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Pressure Influence on the AlCrFeNiMn/Graphite High Entropy Composite Microhardness

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Abstract. During the last years, mechanical alloying technique for high entropy alloys (HEAs) has been more often approached due to the good homogenous chemical distribution and near net shape technology provided by the respectively process. A new composite material having the matrix as HEA reinforced with graphite particles was designed. The graphite particles addition in the high entropy matrix (AlCrFeNiMn) improves the particles weldability during mechanical alloying and assures a good creep behavior for the final product. The aim of this paper is to investigate the pressure influence on the microhardness as dependence of sintering parameters which can be reflected also on the microstructure. The high entropy composite was completely alloyed after 40 hours of milling. The obtained composite was pressed using different pressures values in order to investigate the pressure influence on the microhardness and microstructure. The samples were investigated using optical microscopy, scanning electron microscopy, X-rays diffraction and microhardness tests. The microhardness values for all the samples were between 300 – 700 HV.

Introduction

High entropy alloys (HEAs) are defined as alloys which consist of at least five principal elements, and concentration of each constituent element ranges from 5 to 35 at. % [1], with unique properties, like: high hardness and strength, high corrosion and wear resistance, good thermal stability [2-6]. These promising properties offer many potential for engineering applications, in various fields, such as tools, coatings, magnetic films [1-5]. Al, Cr, Fe, Co, Ni, Cu, Ti, Mn, etc. are the most commonly used metallic elements for HEA production [1, 7]. An improvement in mechanical and tribological properties of HEAs can be attained by introducing ceramic particle such as, graphite, silicon carbide, aluminum oxide, titanium oxide or diboride etc. [8]. Till now, was investigated only the influence of graphite content on the aluminum composites [9-12]. The obtaining of a new composite based on HEAs reinforced with graphite particles represents a new challenge.

The processing routes for HEAs can be summarized according to the starting states for the alloy preparation in: (i) liquid state, (ii) solid state, (iii) gas state, (iv) electrochemical process. During the last years, mechanical alloying technique for HEAs has been more often approached due to the good homogenous chemical distribution and near net shape technology provided by the respectively process. Processing in solid-state, known as mechanical alloying (MA), involve repeated cold welding, fracturing, and re-welding of powder particles in a high-energy ball mill [13] and has been shown to be capable of synthesizing a variety of equilibrium and non-equilibrium alloys starting from blended elemental or pre-alloyed powders. The addition of graphite particles in the high entropy matrix (in this case AlCrFeNiMn) improves the particles weldability during mechanical alloying and assures a good creep behavior for the final product. About HEAs used as matrix for composite materials there is no more information, the scientific research being very poor. Fang and colab. [14] investigate for the first time the influence of a non-metallic element addition in HEAs. Starting from their results and for a better understanding of this new class of materials, the present paper investigate the pressure influence on the microhardness, which can be reflected also on the microstructure of the AlCrFeMnNi/Gr composite.

Experimental procedure

High purity metallic powders Al, Cr, Fe, Ni, Mn (>99%) with different morphologies and particle size ≤45 μm (325 mesh) and graphite powder (Gr) with particle size 250 μm (60 mesh) were used to obtain AlCrFeMnNi/Gr composites by mechanical alloying (MA). The graphite content used to obtain the composites was 3wt.%. The MA process was carried out in a high speed planetary ball mill (RETSCH PM400), in stainless steel vials in which the powders were sealed together with stainless steel balls (10 mm diameter). The ball to powder weight ratio (BPR) was 10:1. The milling was realized under argon atmosphere and using N-heptane as PCA agent in order to avoid cold welding of particles, as well as to prevent the alloy oxidation. The milling time was chosen for 40 hours and the milling speed was 300 rpm. The obtained composite was consolidated by pressing in an RETSCH 40T Hydraulic Press, at room temperature, using pressing forces of 780 MPa and 880 MPa. The consolidate samples were then sintered in a NABERTHERM furnace at 900°C for 1 hour. The sintering temperature was established after a series of experiments performed to determine the best sintering temperature [15]. The influence of pressing force was studied on the microstructure using Reichert-UuniVAr optical microscope (OM) and a FEI Quanta 3D FEG, scanning electron microscope (SEM). X-ray diffraction technique was used for phase analysis of the samples using an XPert Pro Panalytical instrument operated at 40 kV, 30 mA, θ-2θ scan mode, angular range of 30°- 70°, step size 0.02°, counting time 1 sec/step). The sintered composites were then subjected to measuring Vickers hardness using an INNOVA hardness tester, applying a normal load of 100 g and a dwell time of 10s. The hardness values were calculated as the average of 7 tests.

Results and Discussion

As can be seen in the micrographs presented in Figure 1, two different phases can be identified, the matrix and intermetallic compounds (IMCs). IMCs have different sizes and roundness as well as porosity degree. Composite pressed at 780 MPa (Figure 1,a) presents irregular distributions as well as a small relative sizes of pores compared with IMCs. From Figure 1,b can be observed that increasing the pressure, the IMCs sizes decrease while the pores have small dimensions and are redistributed surrounding the IMCs. The relative density of the sintered composites increased with 8.2% in comparison with the pressed samples.

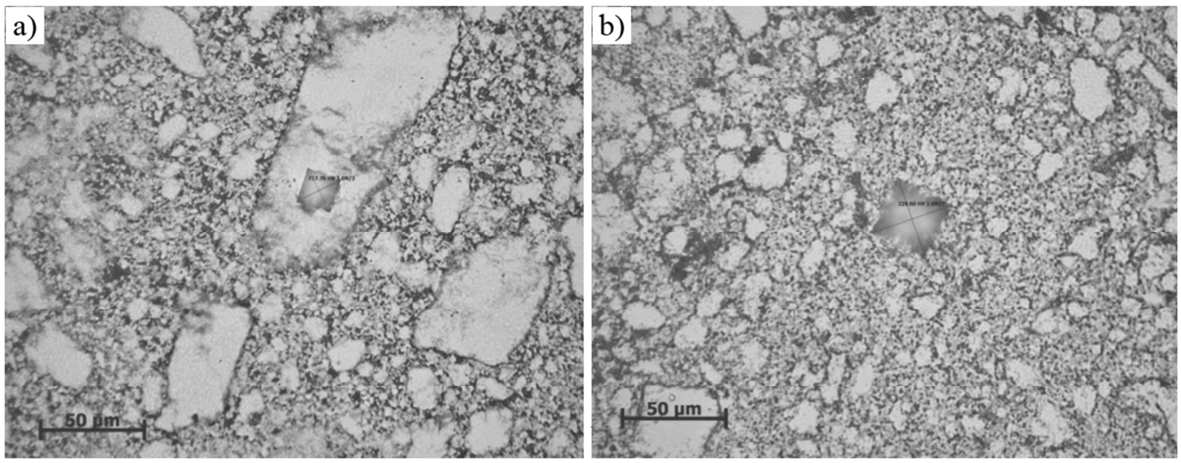

Figure 1. Optical metallography of composite pressed at: a) 780 MPa b) 880 MPa.

It was remarked that microhardness matrix values were influenced by the vicinity pores presence. The same phenomena was observed on microhardness ICMs test, because these can “moved” in the matrix during the indenting. Further microhardness measurements will take in consideration these phenomena and also will be redesigned the technological parameters in our HEAs composite processing. In this respect, obtained microhardness testing values are presented in Figure 2. It can be remarked an increase with around 15% of microhardness both for the matrix and the IMCs. The obtained values are in concordance with the values presented in specialty literature [14-18].

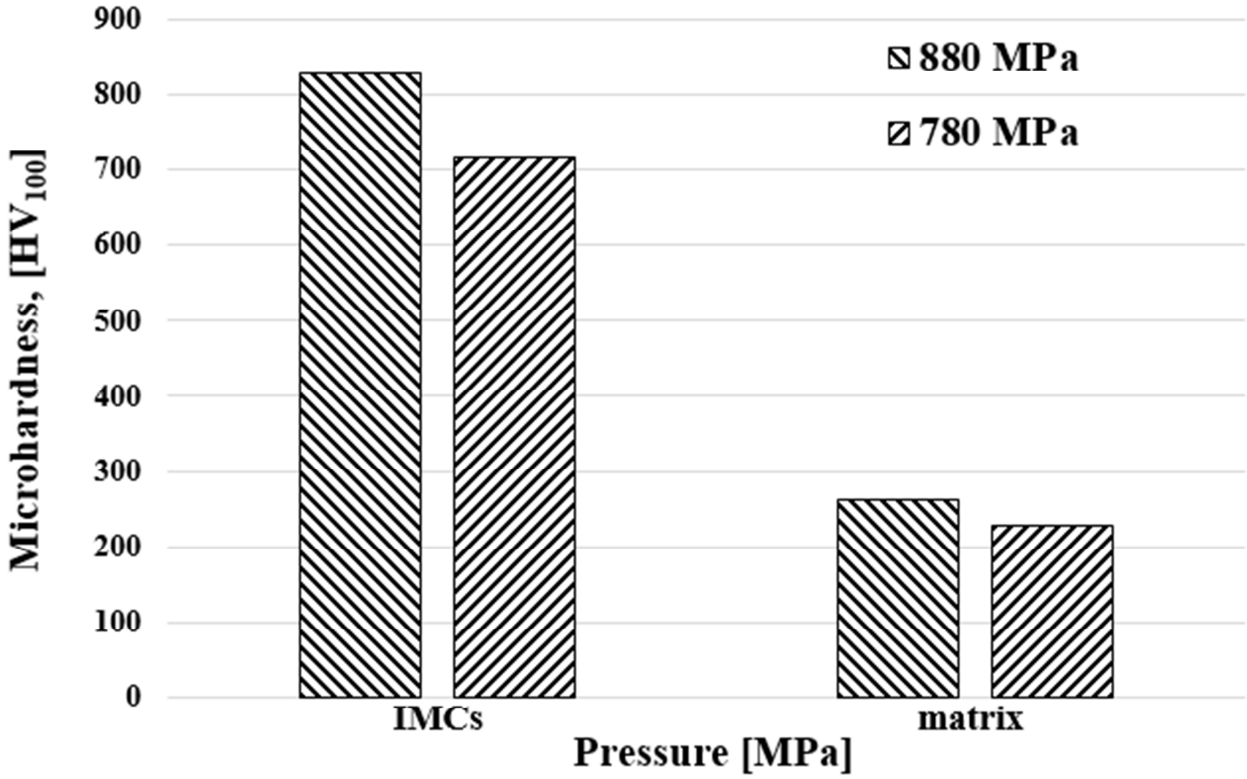

Figure 2. The influence of pressure on the microhardness matrix and composite.

The effect of pressing force on the microstructure is an important issue, which could help in understanding of each element behavior during mechanical alloying. The SEM studies of the AlCrFeNiMn/Gr composite are presented in Figure 3. It can been seen that with increasing the pressure results: i) the number of intermetallic compounds (IMCs) increase; ii) the number of IMCs decreases in size and have an uniform distribution in the matrix; iii) compactness degree increase and the shape of IMCs aim at spheroidal shape; iv) IMCs surface becomes smoother; v) appear a crack tendency inside the IMCs.

The high entropy composite is a new material with a behavior different than a conventional alloy. Thus the pressing force for the composite should be determined in order to obtain consolidated samples that will be further subjected to sintering. The pressing force is important in achieving a proper compaction degree. The specialty literature revealed [3 – 5] as main consolidating method for high entropy alloy spark plasma sintering. We developed a new high entropy composite using powder metallurgy classic route. The idea of keeping the simplest way of processing these materials would be helpful for industry application.

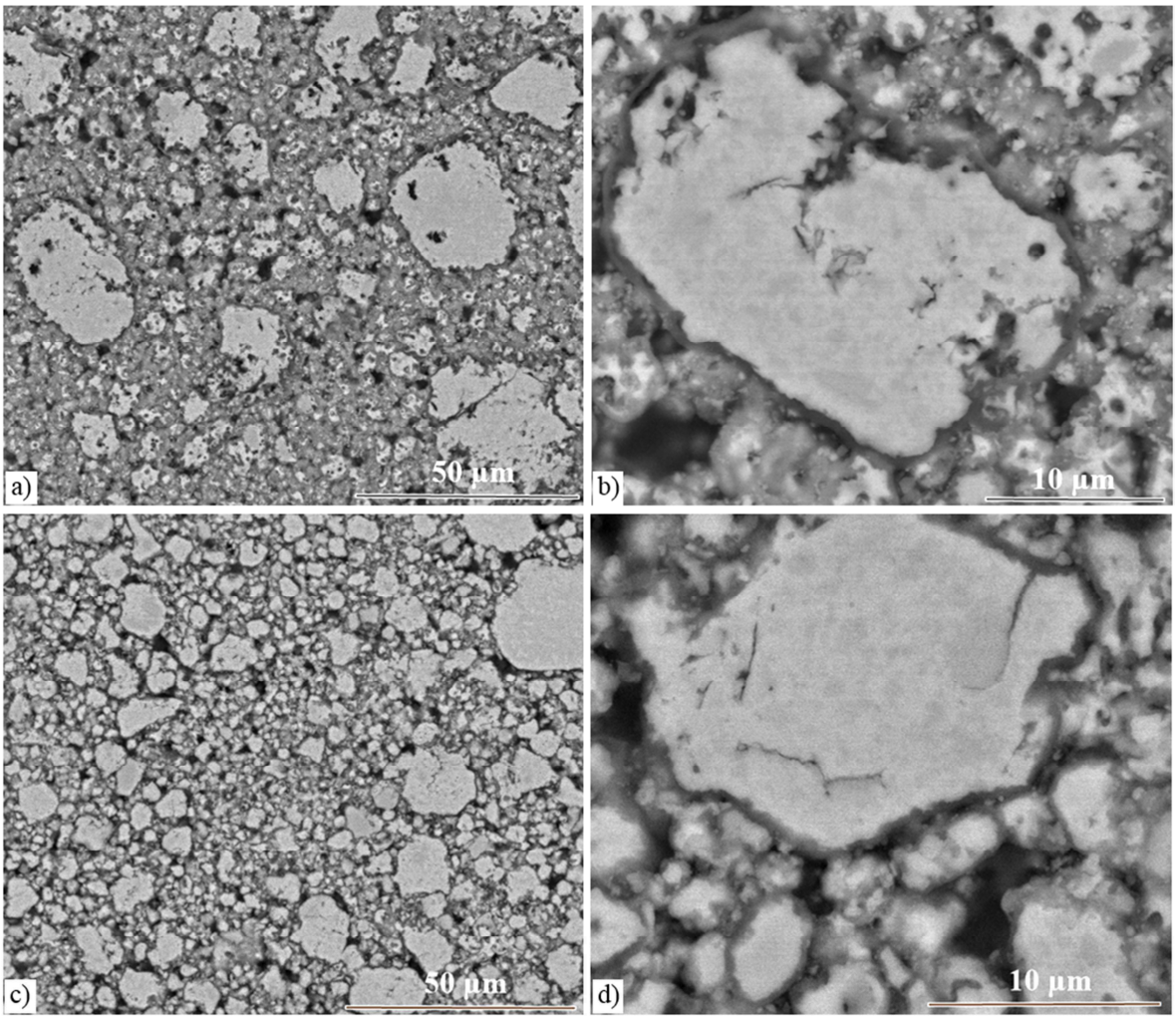

Figure 3. SEM micrographs of AlCrFeNiMn/Gr high entropy composite:
a) pressed at 780 MPa; b) detail of IMCs from composite pressed at 780 MPa;
c) pressed at 880 MPa; d) detail of IMCs from composite pressed at 880 MPa.

Figure 4 shows the XRD patterns of AlCrFeNiMn/Gr composite pressed at 780 MPa and respectively 880 MPa. The diffraction peaks corresponding to an face-centered cubic (FCC), a body-centered cubic (BCC) crystal structure and carbides are presented in the composite pressed at 780 MPa. The BCC phase seems to be more present in the high entropy composite. Thus the increased value of hardness could be explained. No diffraction peak of C can be identified in the XRD patterns, this may result from its smallest atomic fraction in the alloy. As the pressure extends from 780 MPa to 880 MPa, the diffraction peaks exhibit no change except for a minor broadening and a significant increment of diffraction intensity.

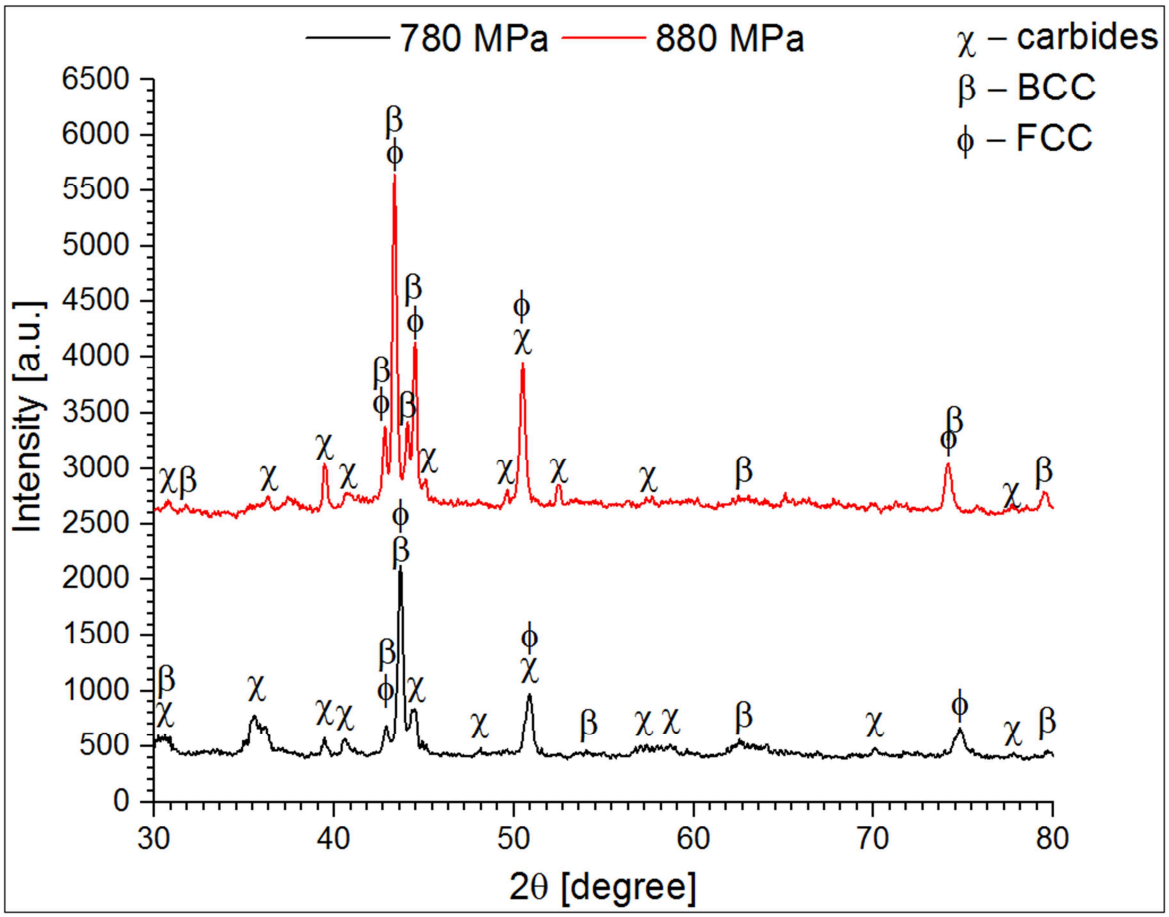

Figure 4. XRD pattern of AlCrFeNiMn/Gr composite.

Conclusion

AlCrFeNiMn/Gr composite was successfully processed by mechanical alloying, a fully alloyed composite material being obtained after 40h. This composite was pressed with different pressures and then sintering at 9000C for 1 hour. The sintered composite was investigated by optic and electron microscopy, being identified two phases: the matrix and intermetallic compounds (IMCs). Optical metallography revealed small sizes of IMCs and a more homogenous structure with pressure increasing. SEM micrographs revealed also the decrease of IMCs number as well as their dimensions with pressure increase. XRD results indicate a good structure of the composite with two phases BCC and FCC, and also carbides. Vickers microhardness measurements revealed also an increase with around 15% both for the matrix and the IMCs with pressure increase.

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