

Microstructural Characterization of NiCoAlFeCuCr High-Entropy Alloys.

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High-entropy alloys (HEA's) contain at least five principal elements in equiatomic ratio or near-equiatomic ratio [1]. They usually form simple FCC and BCC structures, and even amorphous phases [2]. Excellent physical and chemical properties are reported for HEA's, like mechanical resistance, thermal stability and corrosion resistance to mention some [3]. These properties can be affected by the number, contents, and nature of the component elements. The effect of milling time in a multi-component quinary (NiCoAlFeCu) and hexanary (NiCoAlFeCuCr) systems was studied by scanning electron microscopy (SEM), X-ray diffraction (XRD), and transmission electron microscopy (TEM).

Raw materials were commercial Nickel, Cobalt, Aluminum, Iron, Copper and Chromium, pure elemental powders supplied by Sigma-Aldrich. Equiatomic mixtures were mechanically alloyed to form quinary (NiCoAlFeCu) and hexanary (NiCoAlFeCuCr) systems. The milling process was carried out from 0 to 30 h in a high energy shaker ball mill (SPEX-8000M). An argon atmosphere was used to avoid oxidation and methanol (1 ml) was employed as the control agent process. The milling ball-to-powder weight ratio was set at ~5:1.

In figure 1, it is observed a characteristic lamellar structure at 10 h of mechanical alloying. With increasing milling time, the layer spacing becomes finest, conducting to homogeneous microstructure and chemical distribution.

The images in Fig. 2 show non-homogeneous microstructures formed at 10 h of milling (the lowest milling time). In the NiCoAlFeCu alloy were observed three well-defined phases: *A* is a Co-rich phase, *B* is a Ni-rich phase and *C* is an apparent hexanary solid solution. The NiCoAlFeCuCr alloy also presents the three same phases mentioned before, and just chrome zones.

From the analyses done in several system (do not shown), was observed that Fe is a BCC solid solution former, Cu is a dominant element and favor the FCC solid formation which present structural characteristics similar to Cu solid solution. However, Cr apparently is Ni solid solution stabilizer.

On the evidence of mechanically alloyed systems XRD spectra, elemental characteristic peaks disappear as the milling time increases while a solid solution peaks appear.

TEM samples were prepared by powders deposition over a copper grid. No evidence of amorphous phases was observed. Crystal size observed in TEM samples were a little different with those values calculated from XRD spectra. During TEM observations were found small concentration gradients in different particles. However because the particle size is in the range of 10 to 30 microns, it is not considered important.

At this moment, deep microstructural characterization is carrying out to know the effect of different elements additions in the multi-component systems evolution.

References.

- [1] Y.L. Chen, Y.H. Hu, C.W. Tsai, C.A. Hsieh, S.W. Kao, J.W. Yeh, T.S. Chin and S.K. Chen, *Alloys Compd* **477** (2008) 696-705.
- [2] T.T. Shun and Y.C. Du, *Alloys Compd* **479** (2009) 157-160.
- [3] C.P. Lee, Y.Y. Chen, C.Y. Hsu, J.W. Yeh and H.C. Shih, *J. Electrochem. Soc.* **154** (2007) C424-C430.

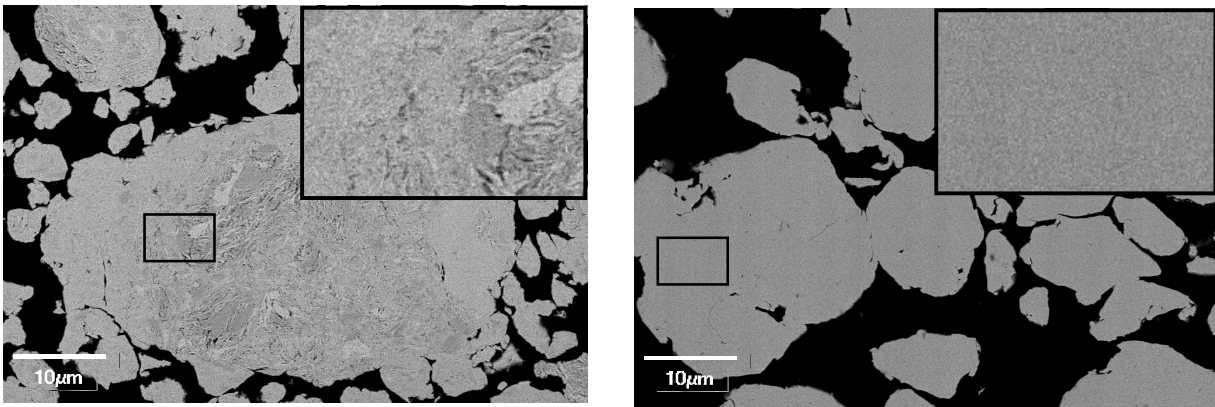


Figure 1. Microstructural evolution during milling of NiCoAlFeCuCr powders at different milling time (left at 10h and right at 30 h).

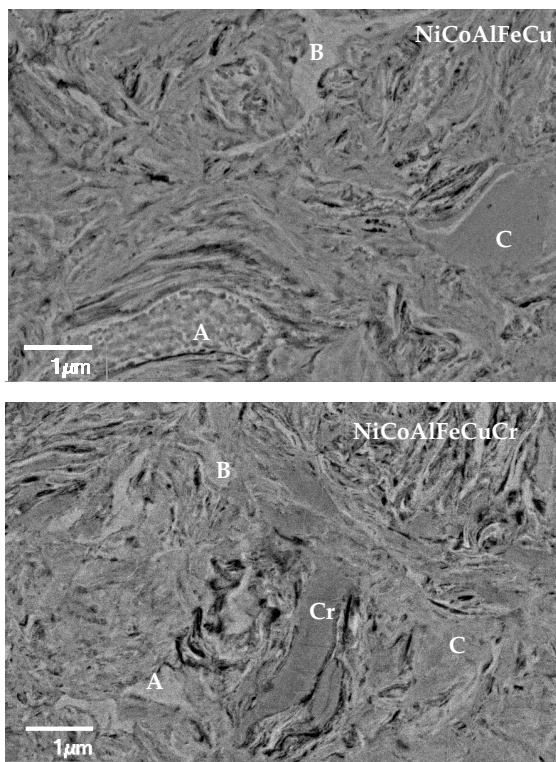


Figure 2. Scanning electron microscopy images of alloys microstructure at 10 h of milling time.

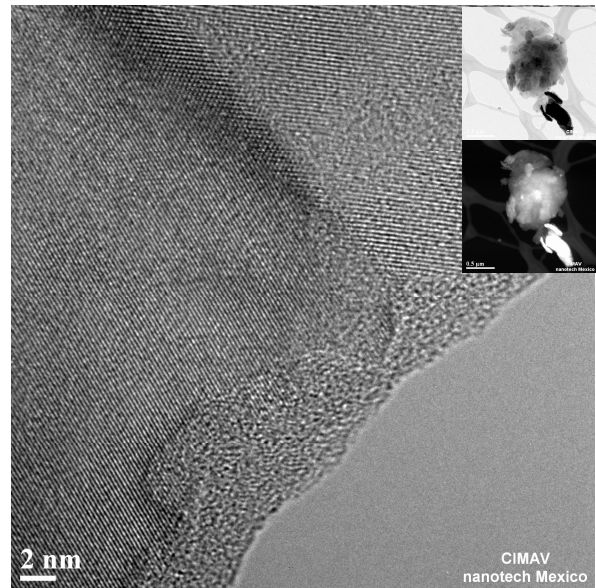


Figure 3. Transmission electron microscopy image of NiCoAlFeCuCr alloy with 30h of milling time.

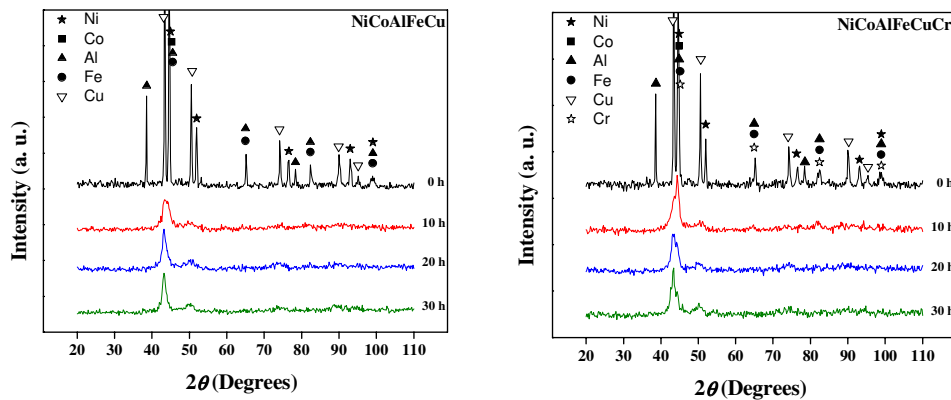


Figure 4. XRD spectra of the equiatomic quinary NiCoMoAlFe, and hexanary NiCoMoAlFeCu systems as a function of milling time.