STABILITY OF BOROSILICATE GLASSES
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MOTIVATION

SiC-SiC ceramic matrix composites (CMCs) are being developed to replace heavier superalloy materials for turbine engine applications. Borosilicate glasses form in these materials upon oxidation of the BN fiber/matrix interphase [1]. These glasses provide protection from further oxidation of the interphase regions, however under some conditions they can have deleterious effects on the longevity of these components. The study of these borosilicates and their interaction with the composite system is crucial for the safe use of CMC turbine materials.

In this research, the main focus is on determining the stability and ease of manufacturing of the borosilicate mixtures. The knowledge gained in this step will help with characterizing the attack that occurs on the SiC fibers in-situ.

MATERIALS

For the thermal analysis mixtures of silica (SiO₂) and boria (B₂O₃) powders were utilized. The silica-boria phase diagram can be seen to the right. A 1:1 ratio (by weight) of boria to silica was used, as was a 3:1 ratio of boria to silica. Both mixtures should begin melting at the eutectic temperature of ~450°C. The 1:1 mixture should complete melting near 790°C while the 3:1 mixture should complete melting near 680°C.

PROCEDURE

Powder mixtures of two compositions were analyzed using a Netzsch STA 449. Combined DSC/TG data were collected from room temperature to 1200°C heating at 20°C per minute in an argon atmosphere.

Additionally, using TG measurements only, a single sample of the boria-rich powder was heated from 1200 degrees, with 30 minute hold periods at 800, 900, 1000, 1100, and 1200 degrees in order to help determine the steady-state weight loss at each temperature.

RESULTS

The scan performed on the 1:1 powder mixture can be seen in Figure 2. In this experiment the DSC data showed a large amount of endothermic activity between 100 and 200°C. This reaction is consistent with the decomposition of the mineral sassolite (H₂BO₃) which can be thought of as a hydrated mixture of 56.3% B₂O₃ and 43.7% H₂O [3]. Sassolite melts near 170°C, and then decomposes to boria and water vapor near 196°C. There are no more distinct peaks or glass transitions on the DSC curve, although there is a large, broad endothermic hump in the curve.

The scan performed on the 3:1 powder mixture is shown in Figure 3. In this case there are four additional features worth noting on the DSC curve. There is a separate water peak at 100°C, and then three additional features at 245, 304, and 450°C. The feature at 450°C correlates nicely with the eutectic temperature, while the other two are unidentified. Figure 4 shows the same sample plotted against time with cooling data shown as well.

Figure 5 shows the results of the stepwise heating, and the weight loss at each temperature for the 3:1 powder mixture. Linear trend lines were fit to give an indication of weight loss in mg/min at each temperature.

IMPLICATIONS

From these data it is indicated that the weight loss due to boria volatilization is minimal, but might still affect the final melt chemistry and provide a silica-rich glass. Additionally, the melting and homogenization of the mixed powders would appear to be a sluggish reaction, and this could be much of the reason that the large endothermic peak at high temperature is so broad [4]. Due to the relative difficulty of creating a borosilicate melt, aqueous or sol-gel processing will be investigated as alternative routes to generate these melts.

FUTURE WORK

One of the next steps of this research will be to do a chemical analysis on the glasses after melt processing to determine the final melt chemistry and homogeneity. This should help to determine boria lost to volatilization as well as the kinetics of the melt reaction. Performing DSC measurements at a slower heating rate might also help to allow for the sluggish melting of the powders and give a greater understanding of the reaction taking place [5]. Instigating methods of aqueous or sol-gel processing will be another step to potentially simplify the creation of borosilicate melts [6].

Once satisfactory borosilicate melts can be made, testing will begin with fibers in the molten glass to study the attack on the fibers. This research will lead into oxidation testing of model composites and actual SiC/SiC materials. In addition to characterizing the oxidation and borosilicate attack on the fibers in the composite, hot corrosion testing of SiC, model composites, and actual SiC/SiC materials will be carried out as well.

REFERENCES