

Oxidation Behavior and Microstructure Evolution of Nuclear-Grade C/C Composites Oxidized in Air and He

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1. Introduction

Carbon-carbon (C/C) composites have been widely used for high-temperature structural applications because they possess excellent mechanical properties such as high specific strength and thermal shock resistance [1]. In the nuclear industry, the composites have been also considered for plasma facing materials in fusion reactors and high-temperature structural parts in gas cooled reactors [2,3]. In the high-temperature gas cooled reactor, the composites are being considered for the application of various high-temperature structural parts such as control rod components, core restraint belts, tie rods, upper plenum shroud, hot duct insulation cover sheets, and floor blocks [3]. However, the carbon based materials are susceptible to oxidation at temperatures above 400°C. Therefore, the oxidation behavior and property degradation at high temperature under impure He or in air in case of accidental air ingress should be evaluated before the deployment of composites.

In this study, the oxidation behavior and microstructure change of nuclear-grade C/C composites, Toyo Tanso CX-270G and SGL Sigrabond 1501YR, were evaluated after oxidation at various temperatures. The oxidation experiments were performed in range of 500 to 1200°C under air and He atmosphere.

2. Experimental Procedure

Samples used in this study were machined from CX-270G (Toyo Tanso Co., Ltd., Japan) and Sigrabond 1501YR (SGL Co., Ltd., Germany) C/C composite plates. Some features of the composites supplied from vendors are shown in Table 1.

Table 1. Characteristics of C/C Composites Used in This Study.

Grade	1501YR	CX-270G
Manufacturer	SGL	Toyo Tanso
Type of fiber	PAN 3K satin weave	PAN 6K plain weave
Matrix formation, Raw material	Resin infiltration and baking, Phenolic resin	Resin infiltration and baking, Phenolic resin
Density (g/cm ³)	1.58	1.63
Impurities	40-60 ppm	< 5 ppm
Heat treat temp.	2200°C	>2800°C

Oxidation experiments for the composite samples with a dimension of 10 x 5 x 2 mm were performed in a thermogravimetry (TG) apparatus. Samples were hung on Pt wire and the oxidation temperature was varied from 550° to 1200°C. The oxidation atmosphere was a synthetic air or high purity He with a gas flow rate of 50 cm³/min. The purity of He gas was 99.999% which contained <1 ppm O₂, <2 ppm N₂ and <2 ppm H₂O by volume as impurities. Surface and cross-sectional microstructures of the oxidized composites were observed using a scanning electron microscope (SEM).

3. Results and Discussion

Fig. 1 shows the weight change of C/C composites as a function of oxidation time in air at various temperatures. The oxidative weight loss increases rapidly above 650°C for both composites.

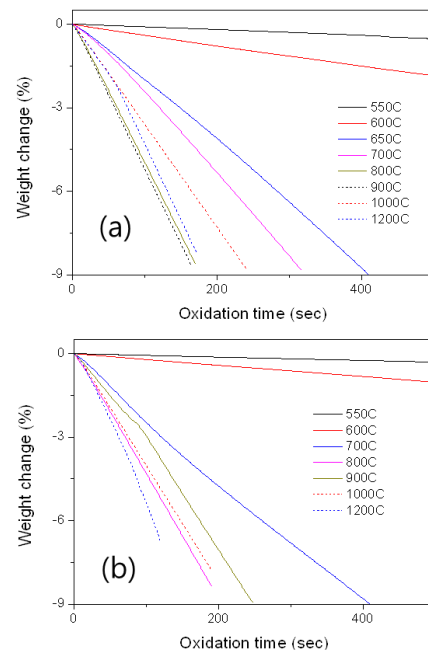


Fig. 1. Weight change of (a) 1501YR and (b) CX-270G composites oxidized in air at various temperatures.

Fig. 2 shows Arrhenius plots of oxidation rate of the composites. It has been known that the oxidation of C-based materials can be divided into three regimes [4]. At low temperature, oxidation kinetics is controlled by the surface chemical reaction and the oxidation occurs homogeneously in the porosity. When temperature

increases, the reaction rate is controlled by oxidant diffusion through pores. At higher temperatures, a mass transport through the boundary layer controls the oxidation reaction, i.e., the overall reaction rate depends on the reactant inflow to the surface and reaction products outflow. It can be seen in Fig. 2 that the oxidation kinetics are controlled by the surface reaction below 650°C although the in-pore diffusion and the mass transport regimes at higher temperatures are not clearly distinguished. The activation energy of oxidation reaction at the surface reaction controlled regime is around 165~200 kJ/mol similar to nuclear graphites.

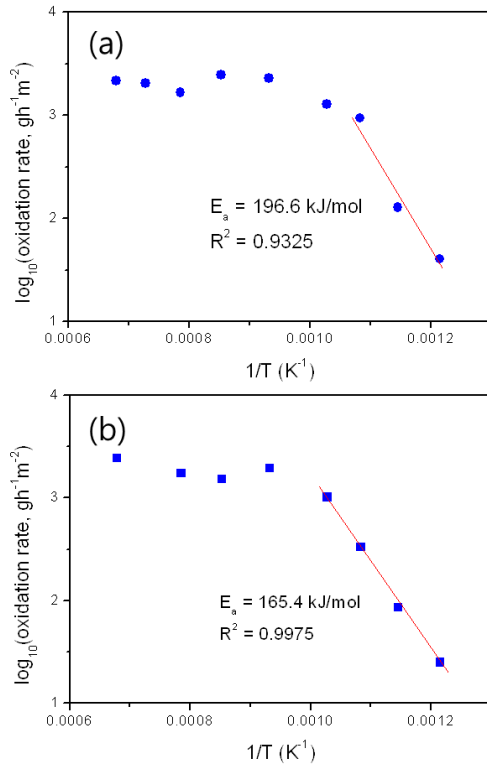


Fig. 2. Arrhenius plots of oxidation rate for (a) 1501YR and (b) CX-270G composites oxidized in air.

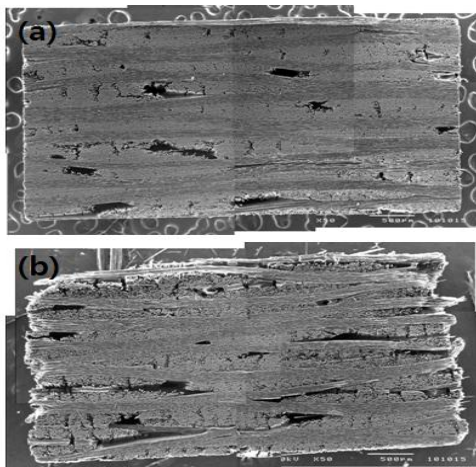


Fig. 3. SEM microstructures of CX-270G composite oxidized at (a) 550°C and (b) 700°C.

Microstructures of the CX-270G composite oxidized at 550° and 700°C are shown in Fig. 3. The sample oxidized at 550°C does not show a significant alteration of the specimen size and depicts a homogeneous oxidation throughout the specimen volume, supporting the surface reaction controlled oxidation reaction. The sample oxidized at 700°C shows, however, a severe degradation of surface regions and a reduction of the specimen size, implying the oxidation in pore diffusion controlled regime. The microstructure evolution of 1501YR composite also showed a similar trend.

Fig. 4 shows the weight change of both composites as a function of time in a high purity He at various temperatures. Detailed analysis of oxidation behavior in He and microstructure change will be shown in the presentation.

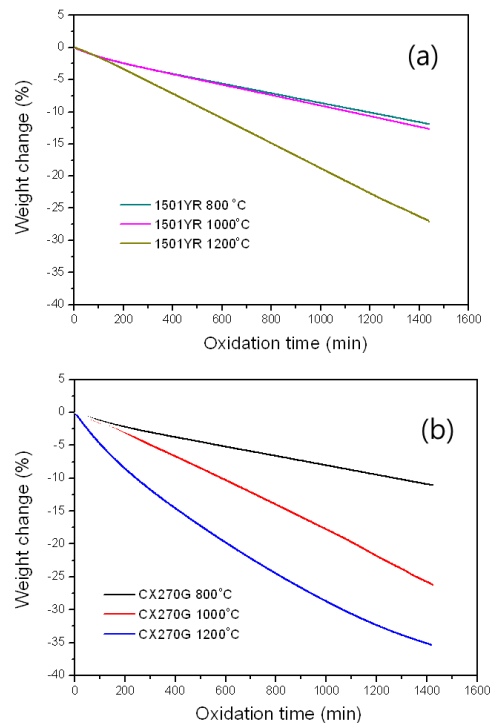


Fig. 4. Weight change of (a) 1501YR and (b) CX-270G composites oxidized in He at various temperatures.

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