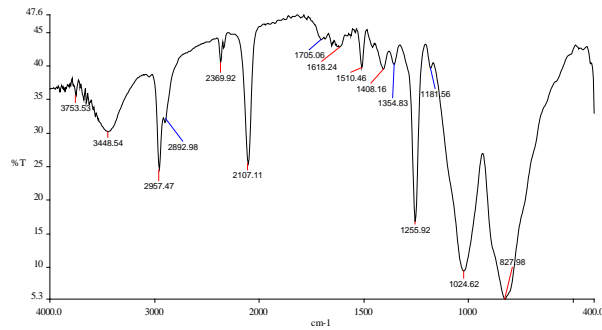


Fig. 1a. FTIR spectra of epoxy poycarbosilane

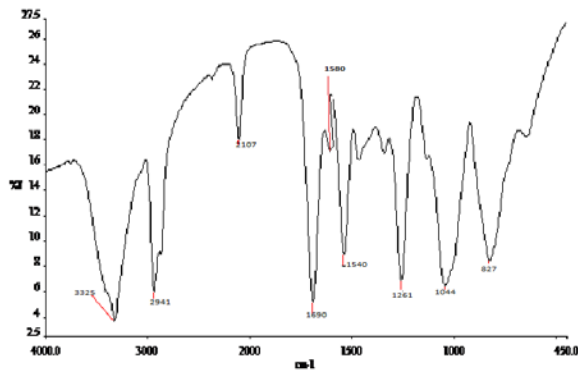


Fig. 1b. FTIR spectra of preceramic foam

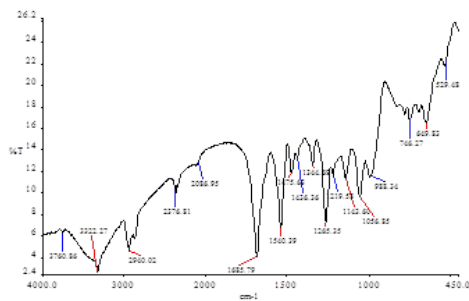


Fig. 1c. FTIR spectra of preceramic foam

XRD spectrum (fig. 2) shows the presence of 20 peaks at 27° (111) Fe₃Si, 36° (110) SiC, 45° (220) Fe₃Si, 60° (220) SiC and 72° (311) SiC which confirms the presence of formation of silicon carbide foam containing iron.

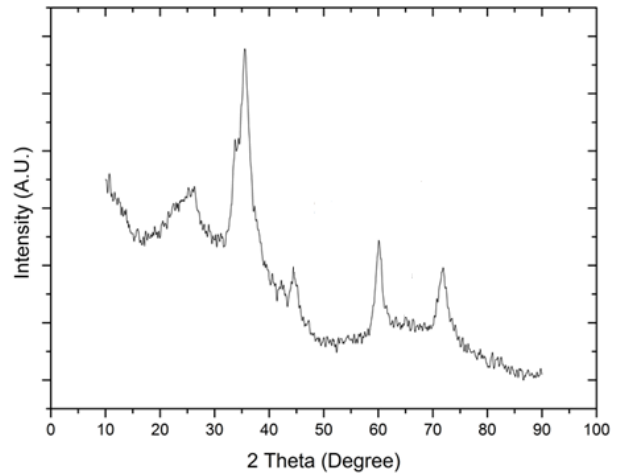


Fig. 2: XRD pattern of Fe-SiC foam

Magnetic Property

Addition of Fe (2 wt%) into SiC prompts change of diamagnetic behavior to ferromagnetic behavior even at room temperature. Low coercivity in case Fe-SiC shows that Fe-SiC can be utilized as soft Diluted Magnetic Semiconductor (DMS). Nearly addition of 2 wt% Fe in SiC enhances the magnetization by several orders. Magnetization vs temperature curve for Fe-SiC is shown in the Figure 3. Figure 4 confirms the existence of ferromagnetism in Fe-SiC.

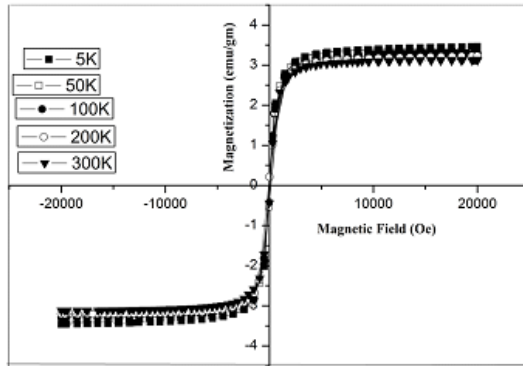


Fig.3: M-H curve of Fe-SiC foam at different temperature

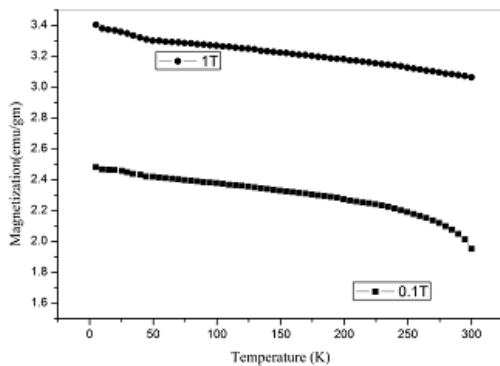


Fig.4: M-T curve of Fe-SiC foam at 0.1T and 1T.

The magnetic property of Fe-SiC at different temperature is shown in the following table:

Temperature (K)	M_s (emu/gm)
5	3.47
50	3.34
100	3.29
200	3.23
300	3.11

Fig.5 shows the SEM image of the Fe-SiC sample obtained after pyrolysis, which shows the presence of open cell structure of the foam. Sample shows a

wide variation of pore size ranging from ~10-250 μ m.

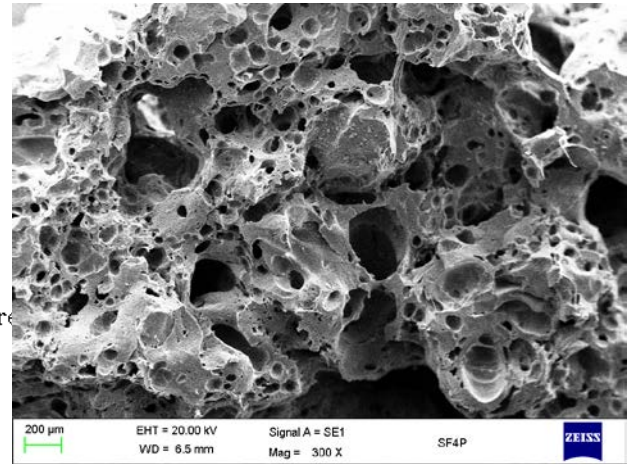


Fig.5: SEM image of Fe-SiC foam

4. Conclusions

Silicon Carbide has been hailed as diluted magnetic semiconductor (DMS) with large band gap ~3.0eV for 6H-SiC. XRD confirmed the the formation of Fe-SiC with some traces of Fe₃Si. Microstructure of Fe-SiC reveal the foam like structure with large variation in pore size. Addition of 2wt% Fe into SiC matrix induces ferromagnetism. XRD exhibits traces of Fe₃Si which may be source of ferromagnetism in Fe-SiC .

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