

Crucial roles of egg white liquid in preparing excellent SiC anti-oxidation coatings on carbon felt fibers

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SiC anti-oxidation coatings on polyacrylonitrile (PAN) based carbon felt fibers were prepared through sintering a pre-immersed felt in a vacuum. The crucial act here was to add egg white into the immersion liquid. Scanning electron microscope (SEM), energy dispersive X-ray (EDX) and differential scanning calorimetry-thermogravimetric (DSC-TGA) analyses for the coated felt fibers as well as for another a uncoated sample were made to basically study the coating effects. The egg white liquid acts both as a favorable natural agglomerant and an active carbon source during the sintering; this leads to an excellent coating.

Key words: Egg white, SiC, Coating.

Introduction

Carbon materials or their correlative products (carbon fibers here) are easily oxidized at above 400 °C in air [1], and this seriously cripples their existence so that the good properties owned by them can not be exploited. It has been reported that a SiC coating on carbon fibers could protect them from being oxidized to a certain degree, improve their poor wettabilities and compatibilities with metals and lower the possibilities of chemical reactions with them at high temperature [2].

Many advanced technologies to obtain various coatings, such as chemical vapor deposition (CVD), physical vapor deposition (PVD), high velocity oxy-fuel flame (HVOF) spraying, etc., are all popular, but they need special equipment to fulfill the coating process, involving high cost. The skill used here to get ultrathin SiC coatings is simple at low cost, and it has the possibility to spread industrially. The impressive thing about the SiC coating is its nearly perfect appearance and evident functioning of improving the anti-oxidation capability for the carbon fiber at high temperatures in air.

Experimental Procedure

The PAN based carbon felt adopted was made in the Lanchow (in China) carbolic products factory; the chemicals adopted were from Shanghai chemical products factory; the vacuum furnace adopted was a home-made one. Detailed procedures of the preparation were as follows: (1) 100 ml precursory liquid of silica sol (liquid 'S' hereafter) was prepared by reference to specific research

[3] (a 20 ml mixture of liquids of deionized water, a few of absolute alcohol and less mineral acid HCl were mixed into 80 ml tetraethyl orthosilicate (TEOS) with stirring. The ratio of the water, the alcohol and the TEOS was about 8 : 1 : 2; the pH value of this liquid was 4.) Another liquid mixture (liquid 'E') was made by mixing 50 ml liquid 'S' with 50 ml egg white liquid which was diluted with deionized water. (2) Two pieces of carbon felt were fully immersed in the liquid 'S' and liquid 'E' in a vacuum (respectively the felt 'FS' and felt 'FE' hereafter), then they were dried. (3) The felt 'FS' and felt 'FE' were sintered at 1,200 °C for 30 minutes with the vacuum pump working continuously all through the process (respectively the felt 'SS' and felt 'SE' hereafter).

SEM and EDX analyses for the coated fibers were made by a stereo-scan equipment QUANTA200; DSC-TGA analyses were made by a simultaneous DSC/TGA analyzer SDT-Q600, and its experimental conditions were as follows: the working atmosphere was 20% oxygen and 80% nitrogen by volume, the gas feed rate was 90 mlminute⁻¹, and the heating rate was 20 Kminute⁻¹.

Results and Discussion

Fig. 1a shows that the fibers are not fully enwrapped by the coating. Some parts of the coating are coming off the fibers, and some agglomerates are seen as knobby attachments. One comparatively well developed part of the coating and a magnified region are shown in Fig. 1b and Fig. 1c. This part is comparatively regular; nevertheless, it still shows loose and uneven distribution of the substance as shown especially in Fig. 1c. Fig. 1d shows that the fibers are fully and neatly enwrapped by the coating. The coating is very thin so that it can not even enshroud the concave longitudinal traces always found on the fiber surfaces. Fig. 1e shows the coating is tidy and compact;

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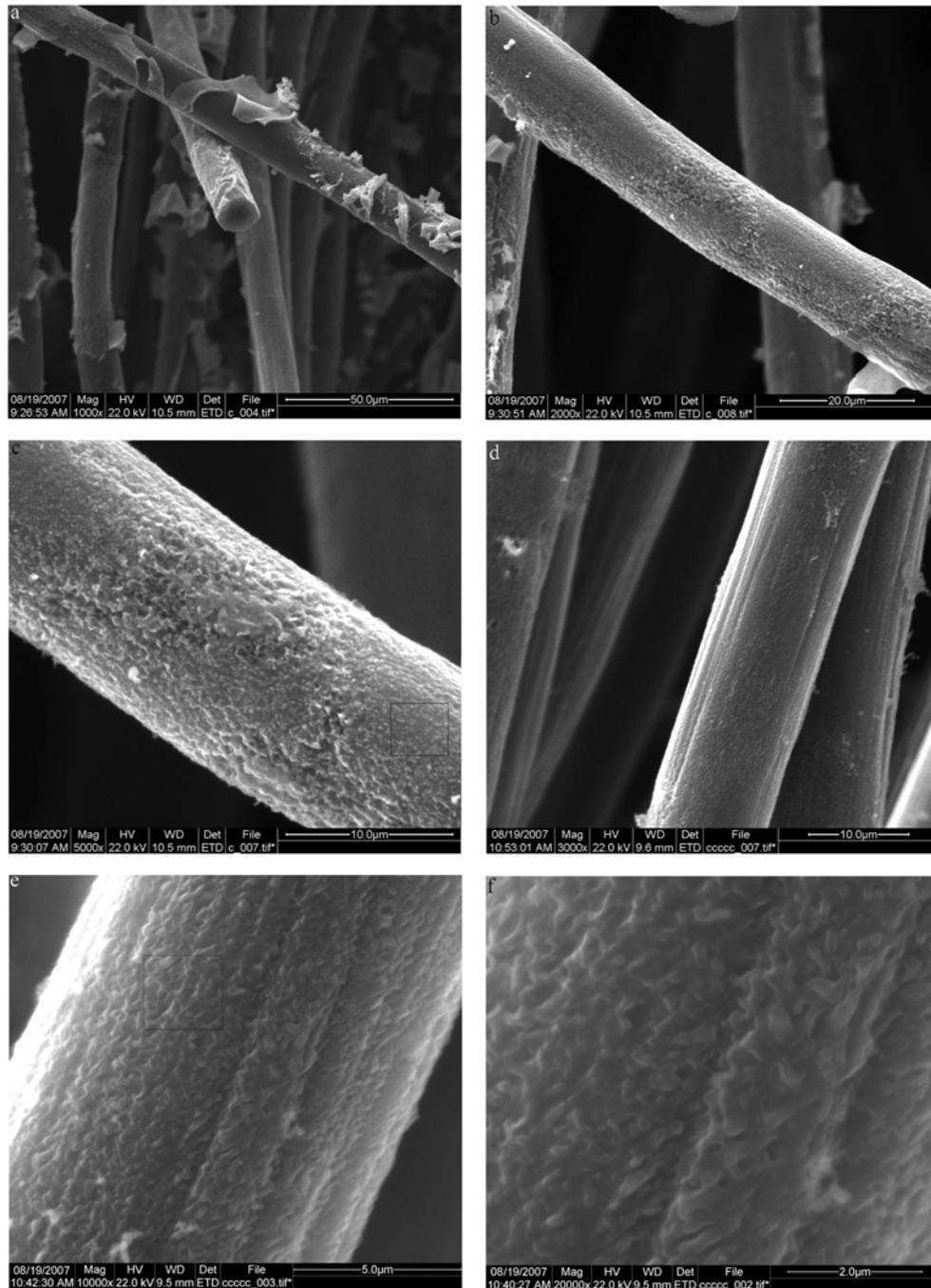


Fig. 1. SEM images of the coated fibers from the felt 'SS' (a, b, c) and felt 'SE' (d, e, f).

the substance of the coating seem to be deployed in a regular manner, and this is evident in the enlarged image of the coating in Fig. 1f in which these tiny contorted coating particles are of nano size. The coating of the fibers from the felt 'SE' (abbreviated as coating 'CSE' hereafter) covers all sides of the fibers neatly, it is much better than that on the fibers from the felt 'SS' (coat 'CSS').

The main reason for the above differences of the coatings is the egg white involved during the sintering. The crucial roles of egg white here are as follows: firstly, it acts as a favorable natural agglomerant to firmly attach the reactants on the surfaces of the fibers.

The firmly settled reactants can naturally be turned into the firmly settled solid outgrowth after sintering, just as seen with the 'CSE' coating. It is conversely deduced that the reactants can be lost and the solid outgrowth can be loosely settled or even incomplete without adhesive role of the egg white, especially under vacuum, as is seen with the 'CSS' coating. Secondly, the egg white acts as an active carbon source to join in the sintering reaction, and this will be referred to later.

Fig. 2 shows elemental contents within the rectangular areas on the surfaces of the coatings shown respectively in Fig. 1c and Fig. 1e. Both the coatings contain carbon

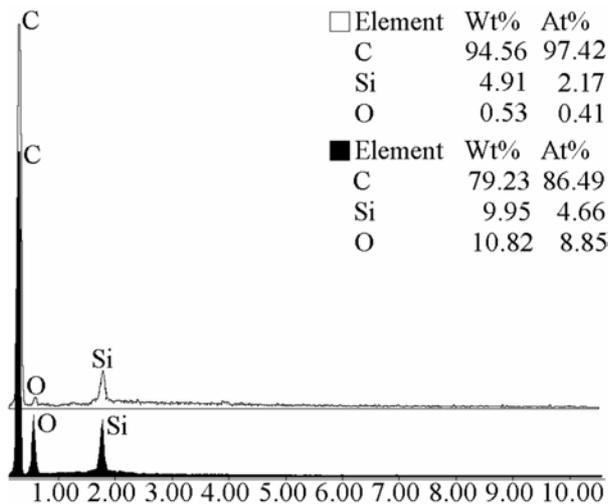
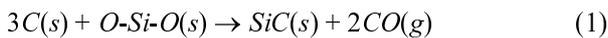


Fig. 2. EDX analyses for the coating 'CSE' (□) and coating 'CSS' (■).

('C'), oxygen ('O'), and silicon ('Si'). The contents of C in both the coatings are high possibly because the detection electrons penetrated through the coatings and contacted the carbon fibers (both the coatings are thin as Fig. 1 shows). The content of O in the coating 'CSE' is much lower than that in the coating 'CSS'. This can be explained by the fact that many proteins contained within the egg white can split to generate active carbon ('C(s)' in formula (1) listed below) which joins in the carbon thermal reduction sintering at high temperature. Such a reaction principally depicted in formula (1) consumes quantities of O existing in the form of 'O-Si-O' which is the microscopic three-dimensional network of silica gel [4].



The network 'O-Si-O' is chemically active and can be sintered to obtain nano sized silica, and it is the main substance of the coating 'CSS'. The carbon fibers are comparatively inactive in such reactions. The content of Si in the coatings on the whole accords with that contained in the immersion liquids prepared. It can be seen that main substance contained in the coating 'CSS' is SiO₂ and that contained in the coat 'CSE' is SiC.

Curves in Fig. 3a are the results of DSC-TGA analyses for the original carbon felt fibers, and curves 'SS-D', 'SS-T' and curves 'SE-D', 'SE-T' in Fig. 3b are those for the felt 'SS' and felt 'SE'. Temperature 'T1' (618 °C) and temperature 'T2' (761 °C) can be respectively regarded as the beginning oxidation temperature (BOT) of the felt 'SS' and felt 'SE' in air (the corresponding 'weight %' value is 100). As compared with the BOT of the original fibers in air (about 210 °C in Fig. 3a), the BOT of felt 'SE' has been improved by several hundred degrees Celsius. The original fibers begin being oxidized by air to lose substantial weight when the temperature is higher than 541 °C, at which the felt 'SE' and even the felt 'SS' are in a good condition as shown in the Fig. 3b. When temperature goes higher than 827 °C, the original fibers are nearly burned out, but

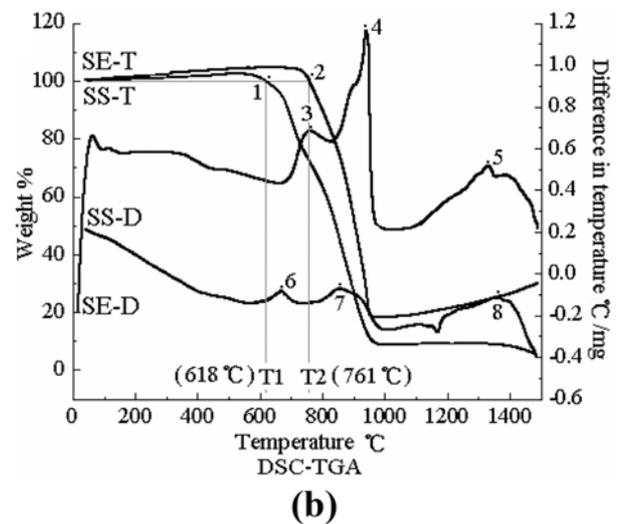
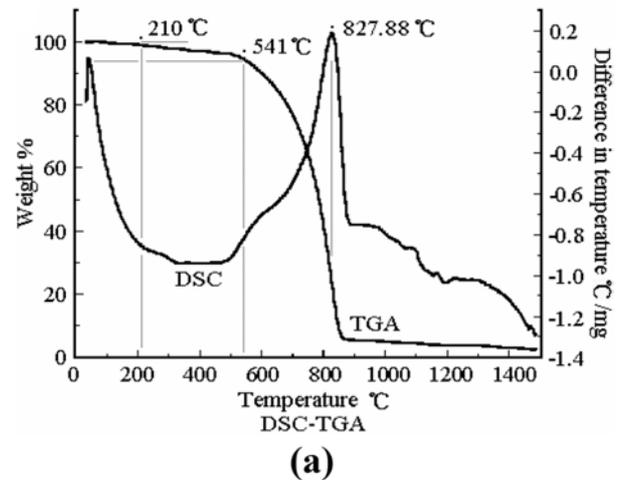


Fig. 3. DSC-TGA analyses for the original carbon felt fibers (a) and the felt 'SS' as well as felt 'SE' (b).

then the felt 'SE' can still preserve most portion of its weight. The felt 'SE' also has a better anti-oxidation capability than that of the felt 'SS'. When temperature is below 'T1' and 'T2' respectively, there are tiny weight increases for both the felt fibers, and this is evident for the felt 'SE'. This suggests the possibility that vitreous silica can be generated by mild oxidation of the coatings and this silica will resist further oxidation by enveloping the possibly uncovered parts of the fibers. When the temperature is higher than 'T1' and 'T2' respectively, there are sharp declines in the weights of the felts, which means serious oxidation of the felts and many gaseous outgrowths can be generated. There are six radiative peaks of oxidation reactions (oxidation peaks) indicated from point 3 to point 8 in Fig. 3b. Oxidation peaks at point 3 and point 6 are destructive to the coatings which will be crippled after the oxidations; the peaks at point 4 and point 7 are destructive to the carbon fibers which have lost the protection from the coatings, and both the fibers are seriously damaged losing more than 80% to 90% weights after the oxidations; other oxidation peaks have no decisive relationships with the

coated fibers which have already been damaged by previous oxidation events.

Summary

The coating 'CSE' and coating 'CSS' were prepared through sintering the pre-immersed carbon felts in a vacuum. Crucial roles of the egg white involved in the coating 'CSE' gave it a tidy, compact and enveloping structure of a nano subscale deployed approximately in a regular array. The coat 'CSS' was comparatively uneven, loose and fragmentary. As compared with the BOT of the original carbon felt fibers in air, the BOT of the fibers from the felt 'SE' was higher by several hundred degrees Celsius. The felt 'SE' also had a much better anti-oxidation capability than that of the felt 'SS'.

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