Synthesis of Ultrafine Fe-W-Al-Ti-Ni-C-B Powders by Mechanical Alloying

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Abstract. The goal of the presented research was syntheses of the ultrafine and nanopowder blends by Mechanical Alloying from Fe-W-Al-Ti-Ni-B-C compositions for further application as a precursor for fabrication bulk High-Entropy Alloy (HEA) by explosive compaction technology. The presented paper describes the preliminary investigations of mechanical alloying of Fe-W-Al-Ti-Ni-B-C powders. Laboratory vibrating sieve equipment was used for sorting the initial powders for the reduction of particle size. For the experiments were used powders of Fe-W-Al-Ti-Ni-B-C system consisting of five major metallic and two minor non-metallic B and C elements were used for mechanical alloying. The crystalline coarse Aluminum, Titanium, Carbon and Nickel, fine-grained iron and tungsten and amorphous Boron powders were used as initial components. The Fe,W,Al,Ti and Ni powders were preliminary checked by “Explorer 5000 XRF” by Skyray instrument and verified. Based on the literature review, theoretical investigation and phase diagrams for mechanical alloying were selected two compositions with the following molar ratio: Fe20W15Al15Ti20Ni20B5C5; Fe25W25Al10Ti15Ni15B5C5; The mechanical alloying was realized on the high energy ball mill with zirconium oxide jars and balls. The ball to powder mass ratio was 10:1, the rotation speed was 500rpm. The process was carried out in dry conditions. The time of mechanical alloying was 5h, 10h and 14 h. The powders were prepared for preliminary analyses and assessment of particle size. The SEM and optical microscopy results of mechanical alloying are presented in the paper.

1. Introduction

High-entropy alloys (HEAs) are known as multi-element alloys or multi-component alloys, which has been the subject of numerous investigations during the last 15 years. HEAs found to have great potential of application because of the novel and improved properties.

These alloys are currently the focus of significant attention in materials science and engineering because they have potentially desirable properties [1]. Research indicates that some HEAs have considerably better strength-to-weight ratios, with a higher degree of fracture resistance, tensile strength, as well as corrosion and oxidation resistance than conventional alloys.

High entropy alloy consists of at least five principal elements with the concentration of each material between 5 and 35 at.%. HEAs now include a broad class of metallic and ceramic systems.
Because of the unique multiprincipal element composition, HEAs can possess special properties. These include high strength/hardness, outstanding wear resistance, exceptional high-temperature strength, good structural stability, good corrosion and oxidation resistance. Some of these properties are not seen in conventional alloys, making HEAs attractive in many fields. The fact that it can be used at high temperatures broadens its spectrum of applications even further. Moreover, the fabrication of HEAs does not require special processing techniques or equipment, which indicates that the mass production of HEAs can be easily implemented with existing equipment and technologies.

HEAs are processed by a variety of processing techniques, involving solid/powder, liquid, and gaseous states. One of the most convenient synthesis routes for the HEAs is ball milling and Mechanical Alloying (MA) in dry conditions [2]. MA involves repeated cold welding, fracturing, and re-welding of powder particles in a high-energy ball mill. Because of the specific advantages, MA is used to synthesize a variety of ultrashort grained materials and nanocomposites. Nanocomposites have also been obtained when the amorphous phases obtained by MA are crystallized at relatively low temperatures. An important attribute of these nanocomposites is in preventing or minimizing grain growth until very high temperatures [3].

In this study are presented syntheses of the ultrafine and nanopowder blends by Mechanical Alloying from Fe-W-Al-Ti-Ni–B-C compositions for further application as precursors for fabrication bulk HEA by explosive compaction technology [4]. Majority of the HEA and bulk materials are attractive for application in modern machine building, airspace, chemical and metallurgical industry. Some of the mentioned methods require the use of high temperature for an extended period of time. Because of significant coarsening of the ultrafine grains, nanostructure effects are decreased. Therefore the presented investigations are aimed to produce a large quantity of ultrafine and nanopowders, which will be used for further consolidation in order to obtain bulk HEAs by explosive compaction technology.

2. Experimental and SEM Investigations
For the experiments were used powders of Fe-W-Al-Ti-Ni-B-C system consisting of five major metallic and two minor non-metallic B and C elements were used for mechanical alloying. The crystalline coarse Aluminum, Titanium, Carbon and Nickel, fine-grained iron and tungsten and amorphous Boron powders were used as initial components. The Fe,W,Al,Ti and Ni powders were preliminary checked by “Explorer 5000 XRF” by Skyray instrument and verified.

Theoretical investigations, phase diagrams and thermodynamic investigations were carried out in order to select Fe-W-Al-Ti-Ni–B-C compositions. At the initial stage, 2 different compositions with different molar ratio were selected. Those compositions are: Fe_{20}W_{15}Al_{15}Ti_{20}Ni_{20}B_{5}C_{5} and Fe_{25}W_{25}Al_{10}Ti_{15}Ni_{15}B_{5}C_{5}.

Precursors were classified by vibratory sieves. The particles size of Ti and Al powders was less than 200 µm. For MA, amorphization and nanopowder production, the high energetic “Fritsch” Planetary premium line ball mill was used. The mill was equipped with Zirconium Oxide jars and balls. Ball to powder mass ratio was 10:1 during the MA process. The time of the processing was varied in the range: 5, 10; 15 hours. The rotation speed of the jars was 500 rpm.

After the MA process, the Scanning Electronic Microscope (SEM) investigations have been carried out for the compositions in powder form. The SEM images in Figure 1, Figure 2 and Figure 3 show the tendency of reduction of brain sizes during the mechanical alloying process according to the increase of processing time.
Figure 1. Microstructure of Fe-W-Al-Ti-Ni-B-C powder composition after 5h MA process

Figure 2. Microstructure of Fe-W-Al-Ti-Ni-B-C powder composition after 10h MA process
The SEM images show the tendency of the reduction of particle size after increasing the processing time. After 14 hour processing time the particles less than 1µm is observed in the microstructure. The tendency still shows the potential for the nanopowder formation with the increase of time of MA.

3. Results and conclusion
The SEM investigation shows the reduction of particle size after processing the initial mixtures in planetary ball mill. In order to assume the content and distribution of elements in the multi-component system, the XRD investigations of powders were carried out.

The selected area of the processed mixture has been analysed and spectrum analysis has been carried out. The pictures of spectrum analysis with element content are presented on figure 4.
Figure 4. Microstructure and Spectrum analysis of Fe-W-Al-Ti-Ni-B-C powder composition after 14h MA process

The analysis shows the practically equal distribution of elements in all spectrum locations, which indicates the homogenous character of the mixture. Besides, the selected element Zirconium is also observed during the analysis. The existence of Zirconium is provoked by the contact of the reaction mixture with zirconium oxide jar and balls.

Table 1. Results of Spectrum analysis of Fe-W-Al-Ti-Ni-B-C powder after 14h MA process

| Result Type | Weight % |
|-------------|----------|
| Spectrum Label | Spectrum 19 | Spectrum 13 | Spectrum 14 | Spectrum 15 | Spectrum 16 | Spectrum 17 | Spectrum 18 |
| B           | 15.64     | 13.89     | 15.26      | 9.41       | 11.03      | 13.93      | 11.34       |
| C           | 25.01     | 17.28     | 26.45      | 19.33      | 18.81      | 17.59      | 22.97       |
| O           | 13.36     | 11.12     | 13.71      | 13.69      | 11.65      | 13.38      | 10.96       |
| Al          | 10.80     | 10.60     | 10.50      | 11.23      | 10.42      | 10.17      | 12.60       |
| Ti          | 8.47      | 11.04     | 8.63       | 10.29      | 10.15      | 10.18      | 11.97       |
| Fe          | 4.29      | 11.49     | 5.78       | 12.26      | 15.10      | 11.88      | 5.89        |
| Ni          | 9.14      | 12.89     | 9.42       | 9.76       | 10.79      | 10.88      | 10.96       |
| Zr          | 2.36      | 1.88      | 1.32       | 2.37       | 1.77       | 1.60       | 1.28        |
| W           | 10.92     | 9.83      | 8.92       | 11.65      | 10.28      | 10.38      | 12.02       |
| Total       | 100.00    | 100.00    | 100.00     | 100.00     | 100.00     | 100.00     | 100.00      |

Figure 5 below shows the XRD analysis of the selected composition. The increase of the MA time leads to the reduction of peaks and therefore, we can predict the tendency of amorphization of the powder structure.
Figure 5. XRD analysis of Fe-W-Al-Ti-Ni-B-C powder after 14h MA process

Preliminary investigations can be summarized as followed:

- The theoretical and experimental investigations were carried out to select the composition in Fe-W-Al-Ti-Ni-B-C system for MA process and synthesis as a precursor of Bulk High Entropy Alloys;
- Ultrafine powders of Fe-W-Al-Ti-Ni-B-C system were prepared for Mechanical alloying and realized in a planetary ball mill with Zirconium oxide chars and balls. The duration and optimal rotation speed were established experimentally;
- Scanning electronic microscopy was carried out for the processed powders to evaluate the tendency of grain size reduction;
- XRD analysis was carried out for the powders to check the uniform distribution of elements; the dry milling process influenced the existence of oxygen and Zirconium in the selected compositions.

Based on the preliminary investigations, we can conclude that the MA process gives the desired result after processing the powders more than 14 hours. The SEM and XRD investigations give promising result to predict the amorphization of the composite structure. The Phase and chemical analysis are required to check the phase formation at the powder processing stage. The processing time is predicted to be increased before the explosive compaction technique.

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