AlCrCuFeNiMn HIGH ENTROPY ALLOY OBTAINED BY POWDER METALLURGY ROUTE

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The paper presents the experiments realized to process an equiatomic AlCrCuFeNiMn high entropy alloy (HEA) by mechanical alloying (MA), compacting and sintering. The alloy was characterized using Scanning Electron Microscopy (SEM) underlining the cocktail effect of high entropy alloys. Also, the microhardness was investigated and the mean value of the hardness was compared with the values of other high entropy alloys and stainless steel found in literature. The value obtained for the AlCrCuFeNiMn alloy was higher with 95% in comparison with stainless steel and with 35% in comparison with AlCrCoFeNi high entropy alloys.

Keywords: high entropy alloy, mechanical alloying, microstructure, hardness

1. Introduction

High entropy alloys emerge as a new type of advanced materials that contain five or more elements in equiatomic or near-equiatomic ratio, each element having the atomic percentage between 5% and 35%. The atomic percentage of each minor element, if any, is less than 5%. The main principle of HEAs is the high mixing entropies of solid solutions which enhance their stability, especially at high temperatures, facilitating their processing, synthesis, manipulation and utilization.

Fig. 1 shows the materials hyper tetrahedron for the HEAs revealing the broad spectrum of research and development that is taking place in this field [1].

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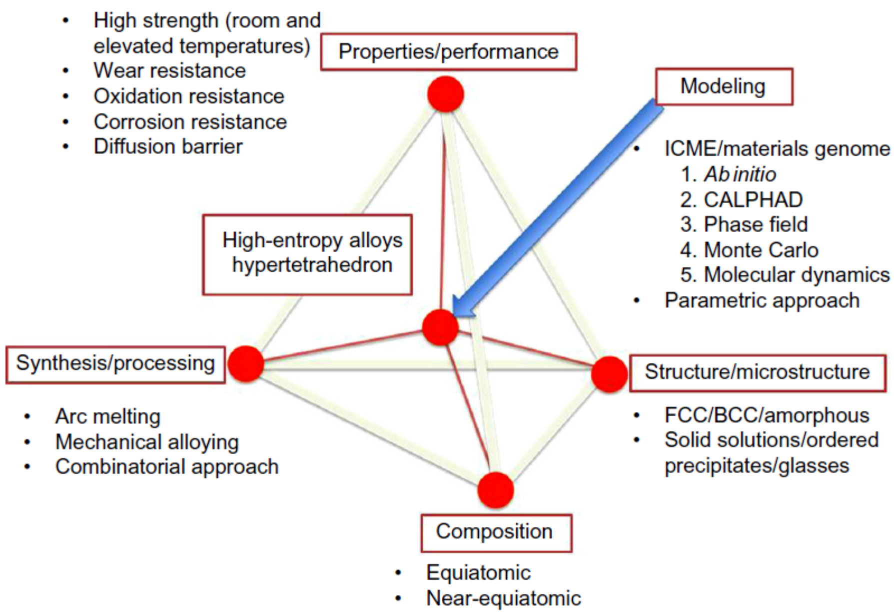

Fig.1.The material hyper tetrahedron for HEAs [1]

HEAs have four core effects due to the composition complexity: (i) high entropy effects, (ii) sluggish diffusion, (iii) severe lattice distortion and (iv) cocktail effects. Each constituent element has a contribution in defining the mechanical properties of high entropy alloys [2].

High entropy alloys are essentially multicomponent equiatomic or near equiatomic alloys which form mostly solid solutions including random solid solutions and partially ordered ones. In few cases they form amorphous alloys. Although composed of multi-principal components, HEAs exhibit simple solid solutions with BCC and/or FCC instead of complicated intermetallics. With proper element constitutions, HEAs exhibit high hardness, excellent strength as well as promising resistances to wear, oxidation and corrosion resistance [3, 4, 5].

Several processing routes are used to obtain these alloys, among them being casting, sputtering, splat quenching, mechanical alloying (MA), semisolid forming, stir friction welding, or spark plasma sintering. Considering the fact that by casting route there are disadvantages such as diseconomy and limitations in shape and size, mechanical alloying (MA) is increasingly used due to smaller costs for preparing the nanocrystalline materials and for giving more homogenous microstructures and better densification characteristics [6,7].

Chao Wanga et al. have synthetized CoCrFeNiMnAl HEA by MA route. They reported a simple structured solution obtained after 30h milling. The HEA alloy resulted showed high Vickers hardness of 662 HV and compressive strength of 2142 MPa [8]. S. Varalakshmi et al. obtained equiatomic CuNiCoZnAlTi HEA using MA route. The alloy has been consolidated by vacuum hot pressing at 800^0C with 30 MPa pressure and the characterization of the material revealed mainly a BCC phase and two minor FCC phases. The hardness of the material and the compressive strength, 7.55 GPa, respectively 2.36 GPa, providing a promising future of the material [9].

High entropy alloys good properties over conventional materials and alloys recommend them in a very large area for the modern industry which includes aerospace, automotive, metallurgy, electronics a.s.o. These properties offer the possibility to use HEAs for extreme conditions working parts such as rolling mill bearings. A cylinder rolling mill bearing stand working loads exceeding several times the working load on other bearings used in other branches of machinery constructions. For example the pressure on a bearing reached 37.3 MPa and pressure multiplied with mandrel circumferential speed reached 65.2 MPa.

The high pressure build on bearings is due to relative small length of the rolling mill cylinder mandrels and significant bending efforts. Rolling mill bearings works in conditions of high temperatures, high pressure, very high pressure per unit area and needs to have very good bending resistance. HEAs can replace with success the conventional materials and alloys used for bearings due to a smaller production cost and higher hardness. Existing literature shows that HEAs have better hardness than Hastelloy and 316 stainless steel, steel alloys used in modern industry [5].

In this work, we focused on a high entropy AlCrCuFeNiMn alloy system synthesized by mechanical alloying followed by compactation and sintering. We studied the powders alloying behavior, and investigate the HEA microstructure and mechanical properties.

2. Materials and technology

Elemental high purity powders of Al, Cu, Cr, Fe, Ni and Mn with particle size less than 45 μm were used as raw materials. Mechanical alloying process was realized in a RETSCH PM 400 planetary ball mill for obtaining HEA powders with micro and nanometric sizes. Very high centrifugal forces of the planetary ball mill result in high energy dispersion so that the milling time is reduced. Stainless steel vials and 10 mm in diameter balls were used as the milling media with a ball-to-powder mass ratio of 10:1. Experiments were carried out in dry conditions using zinc stearate as process control agent (PCA) to prevent agglomeration of the powder and depositing it on the enclosure walls and the balls used for grinding. The samples were milled for 5 h at 250 rpm. After mechanical alloying process the samples were pressed with a laboratory press using 250 MPa uniaxial pressure and sintered in a sintering furnace with Ar atmosphere using a sintering cycle as presented in Fig. 2.

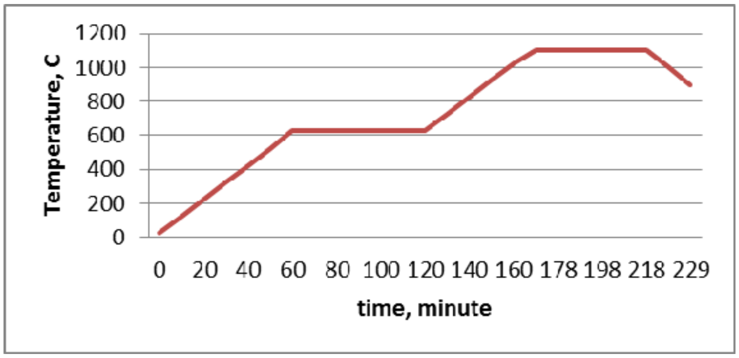

Fig.2. Sintering cycle for obtaining AlCuCrNiMnFe

Among mechanical properties was investigated the microhardness of the obtained alloy on a Shimadzu Vickers microhardness. The obtained microhardness value was compared with the values of other high entropy alloys and stainless steel.

3. Result and discussions

The samples were characterized using Scanning Electron Microscopy (SEM). The SEM specimens were prepared for examination to mirror-like surface. They were examined in plan-view.

This examination revealed the cocktail effect of high entropy alloys. Due to the non-equilibrium state of the MA process, a large amount of internal stress would be stored in the lattice, as well as defects, i.e. lattice distortion and twins. The internal stress was released when the HEA was sintered at high temperature and the metastable state transformed to stable one. Both would contribute to the expansion of the crystal lattice.

The microstructures of obtained AlCrCuFeNiMn HEA powders after 5h MA are shown in Fig. 3. As shown in this figure milled powder agglomerates into elliptical shape of ~2.36 μm and less than 1 μm in thickness.

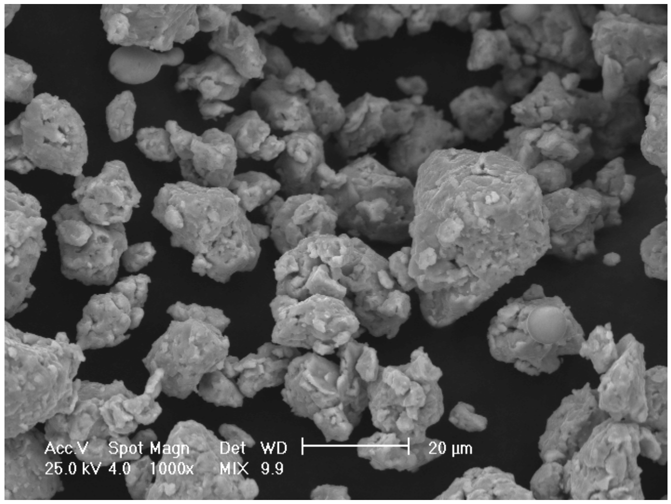

Fig. 3. SEM image of AlCrCuFeNiMn high entropy alloy

The microstructure of sintered AlCrCuFeNiMn HEA is presented in Fig. 4. The distribution images for each element in AlCrCuFeNiMn high entropy alloy after sintering are presented in Fig. 5 (a) to (f).

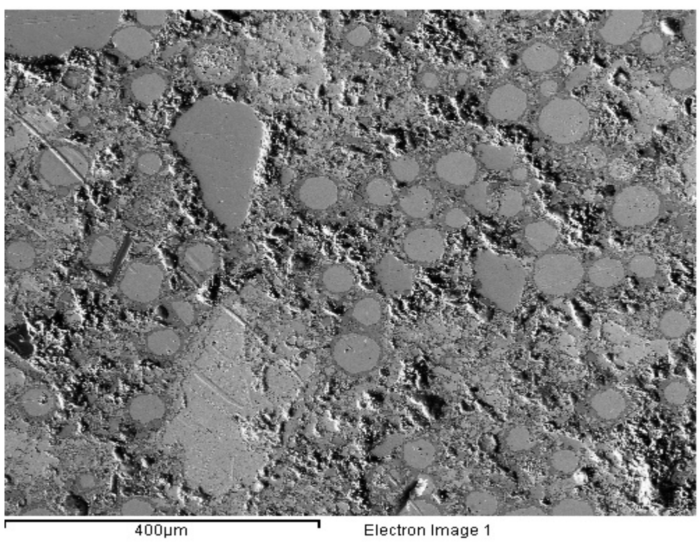

Fig. 4. SEM image of AlCrCuFeNiMn high entropy alloy after sintering.

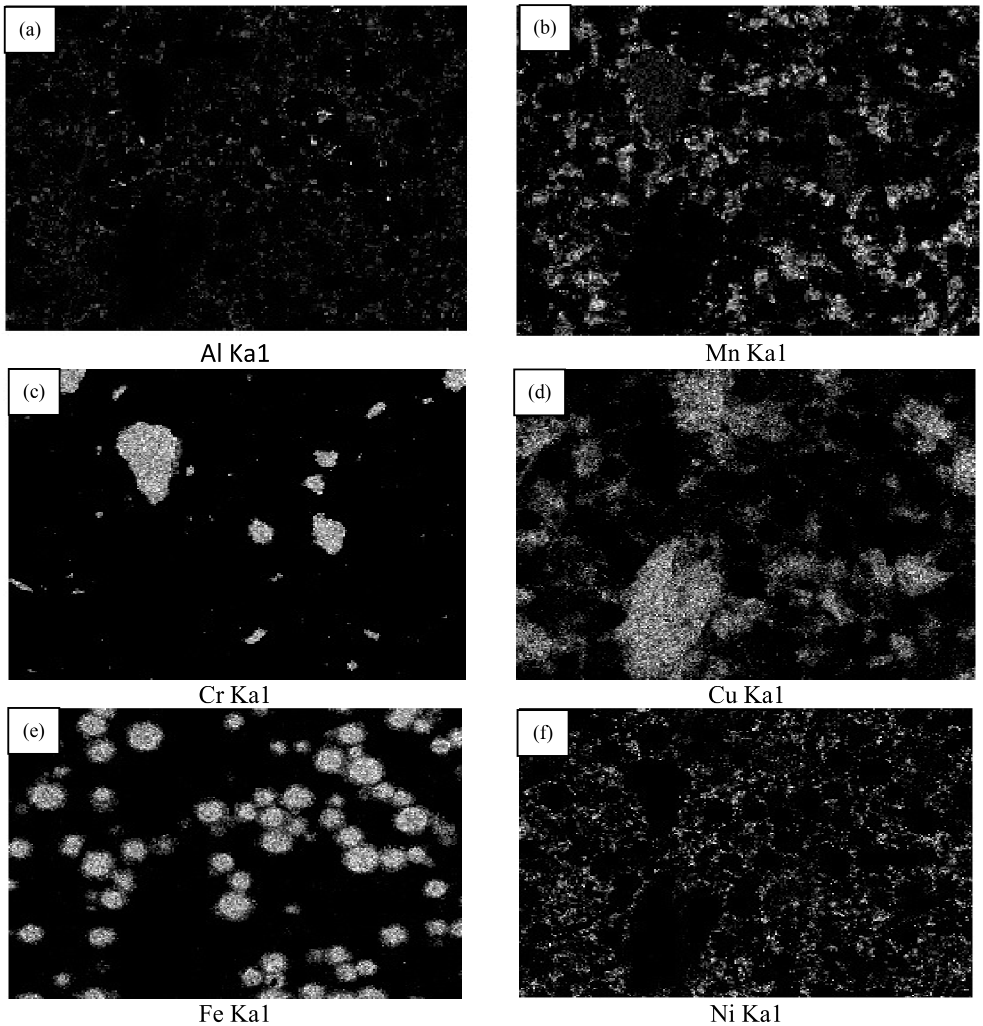

Fig. 5. Distribution images for AlCrCuFeNiMn high entropy alloy after sintering

The images have shown that the cocktail effect of high entropy alloys was started but not all the alloying elements were mixed in the best way. The Cr effect was very intense due to the fact that Cr particles are very hard. Some particles segregated and became clusters of different compounds as we can see in Table 1. These types of clusters will be avoided in future experiments by using heptane as process control agent instead of zinc stearate.

Table 1

Chemical analysis of the AlCrCuFeNiMn after sintering

Element, %wt	Al	Fe	Cr	Cu	Ni	Mn	O
Content	13.8	15.66	16.21	15.17	18.65	17.67	2.84

The particles were slightly oxidized due to the fact that all the experiments were developed in air atmosphere and this was the main reason for the oxygen presence in our high entropy alloy.

Hardness was measured with a Shimadzu Vickers hardness device, with 0.1kg load, and the mean value was calculated. The hardness value of the obtained alloy (575 HV) was compared with literature data for other HEAs and stainless steel (Fig.6). We can see from this graph that the mean value of the hardness is placed near the value of other high entropy alloys and is obviously increased in comparison with other multi component alloys.

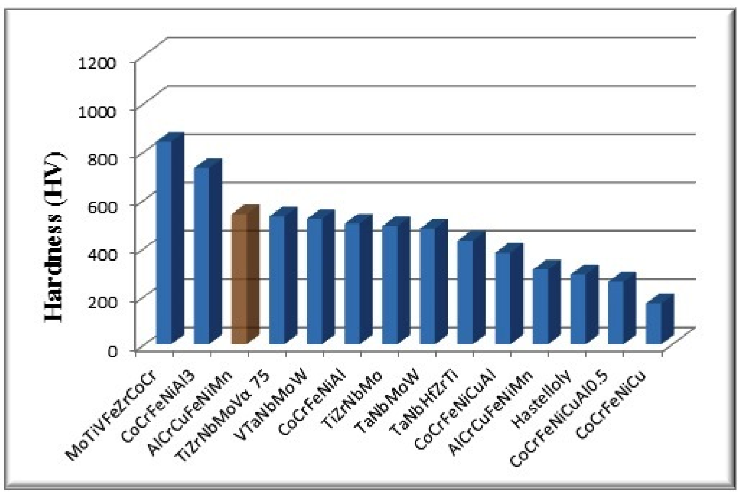

Fig. 6. AlCrCuFeNiMn hardness in comparison with other high entropy alloy hardness found in literature

4. Conclusions

It was obtained a high entropy alloy in the system AlCrCuFeNiMn using a mechanical alloying route.

The microstructure revealed a good homogeneity and the cocktail effect. Also the microstructure revealed the fact that Cr segregated and formed some clusters that hopefully will be avoided in future experiments using other process control agent. From our investigation we observed that we have to increase the milling time to achieve the best results with these kinds of high entropy alloys.

The mean value of the hardness measured for this alloy is 575 HV, a result improved with 95% in comparison with other material composite alloys.

This alloy is a good promise for obtaining bearing roll mill that works in hard conditions.

Acknowledgements:

This research was supported by a grant of the Romanian National Authority for Scientific Research, CNDI– UEFISCDI, project number PCCA 270/2014, HEADURCOR.

The work has been funded by the Sectoral Operational Programme Human Resources Development 2007-2013 of the Ministry of European Funds through the Financial Agreement POSDRU/159/1.5/S/132395.

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