doi:10.1017/S1431927611010452
Microsc. Microanal. 17 (Suppl 2), 2011

Electron Microscopy Study of Co-Continuous Al-Fe/Al_2O_3 Composite Synthesized by Reactive Melt Penetration

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Co-continuous Al-7.5wt.%Fe/Al_2O_3 composites were produced by submersion of silica preforms in a molten Al-Fe alloy bath. The process, known as reactive metal penetration (RMP), proceeds analogous to the known *in situ* transformation of solid silica in pure molten Al [1] and occurs as follows: $3SiO_2$ (solid) + 4Al (liquid) → $2Al_2O_3$ (solid) + 3Si (in Al). If the preform is kept long enough in the molten metal bath the reaction continues until the silica is completely transformed into Al_2O_3 [2]. During the reactive metal penetration, the preform shape is preserved with only negligible volume change, but the microstructure and chemical composition are completely transformed when compared with the starting material. Consequently, by controlling the process parameters such as the metal bath composition, materials with various macroscopic properties can be obtained that can be tailored for applications in aerospace, automotive or the electronic industries. The purpose of this work is to investigate the micro- and nano-structure of Al-7.5wt.%Fe/Al_2O_3 composites using analytical SEM and TEM techniques, for a better understanding of their properties. The electron microscopy samples were prepared using a combination of metallographic and FIB techniques. The investigations were carried out using two JEOL instruments: a JIB-4500 Multi Beam SEM/FIB and a JEM 2100 S/TEM equipped with an EDAX^{TM} EDS system.

Figure 1 shows stereo optical micrographs of a cross-sectioned sample. Three distinct features can be observed: dark and bright columnar regions oriented perpendicular to the sample surface; a Y-shaped boundary symmetrically positioned relative to the edges and corners of the rectangular-shaped cross-section; and small dark precipitates. The nature of the columnar regions in Fig.1 was determined to be preferentially oriented Al_2O_3 and Al phases, as indicated by XRD investigation. SEM imaging showed that the Y-shaped boundary consists of agglomerates of interconnected voids. The nature of the third feature observed by optical microscopy, the small dark precipitates, was identified using high resolution SEM to be plate-like regions consisting of Al-Fe alloy, Al metal and Al_2O_3 ceramic embedded into a dual matrix consisting of interpenetrating Al metal and Al_2O_3 ceramic phases, Figure 2. The EDX maps in Fig. 2 also indicate the presence of small Si particles precipitated in the metal matrix. For nanoscale investigations samples from both the matrix and the plate-like regions were prepared using FIB. Figure 3 shows the results of an S/TEM investigation on the plate-like feature. Three different phases were observed and confirmed by EDS and XRD analysis: Al, Al_2O_3 and Fe_3Al. Two other distinct volumes having an Al-Fe-Si and an Al-Fe-Si-O chemical composition have been observed on the dark-field STEM micrograph. Work on clarifying the crystal structure of these two phases is in progress. The crystallinity of the Al_2O_3 ceramic phase was also investigated using electron diffraction techniques.

References
[1] M.C. Breslin *et al., Mat. Sci. Eng.* A195 (1995) 113.
[2] Y. Gao *et al., J. Mater. Sci.* 31 (1996) 4025.
[3] This research was supported by WP 09-016: "Center for Excellence in Advanced Materials Analysis", Ohio Third Frontier, Wright Projects Program. The use of EM and XRD Facilities within the College of STEM at YSU is gratefully acknowledged.

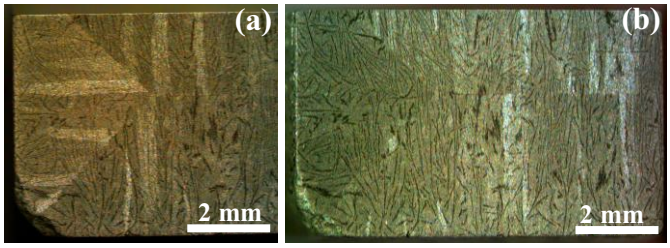

Figure 1: Stereo optical micrographs of a cross-sectioned Al-Fe transformed sample recorded under two different illumination conditions. Bright and dark columns indicate Al_2O_3 crystallographic domains with pronounced preferred orientation.

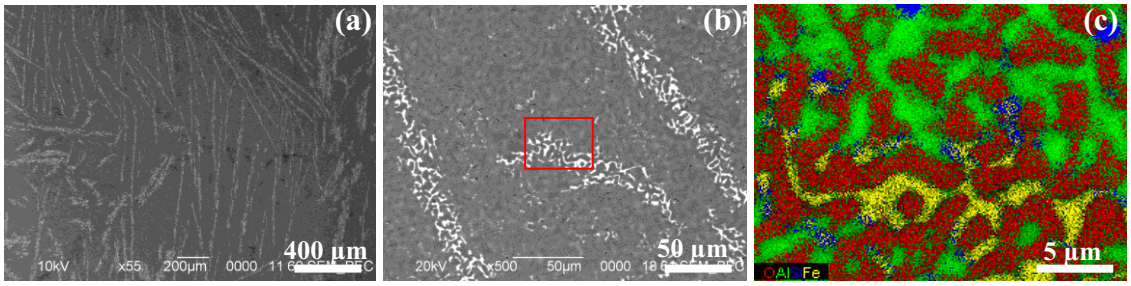

Figure 2: (a), (b) Backscattered electron micrographs of a cross-sectioned Al-7.5wt.%Fe/Al_2O_3 composite indicating the existance of Fe-rich volumes embeded into a Al/Al_2O_3 co-continous interpenetrating matrix. (c) EDS map of the area marked in (b).

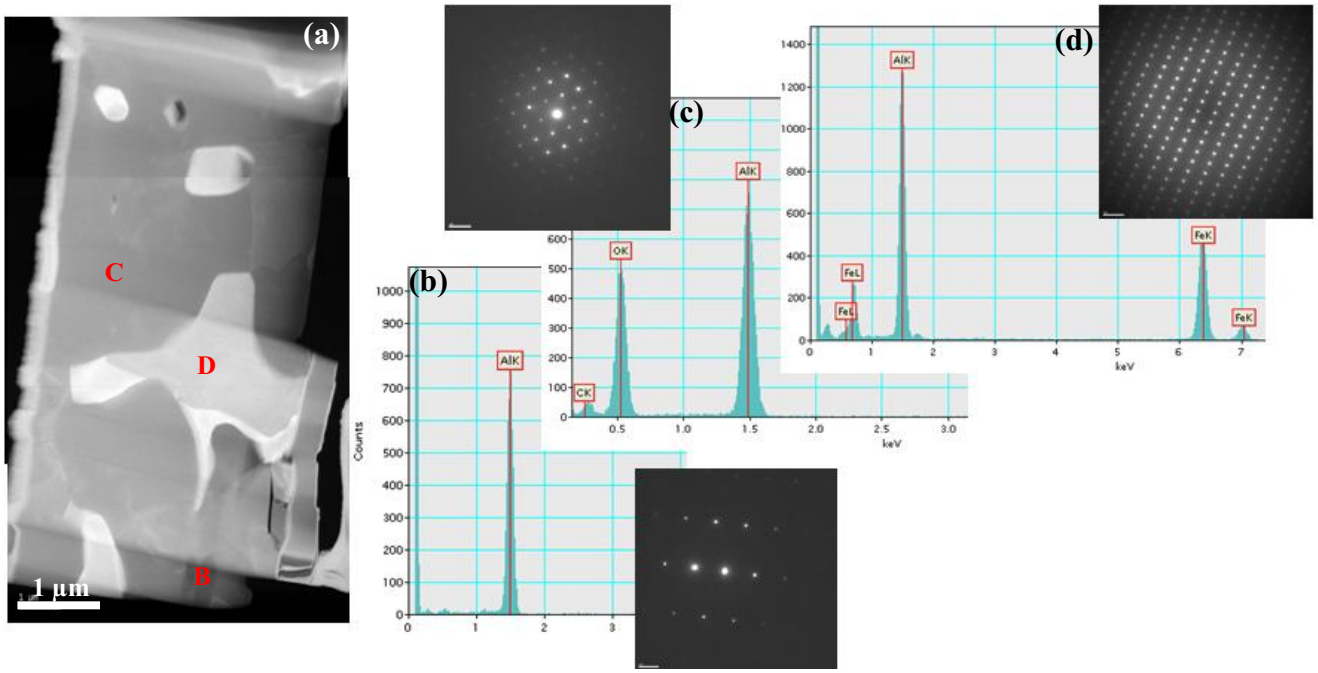

Figure 3: (a) Dark-field STEM micrograph of a sample prepared by FIB technique from the Fe-rich volume indicated in Fig. 2(b). (b) – (d) EDS spectra and corresponding SADPs from the areas B, C and D marked in (a).

https://doi.org/10.1017/S1431927611010452 Published online by Cambridge University Press