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Materials Letters 61 (2007) 956–959
materials letters
www.elsevier.com/locate/matlet

In-situ synthesis of oxygen free SiC fibers and ropes by electron beam melting

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Received 29 March 2006; accepted 11 June 2006
Available online 31 July 2006

Abstract

The SiC fibers, ropes and columnar-like fibers have been synthesized on the substrate of Co-based DSX40M alloy by electron beam (EB) melting. The fibers have been prepared from the SiC particles under high vacuum. The microstructure of the SiC fibers, ropes and columnar-like fibers has been investigated by scanning electron microscope (SEM) having energy dispersive spectroscopy (EDS) as an attachment. The coating of Co, Cr and Ni is observed on the surface of the SiC fibers, ropes and columnar-like fibers. A slight charging is observed in the case of the ropes while the columnar-like fibers and small size woven fibers did not show this effect suggesting the semiconducting nature of the ropes.

Keywords: SiC fibers; Electron beam; Microtubular; Columnar-like structure; Scanning electron microscopy; Co-based superalloy

1. Introduction

SiC fibers have attractive applications in the field of materials science due to their high stiffness, high strength, high creep resistance, chemical stability and high temperature resistance in inert atmosphere, high corrosion resistance and low thermal expansion coefficient [1]. The fibers are used for the formation of composites to enhance the mechanical and thermal prosperities of the materials. The most frequently used SiC fibers (Nicalon) and thread-like crystal (Whiskers) are commonly synthesized from fibrous organo silicon precursors [2–7]. The fibers have been prepared by spinning of molten polycarbosilane (PCS), the curing of the green filaments in an oxidizing atmosphere and pyrolysis in an inert atmosphere [8,9]. Therefore the oxygen content remains in the fibers, which affects their mechanical, chemical and thermal properties. The EB irradiation process has enabled the researchers [10,11] to reduce the oxygen content of SiC fibers prepared from the blend of PCS and polyvinylsilane (PVS). The SiC fibers irradiated with EB have low oxygen content, excellent thermal stability and greater creep resistance at high temperature compared with fibers prepared without curing with EB irradiation [12,13].

In ceramic fiber/ceramic matrix composites, ceramic fibers are required to be as fine as possible to attain high strength and stiffness, since fine ceramic fibers have high tensile strength and fracture toughness as compared to the coarse fibers [14]. Idesaki et al. [15] succeeded in synthesizing the fibers with the diameter of about 8.5 μm by improving the melt spinning technique and curing the SiC fibers with EB irradiation. It is well known that the SiC fibers prepared from PVC and PVS by spin melting technique contain some oxygen content as well. Oxygen content has been reduced by EB irradiation and annealing the fibers at high temperature [11]. The average diameter of the SiC fibers was achieved to be 9 and 16 μm for the SiC prepared from PCS and PCS–20% PVS respectively

Table 1
Nominal composition of the DSX40M alloy

Element	Cr	Ni	W	Al	Ta	Mo	Ti	C	Zr	B	Co
Wt.%	25.0	11.0	7.5	0.8	0.25	0.2	0.15	0.45	0.15	0.05	Bal.

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doi:10.1016/j.matlet.2006.06.023

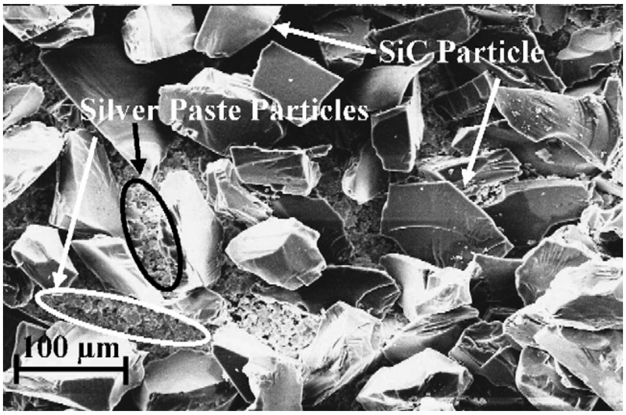

Fig. 1. SEM micrograph (SEI) shows the SiC particles pasted on the surface of Al metal by silver paste.

and was successfully reduced to 6.9 and 11.3 μm using EB irradiation [16]. EB is also being used for the cross-linking of the fibers [1].

The role of the transition elements as catalysts in the synthesis of high purity, single crystal β-SiC fibers has been highlighted using the vapor–liquid–solid (VLS) process [17–19]. DSX40M is a Co-based superalloy containing transition elements and is used as a nozzle guide vane material in gas turbines [20]. This alloy is used as a catalyst for the preparation of SiC fibers by EB melting in the present study. The main aim of the present study is to synthesize oxygen free and small diameter SiC fibers and ropes using the SiC particles on the DSX40M alloy substrate.

2. Experimental procedure

The nominal composition of the substrate (DSX40M) is given in Table 1. Samples of the size $10\times20\times3$ mm^3 were cut from the sheet material. The specimen was polished mechanically down to 0.25 μm on a lapping machine using diamond paste. Grids of the size 2×3 mm^2 having a depth of about 1 mm were made on the surface of the substrate by spark erosion machine. SiC particles were mixed with hexane (C_6H_{14}) to form a slurry and the slurry was put into the grids. An EB, with its direction normal to the surface, was used to melt the surface of the substrate. The parameters used for surface melting were, voltage = 20 kV, beam current = 20 mA, beam travel speed = 400 mm/min, distance of the beam from the sample = 160 mm and the beam was used in vibrational mode. The experiment was carried out under high vacuum ~ 10^{-5} mbar. The sample was kept in contact with a Cu mold of size 50×60 mm^2 during melting to achieve a higher cooling rate. Microstructural analysis of the SiC fibers produced in the grooves as a result of the interaction of the molten metal with SiC, was carried out using SEM having EDS with ultra thin window detector used for the detection of light element up to B as an attachment.

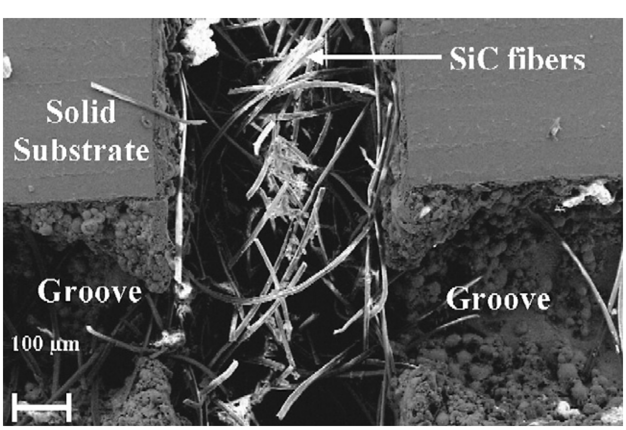

Fig. 2. SEM micrograph (SEI) shows the formation of continuous SiC fibers.

Fig. 3. SEM micrograph (SEI) shows the rope-like structure consisting of several fibers.

3. Results and discussion

Fig. 1 shows the size and shape of the SiC particles used in the experiment for the synthesis of SiC fibers. The size of the SiC particles ranges 70–100 μm with irregular shape. Fig. 2 shows the formation of continuous SiC fibers in the grooves of the substrate at low magnification. Fig. 3 shows the long and continuous fibers at high magnification, which reveals that the long fibers are actually ropes consisting of several fibers. Fig. 4 shows a rope consisting of several fibers. The average diameter of the fiber is about 2 μm and the interface between the fibers is very thin and smooth. The fibers are linked in the form of the ropes to achieve good strength. The

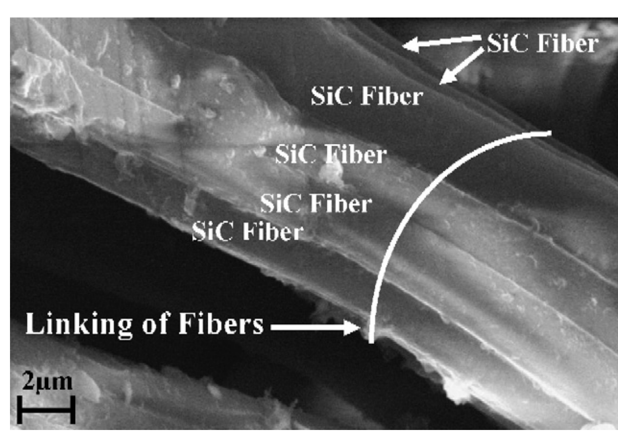

Fig. 4. SEM micrograph (SEI) shows the linking of fibers to form a rope.