Study of the structural evolution of Ni50%at Al50%at mixture during mechanical alloying

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***Abstract*—In this study elemental powder of 50 %.at Ni and 50 %.at Al mixture was mechanically alloyed in planetary ball mill Retsch PM400, for a predetermining time until 48h. Using X-ray diffraction and scanning electron microscopy (SEM) it was observed that milling of powder leads to the formation of NiAl phase. By the investigation of results from different technique microstructural and morphology of the powder was investigated over time of milling.**

Keywords—mechanical alloying; NiAl alloy; x-ray diffraction; SEM; laser granulometry.

I. Introduction

Nanomaterial attracted the attention of many researchers in the past decade, with the development and the progress on tools of characterization and manipulation, they will be able to control size, shape and structure conducting to unique physical and chemical properties which differ significantly and can be superior to those of conventional materials of the same composition [1, 2].

One of methods of synthesis of nanocrystalline materials is mechanical alloying (MA), which is a dry and high energy ball milling process where constituent powder are repeatedly deformed, fractured and welded by grinding media. During milling the mechanical energy is transferred to the powder, resulting in the generation of dislocation and refining of particles size which consequently favors the diffusion. Also a slight rise in powder temperature occurs during milling, lead to alloying of the blended elemental powders during the milling process. This technique enable the production of several advanced materials, including equilibrium, nonequilibrium and composite materials [3, 4].

NiAl alloy present an interesting proprieties such as their corrosion resistance, low density and high melting point accord him a great interest for industrial applications especially in high temperature applications [4, 5].

The aim of this paper is to produce NiAl alloy by mechanical alloying and ascertain the structural evolution during milling.

II. Experimental

Mixture of elemental powder of 50 at.% Ni and 50 at.% Al was milled in planetary ball mill Retsch PM400 with a hardened steel vial and stainless steel ball with 9 mm of diameter and 15:1 ball-to powder weight ratio for different predetermined hours until 48h. The speed of milling was 200 rpm with interval operation of 15 min pause time after each 15 min running time. All the preparation was done under inert argon atmosphere in a glove box.

Phase identification and transformation, and structural characterization was determined by X-ray diffraction performed in a Panalytical X'Pert PRO diffractometer (45 kV, 40 mA) with Cu Kα radiation (k = 0.15406 nm). Crystallite size was calculated using the Scherrer formula (1)

$$D = K.\lambda \;/\; \beta L \cos\theta \qquad (1)$$

Microstructure observations was done using a JEOL 6360 microscope equipped with EDX analyzer.

Particle size and distributions of resulted powder of milling were measured by using a Malvern Master Sizer E version 1.2b laser scattering machine.

III. Results And Discussion

A. X-ray diffraction

Fig. 1, represents the spectral of mechanical milled powder of Al 50%.at Ni mixture over time of milling. At the beginning after 0.5h of milling we can clearly observe the diffraction pics of the (fcc) Al and (fcc) Ni of the raw materials, with progress of milling the pics of Al disappear and the pic of Ni persist with a reduction of intensity and a displacement to the low angles, enabling the formation of new phase corresponding to the (bcc) NiAl.

From this observations we can say that the impact of milling conduit to a distraction of arrangement in aluminum structure, while the lattice of Ni undergo a dilation with increasing of milling time. The atoms of Al are incorporated into the Ni lattice resulting in the formation of NiAl alloy.

Fig.1. X-ray diffraction patterns of mechanical milled powder over time of milling.

As presented in Fig. 1, the pics width broaden with increasing of milling time, which can be attributed to a reduction in crystallite size and an increase of the atomic strain. Where the NiAl alloy present 8.6 nm at 20h of milling and 7.5 nm after 48h.

B. Electron microscopy and analysis

1) EDS

The global chemical analysis obtained by EDS for the mechanical milled powder over time of milling is presented in Fig. 2, we can clearly observe that the composition approach to an equilibrium proportion after 6h of milling.

Fig. 2. EDS results over time of milling.

2) SEM

As presented in Fig. 3, the particles of Al and Ni are clearly visible after 0.5h of milling with a bright and dark contrast appearance respectively, as confirmed by EDS analysis. However the particles take flaky and spherical shape.

Fig. 3. SEM micrograph and EDS analysis of Ni50%atAl mixture after 0.5h of milling time.

After 6h of milling (Fig. 4), the mechanical milled powder presents an aggregate of spherical particle with homogeneous composition of Ni and Al.

Fig. 4. SEM micrograph and EDS analysis of Ni50%atAl mixture after 6h of milling time.

With progress of milling the particles are more refined and touch the micrometer scale as presented in Fig. 5.

Fig. 5. SEM micrograph after: (a) 8h; (b) 20h; (c) 36h and 48h of milling time.

C. Laser granulometry

Fig. 6, presents the granulometry analyses of the resulted powder of milling after 48h, we observe the presence of two population: one with size between 0.8 and 40 µm, second with low population of size varying between 30 and 300µm.

Fig. 6. Particle-size distributions of mechanical milled powder after 48h.

IV. Conclusion

The NiAl alloy was successfully synthesized by mechanical alloying. From SEM and EDS results, we have noted that the composition was homogenous after 6h of milling while the particle shows spherical shape. After 48h of milling, the most particles present a size between 0.8 to 40 µm, moreover the crystallite size attain 7.5 nm.

References

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