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Zinc Addition on AlCoFeNi Multicomponent System Produced by Mechanical Alloying, Microstructural and Morphological Effect

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High entropy alloys (HEAs) are a recent class of metallic materials and a research topic in the field of materials science because of their attractive properties, among which are high mechanical resistance, high corrosion resistance, and high thermal stability. HEAs have unusual chemistry, outside conventional metallurgy, since they contain at least five elements in concentrations of between 5 and 35%, and they tend to form simple BCC and/or FCC type solutions [1]. Conventional metallurgy indicates that for alloys development, one or two elements are selected that provide the main characteristics and attributes, and elements secondary in smaller quantities are added to modify the microstructure and definitive properties [2]. Different authors have reported how each element has a direct effect on different HEAs physical and chemical properties. HEAs most studied contain transition metal elements such as Ni, Co, and Fe. There are few reports on the effects of elements such as Zn, Si, Sn, or Li on the microstructure and properties of HEAs [3]. Among the most commonly used methods to produce alloys in powder form is mechanical alloying, a powerful solid-state technique capable of obtaining nanocrystalline materials with attractive properties. It is well known that alloys produced by mechanical alloying achieve a homogeneous chemical distribution and have a greater tendency to create solid solutions [4]. However, in the field of HEAs, new alloys synthesis is focused on other metallurgical techniques. The present work studies the effects of Zn addition on the microstructure of the equiatomic alloy AlCoFeNi by mechanical alloying.

Elemental powders of Al, Co, Fe, Ni, and Zn with a purity greater than 99.5% and a size of less than 75 μm, were used in this research for the synthesis by mechanical alloying of AlCoFeNi and AlCoFeNiZn equiatomic systems. The times of alloy were 5, 10, and 15 hours in a SPEX 8000M mill, using a vial and hardened steel balls as the grinding medium; a ratio weight 10:1 (ball: powder), an argon atmosphere used to prevent oxidation, and methanol as a process control agent. Microstructural and morphological evolution were analyzed by XRD (Bruker D8 Advance) and SEM (JEOL JSM-7401F). To determine crystallography and elemental composition after the mechanical alloying process, TEM (JEOL JEM 2200FS+CS) and EDS were used.

From the first 5 hours of mechanical alloying, the microstructural change suffered by the elemental powder mixtures is evident. In both alloys, the new signals show a widening and a decrease in intensity, resulting from the reduction of the grain size to a nanometric level and an increase in network microdeformations, a direct consequence of mechanical alloy synthesis. After 15 hours of grinding, the AlCoFeNi alloy showed two phases. One of the predominant FCC types has an approximate lattice parameter of 0.360 nm, which resembles a Fe, Ni type structure, and a second phase of the BCC type with less marked signals. AlCoFeNiZn showed a single phase of type BCC (Fig. 1). Thus, a phase

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refinement effect can be attributed to the presence of Zn within the alloy. The approximate lattice parameters of the AlCoFeNi and AlCoFeNiZn BCC structures are 0.287 and 0.289 nm respectively, suggesting that elements diffuse within the crystalline structure of iron, as iron has a lattice parameter similar to that of these BCC structures. Using SEM (Fig. 2 a-d), mechanical alloy powerful influence on material morphology could be observed. Initially, all powders show a uniform and regular surface, which is characteristic of each element. Due to the grinding media's constant impact on the initial mixture of elementary powders, the characteristic shape of each element is lost, giving rise to powders with irregular shapes and rough surfaces. This leads to the formation of a solid solution at the end of the process of mechanical alloying. The main morphological difference between the synthesized alloys lies in the final particle size. In the case of the AlCoFeNi alloy, sizes between 10 and 30 µm were observed, while for the AlCoFeNiZn alloy, sizes between 5 and 20 µm were seen. Smaller particle size indicates that the addition of zinc favors the fracture mechanism over that of cold welding within the mechanical alloying process. This is yet another zinc direct effect observed in the alloys investigated. TEM/EDS after mechanical grinding of both alloys indicates a homogeneous distribution of elements. The elemental compositions are very close to the equimolar, initial composition. Fig. 2 e-h shows dark field images and the corresponding SAED pattern of both alloys after 15 hours of grinding. It was possible to observe that the crystals that compose the different alloys had a size that was within the nanometric level, as indicated by the diffraction patterns in Fig. 1. After analyzing the SAED patterns of each alloy, it was possible to find signals of main crystallographic planes that matched with the different signals of crystalline structures obtained from powder X-ray diffraction patterns..

In this research, it was possible to synthesize an HEA from AlCoFeNi plus zinc addition as a fifth element, leading to the formation of a nanocrystalline phase BCC. In addition to a homogeneous elemental distribution near to the equiatomic ratio from which it started, two conclusions are obtained from this work. First, zinc promotes BCC phase formation. Second, zinc addition to the equimolar AlCoFeNi alloy reduces particle size after the mechanical alloying process.

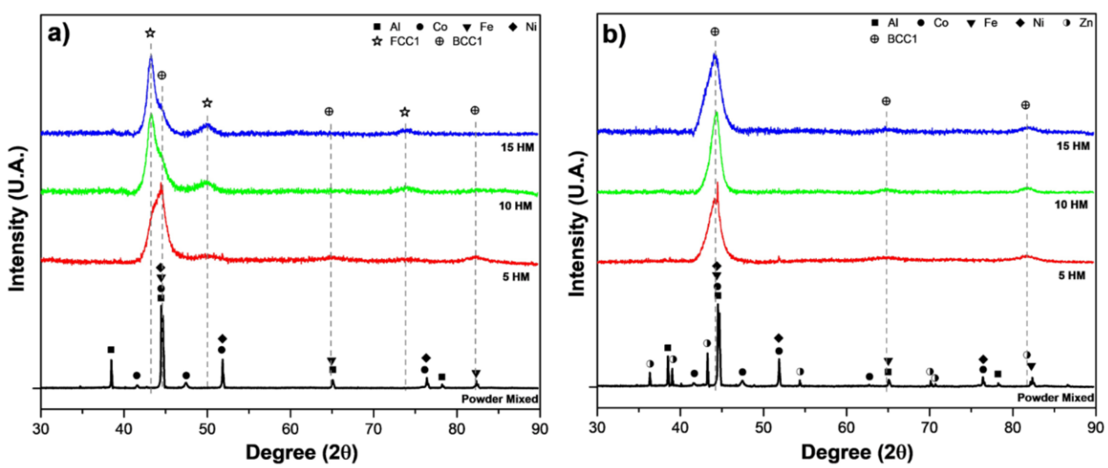

Figure. 1. X-ray diffraction patterns: a) AlCoFeNi and b) AlCoFeNiZn, different grinding conditions.

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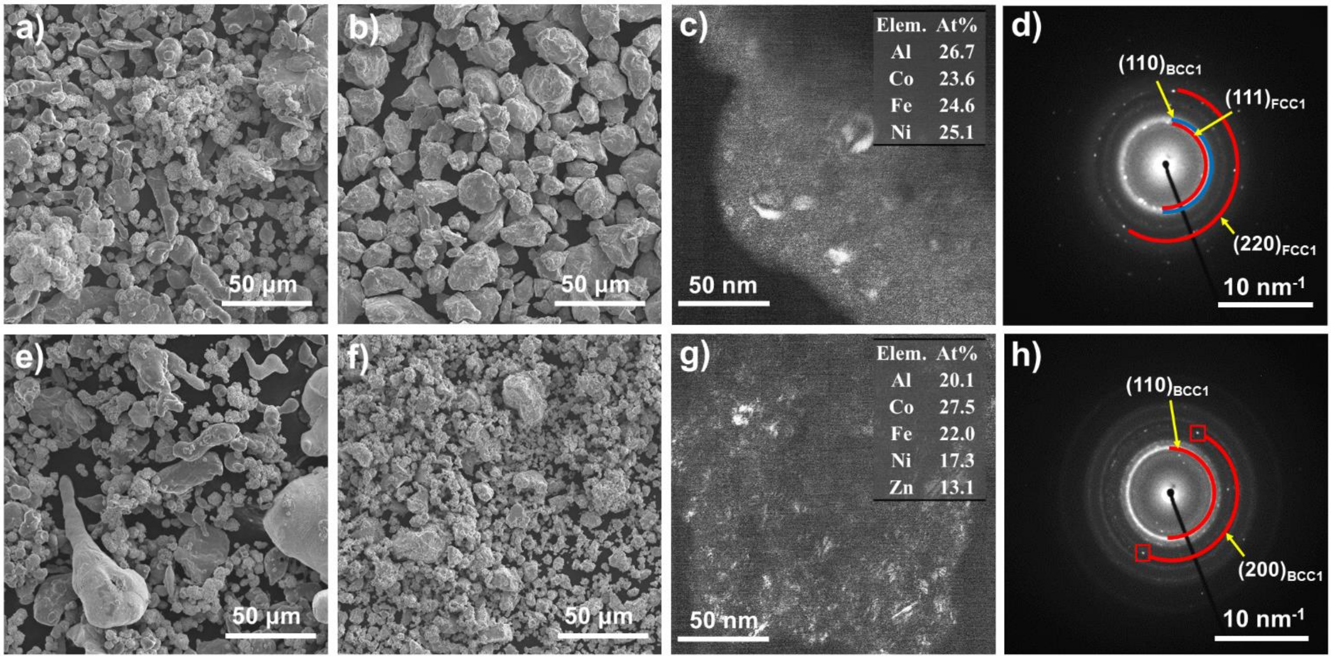

Figure. 2. a) SEM AlCoFeNi mix powder, **b)** SEM AlCoFeNi 15 hours grinding alloys, **c)** TEM/EDS AlCoFeNi particle, **d)** AlCoFeNi SAED patterns, **e)** SEM AlCoFeNiZn mix powder, **f)** SEM AlCoFeNiZn 15 hours grinding alloys, **g)** TEM/EDS AlCoFeNiZn particle, **h)** AlCoFeNiZn SAED patterns.

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