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Experimental and Theoretical STEM X-Ray Composition Images of Cu-Al_2O_3 Nano-Scale Composites Prepared by In-Situ Reduction

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It has been experienced that the physical and mechanical properties of particle-dispersed metal-ceramic composites are improved significantly if the dispersion is homogeneous and the particle sizes are in nano-scale. Particularly, the uniform dispersion of Al_2O_3 particles in a copper matrix enables the use of these materials in high temperature applications. In the present work, the Cu-Al_2O_3 nanocomposites have been prepared by a chemical route [1]. Microstructural characterization has been carried out by means of conventional and scanning transmission electron microscopy (CTEM and STEM). A Jeol 2010 TEM and a VG603 FEG STEM were used, the instruments being operated, respectively, at 200 kV and 300 kV. TEM samples were prepared by mechanically polishing 3 mm disks removed from the bulk down to about 200 μm, dimple ground to about 10 μm and, finally, ion beam milled (PIPS) with argon source at 4 kV.

Previous results [1] by conventional TEM have indicated that the microstructure of the Cu-Al_2O_3 (0.5 wt%) nanocomposite is composed of a Cu matrix and a uniform dispersion of Al_2O_3 particles ranging from 5/10 to 60/70nm. Also, there is a detectable reaction layer at the particle/matrix interface, suggesting the formation of a third phase, which is likely to be $CuAlO_2$ and/or $CuAl_2O_4$ [1]. Figure 1 presents the X-Ray STEM elemental mapping of Cu, Al and O of the Cu-Al_2O_3 (0.5 wt%) nanocomposite. The composition images as well as the bright field (BF) and dark field (DF) images show an approximately rounded Al_2O_3 nanoparticle inside the Cu matrix. It should also be mentioned that these regions are very small and extremely thin, making it virtually impossible to obtain information through convergent beam electron diffraction.

Due to the curvature associated with the Al_2O_3 particle, the metal/ceramic interface will never appear as a sharp interface in STEM composition map images. In order to assess this effect, a theoretical composition image of a spherical particle contained inside a matrix has been calculated, as shown in figures 2a and 2b. If either situation 1 or situation 2 (Fig. 2a) occurs during scanning of the electron beam, then it is inevitable to detect Cu, Al and O at the same scanning point at the particle/matrix interface. Fig. 2b presents a simulated composition image of quarter of a second phase particle inside a matrix, according to the “situation 2” described in Fig 1. The particle is actually imaged as if there is a smooth decrease of composition of second phase from the center of the particle towards the matrix. This simulation considers a beam diameter of 1.6 nm, but does not take into consideration any beam broadening inside the specimen. Further studies are necessary in order to compare truly quantitative experimental and theoretical images and therefore assess the composition of the third phase reaction layers.

References

[1] M.S. Motta. PhD Thesis (2002) 71.
[2] This research is partially supported by CNPq, Brazil.

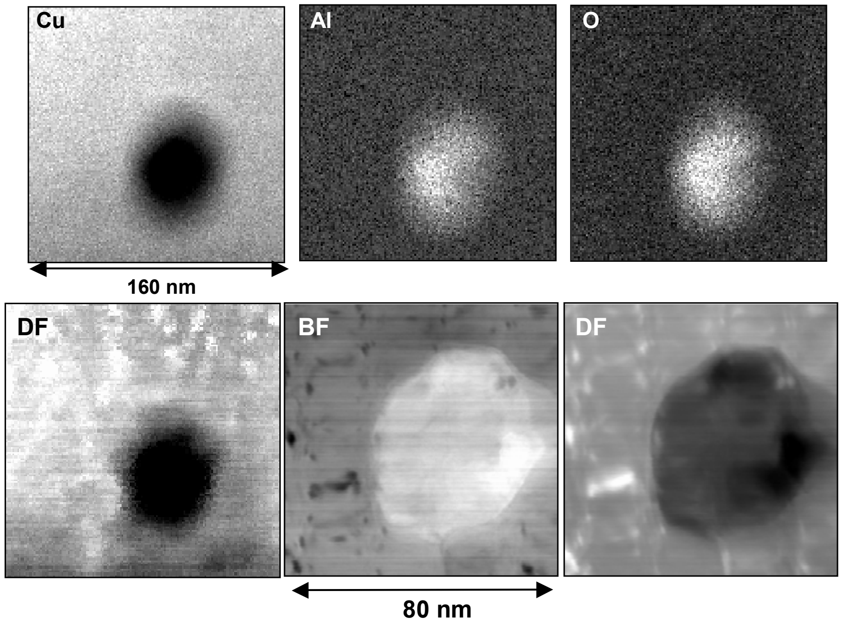
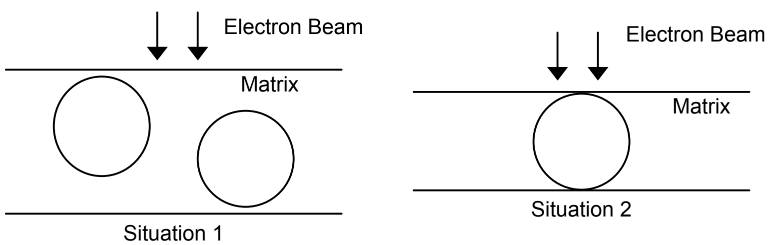


Fig. 1. Upper row: STEM composition maps for Cu, Al and O. Bottom row: from the left, a DF image at a magnification of 500,000x and a STEM BF/DF pair at 1000,000x.



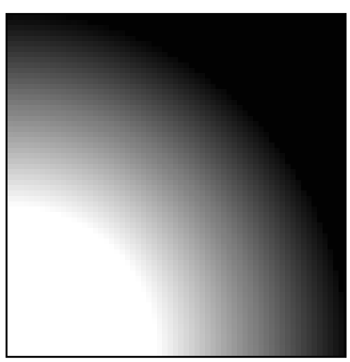


Fig 2. (2a) Schematic drawing of two possible situations for spherical second phase particles dispersed on a matrix. (2b) Simulated composition image of a second phase particle inside a matrix (situation 2).

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