OXIDATION PROTECTION OF GRAPHITE FOAMS

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Introduction

Graphite foams have been shown to be excellent heat transfer mediums and the basis for very efficient heat exchangers (1-4). However, many heat exchangers will need to operate at elevated temperatures between 500°C and 1200°C and will be exposed to oxidizing conditions. Also, other carbon-based materials are being examined for use in liquid metals processing and are being evaluated for extremely high-temperature applications such as un-cooled rocket nozzles. These highly corrosive environments would benefit greatly from the high conductivity to weight ration of the graphite foam, however, methods to inhibit the corrosion must be found. The ability to introduce a CVI ceramic coating of certain carbides and borides has shown promise in protecting the graphite from oxidation. The low, but positive, coefficient of thermal expansion and the low modulus of the foams combine to minimize thermal stresses in the coatings when compared to coatings on carbon-carbon composites and bulk graphites and, therefore, minimize microcracking in the foam.

Experimental

In order to protect the carbon from oxidation and other corrosive environments, silicon carbide (SiC) was deposited on the foam at from the vapor phase using a standard SiC deposition technique (methyl trichlorosilane (MTS) in a hydrogen atmosphere). Specific run conditions were: pressure of 30 torr, temperature of 1100°C, hydrogen flow of 500 cm^3/min, MTS flow of 0.3 g/min, and a duration of 8 hours.

Samples were mounted in epoxy, polished, and analyzed under optical microscopy and SEM. Samples were evaluated for oxidation protection in a TA Instruments SDT 2960 Simultaneous DTA/TGA. The samples were heated at 5°C/min to 1000°C and the weight change was monitored with a microbalance. In addition, separate samples were heated at 5°C/min to 650°C and held at 650°C for 8 hours.

Results

Figure 1 is an optical micrograph of the graphite foam before and after coating with the SiC. As can be seen, the coating thickness appears to be very uniform. Detailed analysis revealed an overall coating thickness of approximately 31.5 microns. The samples gained 124% mass gain and the density changed from 0.46 g/cm^3 to 1.04 g/cm^3. There are no apparent microcracking in the coating layer and the coating appears to be well bonded to the carbon foam ligaments (a necessary result to attain good corrosion resistance). In higher magnifications, Figure 2, it is evident that there are actually two layers of the coating. The first layer (A), and interface layer, appears to be approximately 1.6 microns thick. The second layer (B), the coating layers, appears to be approximately 30 microns thick. Elemental analysis using the SEM EDAX revealed that the coating layer is, as expected, only carbon and silicon. However, detailed analysis indicated that the inner layer is carbon rich. However, this is inconclusive since the resolution of the SEM EDAX is not accurate enough to differentiate completely from the carbon ligament and a 1.6 micron coating. Currently, TEM analysis is being conducted to perform a more detailed study at this interface.

Figure 3 is a high magnification image of the foam after heat treatment in air at 650°C for 8 hours. Most of the foam showed no corrosion effects. However, this selected region is shown to illustrate the appearance of oxidation of the foam just below the surface of the coating. As there are no microcracks in coating in this region, it is believed that the oxygen intrusion came from a nearby crack that was either above or below the plane of the polished surface.

Figure 4 is the plot of weight yield versus temperature of the coated foams compared to the uncoated foam when heated to 1100°C. As evident from the plot, the coating increases the resistance to oxidation. The uncoated foam began to oxidize at roughly 700°C and burned-up completely by 1000°C, while the coated foam started to oxidize at about 900°C and only lost 21% by 1100°C, a significant increase in corrosion protection. This oxidation protection is also observed when the foam is heated in air to 650°C and held at temperature for 8 hours (Figure 5). In this case, the foam with the coating had a 0.72 % weight loss after an 8 hours exposure, and the uncoated foam had nearly 20%.

Conclusions

Clearly, coating the foam with SiC enhances the oxidation resistance of the graphite foam. However, it is also

apparent that better resistance will be needed to operate the foam in heat exchangers at elevated temperatures. In future work other coatings such as silicon-boron-carbide, mullite, hafnium compounds, and metal carbides will be examined.

Acknowledgments

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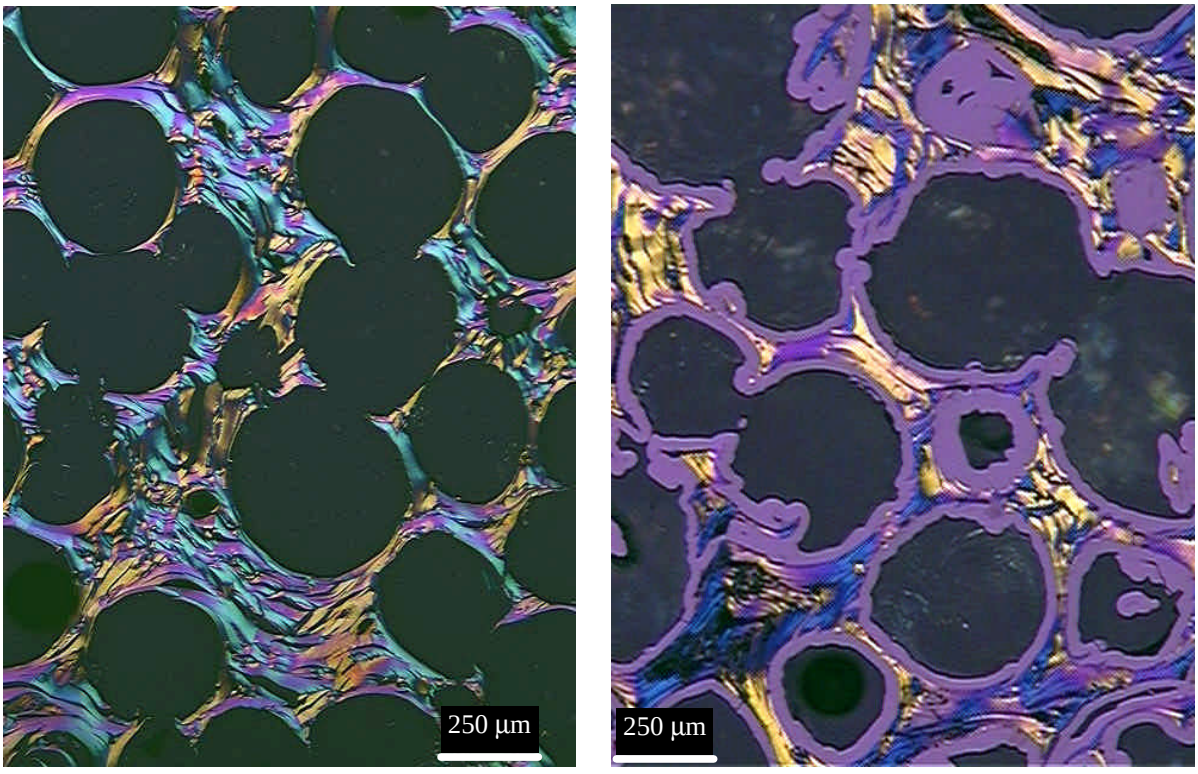

Figure 1. Optical Micrographs of carbon foam before and after coating with silicon carbide.

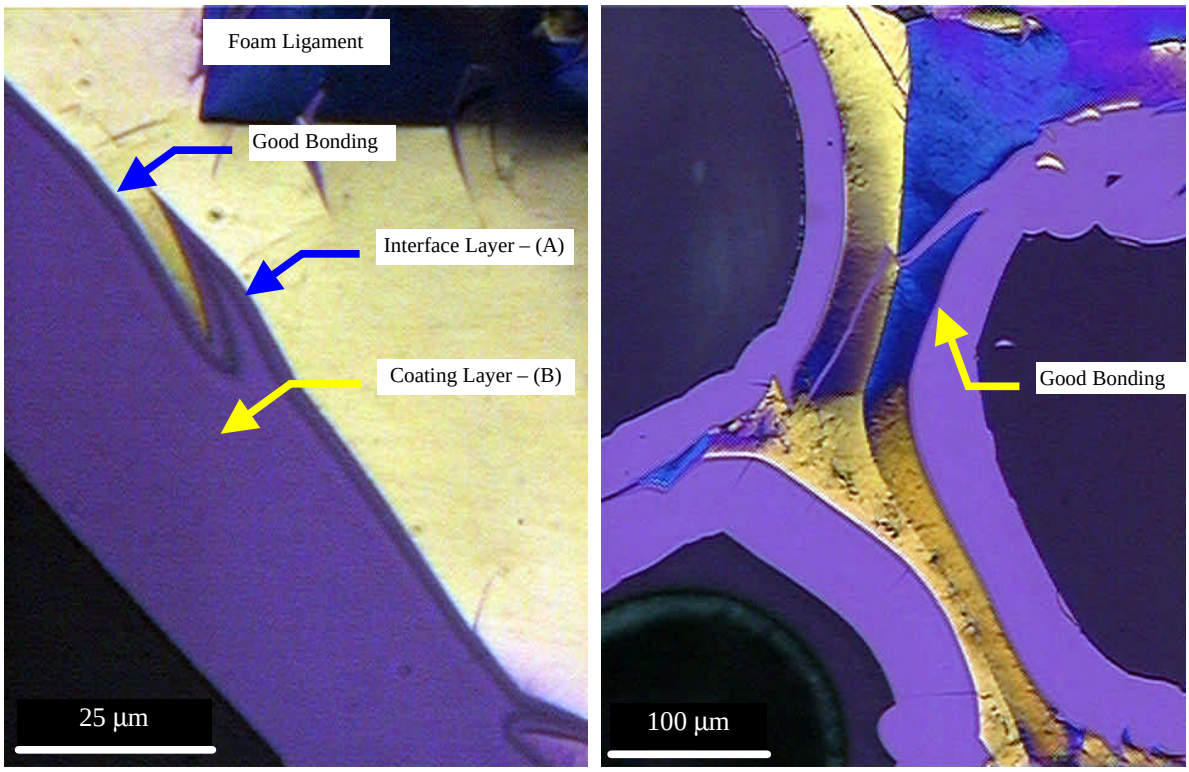

Figure 2. High magnification images of the interface between the foam and the coating illustrating a 3 layer coating.

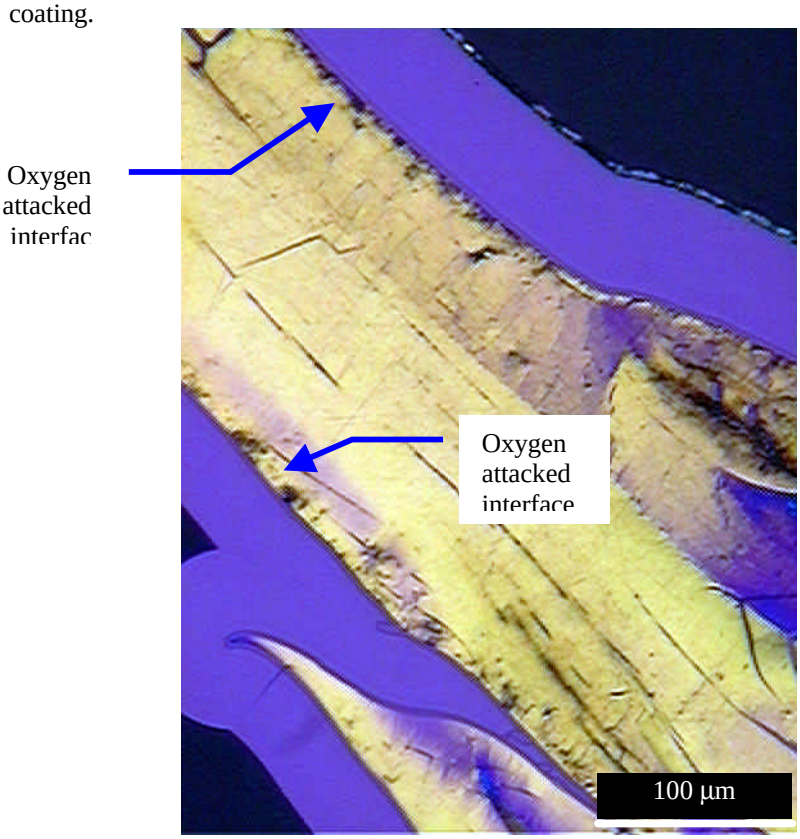

Figure 3. High magnification image of interface after oxidation in air at 650°C for 8 hours.

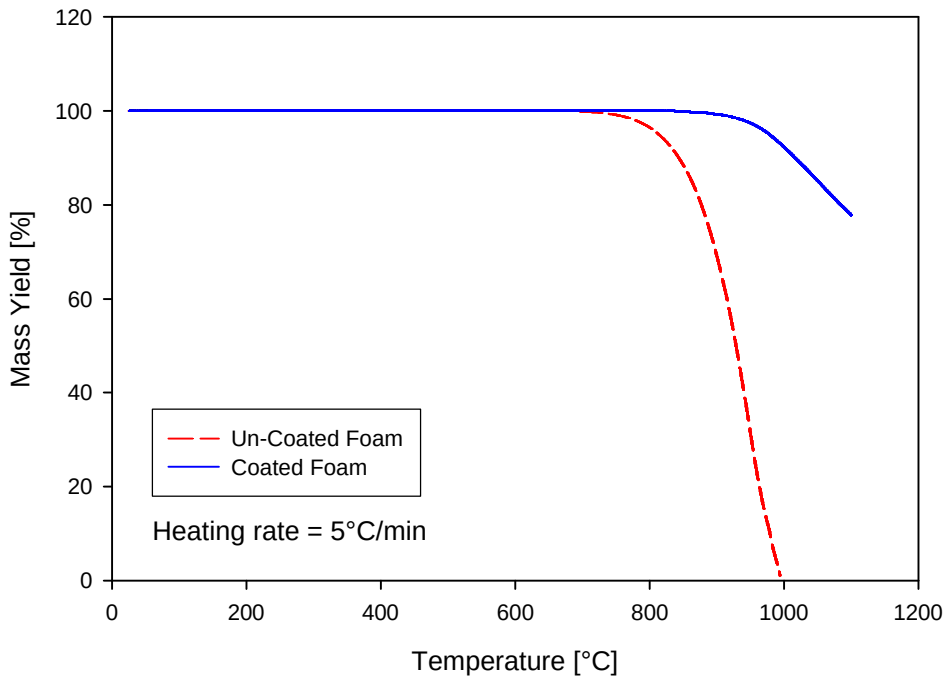

Figure 4. TGA plot of the uncoated and coated foams in air and temperatures up to 1100C.

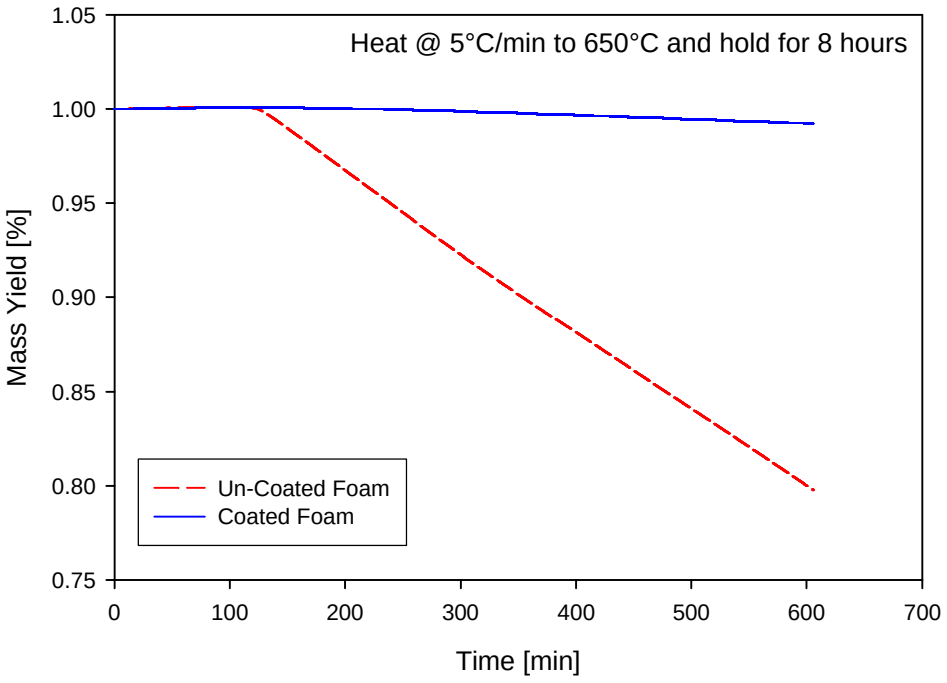

Figure 5. TGA plot of the uncoated and coated foams held in air at 650C for 8 hours.