946 Microsc. Microanal. 16 (Suppl 2), 2010
doi:10.1017/S1431927610060824

Characterization of Structure Evolution in Aged Al_2O_3/SiC Composite Refractories by Electron Microscopy

Virgil C. Solomon,* Klaus-Markus Peters,** Brian P. Hetzel,** and Tim R. Wagner ***

* Department of Mechanical & Industrial Engineering, Youngstown State University, Youngstown, OH 44555
** Fireline, Inc., Youngstown, OH 44505
*** Department of Chemistry, Youngstown State University, Youngstown, OH 44555

Advanced refractory materials are of great interest in metallurgy mainly due to their higher resistance to mechanical degradation and better thermal management, which increases the energy efficiency of melting and casting processes. In the present experiment preforms containing sacrificial oxides (SiO_2) and inert phase (SiC) are fully immersed in molten metal (such as Al-Ti alloy, in this case) for various periods of time, depending on specific processing conditions [1]. Although the refractory characteristics of the composites are improved, the materials kept in atmosphere at room temperature show the degradation of the mechanical properties. The purpose of this report is to present the results of the investigation of the structural changes in a refractory Al_2O_3/SiC composite due to aging in atmosphere at room temperature using scanning and transmission electron microscopy techniques.

Figure 1 shows optical images of a cross-sectioned sample after being exposed to air for one and three months. The cracks in the sample can be readily observed, as well as the multi layered structures due to the inhomogeneity of the SiC distribution through the preform and the in homogeneity of the Ti distribution throughout the transformed sample.

The SEM samples have been prepared using metallographic techniques, while TEM samples were prepared by a combination of metallographic and FIB techniques. The investigations were carry out in two JEOL instruments a JIB-4500 Multi Beam SEM/FIB and a JEM 2100 S/TEM. The results of SEM investigation are presented below.

Figure 2 shows the XEDS maps of the areas containing the interfaces between layers 1 to 4. Layer 1 is about 50 μm thick and is excess Al-Ti alloy from the melt. Layer 2 contains a TiC phase formed around the SiC grains. Layer 3 was the one most drastically degraded over the observed time. No TiC phase was observed in this layer, although using PXRD, the existence of an Al_4C_3 phase was confirmed in Layer 3. It is known that Al_4C_3 forms at liquid Al/SiC interface, according to [2]:

$$3SiC + 4Al \leftrightarrow Al_4C_3 + 3Si$$

The Al_4C_3 phase is highly unstable and reacts with the moisture in the atmosphere, as follows:

$$Al_4C_3 + 18H_2O \rightarrow 4Al(OH)_3 + 3CO_2 +12H_2$$

It seems that a secondary effect of this reaction is the volume increase of layer 3. The stress created by increased volume of layer 3 cannot be accommodated between layer 2 and core 4, leading to the failure of the sample, as observed in Fig. 1. On the other hand the formation of Al_4C_3 phase in layer 2 was prevented due to the preferential formation TiC around SiC grains [3]. The core 4 contains a mixture of SiC and Al_2O_3 phases.

Figure 3 shows the failure surfaces of layer 2 and core 4 on the five months old sample. Figure 4 (a) and (b) shows the complete disintegration of layer 3 after three and five months, respectively. It should be mentioned that samples stored for five months in vacuum show no defects.

Based on these observations several solutions have been proposed to prevent refractory material disintegration: longer immersion time, higher melt temperature and/or pressure impregnation.

References
[1] J. Xu et al., *Int. J. Appl. Ceram. Technol. 4 [6] (2007) 514.*
[2] J.C. Lee et al., *Acta Mater.* 5 (1988) 1771.
[3] Y.C. Lei et al., *Trans. Nonferrous Met. Soc. China* 18 (2008) 809.
[4] This research was supported by WP 08-030: “Center for Excellence in Advanced Materials Analysis”**,** Ohio Third Frontier, Wright Projects Program. The use of Electron Microscopy Facility within STEM College at YSU is gratefully acknowledged.

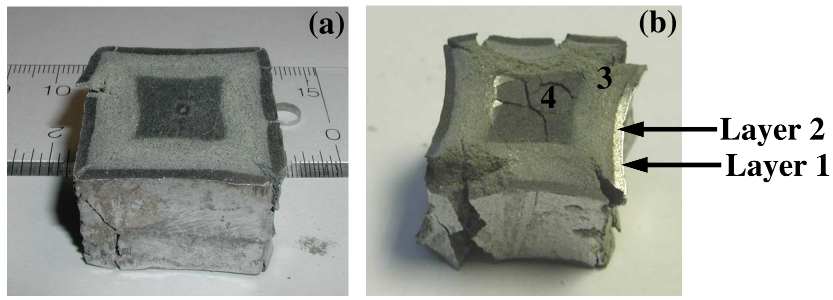

FIG 1: (a) Appearance of the cross-sectioned sample stored for one month at room temperature and atmospheric pressure. (b) Same sample after two months of storage.

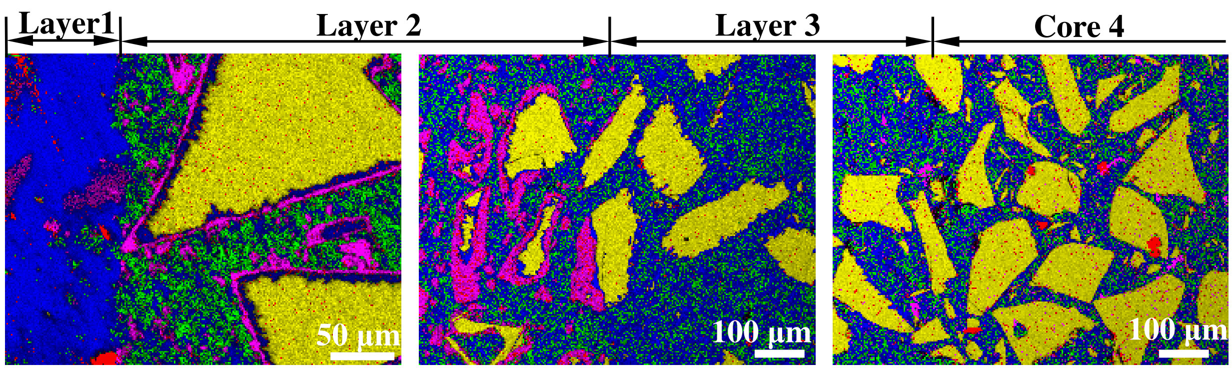

FIG 2: XEDS maps from cross-sectioned sample, layers 1 to 4. Color code: Al-blue, C-red, O-green, Si-yellow, Ti-magenta.

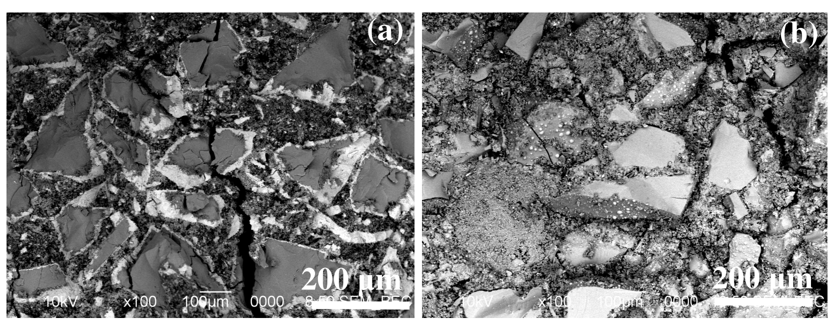

FIG 3: (a) and (b) BSE micrographs of fracture surfaces of layer 1 and core 4, respectively. Note in (a) the presence of TiC phase surrounding SiC grains.

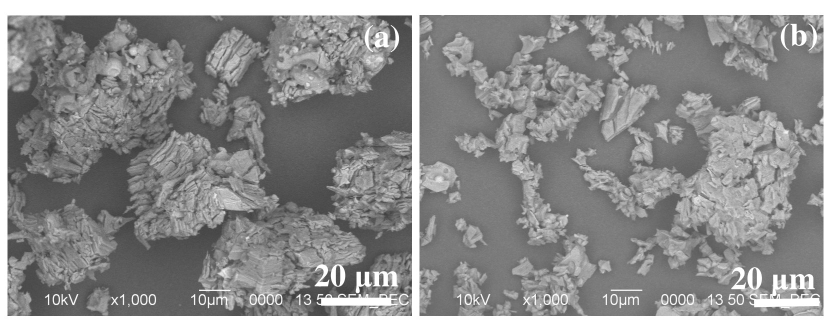

FIG 4: (a) and (b) BSE micrographs show the appearance of layer 3 in samples stored at room temperature and atmospheric pressure for three and five months, respectively.

https://doi.org/10.1017/S1431927610060824 Published online by Cambridge University Press