

Article Study of Al Addition on Sintered CuCrFeNiTi as a Potential Alloy for Automotive Components

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Abstract: CrCuFeNiTiAl_x high-entropy alloys (where x = 0, 0.5, 1.0, 2.5 and 5.0 mol percent or mol %) were processed through powder metallurgy. Aluminum concentration was varied in the alloy to determine its effect on the microstructure and phase formation within the CrCuFeNiTiAl_x system. X-ray diffraction (XRD) studies revealed the presence of structures mainly composed of FCC and BCC solid-solution (SS) phases in the CrCuFeNiTi alloy. The addition of aluminum content is responsible for an increased volume fraction of the BCC phase on the sintered alloys. XRD results also indicate the formation of compounds of a chemical composition and crystalline structure different from those of FCC and BCC SS phases. The presence of these compounds was also confirmed through mapping of elements and punctual chemical analysis through energy dispersive spectroscopy (EDS). Bulk samples exhibited microstructures with multimodal grain size. From the microhardness test results, it was determined that addition of Al is proportional to an increase in hardness.

Keywords: HEAs; powder metallurgy; mechanical properties; phase composition; vehicle components

1. Introduction

High-entropy alloys (HEAs) are advanced materials with at least five elements present in equimolar or near-equimolar amounts, and have attracted significant attention from the scientific community due to their potential applications [1–3]. These alloys often exhibit unusual physical and mechanical properties due to their different chemical designs and processing methods. In these alloys, the metal atoms which are added in the same proportion have the same probability of populating a specific/particular place in the lattice, thus achieving a unique crystalline structure.

Since the discovery of HEAs two decades ago [4–6], several studies have been carried out around the world to explore their physical characteristics and properties, as well as their usefulness in different industrial applications. This has resulted in a newly emerging field for their production, development, and applications. For example, in the automotive sector, vehicle moving parts wear out over time so they must be replaced before failure. This generates (in the best of cases) additional costs in the maintenance of the vehicle(s) and requires time investment so the worn parts can be replaced. Some engine parts are unknown or can go unnoticed by most car owners; however, they are vital components for the engine's proper functioning. Examples are internal combustion engine valves and their seats [7], aluminum cylinder liners and cast-iron piston rings [8] or rocker arms (a mechanical lever) which are pivoted and transmit camshaft motion to the valves [9]. These



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Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). components are usually made of alloys that provide an adequate mechanical strength-toweight ratio. The weight is significant since excessive mass limits the engine's ability to reach high revolutions. However, most cars have traditional alloys, making them stiffer and less resistant to wear.

Many high-entropy alloys have been developed with exceptional mechanical response such as increased strength, good ductility, excellent fracture toughness, good creep, wear resistance, chemical and thermal stability [10–14] in comparison to traditional alloys. HEAs mainly have been processed through traditional melting methods that have the disadvantage of microstructural heterogeneity and heavy element segregation [15]. On the other hand, using powder metallurgy (involving mechanical milling), the tendency toward random distribution of atoms (chemical disorder) is favored and leads to extending the solid solubility limit and improving their mechanical properties [16–18]. Therefore, there are great expectations for high-entropy alloys that can be used in the automotive sector.

The CuCrFeNiTiAlx system has not been studied in detail; the effect of adding aluminum to the system has yet to be reviewed. However, it could represent interesting prop-erties due to the additional phase changes that could be generated in addition to the ten-dency of the alloy to form a mixture of FCC + BCC phases [16]. A system with similar properties that has been widely studied is CoCuCrFeNi, which has been used for magnetic applications [19,20]. The Cr and Fe promote the formation of BCC solid-solution (SS), while Cu and Ni promote the formation of FCC phases. If Ti replaces Co, the alloy gains in mechanical aspects. On the other hand, it has been reported that Al addition to this type of HEA favors the formation of BCC SS.

The objective of this work is focused on the characterization of the CrCuFeNiTi system with Al_x (x = 0, 0.5, 1, 2.5, 5 mol %) produced by powder metallurgy to evaluate the effect of Al on the phase and microstructural evolution of the alloy, and determine its potential application in vehicle parts.

2. Materials and Methods

Elemental powders of Al, Cr, Cu, Fe, Ni, and Ti with purity greater than 99% and sizes of -325 mesh were mechanically processed with a planetary mill (Retch, PM100, Germany) using ZrO_2 balls (3 mm in diameter) as the milling medium with a ball to powder ratio of 12:1 (in weight). Milling was conducted at 300 rpm for 360 min. For control particle size distribution reducing the agglomeration of the powder particles during the ball milling action, 3 mL of isopropyl alcohol was used. The studied system was: CrCuFeNiTiAl_x, where x ranged from 0 to 5 mol % and the rest of the metals were added in equiatomic 2.5, and 5.0 mol %, respectively. With the help of a uniaxial press (Montequipo, LAB-30-T, Mexico), the resulting powder from the milling stage was conformed in cylindrical samples of 10 by 4 mm in diameter and thickness, respectively, using 300 MPa. The cold-conformed samples were sintered in an electrical furnace (model FB1315M from Thermo Scientific Thermolyne, Waltham, MA, United States) at 1300 °C for 2 h. Before characterization of the sintered samples, they were ground with SiC sandpaper and later polished using alumina and diamond suspensions. Samples were etched with a metallographic reagent for the microstructure enhancement (which was prepared using equal parts in volume of concentrated HNO₃, HF, deionized water and few drops of H_2SO_4). Etching time was 15 to 20 s. Crystalline phases of sintered alloys were determined using X-ray diffraction analysis (XRD) under CuK_{α} radiation in an X'Pert PRO PANalytical device, and diffractograms were interpreted with the X'Pert Highscore Plus PANalytical software using patterns in the ICDD PDF2 database. The microhardness of specimens was determined according to the ASTM C1327 standard [21]. Meanwhile, the obtained microstructure was analyzed by scanning electron microscopy (SEM) and an energy dispersion spectrometer detector (EDS) on a HITACHI SU3500 microscope.

3. Results

3.1. Phase Analysis

The XRD diffractograms shown in Figure 1 indicate that the samples contain phases related to metallic compounds, intermetallics and oxide traces. XRD shows the presence of solid solutions in the form of a combination of FCC and BCC crystal structures and traces of chromium and copper oxides, their signal increasing as a function of Al content. In addition, some compounds formed by the alloying elements were detected. They are summarized in Table 1. Formation of these compounds is due to the type of crystal structure of the interacting elements influencing the alloying mechanism, so elements of the same crystal structure are readily dissolved among themselves, as was previously documented [22,23]. Previous work exposed how the alloying order is associated with the elements' melting point, i.e., the lower the melting point, the easier it is to be alloyed [24]. Figure 1 shows two small peaks at 40.3 and 56.2 (20 angles), corresponding to an HCP (hexagonal closepacked) structure. The presence of this phase is due to the addition of Ti, Ti being the metal with the highest melting point, only below Cr. Furthermore, it is also the metal with the largest atomic radius with an HCP crystalline structure. Both conditions of Ti enhance its solubility in the alloy; hence, the two peaks with the HCP phase that appear in the pattern are related to the presence of Ti. As seen in Table 1, Cr and Cu oxides are generated during processing. Formation of these oxides is not necessarily negative. Studies have established that the configurational disorder can be compositionally engineered into mixed oxides by populating a single sublattice with many distinct cations. The formulations promote novel and entropy-stabilized forms of crystalline matter where metal cations were incorporated in new ways [24,25]. In addition, an increase in the intensity of the peak located at the 42.9 20 angle corresponding to the BCC phase is evident and can be associated with increased aluminum content. The increase in the BCC phase can be attributed to the BCC structure generally having a lower atomic packing density (68%) than the FCC structure (74%), and therefore, being able to more easily accommodate larger solute atoms like Al = 1.43 Å (Cu = 1.28 Å, Cr = 1.249Å, Fe = 1.241Å, Ni = 1.246 Å, Ti = 1.45Å) [26]. Furthermore, the excessive addition of aluminum changes the FCC phase to a BCC phase due to a considerable lattice distortion energy that destabilizes the FCC structure [27].



Figure 1. XRD patterns of sintered high-entropy CrCuFeNiTiAl_x alloys as a function of Al content.

Phase	JCPDS Card Numbers	Chemical Formula	0	0.5	1.0	2.5	5.0
FCC	03-065-6291	Solid-solution	х	х	х	х	х
BCC	00-019-0850	Solid-solution	х	х	х	х	х
HCP	00-018-0388	Solid-solution	х	х	х	х	х
Chrome oxide	00-006-050	Cr_2O_3	х	х	х	х	
Copper oxide	00-034-1354	Cu ₂ O				х	

Table 1. Indexed phases found in the diffraction patterns of Figure 1.

3.2. Lattice Constants and Microhardness

The lattice constants of the synthesized alloys were calculated using Bragg's law equation through the interplanar distance (d_{hkl}) , using peaks at $2\theta = 43.2^{\circ}$ and 43.8° , that correspond to the (111) and (110) planes for both FCC and BCC structures, respectively. The lattice parameter was determined using the value of d_{hkl} , and Equation (1) that relates the interplanar distance to the lattice parameter in cubic systems. The results are plotted in Figure 2, as a function of the aluminum content in the alloy.

$$a = d_{hkl}\sqrt{h^2 + k^2 + l^2}$$
(1)



Figure 2. Microhardness results in Vickers scale (average and standard deviation) and lattice constant of the CrCuFeNiTiAl_x alloy, both presented as a function of aluminum content.

In Figure 2, a considerable increment in lattice constants of both structures (FCC and BCC) is observed as the proportion of aluminum increases. However, the increase of the lattice constant is more significant for aluminum contents between 0.5% and 2.5% in the alloy. Its increase is associated with the atomic radius of Al and its solid solubility into the FCC and BCC phases present in the CrCuFeNiTiAlx system. As previously discussed, the presence of aluminum plays an essential role in the random formation of a solid solution composed of several metals presenting a mixture of FCC and BCC structures.

Figure 2 also shows the results obtained from microhardness measurements on prepared alloys as a function of aluminum content. This figure clearly shows the substantial effect of the aluminum content on the hardness of the alloy, since as the amount of aluminum increases, so does the hardness. It could also be due to the formation of hard particles of metal oxides during the powder metallurgy process, which acts as a second hardening phase. A list of formed phases and solid solutions is presented in Table 1.

3.3. Morphology and Porosity

The general microstructure of sintered HEAs is depicted in Figure 3. It is composed of irregular grains of varied sizes and particle shapes. Samples exhibited a wide distribution of particle sizes, with a multimodal behavior ranging from 0.5 to 50 microns. Their microstructure is composed of larger grains with different contrast (brighter zones) and various morphological features. These bright grains with different aluminum concentrations are associated with other metallic compounds formed during the alloy-processing, as detected by XRD and SEM-EDS. Micrographs at low magnification (100x) were processed using an image processor (AxioVision SE64) and porosity was calculated as an image percentage (the values are illustrated by green circle inserts and the results are shown below the circle of each micrograph of Figure 2). When intermediate amounts of Al are added in the HEA, there is an evident reduction in porosity; thus, a higher densification is obtained.



Figure 3. SEM-SE micrographs of CrCuFeNiTiAlx alloys with different porosity levels as a function of added Al.

3.4. Chemical Analyses by EDS-SEM

Through the observations and analyses with scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS), it was evident that sintered CrCuFeNiTiAlx alloys possess a multi-phase microstructure (Figure 4). Figure 4a shows the microstructure corresponding to the CrCuFeNiTi sample, where different morphology zones were observed. Spots enclosed in a white box indicate the specific area where the chemical analyses were carried out. Figure 4b corresponds to the CrCuFeNiTiAl_{0.05} sample.

Some chemical analyses were conducted through EDS-SEM on regions with different tonalities. EDS results with their corresponding standard deviation values are shown in Table 2. The constituent phases have a significant difference in chemical composition.

Table 2 shows punctual chemical analysis indicating the presence of phases with different chemical compositions, where some oxidation occurred during the alloy processing (as detected by XRD). The composition of each grain certainly varies, which is indicative of the formation of a high degree of entropy alloy. In specific grains, more atoms of certain elements compete between them to occupy specific spaces in the crystalline structure. However, there was always an original balance in the alloys' chemical compositions, which was verified by a general mapping of the distribution of the elements in the alloy with 1% at. of aluminum. The result of this mapping analysis is shown in Figure 5. The obtained spectrum, which presents the components of the alloy, and a chart with the result of the chemical analysis, indicate that the resulting chemical composition is close to the hypothesized composition. Chemical analysis also showed the presence of oxygen, which partially oxidized elements (such as Fe and Cu) during processing. Finally, the spatial distribution of the elements within the alloy is homogeneous.

Table 2. Summary chart with chemical analyses (in at. % & deviation) of spectrum shown in Figure 4 (ND = non-detected).

Element	Spec. 40	Spec. 41	Spec. 44	Spec. 71	Spec. 73	Spec. 74	Spec. 76
Al	ND	ND	ND	1.1 ± 0.1	ND	19.6 ± 0.1	20.1 ± 0.2
Cr	15.2 ± 0.1	9.5 ± 0.4	17.7 ± 0.3	7.7 ± 0.3	90.3 ± 0.2	5.3 ± 0.2	6.4 ± 0.1
Cu	12.8 ± 0.4	9.7 ± 0.3	4.6 ± 0.2	10.7 ± 0.3	ND	2.9 ± 0.1	3.9 ± 0.2
Fe	38.8 ± 0.4	3.5 ± 0.2	2.0 ± 0.1	57.7 ± 0.2	1.9 ± 0.1	0.9 ± 0.1	2.5 ± 0.1
Ni	30.5 ± 0.3	5.2 ± 0.3	2.2 ± 0.1	19.5 ± 0.3	1.4 ± 0.3	1.2 ± 0.2	3.4 ± 0.2
Ti	0.5 ± 0.1	47.6 ± 0.3	25.9 ± 0.3	0.4 ± 0.1	ND	26.3 ± 0.4	16.3 ± 0.2
0	2.2 ± 0.1	24.5 ± 0.1	47.6 ± 0.4	3.0 ± 0.2	6.4 ± 0.4	43.8 ± 0.4	47.5 ± 0.3



Figure 4. SEM-BSE micrographs and studied zones of x = 0 (**a**) and 5% (**b**) Al samples.



Figure 5. Elemental mapping of CrCuFeNiTiAl_{0.01} sample.

According to the above results, it may be said that oxidation occurs during the solidstate processing (as detected by XRD), and the microstructure of the CrCuFeNiTi alloy synthesized by powder metallurgy is composed of two main phases. In comparison, the CrCuFeNiTiAl_{0.05} alloy is composed of three main phases. Elemental mappings were obtained to analyze each sample's elemental distribution. Figure 6 shows an SEM micrograph and the CrCuFeNiTi alloy's elemental mapping separated by element distribution. From these results, it can be inferred that the microstructure of this alloy is composed of a high Cu, Fe and Ni solid solution (light region), a dark region composed of Ti and Cr, and a dispersed chromium oxide phase.



Figure 6. Elemental mapping of CrCuFeNiTi sample.

Figure 7 shows an SEM micrograph and the elemental maps of the CrCuFeNiTiAl_{0.05} alloy. According to the results, addition of Al promotes the increase of the volume fraction of the chromium oxide phase.



Figure 7. SEM micrograph and elemental mappings of the $CrCuFeNiTiAl_{0.05}$ sample.

Furthermore, it was observed that the Al distribution is preferentially in the high Ti phase, while the amount of Cr in the Ti-rich phase decreases as the Al content in the alloy increases. It was also observed that the amount of Fe in the Cu-Ni phase decreases while exhibiting a preference for the chromium oxide phase.

4. Conclusions

 \bigcirc High-entropy alloys of the CrCuFeNiTiAlx (x = 0%, 0.5%, 1%, 2.5%, 5%) type were synthesized following a powder metallurgy method. The solid-state process was proved

as an effective experimental route to produce high-entropy alloys since milling induces finer particle size and high lattice distortion, facilitating the metal atoms' diffusion during sintering. Furthermore, sintering promotes metal diffusion during sintering.

- Based on microstructural results, the CrCuFeNiTi alloy synthesized by solid-state route is composed of a Cu-Fe-Ni SS, Ti-enrich phase and chromium oxide-type phase. The Al content increases the volume fraction of the chromium oxide-type phase. In addition, the preference distribution of Al in the Ti-enrich phase was observed.
- The HEA alloy that achieved the highest hardness is the one with the highest Al content. These alloys harden significantly with the addition of Al due to the increase of the chromium oxide-type phase, and the strong atomic bonding between Al and other elements, due to the larger atomic radius of Al. This phenomenon is also related to the reduction in porosity as a function of aluminum content.

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