

EBSD Analysis of Solid-State Reactions Between Al₂O₃ and Cu₂O

J.K. Farrer* and M.M. Nowell**

*Department of Physics and Astronomy, Brigham Young University, N283 ESC, Provo UT 84602

**TexSEM Laboratories (TSL), 392 East 12300 South, Draper UT 84020

The crystallography of a material and especially crystal imperfections have a significant influence on the transport of structural elements and other kinetic processes that affect the behavior of solid-state reactions[1]. In some spinel-forming reactions, where the structural and chemical relationships are complex, other reaction products may be formed aside from the spinel. For example, in the Cu–Al–O system there are two possible copper oxide reactants: CuO, which is the room-temperature stable oxide, where the Cu ion has a +2 valence state, and Cu₂O, which is stable above 1020°C, and where the Cu ion has a +1 valence state. There are also two stable reaction products that can form in the Cu–Al–O system, namely, CuAl₂O₄ and CuAlO₂. In these more complex reaction couples, it has been shown that the reaction kinetics are influenced by the initial orientation of the alumina substrate [2]. A full understanding of the influence of the crystallography and structural imperfections upon the reaction, requires a complete characterization of the crystallography of the materials present.

The example shown here studies the reactions between copper oxide powder and bulk aluminum oxide, where more than one product phase is formed. Polycrystalline alumina and single-crystal sapphire substrates were pressed in contact with copper oxide powder and heated in air at 1100°C for 192 hours. Results from High-resolution backscattered electron (BSE) imaging as well as from X-ray energy dispersive spectrometry (XEDS) are combined with the electron backscatter diffraction (EBSD) data to obtain complementary chemical and crystallographic information.

Figure 1 is a BSE image of the resulting microstructure showing two product layers between the copper oxide and the polycrystalline alumina. All four layers were identified and confirmed using EDS and EBSD[3]. The materials were identified, from top to bottom in Figure 1, as Cu₂O (cubic), CuAl₂O₄ (cubic–spinel structure), CuAlO₂ (trigonal–delafossite structure), and Al₂O₃ (trigonal–corundum structure). Using an XEDS detector simultaneously with the EBSD detector[4], the reaction region of the (0001) single-crystal sapphire was analyzed. An image quality map in figure 2(a) reveals the grain morphology of the region as it relates to the advance of the reaction front. The phase map, in figure 2(b) indicates the presence and location of the two reaction product phases, and the inverse pole figure map (crystal orientation), in 2(c) shows the orientation of each grain in the region. The Cu₂O powder has sintered into large grains and is in contact with the CuAl₂O₄ layer. These two layers are of the same symmetry point group (cubic 0h m3m) and the lattice parameter of the CuAl₂O₄ phase is almost twice that of the Cu₂O ($a = 8.075\text{Å}$ and 4.25Å , respectively). The CuAlO₂ has formed in contact with the polycrystalline alumina. These two materials also share the same point group (trigonal D3d –3m). This study will focus on the reasons the product layers are not adjacent to the reactant with the most similar chemistry. It is also shown that the use of EBSD allows for a large enough sampling of the grains in the reaction region to understand the texture of the reaction layers, and the orientation of individual grains at the interface can be associated with the localized behavior of the interface.

[1] Schmalzried, H., *Chemical Kinetics of Solids*. 1995, VCH, Weinheim.

- [2] Susnitzky, D.W. and C.B. Carter, *J. of Mat. Res.*, 6 (1991) 1958.
 [3] Michael, J.R., in *Electron Backscatter Diffraction in Materials Science*, 2000, Kluwer Academic/Plenum Publishers, New York, 75.
 [4] Nowell, M.M. and S.I. Wright, *J. of Microsc.*, 213 (2004) 296.

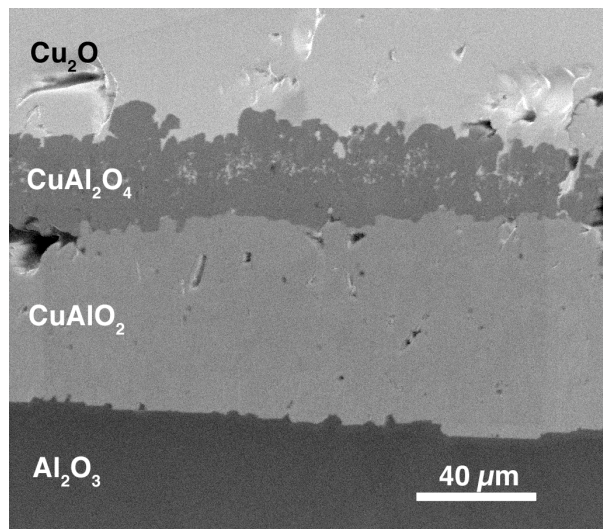


Figure 1. Back-scattered electron image of the resulting microstructure after annealing Cu_2O powder in contact with polycrystalline alumina in air at 1100°C for 192 hours. The image shows the two reaction product layers between the copper oxide and the alumina.

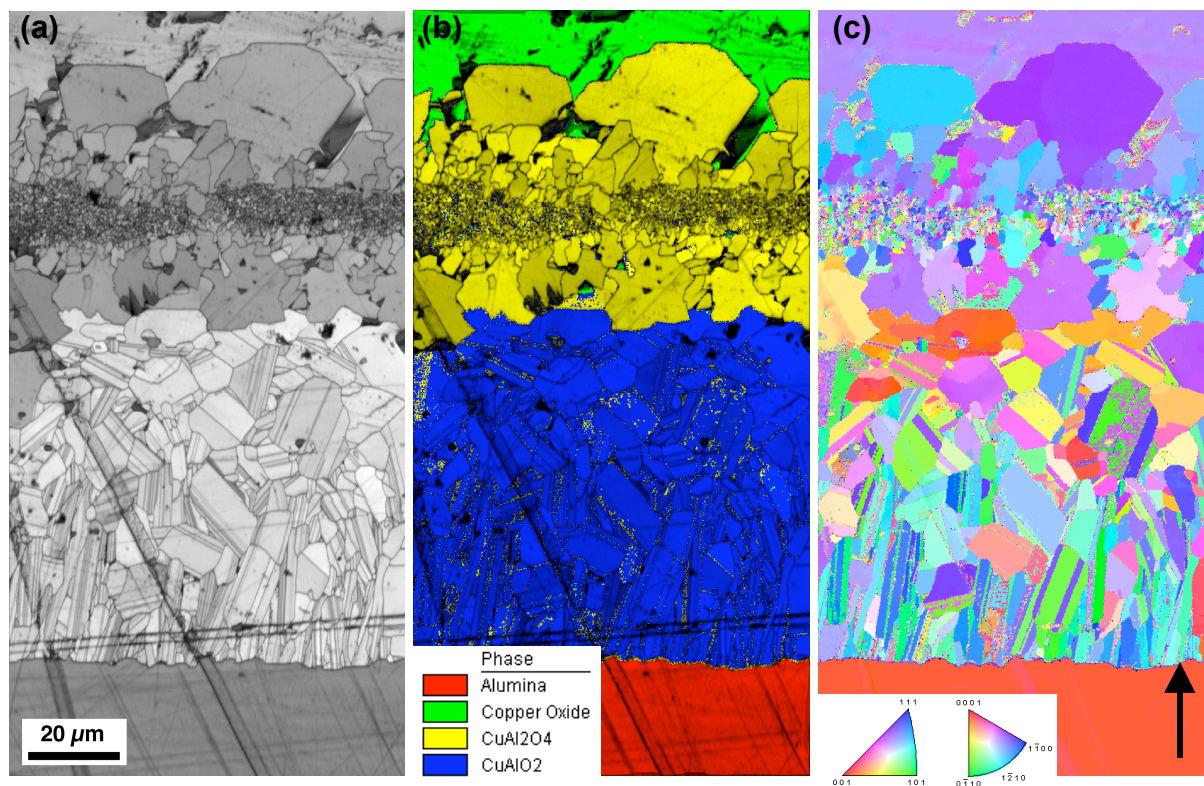


Figure 2. (a) An image quality map of the reaction region of the (0001) single-crystal sapphire, revealing the grain morphology of the region as it relates to the advance of the reaction front. (b) A phase map of the same area indicates the presence and location of the two reaction product phases, and (c) the inverse pole figure map shows, by color, the crystallographic direction of the material that is parallel to the arrow in the image, which is normal to the original reaction interface.