EFFECT OF NITRIDATION ON PROPERTIES OF SiC MONOFILAMENT

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SUMMARY: Pre-nitradation of the TiB_x coating surface of the Sigma SM1240 SiC monofilament can form more stable compounds at the surface and obstruct the release of boron atoms into the Ti-based alloy matrix. The effect of nitridation on the tensile strength of the filament was investigated in this work. Nitridation could degrade the tensile strength of the SiC filament if the treating temperature and time are not optimized. The chemical reaction between the W core and SiC and the modification of filament microstructure during the nitridation are responsible for the degradation in strength . The strength can be maintained by further optimization of the treating temperature and time. Therefore, stabilizing the surface of TiB_x coating and hence the interface of the SiC_f /Ti composite by the nitridation of the SiC filament is a feasible technique for practical applications.

KEYWORDS: SiC monofilament, Surface Coating, Nitridation, Tensile Strength.

INTRODUCTION

CVD Silicon Carbide (SiC) monofilaments are characterized by high specific strength and stiffness as well as excellent high temperature stability and oxidation resistance(1). These are considered as one of the most attractive reinforcements for Ti alloys and Ti-Al intermetallic compounds. It is generally accepted that Ti-SiC system is a non-equilibrium one. When the composites are heated to medium or high temperatures, harmful interface reaction takes place, which degrades the properties of the materials. In order to prevent the occurrence of chemical interactions between SiC and matrixes, protective coatings are applied to the surface of the SiC filament, such as the TiB_x/C double coating on one of the Sigma SiC monofilament, manufactured by the DERA Sigma, UK. Unfortunately, the formation of TiB needles were found at the interface between Ti-based alloys and the TiB_x coating, which may enhance stress concentration and cracking at the interface(2). A method of improving the interface chemical stability by pre-nitradation of the surface of the TiB_xcoating was established, which can form more stable compounds at the surface and obstruct the release of boron atoms into the Ti-based alloy matrix(3). Present work was concentrated on the effect of nitridation on the tensile strength of the Sigma SM1240 SiC monofilament in order to identify optimum treating condition and to determine if the method can be used in practice.

EXPERIMENT

The Sigma 1240 monofilament was supplied by DERA Sigma Ltd. A mixture of NH_3 and Ar gases was used in the study. The experiments were preformed on mercury sealed vertical Pyrex-glass tube reactor with 16 mm in diameter and 300 mm in length. The filament samples were passed through the cylindrical chamber of the reactor, a mixture of NH_3 and Ar gases was introduced from an inlet at the bottom of the reactor. The samples were heated to a desirable temperature by a DC supply applied across the liquid mercury contacts at the top and bottom respectively. In order to keep the temperatures constant, the DC current was controlled at 295, 300, 305mA for 30, 60, 180 sec respectively with the range of temperatures around 1100-1300 approximately. Then both of treated and as-received filaments were tested by an UTM-II-20 tensile testing machine and examined by SEM and X-ray diffraction.

RESULTS and DISCUSSION

The Sigma SM1240 SiC monofilament has a nominal diameter of 100 m with a W wire core and a TiB_x/C double coating about 3m on the filament surface, as shown in Fig.1(a).
By SEM- EDS analysis, it was noted that the nitrogen peak was just located at the surface of TiB_x coating of the treaded filament samples (see Fig.1 (b)).

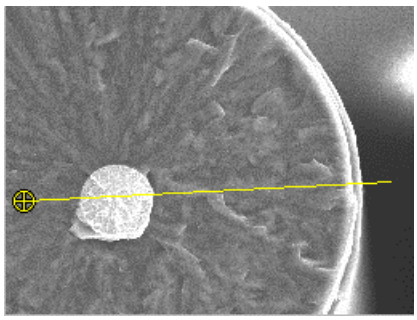


Fig.1 (a): Fractograph of SiC monofilament

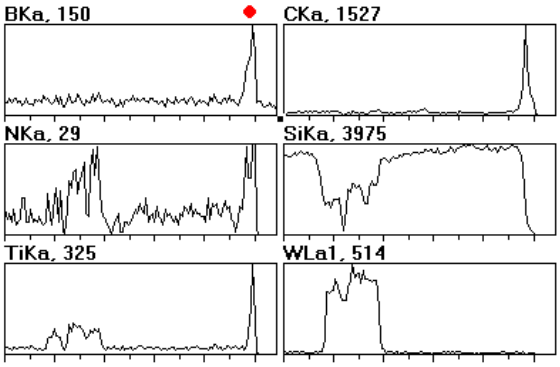
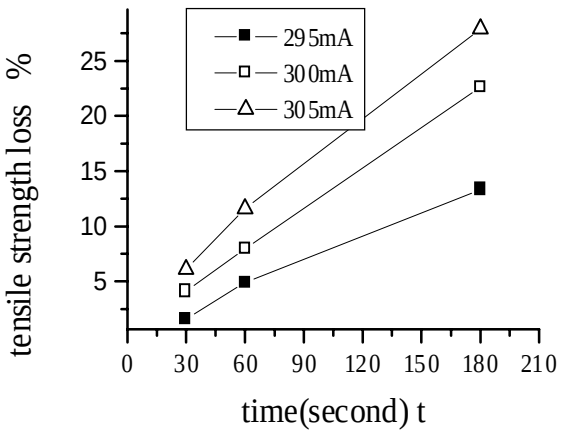


Fig.1 (b):X-ray line scanning by EDS

Ti_3N_2B compound peaks were also found from the X-ray diffraction patterns. It is evident that all the fibers were nitrided after the nitridation. The possible reaction mechanism are as following:

$$3TiB+N_2=Ti_3N_2B+BN$$
$$6TiB_2+7N_2=2Ti_3N_2B+10BN$$
$$2Ti_3B_4+5N_2=2Ti_3N_2B+6BN$$

The tensile strength of the as received filament was measured as 3343MPa. After the nitridation the tensile strength of the filament was degraded by 3% to 27% as the treating



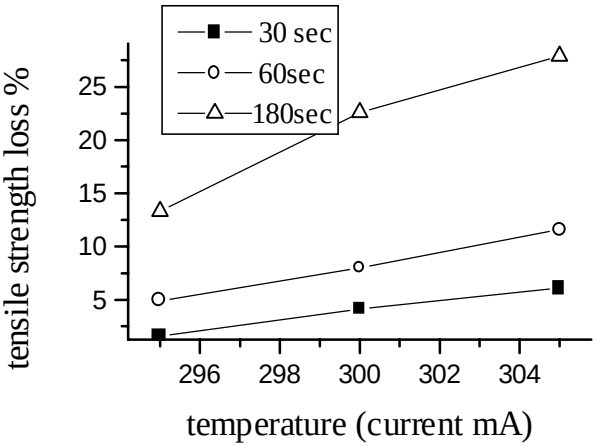


Fig.2 (a): Effect of treating time on strength of the SiC filament

Fig.2 (b): Effect of treating temperature on strength of the SiC filament

temperature (DC current) and time increased (see Fig.2 (a) and (b)). These were consistent with the results of previous studies which annealed SiC(Ø) filaments in vacuum[4] and on heat treated SiC filament in air. The later was produced by Institute of Metal Research (IMR), China[5].

X-ray diffraction examination was also be carried out to identify the cause of the degration of the tensile strength of the treated filament. Apart from peaks attributable to the aforementioned nitrides, additional X-ray peaks were identified, which can be attributed to some high temperature phases, such as W_2C, ↑-SiC.

Table 1: Grain size change of the filaments due to nitradation

Crystal face	Treating condition			
	As received	30sec.,295mA	60sec.,300mA	180sec.,305mA
(311)	7.6nm		7.6nm	7.8nm
(222)	12.9nm	13.2nm	16.2nm	

Furthermore, by measuring the grain size of the treated filament, as shown in Table 1, a trend of recrystallization can be seen quite clearly as increasing the treating temperature and time of the samples. All these results strongly suggest that the reduction in strength may be attributed to (❑) chemical reactions between the Tungsten core and SiC which result in brittle phases W_2C, and (❑) a modification of fiber microstructure (i.e. a grain growth of SiC and occurrence of a high temperature phase↑-SiC).

However, from the Fig.2 it is noted that the strength of the SiC filament only decreased by about 3% if the treating temperature and time were the lowest (295mA and 30 second respectively). These results indicate that the strength of the SiC filament could be maintained with an appropriate combination of treating temperature and time.

CONCLUSIONS

In conclusion, the nitridation of the SiC monofilament (Sigma SM1240) could degrade the tensile strength of the filament if the treating temperature and time are not optimized. The chemical reaction between the W core and SiC and the modification of filament microstructure during the nitridation are responsible for the degradation in strength . The strength can be maintained by further optimization of the treating temperature and time. Therefore, stabilizing the surface of the TiB_x coating and hence the interface of the SiC_f/Ti composite by the nitridation of the SiC filament is a feasible technique for practical applications.

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