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The Microstructure of NiCoAlFeCuCr Multi-Component Systems synthesized by Mechanical Alloying

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Abstract.

Series of binary to senary alloys (Ni, Co, Al, Fe, Cu, Cr) were produced by mechanical alloying. A different answer is observed in all systems studied. Formation of an FCC solid solution is observed in all systems studied. For quaternary to senary systems the presence of a mixture of FCC and BCC solid solution is detected for the shorter milling times. Fe and Cr are promoters of BCC solid solution and present an apparent low dissolution rate in FCC phase. FCC solid solution is stable at least until 1000°C. Heated products present a mixture of FCC and BCC solid solutions with lattice parameters very close to those found in the milled products.µHV).

Introduction.

Within traditional alloy concept, most alloys are composed of one principal metal element. Multi-component systems are characterized by a high entropy and ability to form amorphous phases. High entropy alloys (HEAs) are a new era of materials that consist of various major alloying elements; the system selected in each case is different, and each additional element changes the behavior of the final alloy. The HEAs are quite simple to analyze and control because they tend to form simple solid solution phases, mainly of FCC and BCC structures. They have numerous beneficial mechanical, magnetic, and electrochemical characteristics. These systems can be processed by different routes: conventional casting, thin film deposition and milling process. The mechanical alloying (MA) process has been widely recognized as an alternative route for the formation of nanocrystalline materials, with unusual properties. In this investigation, a multi-component system formed by Ni-Co-Al-Fe-Cu-Cr is studied from the binary to senary alloy; the effect of each element and milling time is reported and discussed..

Experimental.

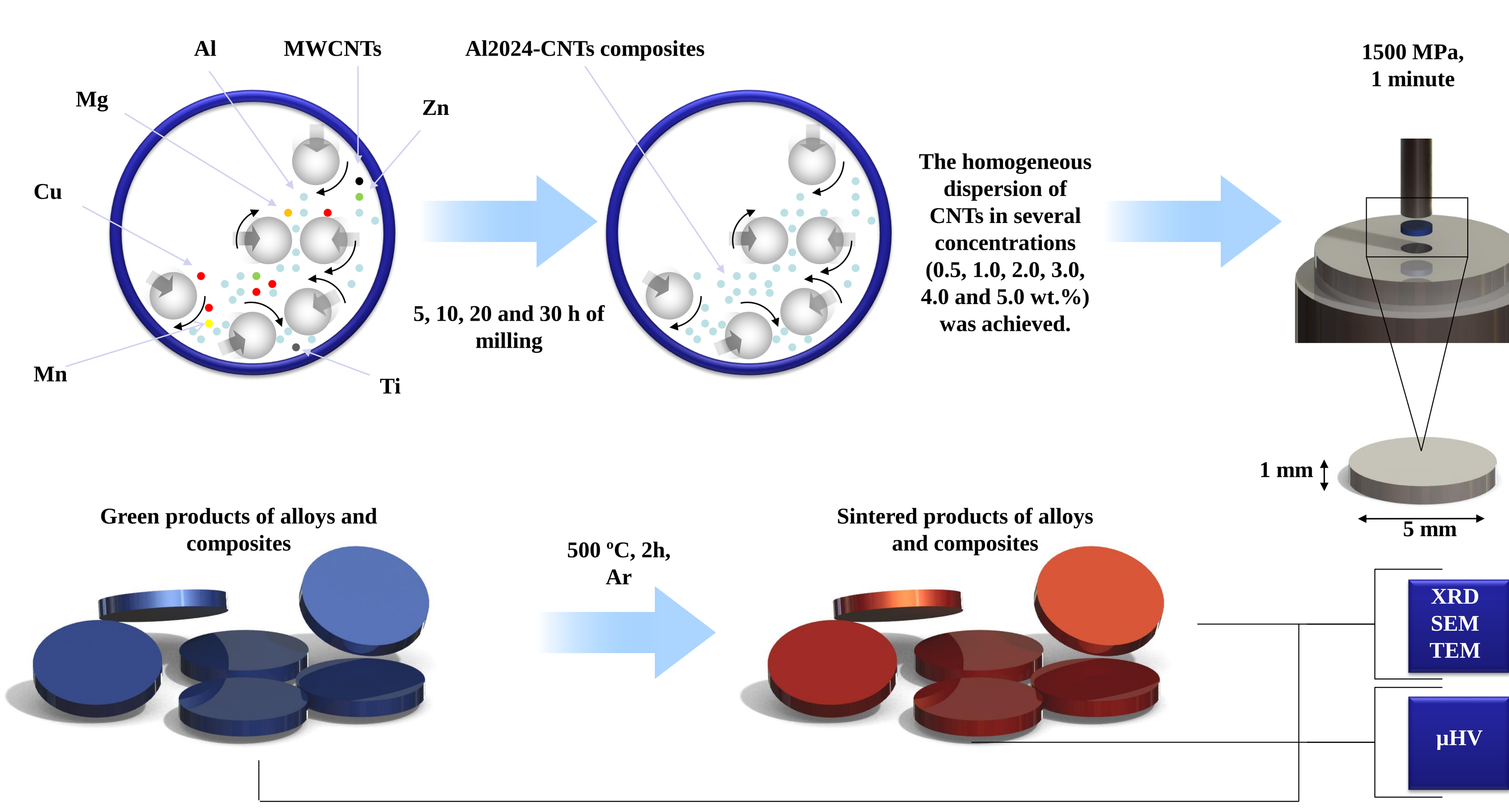
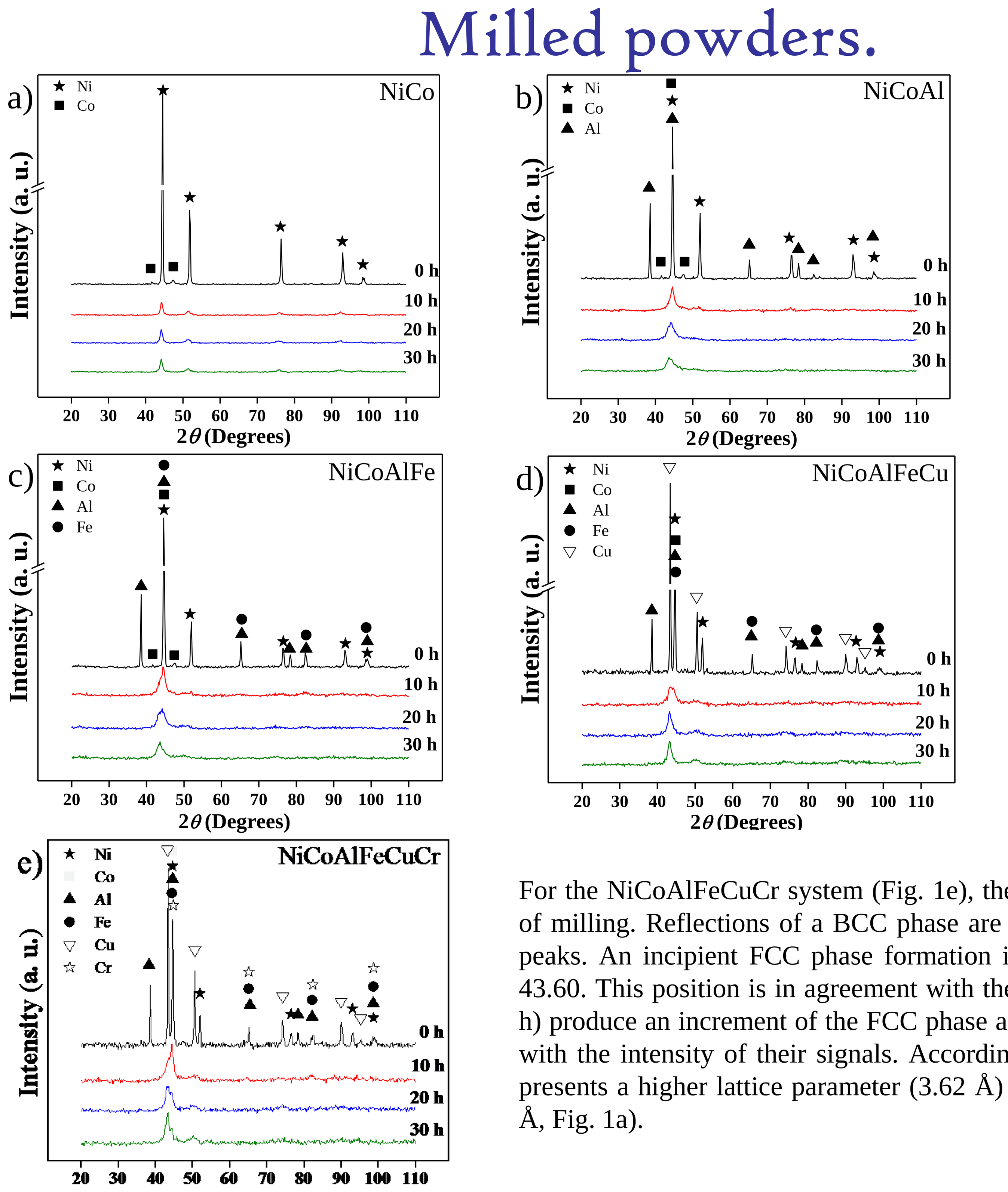


Figure 2. Schematic process of the route followed in the production of Al2024-CNTs. Powders are milled under argon atmosphere, cold consolidated and then sintered in a pressure-less argon atmosphere during 2h. Products milled are designated according to the milling time: A (5 h), B (10 h), C (20 h) and D (30 h). Subscripts indicate the CNTs content: 00 (0.0%), 05 (0.5%), 10 (1.0%), 20 (2.0%), 30 (3.0%), 40 (4.0%) and 50 (5.0%).

Milled powders.



The XRD patterns as a function of the milling time for the NiCoAl system are presented in figure 1b. After 10 h of milling, Co and Al characteristic reflections disappear, remaining broad and small Ni-type peaks; no evident shifting in these peaks was observed. After 20 h of milling, it is observed the incipient shoulder formation on broad principal Ni-type peak and secondary peaks almost disappear. After 30 h of milling, only the most intense Ni-type peak can be seen and an apparent mixture of two solid solutions is evidenced, both corresponding to a FCC structure in agreement with the secondary reflections position; in addition, the Ni-type reflection shifts slightly to lower angles.

For the NiCoAlFeCuCr system (Fig. 1e), there is no evidence of Co and Al peaks after 10 h of milling. Reflections of a BCC phase are observed, which are overlapped with Cr and Fe peaks. An incipient FCC phase formation is observed by the presence of a signal at 2q = 43.60. This position is in agreement with the last spectra (Fig. 1d). Longer milling times (20 h) produce an increment of the FCC phase and a decrement of the BCC phase, in accordance with the intensity of their signals. According to the peaks position, this FCC solid solution presents a higher lattice parameter (3.62 Å) than that found in the inary Ni-Co system (3.54 Å, Fig. 1a).

Figure 1. XRD spectra of the equiatomic (a) binary NiCo, (b) ternary NiCoAl, (c) quaternary NiCoAlFe, (d) quinary NiCoAlFeCu, and (e) senary NiCoAlFeCuCr systems as a function of the milling time.

For all the series, the crystallite size is refined and the lattice strain increases as the milling time increases. This is attributed to the mechanical deformation introduced in the powders; severe plastic deformation can lead to variation in crystallite size and the accumulation of internal stresses. Due to the precision achieved in our evaluations (5-15 Å), difference in crystallite size between samples B, C, D and E (Fig. 2) could be negligible.

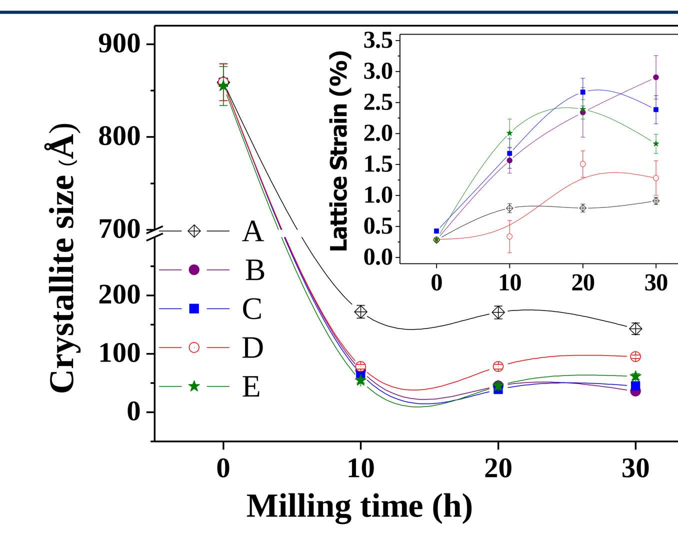


Figure 2. Crystallite size and lattice strain versus milling time calculated from main Ni-type peak.

System	Milling Time (h)	at. % Ni	at. % Co	at. % Al	at. % Fe	at. % Cu	at. % Cr
Binary (A)	0	*50.0	*50.0	0.0	0.0	0.0	0.0
	10	50.9	49.1	0.0	0.0	0.0	0.0
	20	51.5	48.5	0.0	0.0	0.0	0.0
	30	51.0	49.0	0.0	0.0	0.0	0.0
Ternary (J)	0	*33.3	*33.3	*33.3	0.0	0.0	0.0
	10	36.3	35.7	28.0	0.0	0.0	0.0
	20	35.3	35.8	28.9	0.0	0.0	0.0
	30	35.6	35.3	29.1	0.0	0.0	0.0
Quaternary (K)	0	*25.0	*25.0	*25.0	*25.0	0.0	0.0
	10	25.1	27.6	22.0	25.3	0.0	0.0
	20	24.6	27.8	22.3	25.3	0.0	0.0
	30	23.3	27.5	23.3	25.9	0.0	0.0
Quinary (M)	0	*20.0	*20.0	*20.0	*20.0	*20.0	0.0
	10	19.2	23.2	16.8	21.6	19.2	0.0
	20	21.7	23.6	9.1	21.9	23.8	0.0
	30	21.5	23.5	9.6	21.8	23.5	0.0
Senary (O)	0	*16.6	*16.6	*16.6	*16.6	*16.6	16.6
	10	19.2	19.0	8.3	17.7	21.0	14.8
	20	18.3	18.5	8.9	18.0	20.1	16.2
	30	21.1	21.4	8.8	19.2	11.4	18.0

* Nominal values.

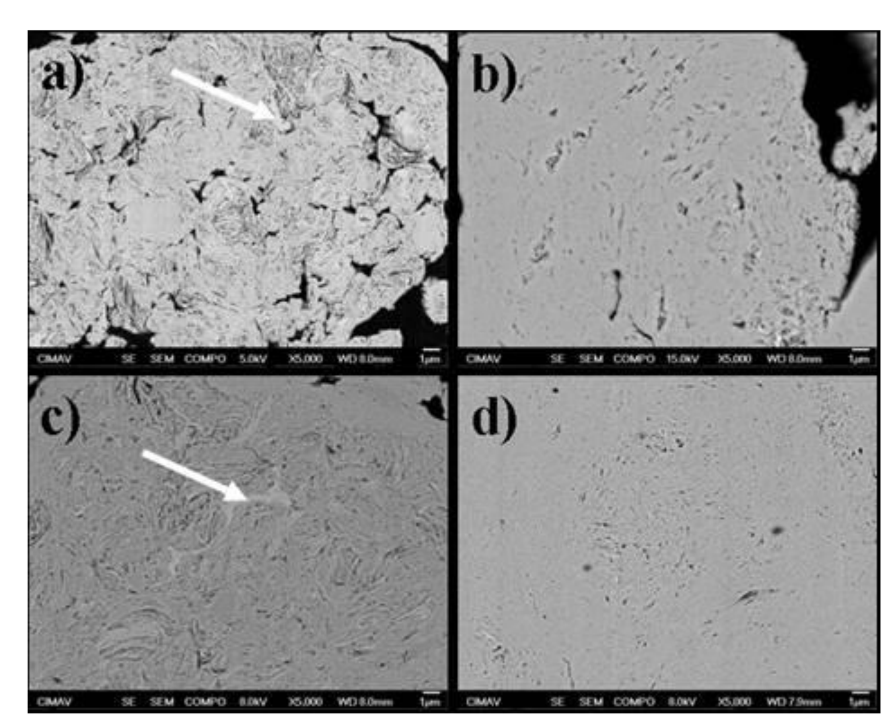


Figure 3 shows the representative cross section microstructures of quaternary and quinary alloys after 10 and 30 h of milling. For lower milling times a quasi-lamellar structure is observed, which is typical in the early stages of MA for ductile components With further milling, a more uniform microstructure (figures 3b and 3d) is observed, but some bright particles are still noted.

Figure 3. Powder cross section backscattered electron images of the quaternary NiCoAlFe alloy after (a) 10 h and (b) 30 h and the quinary NiCoAlFeCu alloy after (c) 10 h and (d) 30 h of milling.

Furthermore, milled products were thermally treated to evaluate the structural stability; figure 4 shows XRD spectra from the quinary system after a thermal cycle (1000°C). A mixture of phases (FCC and BCC solid solutions) is observed after this treatment. The FCC phase formed during the milling process is stable at least until 1000°C. Crystallization or growing of the BCC solid solution during the heating was observed. Milling time has no effect on the crystallized phases: the XRD spectra of the samples milled during 10, 20 and 30 h are practically indistinct.

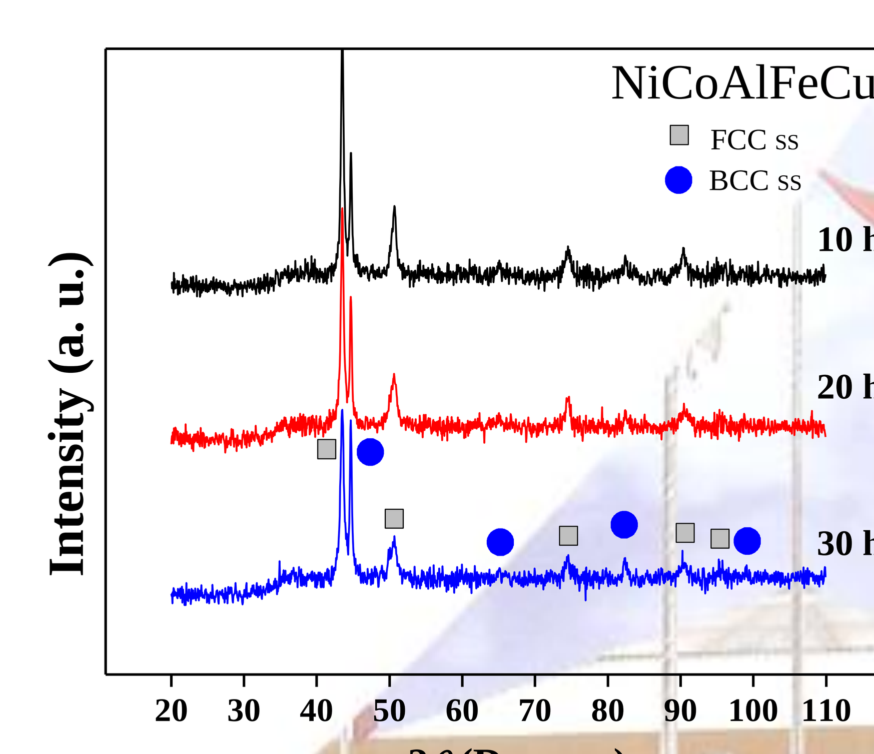


Fig. 4.- XRD spectra of the equiatomic NiCoAlFeCu system after milling for selected time and heated to 1000°C.

Conclusions.

The binary to senary equiatomic high entropy alloys in Ni-Co-Al-Fe-Cu-Cr system have been successfully synthesized by mechanical alloying in a high energy ball mill. Formation of an FCC solid solution structure after longer milling time was observed in binary and senary systems. Presence of Fe and Cr favors the formation of BCC phase, but only for the shorter milling times. FCC phase shown a thermal stability at least until 1000°C. However, there exist a BCC phase after a thermal treatment.