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Influence of Milling Time on Microstructure of AlCoCrMnMo High-entropy Alloy Produced by Mechanical Alloying

G.D. Avila-Rubio[1], M.A. Avila-Rubio[1], J.A. Baldenebro-López[1*], F.J. Baldenebro-López[1*]

[1.] Facultad de Ingeniería Mochis, Universidad Autónoma de Sinaloa. Prol. Ángel Flores y Fuente de Poseidón, S.N., 81223 Los Mochis, Sinaloa, México.
* Corresponding author: jesus.baldenebro@uas.edu.mx ; francisco.baldenebro@uas.edu.mx

High entropy alloys (HEAs), defined as alloys containing multiple elements in equiatomic or near equiatomic percentages, have aroused great interest in the fields of metallurgy and materials engineering due to their excellent properties, as well as their unique ability to form solid solutions (SS) with BCC and/or FCC simple structure crystals [1]. Among the various processing routes, it is becoming more and more common to find mechanical alloying (MA) as a route for the fabrication of HEAs. MA has most of the advantages of powder metallurgy techniques and can be used to significantly increase the solubility of HEAs [2]. However, MA is a complex process that requires the optimization of a number of parameters to achieve the desired microstructure [3]. In the present work, the influence of grinding time on the microstructure of the AlCoCrMnMo high-entropy alloy produced by mechanical alloying is evaluated.

The high entropy equiatomic AlCoCrMnMo alloy was synthesized using elemental powders with a purity greater than 99.5%. The powders were mixed and then mechanically alloyed for 5, 10 and 15 h in a high energy mill (SPEX 8000M), using a vial and hardened steel balls as the grinding medium with a 5:1 balls/powder ratio. To avoid excessive oxidation of the material, an argon atmosphere was used, and methanol was added as a process control agent. Microstructural changes from different grinding times were studied by XRD (Bruker D8 Advance) and SEM (LEICA Stereoscan 440).

Fig. 1 shows the morphological evolution of HEA-AlCoCrMnMo. It is possible to identify the different shapes, sizes, and textures of the elements that compose it. These characteristics are of great importance since they influence the evolution of the MA process. The chemical composition of the final powders is similar to the composition of the starting powders with relative errors in the order of ±6%, which was corroborated by SEM/EDS analysis. Powder size was the main difference between the different grinding times. SEM analysis indicated that at 5 h of grinding there is a reduction in the starting particle size, while at 10 h of grinding a larger particle size is observed, and finally at 15 h there is again a reduction in particle size. Therefore, the material transfer process was successful. Undoubtedly, the formation of SS powder was promoted by the continuous fracturing, welding, and re-fracturing of the material during mechanical milling.

Fig. 2 shows the XRD patterns of HEA-AlCoCrMnMo at different milling times. After 5 h, the peaks alluding to elemental powders disappear. In contrast, peaks appear with less intensity but with a more pronounced width. This phenomenon indicates that atoms of the elements are introduced into the crystal structure formed during the MA. After 10 and 15 h of milling, only the Mo-type BCC and Cr-type BCC phases remain. However, after 15 h of milling, some amorphization was observed in the reflections emitted by the alloy. The patterns suggest that the presence of Mo favors the formation of SS with BCC structures. This is mainly due to two factors. First, Mo has a larger and not-so-compact lattice parameter that facilitates the incorporation of other elements within its unit cell. Second, the low concentration of

valence electrons favors the formation of BCC structures over FCC. A summary of the changes in microstructural parameters is shown in Table 1. It can be observed that the crystallite size of SS with a BCC structure decreases with increasing grinding time, and also the microdeformations are increased due to the mechanical grinding effect.

HEA-AlCoCrMnMo was successfully synthesized by mechanical alloying. The effect of milling time on the alloy was studied. After 10 h of milling, the alloy did not show any substantial changes in its microstructure. Increased milling time produces microstructures with smaller crystal sizes and larger microstrains.

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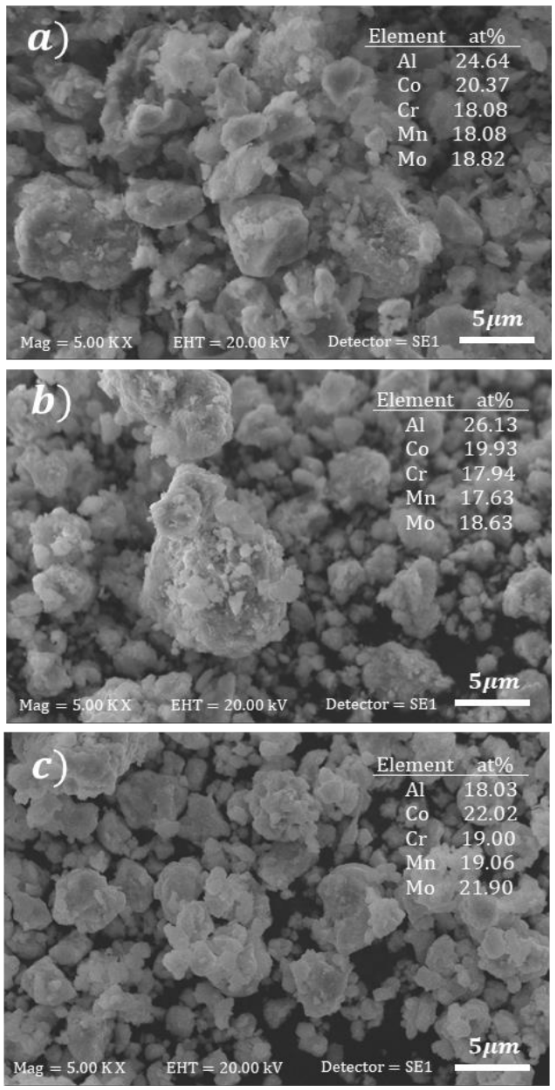

Fig 1. Powders mixed SEM micrographics HEA-AlCoCrMnMo after different milling time: a) 5 h, b) 10 h, c) 15 h.

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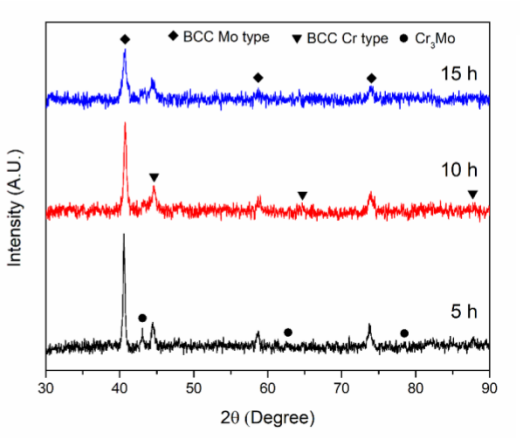

Fig. 2. XRD patterns HEA-AlCoCrMnMo after different milling time: 5, 10 and 15 h.

Table 1. Mechanical alloyed HEA-AlCoCrMnMo microstructural parameters

Alloy	Milling time (h)	Structure	Lattice (nm)	Cristal Size (nm)	Micro-strain (%)
AlCoCrMnMo	5	BCC1	0.314	26.2	0.381
		BCC2	0.288	17.5	0.523
		Cr_3Mo	0.296	26.4	0.357
AlCoCrMnMo	10	BCC1	0.314	18.2	0.542
		BCC2	0.288	8.7	1.049
AlCoCrMnMo	15	BCC1	0.314	12.8	0.778
		BCC2	0.288	7.7	1.180

* It was not possible to calculate based on the observed XRD patterns
* BCC1 = BCC Mo type
* BCC2 = BCC Cr type

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