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Microstructural Evolution of $CuNi/Al_2O_3$ Nanocomposites: from Nanoparticles to Cold Rolled and Annealed Ribbons.

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Nanocomposites ribbons of 89wt%Cu-10wt%Ni-1%wtAl_2O_3 were produced by chemical route synthesis of nanoparticles and thermomechanical processing. The nanoparticles synthesis procedure is reported elsewhere [1]. While the metallic constituent of CuNi is a solid solution, the ceramic Al_2O_3 are dispersed nanoparticles. The present study reports the microstructural evolution of different steps of the processing: from nanoparticles to cold pressed pellets and, finally, ribbons. The final products of CuNi-Al_2O_3 composites were obtained after cold rolling the pellets down to ribbons reaching 40%, 60% and 80% reductions in thickness.

Overall microstructural evolution observations were conducted by scanning electron microscopy using FEI SEM/FIB Helios instrument under 5KV to 20KV voltage and low emission current. Detailed microstructural characterizations and Al_2O_3 particles distribution analysis was performed by transmission electron microscopy (TEM/STEM) using a FEI Tecnai F20 instruments in diffraction and phase contrast modes. SEM specimens were prepared by flat polishing in a Minimet and, finally, submitted to Ga^+ ion polishing in the SEM/FIB. Electron transparent samples were prepared by conventional double jet electropolishing of thin foils and, specifically, selected lamellae prepared by Focus Ion Beam (FIB) using FEI Strata 205 and, finally, thinned in Fischione's 1040 NanoMill system.

A typical STEM bright field (BF) image of the nanoparticles with size ranging from 20 to 100 nm is shown Figure 1(a). The SEM image of Figure 1(b) shows the microstructure of a cold pressed pellet produced from this powder. In Figure 1(c), it is shown a SEM-FIB microstructure of a 80% cold-rolled ribbon follow by annealing at 600ºC for 30 minutes. A fully recrystallized microstructure has been generated. It is clear, however, than such high amount of plastic deformation has promoted not only recrystallization but also grain growth. While some regions in this micrograph, as market by R, represent a fully recrystallized structure with average grain size of 100 to 500 nm, the central region of this micrograph, market by G, exhibit significant grain growth, reaching 10 μm grain size. One can argue that the Al_2O_3 nanoparticles have not been fully effective in limiting the grain growth. In other words, in regions of the ribbon where Al_2O3 is present grain growth is limited and the opposite is true in regions where Al_2O_3 particles are absent. In fact, STEM analysis has proved to be effective in elucidating this finding. In Figure 1(d), a STEM dark field (DF) image, it is observed the microstructure of this ribbon with a defined grain size of about 200 to 500 nm where an alignment of Al_2O_3 nanoparticles is observed across this image. A detail observation of the interface, between Al_2O_3 and the metallic matrix is shown in the HRTEM image of Figure 1(d). The STEM high angle annular dark field (HAADF) image of Figure 1(f) it is revealed, in higher contrast such alignment of Al_2O3 particles, some pinning the metallic grain boundaries. The EDS spectrum of the boxed area show in the same figure corroborates that such particles are in fact Al_2O_3. The nature of the other homogenous particles observed with finer contrast in some grains, probably arising from FIB radiation damage, is currently under investigation.

References:
[1] Ramos, M.I et al. Metall Mater Trans A, V48, (2017).

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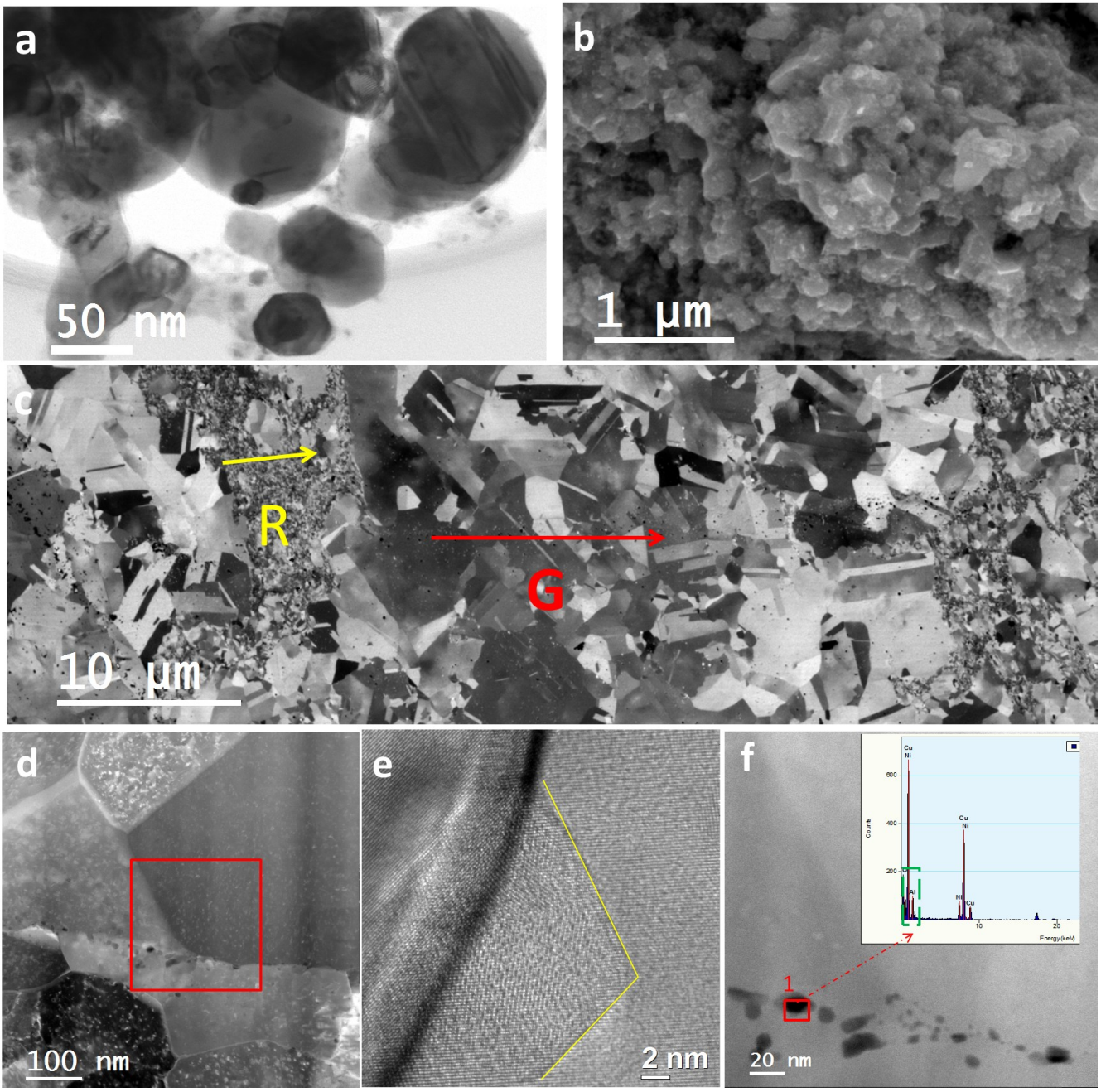

Figure1. a) STEM BF image of the synthesized CuNi-Al_2O_3 nanoparticles; **b)** SEM image of fractured surface of a cold pressed pellet; **c)** SEM image of a 80% cold rolled ribbon annealed at 600ºC for 30 min **d)** STEM DF image at of cold rolled and annealed composite showing the CuNi matrix and Al_2O_3 nanoparticles **e)** HRTEM a faceted Al_2O_3 nanoparticles at a CuNi grain boundary of figure 1 (d); **f)** HAADF showing Al_2O_3 with EDS spectrum nanoparticles.

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