Factorization Method for Failure Mechanism Analysis of C/C and C/SiC Composites

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ABSTRACT

A mathematical method, called factorization method, for failure mechanism analysis of high-temperature materials in various environments was presented and demonstrated with a C/SiC and a C/C composite. No matter how complicated, the experimental relation of any goal property to temperature can always be well described by a continuous function. It can be factorized to a multinomial with different Arrhenius relations. The failure mechanisms are corresponded with the least multinomial terms necessary to fit the experimental relation best. The activation energy and the threshold temperature for each mechanism and whether it is positive to the property can be determined.

Key Words: Failure mechanism, Factorization, High-temperature materials.

1. INTRODUCTION

One of the greatest challenges in developing materials against special environments is to determine the failure mechanisms. Because the damage of materials in a service environment, such as combustion gas, is always produced by multiple factors, it is very difficult to determine the failure mechanisms by experimental simulation based on the controlled environmental parameters. Although the property degradation and the microstructure evolution of materials can be related to the environment parameters

by experimental simulation and forecasted by further computer simulation based on a fuzzy nervous network (FNN), it is still not clear how the design of materials can be improved [1,2]. No exact suggestions can be obtained from the experimental simulation about what kinds of initial microstructures and properties the materials should have. The computer simulation by the Monte Carlo (MC) and the molecular dynamics (MD) method can be used for designing materials by formation of microstructures, but it will take a long time to relate the processing parameters and the environmental parameters to the microstructures of materials [3-6]. Actually, only microstructures at an equilibrium state can be formed by the MC and DC method in the near future. However, the microstructures formed in real processing are far away from an equilibrium state and include defects. Therefore, using microstructure computation without considering the processing parameters and the environmental parameters, it is nearly impossible to rationalize the multiple mechanism of failure for structural materials, especially for the high-temperature structural materials.

The failure mechanisms can be used to revise the processing and change the composition if they are clarified. This is very effective in enhancing properties of materials against environments before the microstructure computation can be related to the processing parameters and the environmental parameters. A method for analysing the failure mechanisms of materials by experimental simulation is presented in this paper, and then demonstrated by analyzing the failure mechanisms of a C/SiC and a C/C composite in different environments.

2. MODELING

If the goal property of a material is not monotonously decreasing or increasing with temperature over the full temperature range, damage to the material is produced by more than one mechanism relating to the environmental parameters. The effect of every mechanism on the goal property should be either monotonously decreasing or monotonously increasing with temperature. Generally, the effect can be expressed by the Arrhenius law:

$$\Delta\Omega_i = A_i \left(1 - \exp(-B_i T^{n_i}) \right) \quad (1)$$

Here $\Delta\Omega_i$ is the partial change in any goal property produced by the mechanism i ; n_i , A_i and B_i are constants. The total change in the goal property produced by all the mechanisms should be the sum of all partial changes

$$\Delta\Omega = \Delta\Omega_1 + \Delta\Omega_2 + \cdots + \Delta\Omega_l + \cdots + \Delta\Omega_N \quad (2)$$

Therefore, the complicated relation of the goal property to temperature, obtained by experimental simulation, can be described by a continuous function. In order to reveal the damage mechanisms, the continuous function should be factorized. If the number of the mechanisms is known, a multinomial function in the form of Equation (2) can be given, and then all the constants in the multinomial function can be determined by fitting it to the experimental results. The constants related to Equation (1) show respectively the activation energy and the threshold temperature for each mechanism and whether it is positive or negative to the goal property. Actually, this method for failure mechanism analysis of materials is a mathematical method. It might be called the factorization method. Although it is very simple, the factorization method is very useful and important in developments and applications of high-temperature materials.

It is most important to make out the number of mechanisms from the experimental relation. Because one experimental relation can be well fitted by different multinomial functions, the mechanisms should be corresponded with the least multinomial terms which are necessary to fit the relation best. If there is more

than one mechanism in a narrow temperature range, it is difficult to separate them by fitting. As a result, the determined number is not correct with certainty. In that case, the goal property change is considered to be controlled by a representative mechanism in factorization. However, this will not constitute any essential obstacle to understanding of the failure process. The physical meanings of the mechanisms can be clarified by the material characteristics and the further microstructure analysis.

3. FAILURE MECHANISM ANALYSIS

3.1. Weight change of C/SiC composite as a function of temperature in salt vapor

A three-dimensional carbon fiber reinforced silicon carbide composite (3D-C/SiC) with a pyrolysis carbon interlayer was employed. The composite was prepared by chemical vapor infiltration (CVI). A two-layer SiC coating was prepared by chemical vapor deposition (CVD). The corrosion tests of the composite were conducted in a Na_2SO_4 vapor environment at a concentration of 300 ppm at temperatures from 900°C to 1500°C [7]. Figure 1 showed that the weight change of the composite could be related to temperature by three Arrhenius functions after 5 hours of corrosion over the

Fig. 1: Weight change of the C/SiC composite as a function of temperature after corrosion in salt vapor for 5 hours.

● ΔW measured, — ΔW calculated, ▲ ΔW_1 , ◇ ΔW_2 , * ΔW_3 .

whole temperature range

$$\Delta W = \Delta W_1 + \Delta W_2 + \Delta W_3 \quad (3)$$

This indicated that the weight change could be considered to be caused by three mechanisms. The small weight gain ΔW_1 produced by the first mechanism confirmed that passive oxidation of the CVD SiC took place

The small weight loss ΔW_2 produced by the second mechanism could only be due to oxidation of the carbon phases in the composite

Above 1300°C, the oxidation of the CVD SiC transformed from passive to active

Hence, the third mechanism led to a large weight loss, ΔW_3 . The threshold temperature for these reactions was, respectively, 1080°C, 1100°C and 1300°C. The activation energy for these reactions was, respectively, calculated to be 114 kcal/mole, 105 kcal/mole and 112 kcal/mole.

3.2. Weight change of C/SiC composite as a function of temperature in air

The same 3D-C/SiC with a two-layer coating was employed. The coating consists of a SiC inner layer prepared by CVD and a Si-Zr prepared by liquid reaction. The oxidation tests of the composite were conducted in air at temperatures from 500°C to 1500°C [8]. Figure 2 showed that the weight change of the composite could be related to temperature by four Arrhenius functions after 5 hours oxidation over the whole temperature range

$$\Delta W = \Delta W_1 + \Delta W_2 + \Delta W_3 + \Delta W_4 \quad (7)$$

It could be seen that the weight change was produced by

Fig. 2: Weight change of the C/SiC composite as a function of temperature after oxidation in air for 5 hours.

● ΔW measured, — ΔW calculated, ▲ ΔW_1 ,
◇ ΔW_2 , * ΔW_3 , ○ ΔW_4 .

four mechanisms. The first mechanism was the reaction of oxygen with the carbon phases in the composite, leading to uniform oxidation of the carbon phases. The second one was the sealing of the SiC matrix cracks formed by mismatch of the matrix and the fiber, leading to non-uniform oxidation of the carbon phases. The third one was the sealing of the coating cracks formed by mismatch of the coating and the composite, leading to superficial oxidation of the carbon phases. The fourth one was the oxidation of the coating. The threshold temperature for these reactions was, respectively, 400°C, 500°C, 850°C and 1250°C. The activation energy for these reactions was, respectively, calculated to be 26 kcal/mole, 18 kcal/mole, 54 kcal/mole and 56 kcal/mole.

3.3. Flexural strength change of C/SiC composite as a function of temperature in air

Figure 3 showed the flexural strength change corresponding to the weight change of the composite in Fig. 2 [9]. Although it was different from the weight change, the strength change could also be related to temperature by four Arrhenius functions after 10 hours of oxidation over the whole temperature range

$$\Delta \sigma = \Delta \sigma_1 + \Delta \sigma_2 + \Delta \sigma_3 + \Delta \sigma_4 \quad (8)$$

Fig. 3: Weight change of the C/SiC composite as a function of temperature after oxidation in air for 10 hours.

● $\Delta\sigma$ measured, — $\Delta\sigma$ calculated, ▲ $\Delta\sigma_1$,
◇ $\Delta\sigma_2$, * $\Delta\sigma_3$, ○ $\Delta\sigma_4$.

Similarly, the strength change was produced by four mechanisms. The weight loss of the composite changed with temperature similarly to the strength loss, because the former three mechanisms for the weight change affected the strength change by damaging the fibers. Above 1050°C, the coating cracks were sealed, and the oxidation took place on the coating surface. Because the surface oxidation decreased the width of the coating cracks due to bulk expansion of the formed oxide film, the strength was increased. However, the strength was decreased above 1400°C for the reason that the oxidation took place on the composite surface after the coating was completely oxidized in 10 hours. Consequently, the fourth mechanism led to the strength loss of the composite when it led to a sufficient weight gain.

3.4. Weight change of C/C composite as a function of temperature in air

A two-dimensional carbon fiber reinforced carbon composite (2D-C/C) with a three-layer coating was employed. The coating consists of a SiC inner layer prepared by siliconization, a SiC barrier layer prepared by CVD and a Si-W outer layer prepared by liquid reaction. The oxidation tests of the composite were conducted in air at temperatures from 500°C to 1700°C /10/. Contrary to the C/SiC composite, the weight

change of the C/C composite was produced by three mechanisms over the temperature range from 400°C to 1500°C (Fig. 4). The sealing mechanism of the matrix cracks took place in the former, but not in the latter, because there were no matrix cracks in it. However, another mechanism was dominant from 1500°C to 1700°C. Considering that the oxidation took place on the coating surface, this mechanism was the interfacial reaction between the oxide film and the coating, leading to rapid weight loss in a short time by producing bubbles in the oxide film.

Fig. 4: Weight change of the C/C composite as a function of temperature after oxidation in air for 5 hours.

● ΔW measured, — ΔW calculated, ▲ ΔW_1 ,
◇ ΔW_2 , * ΔW_3 , ○ ΔW_4 .

4. CONCLUSIONS

1. No matter how complicated, the experimental relation of any goal property to temperature for high-temperature materials in various environments can always be well described by a continuous function. The continuous function can be factorized to a multinomial with different Arrhenius relations. The failure mechanisms are corresponded with the multinomial containing the least terms necessary to fit the experimental relation best. The activation energy and the threshold temperature for each mechanism and whether it is positive or negative to the goal property can be obtained by fitting the multinomial to the experimental relation.

2. As goal properties, the weight change and flexural strength change of a C/SiC and a C/C composite with different coating to temperature in air and in a salt vapor environment were used to demonstrate the factorization method for failure mechanism analysis. The complicated mechanisms for the weight change and the flexural strength change of the composites were revealed by this method.

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