

HYDROGEN EFFECTS ON SiC/SiC COMPOSITES FOR FUSION STRUCTURAL APPLICATIONS

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OBJECTIVE

The purpose of this study is to quantitatively assess the stability of SiC/SiC composites in high-temperature, low-hydrogen environments by the measurement of weight loss, in relation to their potential use as structural materials in fusion reactors.

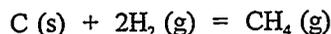
SUMMARY

Two exploratory experiments at 1100°C and 1200°C in an argon + 1% hydrogen environment have demonstrated a relatively slow reaction rate. This slow reaction rate concurs with literature¹ yet a better understanding of kinetics is still needed. Further experimentation will test the reproducibility of these early results and better determine the influence of experimental conditions by varying temperature and the partial pressure of hydrogen.

Introduction

Silicon carbide composites are a leading type of ceramic matrix composite (CMC) being evaluated for use as a structural material in fusion reactors². In this evaluation, positive factors include high strength at elevated temperatures, low density, high stiffness, and low neutron activation, with some negative factors being material cost and fabrication complexity. The mechanical properties of SiC/SiC, such as strength and toughness, have been shown adequate for the design requirements. What needs to be further established for SiC/SiC composites is the predicted performance under the conditions of irradiation and exposure to fusion relevant environments.

This study will focus only on the exposure of SiC/SiC composites with C-interfaces to low concentrations of hydrogen in order to simulate a gas cooled, solid-breeding blanket. The interfacial carbon should react with hydrogen to produce methane gas, with negligible traces of other hydrocarbons⁽³⁾. The matrix and fibers should be stable under the experimental conditions and should not skew the weight loss measurement by other reactions. Therefore, the primary reaction to consider is:



The purpose of this study was to assess the loss of C-interface due to this reaction under various temperatures and hydrogen partial pressures.

Experimental

The samples used in this study were acquired from Refractory Composites, Inc. of Whittier, California. Fabrication was done by the chemical vapor infiltration (CVI) of Beta-SiC matrix into 8-ply Nicalon fiber cloth, pre-coated with C-interface. This formed a 0°/90° weave pattern with fiber diameters of 15 microns and interface thicknesses of 1 micron. Final sample shape was rectangular with average dimensions of 0.4 x 0.4 x 0.5 cm. In order to better isolate the reaction fronts, only two faces were uncoated, while four sides were completely coated with SiC in order to prevent any reaction.

A Netzsch STA409 thermogravimetric analyzer (TGA) was used for these measurements. Both exploratory tests were executed on this unit according to the following procedure:

- 1) The composite sample was weighed and placed in the left furnace crucible such that neither of the uncoated sides faced downward. (Alumina powder rested in the right furnace crucible as an inert reference material used to stabilize the TC rod.)
- 2) The furnace was sealed.
- 3) Gas flow was adjusted to 3.2 standard cubic feet per hour (SCFH) and the system was purged for about 2 hours.
- 4) Cooling water flow was brought to a steady level.
- 5) The balance was released and the scale adjusted to the proper zero level.
- 6) Initiation of the profile controller program brought the furnace to peak temperature in half an hour by ramping.
- 7) Peak temperature was maintained for the experiment duration and was followed by a 2-hour cooldown period to ambient levels.
- 8) The flow of gas and cooling water was stopped.
- 9) The furnace was opened.
- 10) The composite sample was removed and re-weighed.

PROGRESS AND STATUS

The results of the two exploratory tests are shown in Figure 1. Three items are important to bear in mind when examining these results. First, they are merely single points, not curves. The Netzsch stripchart was originally designed to record dynamic weight measurements, but has been found unreliable at lower gas concentrations. Weight losses depicted in Figure 1 were obtained simply by total sample weight change, with weights recorded on a balance separate from the Netzsch unit before and after testing. Second, these weight losses have been normalized with respect to exposed surface area. This normalization assumes that interfacial removal along one fiber axis does not affect interfacial reduction down the perpendicular fiber axis. Finally, since little weight loss was detected on the first test the same sample was used for the second test.

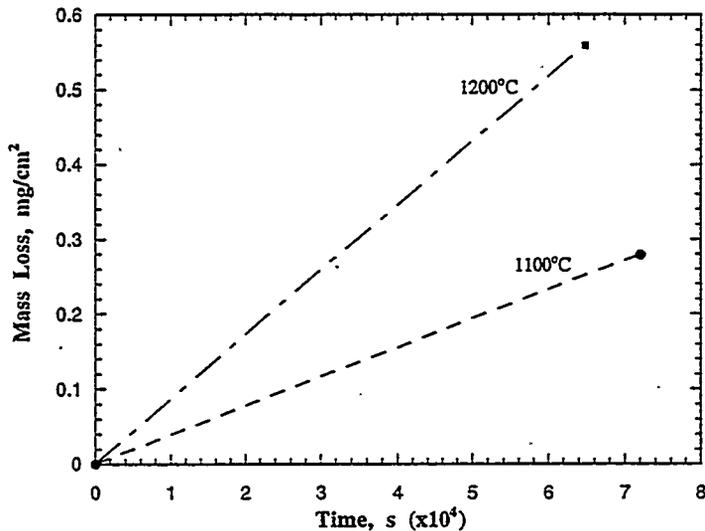


Figure 1: Weight Loss Data for C-Interface SiC/SiC Composites in 1% H₂ (balance He)

The weight losses observed in hydrogen were about an order of magnitude less than those observed in oxygen at equivalent times and even lower partial pressures⁽⁴⁾. Table 1 shows data by Walker et al. for gas-carbon reactions at 800°C and 0.1 atm. Similar to the results reported in this study, Walker observed that the carbon-oxygen reaction proceeded much faster than that of carbon-hydrogen.

A noticeable temperature dependency was observed for the carbon-hydrogen reaction. At similar times, weight losses at 1200°C were about twice as much as those at 1100°C. Worth noting in the 1200°C run was the formation of small white deposits in crevices on the uncoated faces of the sample. If these deposits were substantial enough to affect measured weight loss, they would have increased the sample weight slightly and downplayed the actual effect of temperature on interfacial removal.

Additional tests are being planned with similar conditions, to test the reproducibility of these early results, and at lower hydrogen concentrations, to better simulate the hydrogen concentration expected in a gas cooled blanket.

Reaction	Relative Rate
C - O ₂	1E5
C - H ₂ O	3
C - CO ₂	1
C - H ₂	3E-3

Table 1: Approximate Relative Rates of the Gas-Carbon Reactions at 800°C and 0.1 Atm Pressure. (Walker et al., Advances in Catalysis)

CONCLUSIONS

Exploratory test results have indicated a relatively slow rate of interfacial carbon removal in hydrogen environments for SiC/SiC composites. These results concur with literature, but much more experimentation is necessary to refine the understanding of temperature and hydrogen partial pressure influences.

FUTURE WORK

The reproducibility of preliminary data will be assessed by more studies with 1% hydrogen at high temperatures, between 1100°C and 1200°C. Realistic fusion conditions will be better simulated by additional testing in 1000 ppm hydrogen at various temperatures.

REFERENCES

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4. G. D. Springer, C. F. Windisch, Jr., C. H. Henager, Jr., and R. H. Jones, "Oxygen Effects on SiC/SiC Composites for Fusion Structural Applications," Pacific Northwest Laboratory, DOE/ER-0313/18, Fusion Reactor Materials Semiannual Progress Report for Period Ending March 31, 1995.