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The structural changes of Al-Ti-Fe alloy during mechanical alloying process

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Materials

ABSTRACT

Purpose: The aim of this paper is to determine the influence of the mechanical milling process on the structure of $Al_{67}Ti_{25}Fe_8$ alloy.
Design/methodology/approach: The powders of the $Al_{67}Ti_{25}Fe_8$ alloys were obtained by mechanical alloying method in a planetary Fritsh Pulverisette 5 mill. In order to investigate the structure scanning electron microscopy were used. Phase transformations were determined by means of diffractometer. The cross-sectional microstructure evolution and element distribution of $Al_{67}Ti_{25}Fe_8$ powder alloys were investigated using backscattering electrons of SEM. The distribution of powder particles was determined by a sieve analysis.
Findings: The laboratory test show that, by using the mechanical alloying method, one can produce $Al_{67}Ti_{25}Fe_8$ alloys with intentional chemical constitution and desirable structure. Inside the milling materials didn't find some impurities and undesirable phases.
Research limitations/implications: Property of $Al_{67}Ti_{25}Fe_8$ alloys correction is possible by refinement of grains and modification of phases composition. All of the presented experiments in this article are made on a laboratory scale. Continuation of the investigations in the field of sintering alloyed powders to obtained massive materials is foreseen.
Practical implications: In the nearest future the producing of bulk materials characterized by better properties in comparison with traditional materials will take place not only in the laboratory scale and move to the industry.
Originality/value: : In addition a good microstructural homogeneity and first of all mechanical properties was achieved, also practical application will be possible. The Al-Ti-Fe alloys have been considered to be potentially important for applications at high temperature by reason of their low density end expected high specific strength.
Keywords: Metallic alloys; Powder metallurgy; Al-Ti-Fe alloy; Mechanical alloying

1. Introduction

A growing trend to use new nanocrystalline, functional materials is recently observed world-wide. Within this group of materials particular attention is focused on the nanometric grain size of a aluminum matrix. These materials exhibit higher mechanical properties than microcrystalline aluminum.

The mechanical alloying (MA) technique is a simple method to obtain compound phases from elemental powders. Mechanical alloying method enable to obtain supersaturated solid solution, intermetallic phases, crystalline and amorphous phases. Mechanical alloying is a complex process that require optimizing many parameters to achieve desirable material. The following parameters have an influence on powder structure and properties: process control agent, ball to powder weight ratio, time of milling process, the mill type, atmosphere. The essence of the mechanical alloying process is the action of the grinder balls colliding with powder grains and the interaction of the powder grains. During this process the change of the chemical composition and material microstructure occurs [1-10].

The Al-Ti and Al-Fe alloys have specific properties, for example: low density, high strength, good corrosion resistance at elevated temperature. They are very attractive materials for the aircraft and defense industry. There have been a lot of investigations on the mechanical milling of binary Al-Ti and Al-Fe systems. The mechanical alloying process of aluminium, iron and titanium powders has been studied by Liu et al., Bonetti et al. and Venkatasuwamy et al., separately. The addition of a third element to the binary chemical constitution can improve their properties. For example: Al is very ductile, Ti element has a positive influence on tribological properties, increase the yield strength, raise the transition temperature and produced precipitation strengthening at elevated temperatures [11-21].

The main goal of the present investigation was to study the structural and phase transformations during mechanical alloying of the $Al_{67}Ti_{25}Fe_8$ alloy in a planetary ball mill. This paper is concerned with the structural characterization of mechanically alloyed Al-Ti-Fe powders. Development of a structure during deformation of these materials was investigated.

2. Experimental procedure

The aim of the present work is the characterization of the milled Al-Ti-Fe alloy using XRD (X-Ray Diffraction) and SEM (Scanning Electron Microscopy) methods. The mechanical alloying process of the pure aluminum, titanium and iron powders was realized. Characteristic of powders used to manufacturing materials is given in Table 1. The mechanical alloying process was carried out in a high-energy planetary ball mill Fritsch Pulverisette 5. In this process wasn't added process control agent. The ball to powder weight ratio was 8:1. In order to prevent powder impurities, the samples were sealed in the vial under argon atmosphere. The powders were ground for 10, 45 and 100 hours. The microscopic observation of the shape and size of the powdered material particles was carried out by means of the OPTION DS 540 scanning electron microscope, within the magnification of 500 and 2000 times. The macroscopic observation were carried out too. The changes of the phase constitution were tested by means of the Philips PW 1140 X-ray diffractometer. Powder sample were analyzed by energy dispersion spectroscopy too. The measurements of particles size were carried out by means of the sieve analysis.

3. Results

The as-milled powders consist of a fine mixture of pure elements and amorphous phases. The phase X-ray analysis no exhibit the changes in the phase composition occurring in the 100 hrs of mechanical alloying process. The X-ray diffraction patterns of powder milled for 10 and 100 hrs show the peaks characteristic for Al, Ti and Fe-α (Fig. 1, Fig. 2). A little extending of peaks is connected with the size reduction in the powder grain as well as with the presence of considerable stresses resulting from the intensive plastic strains occurring during the grinding.

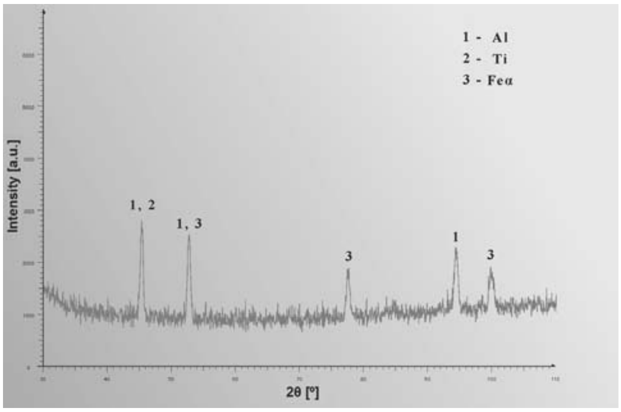

Fig. 1. The X-ray diffraction patterns of $Al_{67}Ti_{25}Fe_8$ powder alloy after 10 hrs of mechanical alloying

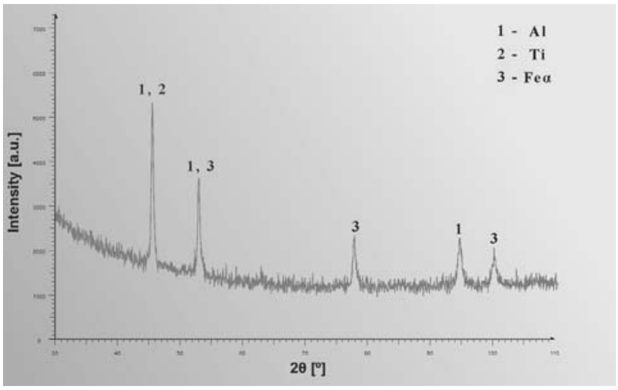

Fig. 2. The X-ray diffraction patterns of $Al_{67}Ti_{25}Fe_8$ powder alloy after 100 hrs of mechanical alloying

Table 1.
Chemical composition, particle size and purity

Chemical element	Particle size [μm]	Purity [%]	at. [%]	wt. [%]
Aluminium	44	99.5	67	18.2433
Titanium	149	99.5	25	7.2475
Iron	74	99.98	8	4.5092

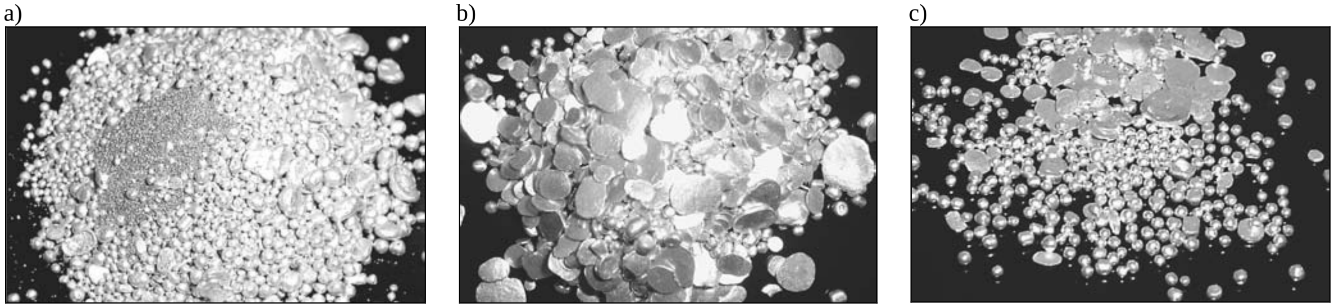

Fig. 3. Powder particles after a) 10hrs, b) 45hrs, c) 100hrs, of mechanical alloying (macroscopic observation)

There is a small broadening of the XRD patterns and increasing the background as mechanical attrition progresses, suggesting the formation of an amorphous phase, fine crystalline grains and high density of defects.

Observation of morphology of powder mixture after 10, 45 and 100hrs mixing process respectively allows that the mechanical milling process have an very intensive influence on morphology changes of the initial powders Figure 3. Extension of mechanical milling process up to 45hrs cause particle flatten. The particle agglomerates are thin and flat. However, after 100hrs of milling, the particles are predominantly globular.

The scanning electron microscopy observations of the initial samples morphology are presented in Figs. 4-5. The shape of particles powder is irregular. Structure' $Al_{67}Ti_{25}Fe_8$ powder alloy after 45hrs of mechanical alloying is presented in Fig. 7.

Powder particle surface structure of Al-Ti-Fe alloy after 10 and 100 hrs of mechanical alloying show Fig. 6 and Fig. 8.

The cross-sectional microstructure evolution and element distribution of $Al_{67}Ti_{25}Fe_8$ powder alloy were investigated using SEM. Typical images of the cross section of $Al_{67}Ti_{25}Fe_8$ powders after 10, 45 and 100 hrs of mechanical alloying are shown in Figs. 9-12.

Bright, grey and dark areas in this micrographs correspond to Fe, Ti and Al, respectively. From this images, it can be seen that the elemental distribution of Fe, Ti and Al was not uniform in this stage of milling.

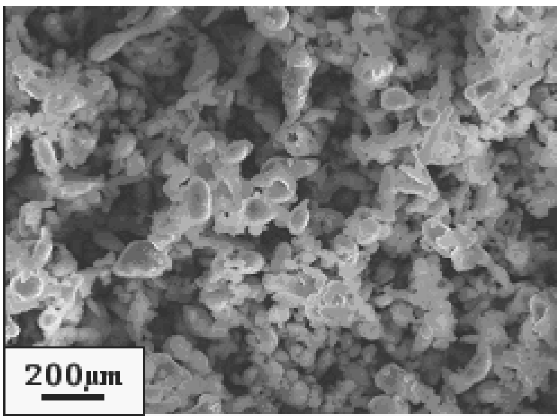

Fig. 4. Structure of initial powder mixture of $Al_{67}Ti_{25}Fe_8$ alloy

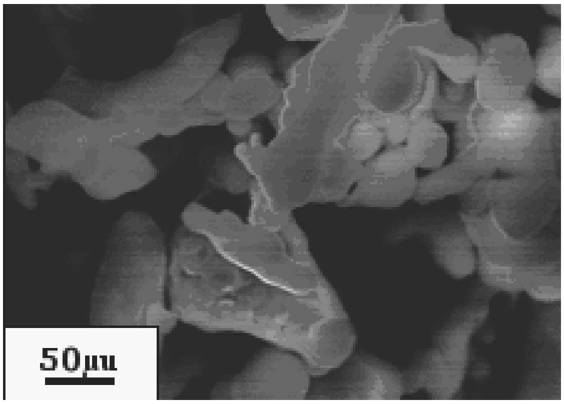

Fig. 5. Structure of initial powder mixture of $Al_{67}Ti_{25}Fe_8$ alloy

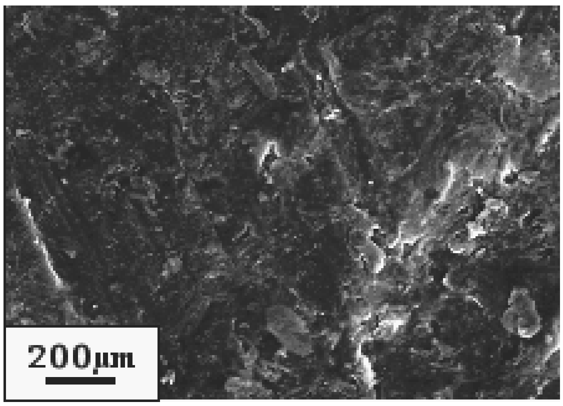

Fig. 6. Structure of powder particle surface of $Al_{67}Ti_{25}Fe_8$ alloy after 10 hrs of mechanical alloying

During the process powders become effectively broken up while the equilibrium between cracking and joining is fixed, which results in the laminar structure. All chemical elements of the investigated alloys are listed in Table 2. Chemical composition of areas' powder simple after 100 hrs of mechanical alloying are shown in Figure 13 and in Table 2.

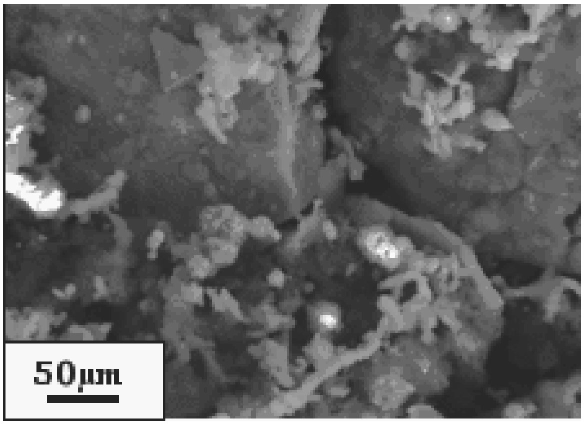

Fig. 7. Structure of powder mixture of $Al_{67}Ti_{25}Fe_8$ alloy after 45hrs of mechanical alloying

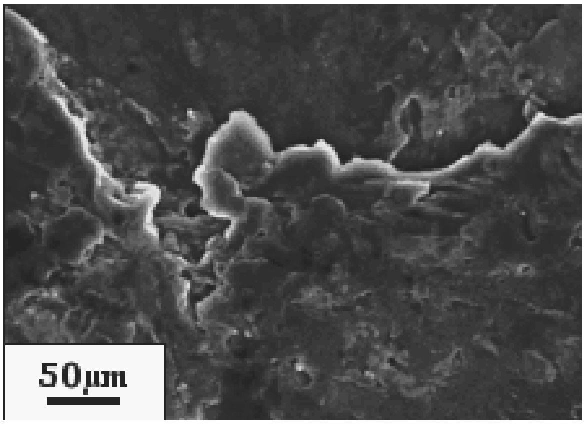

Fig. 8. Structure of powder particle surface of $Al_{67}Ti_{25}Fe_8$ alloy after 100 hrs of mechanical alloying

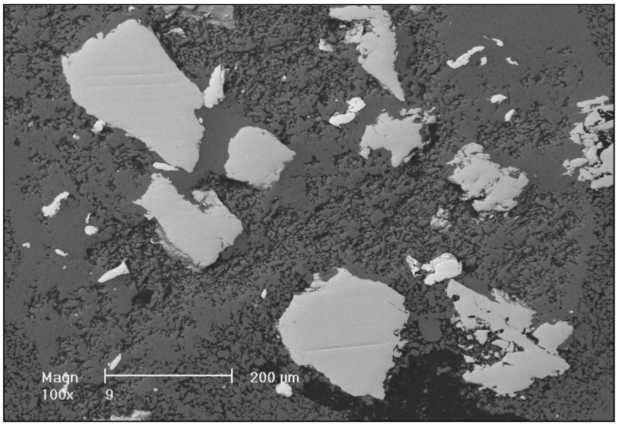

Fig. 9. Cross section of particles of $Al_{67}Ti_{25}Fe_8$ initial powder alloy (SEM)

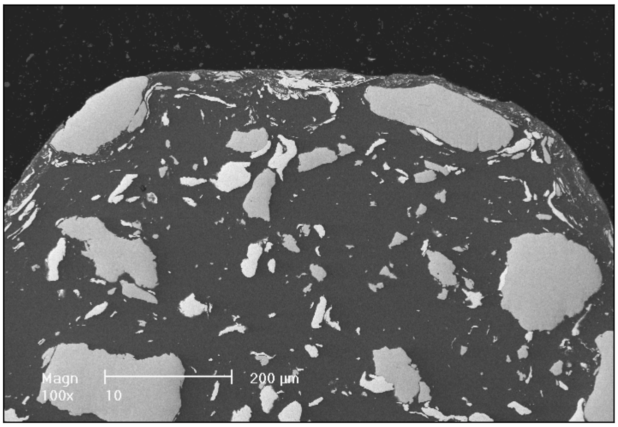

Fig. 10. Cross section of particles of $Al_{67}Ti_{25}Fe_8$ powder alloy mechanically milled for 10 hrs (SEM)

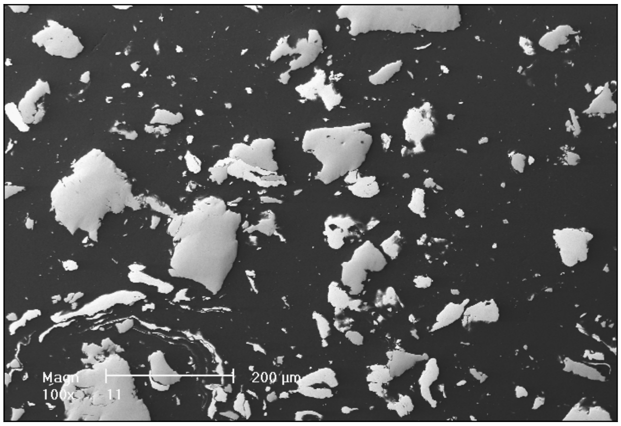

Fig. 11. Cross section of particles of $Al_{67}Ti_{25}Fe_8$ powder alloy mechanically milled for 45 hrs (SEM)

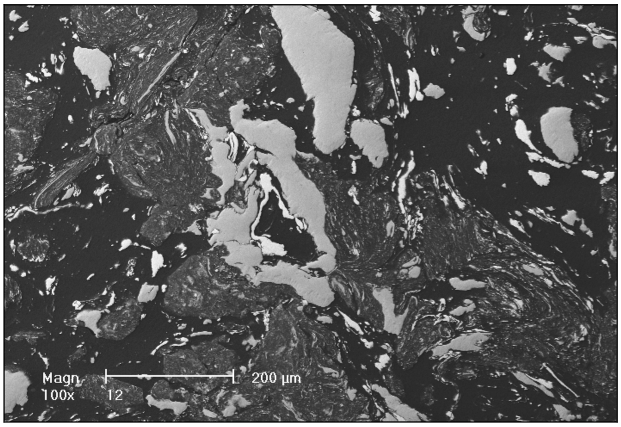

Fig. 12. Cross section of particles of $Al_{67}Ti_{25}Fe_8$ powder alloy mechanically milled for 100 hrs (SEM)

Table 2.
Chemical composition of powders investigated in the present study (EDS)

Test area	Element	at. [%]	wt. [%]
1	Al	2.90	1.42
1	Ti	0.54	0.47
1	Fe	96.56	98.11
2	Al	2.23	1.27
2	Ti	97.77	98.73
3	Al	100	100
4	Al	89.08	80.65
4	Ti	4.17	6.7
4	Fe	6.75	12.65

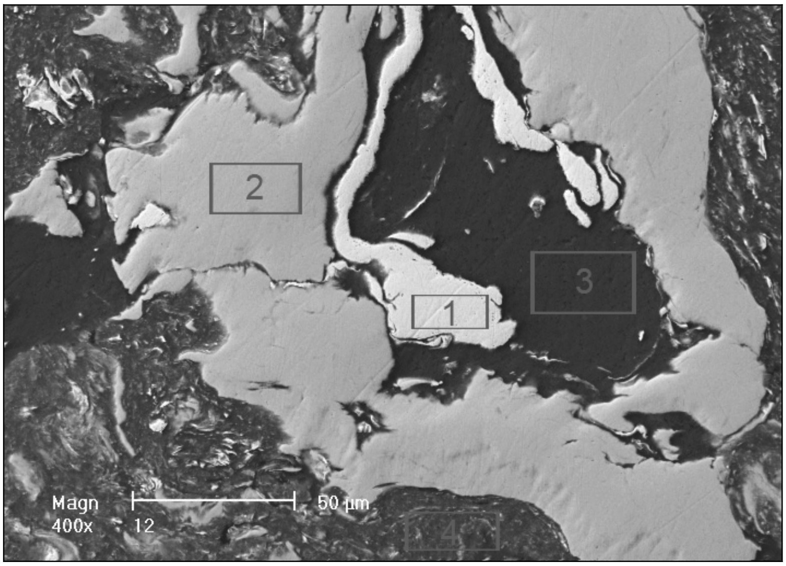

Fig. 13. Cross section of $Al_{67}Ti_{25}Fe_8$ powder alloy mechanically milled for 100 hrs (SEM)

The investigation results have shown that the obtained powder particles are non-homogeneous. Agglomeration of nanopowder particles is a serious problems.

The inhomogeneities (heterogeneity) of chemical composition were observed in some areas.

Examinations of the chemical composition on the section particles made by the X-ray energy dispersive spectrometer (EDS) indicate that iron dominates inside of light area's (1) of these micro-particles. Plot of the X-ray dispersive energy spectrometer measurement from the $Al_{67}Ti_{25}Fe_8$ alloy after 100hrs of mechanical alloying process (point 1) is shown on Fig. 14.

Examination of the chemical composition of the $Al_{67}Ti_{25}Fe_8$ alloy particles made by the X-ray energy dispersive spectrometer indicate that titanium dominate in grey field (2) and aluminum dominate in dark field (3). Plot of the X-ray dispersive energy spectrometer measurement from the $Al_{67}Ti_{25}Fe_8$ alloy after 100hrs of mechanical alloying process (point 2 and point 3) are illustrated in Figs. 15 -16.

Laboratory test of the chemical composition of the $Al_{67}Ti_{25}Fe_8$ alloy (Fig. 17) indicate that the process is still not come to an end (point 4 in Fig. 13). The mechanical alloying process of tested material one should increase.

The important factor determining properties of materials is the particle size. The distribution of powder particles size was determined by a sieve analysis. The powder particles size and their distribution are shown in Figs. 18-20. The influence of mechanical alloying time process on the shape and size of powder particles is great.

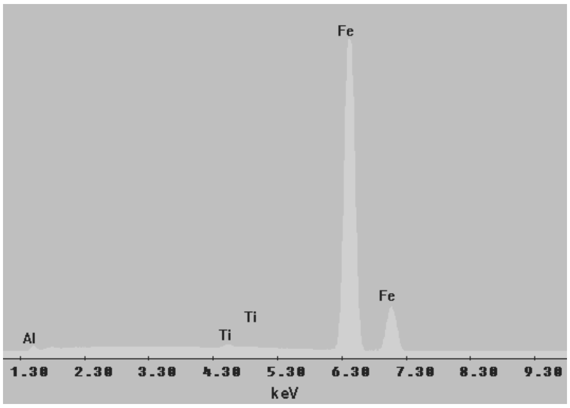

Fig. 14. Plot of the X-ray dispersive energy spectrometer measurement from the $Al_{67}Ti_{25}Fe_8$ alloy after 100hrs of mechanical alloying process (point 1 in Fig. 15.)

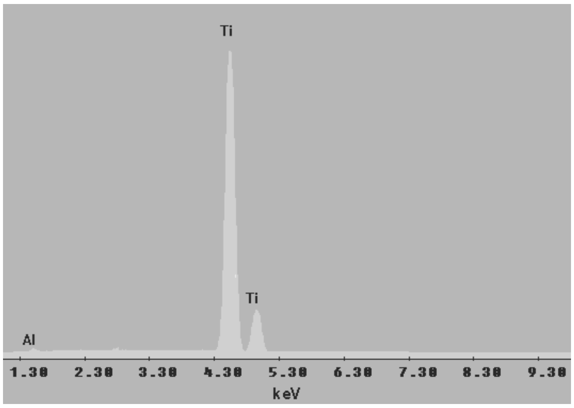

Fig. 15. Plot of the X-ray dispersive energy spectrometer measurement from the $Al_{67}Ti_{25}Fe_8$ alloy after 100hrs of mechanical alloying process (point 2 in Fig. 15.)

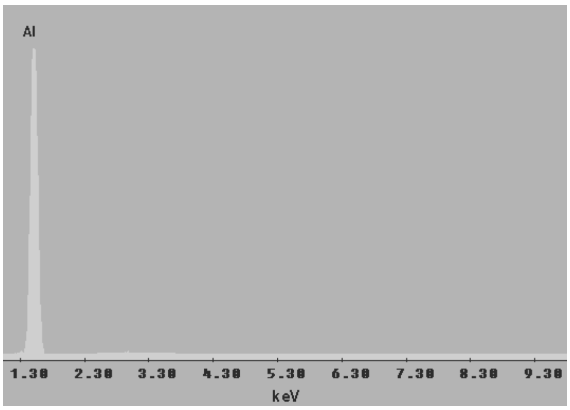

Fig. 16. Plot of the X-ray dispersive energy spectrometer measurement from the $Al_{67}Ti_{25}Fe_8$ alloy after 100hrs of mechanical alloying process (point 3 in Fig. 15.)

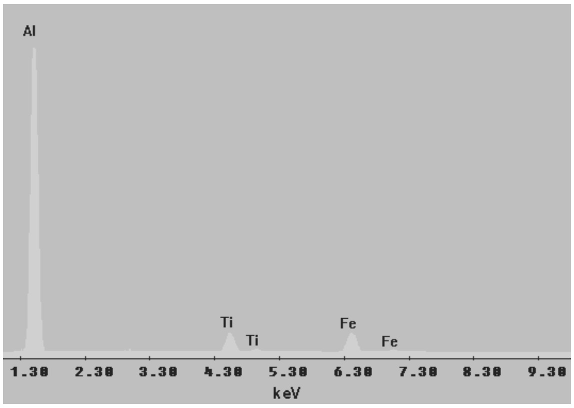

Fig. 17. Plot of the X-ray dispersive energy spectrometer measurement from the $Al_{67}Ti_{25}Fe_8$ alloy after 100hrs of mechanical alloying process (point 4 in Fig. 15.)

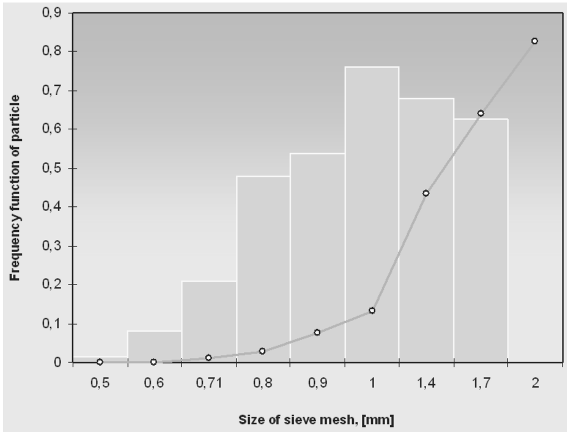

Fig. 18. Particle size distribution of studied $Al_{67}Ti_{25}Fe_8$ powder after 10hrs. of mechanical alloying

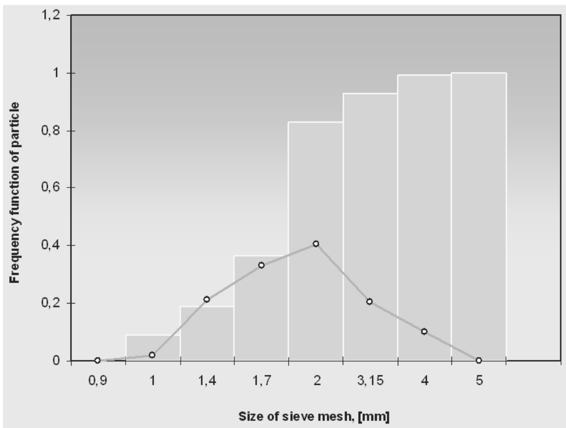

Fig. 19. Particle size distribution of studied $Al_{67}Ti_{25}Fe_8$ powder after 45hrs. of mechanical alloying

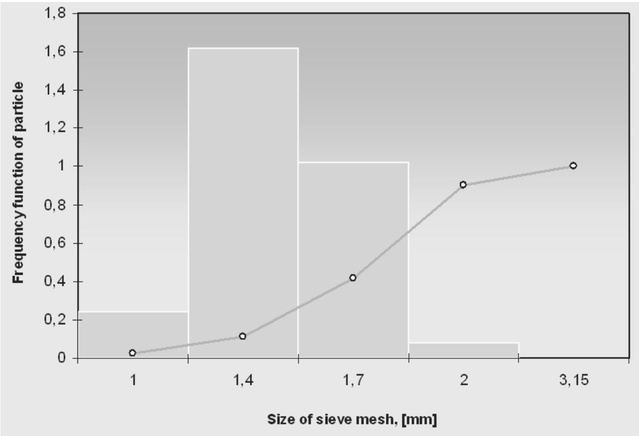

Fig. 20. Particle size distribution of studied $Al_{67}Ti_{25}Fe_8$ powder after 100hrs. of mechanical alloying

4. Conclusions

The relationship between the initial materials, structure and mechanical alloying process was specified. Mechanical alloying method can be used to produce sufficient materials in Al-Ti-Fe systems.

The laminar structure generally result from repeating joining and cracking of powder particles during mechanical alloying process. Probably, the laminar structure will be broken as the process will be continued, a homogeneous structure will be formed.

The process control agents can strongly influence on the course of mechanical alloying process and phase composition of the manufactured $Al_{67}Ti_{25}Fe_8$ alloy. Aluminium is very ductile, therefore a process control agents should be used for preventing of sedimentation of Al powder to the mill's walls and grinding medium. Probably, to avoid the grain growth and sedimentation application of stearine acid or Microvax were performed.

We expect, that samples of the consolidated, tested materials will have higher mechanical properties than those of similar materials with microcrystalline size of grains.

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