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A production attempt of $Ni_{50}Ti_{50}$ and $Ni_{52}Ti_{41}Nb_7$ alloys by mechanical alloying method

W. Pilarczyk [a,*], R. Nowosielski [a], A. Pilarczyk [b], P. Sakiewicz [a]

[a] Division of Nanocrystalline and Functional Materials and Sustainable Pro-ecological Institute of Engineering Materials and Biomaterials, Silesian University of Technology, ul. Konarskiego 18a, 44-100 Gliwice, Poland

[b] Institute of Welding, ul. Bł. Czesława 18a, 44-100 Gliwice, Poland

* Corresponding author: E-mail address: wirginia.pilarczyk@polsl.pl

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ABSTRACT

Purpose: The main aims of this work are the production attempt of $Ni_{50}Ti_{50}$ and $Ni_{52}Ti_{41}Nb_7$ powder alloys by mechanical alloying method, the presentation of the influence of mechanical alloying time on the structure of obtained alloys and the finding of thermal effects during the heating to temperature of 700ºC.

Design/methodology/approach: The test material was the mixture of pure nickel, titanium and niobium powders. The powders were ground for the 5, 25 and 40 hrs. The mechanical alloying process was conducted in a high energy SPEX mill under inert argon atmosphere. The microscopic observation of the shape and size of the powdered material particles was carried out by the scanning electron microscope. The changes of the powder structure were tested by means of the X-ray diffractometer. The thermal properties of the powder alloys were examined by DSC method.

Findings: Based on the presented experiment results it is clear that producing of assumption powder alloys by mechanical alloying method is possible, but special attention is needed during the selecting of process parameters. The application of used method gives possibility to produce crystalline and amorphous phase in Ni-Ti and Ni-Ti-Nb powder alloys.

Research limitations/implications: The experiments in this work are made only on a laboratory scale. Further investigations should be concentrate on the developing of powder consolidation method and refinement particles during high energy ball milling.

Practical implications: Ni-Ti alloys exhibit unique shape-memory effects, good corrosion resistance, high wear resistance, biocompatibility and superplasticity. Ni-Ti intermetallic compounds have been widely used in a different fields: mechanical, electric and biomedical applications, aeronautics and astronautics fields.

Originality/value: The Ni-Ti and Ni-Ti-Nb powder alloys produced by mechanical alloying method can be use to produce bulk materials with desirable mechanical, physical and chemical properties.

Keywords: Metallic alloys; Ni-Ti alloy; Ni-Ti-Nb alloy; Mechanical alloying; Powder metallurgy

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MATERIALS

1. Introduction

The NiTi intermetallic compounds are attractive functional materials because of their excellent shape memory effect, satisfying biocompatibility, good mechanical properties, super elasticity and corrosion resistance [1,2]. Ni-Ti shape memory alloys have been successfully used in engineering, medical and orthopaedic applications due to their above enumerated excellent properties [3,4]. Thanks to the advance in development of material engineering it is possible to make use of porous Ni-Ti shape memory alloys to biomedical applications for hard tissue replacement, in particular for hip implantation and femur repair. From the analysis of literature data it can be concluded that these porous alloys have the adjustable mechanical properties, reduced weight and increased biocompatibility due to their porous structure allowing in-growth of the human tissue, nutrition exchange and medicament transportation within human bodies [3,5]. On the one hand a porous structure of determined mechanical properties as a bone is desired for implant, on the other hand, it requires controlling pore size, morphology and orientation [3].

Ni-Ti and Ni-Ti-Nb based intermetallic alloys have found wide applications in medical implants, intelligent control, aerospace engineering, astronautics field [1,2,6,7,8]. It has been shown that the Ni-Ti-Nb alloys are important functional materials for their unique excellent mechanical properties, wide hysteresis and shape memory effect, that have made good use for applications as coupling and fasteners [5,9,10]. With the widespread development and applications of Ni-Ti based alloys as shape memory materials and as potential high temperature structural materials for example Ni-Ti-Al alloys, the improvement of oxidation and corrosion resistance of these alloys is very important [1,11].

In last decade, mechanical alloying method of elemental Ni, Ti and additional elements powder mixture for fabricating intermetallic compounds has been widely investigated [1,6,12-17]. Currently, there are some, powder metallurgy methods [18-25] for fabrication of porous Ni-Ti shape memory alloys, for example elementary powder sintering, hot isostatic pressing (HIP), spark plasma sintering, self-propagating high-temperature synthesis (SHS), capsule-free hot isostatic pressing, explosive shock-wave compression, metal injection moulding [3]. In accordance with earlier publication it was found that SHS offers more advantages compared to conventional powder metallurgy. Evidence in the literature indicates that the SHS method results in higher levels of porosity and a higher degree of NiTi formation [3].

Y. Zhou et al. [6] have conducted investigations of effect of self-propagating high-temperature combustion synthesis on the deposition of NiTi coating by cold spraying using mechanical alloying Ni, Ti powders. The Ni and Ti powders after mechanical alloying are chemically active and self-propagating high-temperature synthesis reactions is easy ignited. As a consequence of this, the deposition of efficiency of mechanical alloying powder particles during cold spraying may be affected by possible SHS reaction of MA powder in cold spraying.

The addition of Nb elements is interesting because it can remarkably alter the oxidation kinetics and drastically improve oxidation resistance at elevated temperature. X. Zhao et al. [1] carried out a tests in the Ni-Ti-Nb system. They investigated the influence of Nb on the oxidation behavior of single phase Ni-Ti-Nb alloys at elevated temperatures. As a result of the tests it was observed that the addition of Nb can improve the high temperature oxidation resistance of Ni-Ti alloy. In the range (0-7 %at. Nb) of Nb addition of the [1] study, the higher amount of Nb addition resulted in better resistance to high temperature oxidation. They have observed also that during high temperature oxidation of binary Ni-Ti, the oxidation products are composed of TiO_2 and $NiTiO_3$. The addition of Nb changes the oxidation kinetics and the microstructure of the oxide scales. The oxide layer formed on the Ni-Ti-Nb alloys consists of TiO_2 and Nb-rich nonstiochiometric oxide layer [1]. In the opinion of the authors of this publication the Nb-rich oxide layer formed beneath TiO_2 can effectively impede the interdiffusion of oxygen and metallic cations during oxidation process, resulting in a remarkable improvement of oxidation resistance of Ni-Ti-Nb alloys at high temperature [1].

He et al. found that $Ti_{46.9}Ni_{50.1}Nb_3$ alloy has better shape memory effect than $Ti_{44}Ni_{47}Nb_9$ and exhibits wide enough transform temperature hysteresis after deformation [7,26]. Zhao et al. have produced Ni-Ti-Nb alloys with 4.5 at.% Nb. They indicated that Nb dissolved in NiTi matrix is responsible for the hysteresis expansion. In this experiment high strength was obtained [7,26].

The effect of low Nb content (3.5 at.%, 4.5 at.%, 5.0 at.%) on the mechanical properties of Ni-Ti-Nb shape memory alloy has been investigated [9]. It has been observed that it is possible to control of the M_s temperature through adjusting Nb content and Ni, Ti ratio in Ni-Ti-Nb alloys. They have also observed that the yield strength of Ni-Ti-Nb alloys with low content at room temperature grows with the increase of Nb content, while the elongation decreases, but still in a high level. The deformation behavior at 193 K shows the similar trend [9]. As a result of the tests it was measured that the $Ni_{49.8}Ti_{45.1}Nb_{5.0}$ alloy has 640 MPa yield strength, 930 MPa rupture strength, 11% elongation at room temperature and 7.4 % shape recovery strain. It is believed that Ni-Ti-Nb alloys with low Nb content can obtain excellent shape memory characteristics [9,26].

The mechanical alloying of $Ni_{50}Ti_{50}$ powder alloy has been investigated in the literature, but different results have been reported using the same initial powder composition. It has been shown that the MA of $Ni_{50}Ti_{50}$ elemental powder mixture can lead to amorphous phase formation, while other scientists reported that the products are nanocrystalline solid solution and amorphous phase [5].

Y. Zhou et al [6] have conducted investigations of the microstructure of the $Ni_{50}Ti_{50}$ powder particles milled for different times. The high deformation leads to the formation of layered structure of the constituent metals. The authors observed cross-section of powder particles after 4h of mechanical alloying process. The layer structure of Ni and Ti was clearly observed, the distribution of layers was not uniform due to short time of mechanical alloying. In this experiment it was observed that with the continuous mechanical alloying time to 8h, Ni and Ti particles became finer and a uniform lamellar structure was formed. What's more, further mechanical alloying to 14 h resulted in a refined lamellar structure. The authors [6] suppose that the lamellar structure in milled powders could decrease the diffusion distance of the powder constituents to the micrometer even nanometer range, which was beneficial to the formation of Ni(Ti) solid solution. The scientists Y. Zhou et al. [6] also conducted

thermal research. They found out the exothermic reactions occurred with the heating of the alloyed powders. The peak temperatures of the first exothermic peaks on DSC curves of Ni-Ti powders milled for 4 h, 8 h and 14 h were 515ºC, 460ºC and 430ºC, respectively. The scientists stated that those exothermic peaks were attributed to recrystallization of Ni-Ti alloys. The hardness of the powders was increased with the increase of mechanical alloying time. The micro-hardness values of powders milled for 4h, 8h and 14h were 358HV, 388HV and 431HV, adequately [6].

T. Mousavi et al. [5] carried out a tests in the Ni-Ti system, too. They studied the mechanism of NiTi formation using mechanical alloying method. The phase transition, micro hardness and morphology of the powders during mechanical alloying and after annealing process were investigated by this group of researchers. In the opinion of the authors of this publication the mechanical alloying of $Ni_{50}Ti_{50}$ powders leads to the synthesis of disordered nanocrystalline B1-NiTi intermetallic phase. A mechanism has been proposed for the formation of NiTi [5]. According to this mechanism a composite lamellar structure is formed first with the dissolution of Ti in Ni at the same time. With the progress of mechanical alloying process, the obtaining of supersaturated solid solution becomes non-stoichiometric and finally leads to the formation of disordered NiTi [5]. By annealing of the milled powder, grain growth, microhardness decrease and transformation of disordered structure to ordered NiTi with long-range order of 0.94 can be achieved. Small amount of $NiTi_2$ and Ni_3Ti phases was also detected in the annealed NiTi.

Recently, the solid state amorphization reaction of Ni-Ti system has been extensively investigated [27]. Schwarz et al. observed that partial solid state amorphization reaction in $NiTi_2$ during mechanical alloying is completed by successive annealing at about 680 K [27]. Batteazi et al. have observed that the phases crystallized from the amorphous $Ni_{50}Ti_{50}$ change with mechanical alloying time [27].

The structural relaxation in amorphous Ni-Ti alloys prepared by mechanical alloying method was studied by Y. Makifuchi et al. [27]. The structural changes in the amorphous state during mechanical alloying have been revealed with Ni-Ti alloys of a wide composition range. As a result of the tests it was observed that in the $Ni_{33}Ti_{67}$ alloy, two distinct crystallization steps to $NiTi_2$ appeared by prolonged mechanical alloying after termination of amorphization. The authors [27] suppose that the crystallization at a higher temperature is associated with a higher activation energy and predominates with the progress of mechanical alloying. As mentioned above, scientific research of $Ni_{50}Ti_{50}$ shows that a DSC exothermic peak due to the structural relaxation leading to the crystallization to $NiTi_2$ and Ni_3Ti appeared, implying a phase separation in the amorphous phase. In the $Ni_{70}Ti_{30}$ alloy, the amorphous phase transform into crystalline solid solution of Ni by mechanical alloying elongation [27].

2. Experimental procedure

The main aims of the presented work are the production attempt of $Ni_{50}Ti_{50}$ and $Ni_{52}Ti_{41}Nb_7$ powder alloys by mechanical alloying method, the presentation of the influence of mechanical alloying time on the structure of obtained powder alloys and the finding of thermal effects during the heating to temperature of 700ºC.

Ni-based powder alloys with follow compositions of $Ni_{50}Ti_{50}$ and $Ni_{52}Ti_{41}Nb_7$ where prepared by mechanical alloying method of the pure Ni, Ti and Nb elements. Characteristic of powders used to manufacturing materials is given in Table 1 and Table 2. The mechanical alloying process was carried out in a high-energy SPEX 8000 mill of the shaker type. In this process added process control agent (1% mass. stearic acid). 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 5 h, 25 h and 40 h.

Table 1. Characteristic of initial powder

Chemical element	Nickel	Titanium	Niobium
Particle size [μm]	44	149	44
Purity [%]	99.8	99.5	99.8

The structure was tested with X-ray diffraction (XRD) using a Seifert - FPM XRD 7 diffractometer with Co Kα radiation at 35 kV. The data of diffraction lines were recorded by means of the stepwise method within the angular range of 20° to 100° and the counting time in the measuring point was 3 s.

Scanning electron microscope (SEM) by a large depth of view allows to observe the powder particles. It is a primary tool for research which is used to analyze the shape and evaluate its morphology. The microscopic observation of the morphology of studied materials with different time of mechanical alloying was carried out by means of the OPTON DS 940 scanning electron microscope, within the magnification 100-500 times.

Using differential scanning calorimetry (DSC) it is possible to observe fusion and crystallization process. Differential scanning calorimetry was carried out in a Mettler Toledo DSC 822e analyzer. The powder sample (about 30 mg) was taken and used for the DSC measurement. It was heated to 700ºC at a constant heating rate of 50ºC/min.

Table 2.
Chemical composition and time of grinding

Chemical element	Ni [at. %]	Ni [mass. %]	Ti [at. %]	Ti [mass.%]	Nb [at. %]	Nb [mass.%]	Time of grinding [h]
I	50	55.07	50	44.93	-	-	5
II	50	55.07	50	44.93	-	-	25
III	52	53.87	41	34.65	7	11.48	5
IV	52	53.87	41	34.65	7	11.48	25
V	52	53.87	41	34.65	7	11.48	40

3. Results and discussion

3.1. X-ray analysis

The X-ray phase analysis proved the changes occurring during the MA process. The X-ray diffraction patterns of the powders showed also the dependence of changes in the phase composition versus the time of grinding.

Usually, the X-ray peaks characteristic for elements which dissolve in other components fade. Whereas, position of X-ray peaks characteristic for solvent elements are displaced. It proves distinct changes of crystalline structure parameters.

The diffraction records of powders of $Ni_{50}Ti_{50}$ alloy vs. the different time of grinding are shown in Fig. 1 and Fig. 2. The diffraction pattern of $Ni_{50}Ti_{50}$ alloy recorded for the powder after 5 h and 25 h shows the peaks characteristic for Ni and Ti phases, whereas X-ray peaks originating from the NiTi phase were observed. When the grinding time increases, all X-ray peaks become wider and their intensity considerably decreases.

The changes of character of background in surroundings of line Ni and Ti show the beginning of synthesis of amorphous phase.

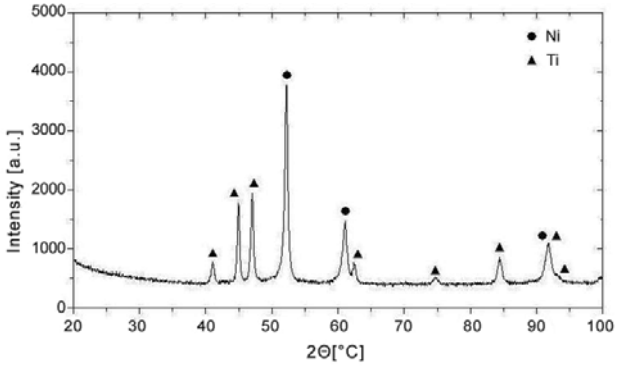

Fig. 1. The X-ray diffraction patterns of $Ni_{50}Ti_{50}$ powder alloy vs. the grinding time 5 hours

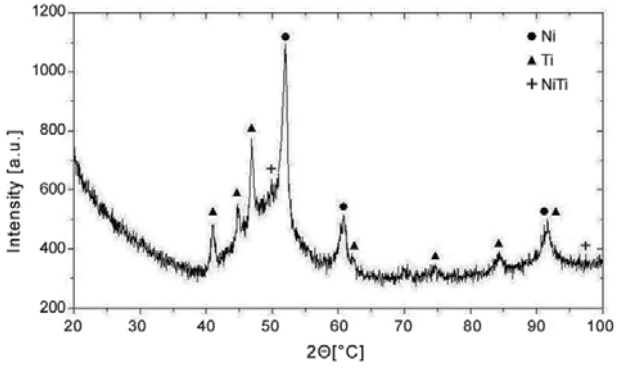

Fig. 2. The X-ray diffraction patterns of $Ni_{50}Ti_{50}$ powder alloy vs. the grinding time 25 hours

The diffraction pattern recorded for the powder of $Ni_{52}Ti_{41}Nb_7$ ground for 5 h, 25 h and 40 h shows the peaks characteristic for Ni, Ti and Nb phases. The diffraction records of powders versus the time of grinding are shown in Figures 3-5.

The presence of diffractive lines characterizing the nickel, titanium and a little of NiTi phase shows, that the synthesis of $Ni_{50}Ti_{50}$ does not take place to the end.

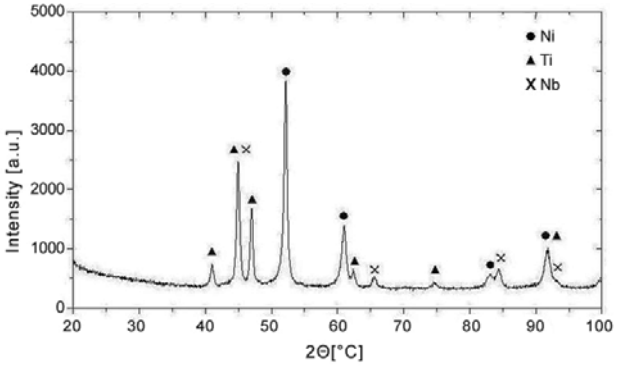

Fig. 3. The X-ray diffraction patterns of $Ni_{52}Ti_{41}Nb_7$ powder alloy vs. the grinding time 5 hours

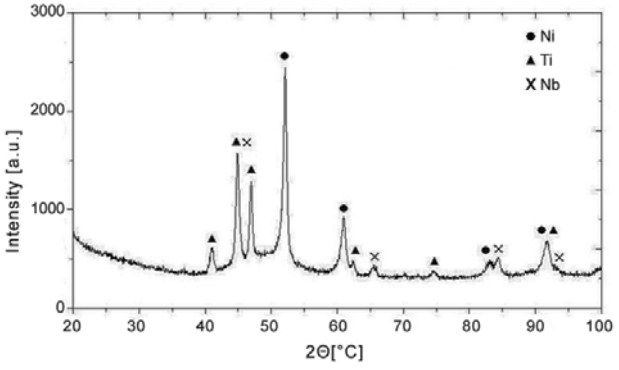

Fig. 4. The X-ray diffraction patterns of $Ni_{52}Ti_{41}Nb_7$ powder alloy vs. the grinding time 25 hours

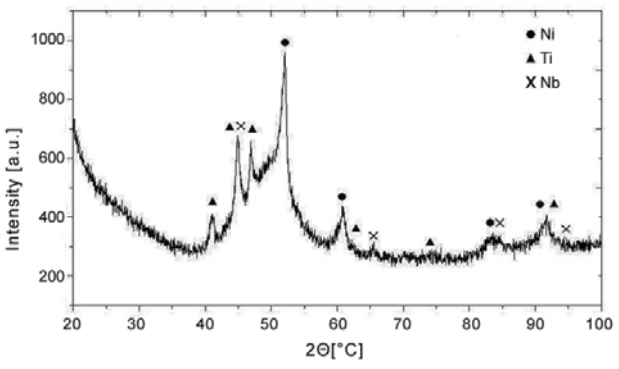

Fig. 5. The X-ray diffraction patterns of $Ni_{52}Ti_{41}Nb_7$ powder alloy vs. the grinding time 40 hours

When the grinding time increases to 25 h, all X-ray peaks become wider and their intensity decreases. The extension of mechanical alloying process until 40 h shows the changes in milled material structure. There is a possibility of observation of titanium diffraction peaks disappear and considerably broadening of all diffraction curves. The widening of peaks in comparison with initial powder is connected with the size reduction in the powder grains and formation of the amorphous phase. Probably, the solid solution was formed, too.

During the mechanical alloying there is not any change of phase constitution in this alloy.

3.2. Microscopic observation

The Ni-Ti and Ni-Ti-Nb alloys were researched in scanning microscope to analyze particle size and shape of the obtained powder material. The pictures show $Ni_{50}Ti_{50}$ and $Ni_{52}Ti_{41}Nb_7$ powder alloy particles after 5 h, 25 h and 40 h of grinding. The samples consist of both big particles of the powder and great number of small ones.

The particle agglomerates are visible in Figures 6-9. The particles of the powder collide with the grinding media, walls of the container and also with themselves. The particles of the powder are cold pressure welded and their morphology changes from irregular, indetermined to more lamellar and spherical one as well as more fine particles are generated which cluster around the larger ones. The large aggregations are crushed whereas smaller ones which can resist deformations without cracking are joined in larger systems.

The Figures 10-15 show the sequent powder structures after 5 h, 25 h and 40 h of grinding. During the process, the powders become effectively broken up while the equilibrium between the cracking and joining is fixed, what results in the relatively stable particle sizes of the powder after the prolonged time of grinding (Fig. 15 - after 40 hours). This picture presents more homogeneous and finely powdered material after 40h of mechanical alloying process.

The scanning microscopy research showed the presence of non-homogenous particles in researched materials.

The pictures show irregular shape of Ni-Ti and Ni-Ti-Nb powder particles consisting of smaller particles after longer time of alloying.

The tests carried out on the scanning electron microscope show that the particles' size of the $Ni_{50}Ti_{50}$ and $Ni_{52}Ti_{41}Nb_7$ powders decrease together with the increased time of grinding.

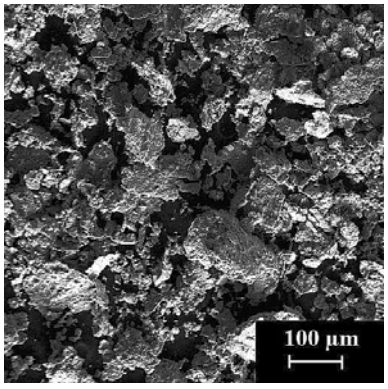

Fig. 6. Structure of $Ni_{50}Ti_{50}$ powder after 5 hours of mechanical alloying

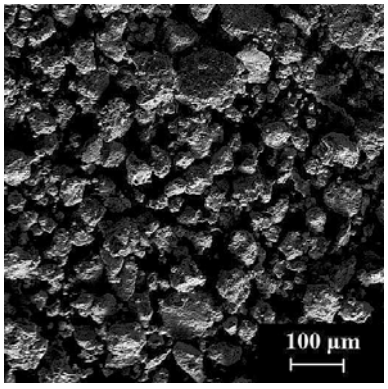

Fig. 7. Structure of $Ni_{50}Ti_{50}$ powder after 25 hours of mechanical alloying

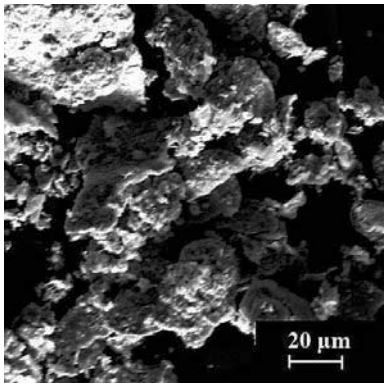

Fig. 8. Structure of $Ni_{50}Ti_{50}$ powder after 5 hours of mechanical alloying

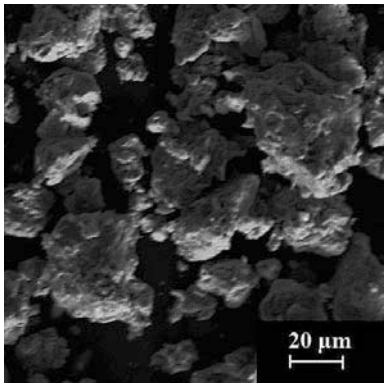

Fig. 9. Structure of $Ni_{50}Ti_{50}$ powder after 25 hours of mechanical alloying

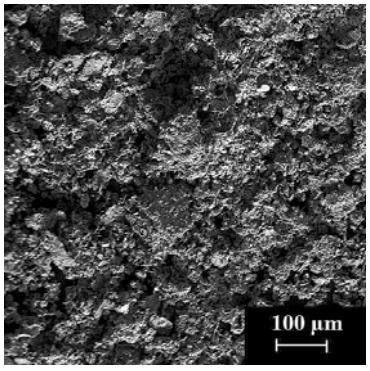

Fig. 10. Structure of $Ni_{52}Ti_{41}Nb_7$ powder after 5 hours of mechanical alloying

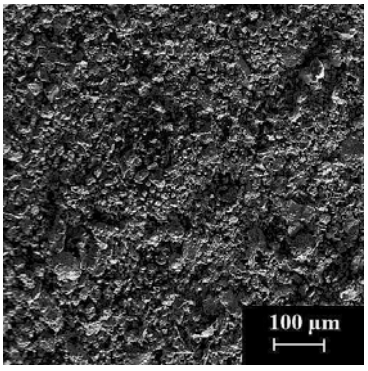

Fig. 11. Structure of $Ni_{52}Ti_{41}Nb_7$ powder after 25 hours of mechanical alloying

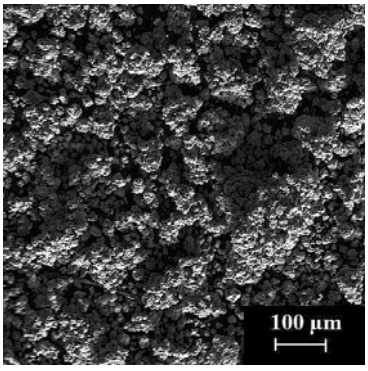

Fig. 12. Structure of $Ni_{52}Ti_{41}Nb_7$ powder after 40 hours of mechanical alloying

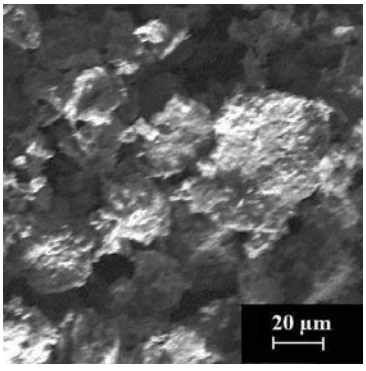

Fig. 13. Structure of $Ni_{52}Ti_{41}Nb_7$ powder after 5 hours of mechanical alloying

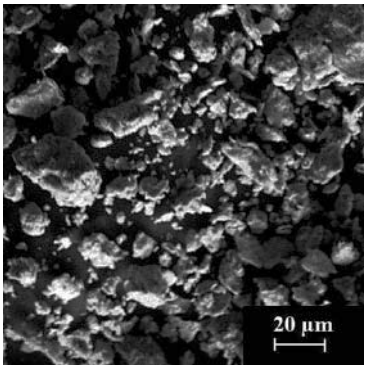

Fig. 14. Structure of $Ni_{52}Ti_{41}Nb_7$ powder after 25 hours of mechanical alloying

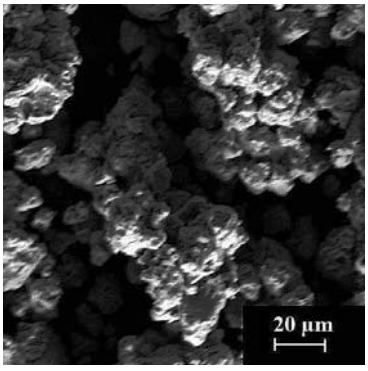

Fig. 15. Structure of $Ni_{52}Ti_{41}Nb_7$ powder after 40 hours of mechanical alloying

3.3. Results of analysis of the crystallization process

The calorimetric measurement enables to detect energetic changes during heating to temperature of 700ºC. The test carried out on the differential scanning calorimeter shows that amorphous structure obtained in MA process is arranged during the heating. During the heating of studied alloys the exothermic peaks were formed. These peaks point to phase transformations and chemical reactions.

Fig. 16 shows DSC curves over the whole temperature range and the enlarged crystalline transition regions of amorphous powders.

The curves recorded for the powder ground for 5 h and 25 h show the exothermic peaks, whereas none of endothermic peak was observed. The DSC curve in Fig. 16 for the sample subjected to short mechanical alloying time show two small exothermic peaks. As research in the previous paper [6,27], the structural changes at the peak 1, are possibly associated with the structural change in the amorphous state during the progress of mechanical alloying. The first peak is due to its crystallization into NiTi. Probably, the structural change taking place at the peak second is associated with the change of the crystallized phase from NiTi into $NiTi_2$ and Ni_3Ti[6,27].

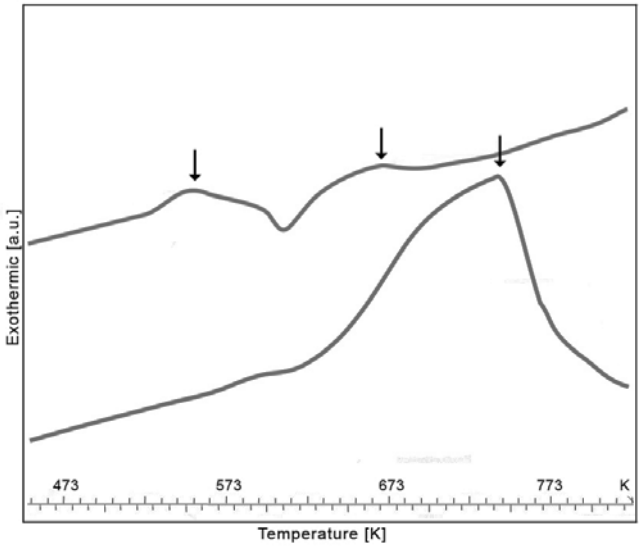

Fig. 16. DSC curve of $Ni_{50}Ti_{50}$ powder after 5 h and 25 h of mechanical alloying process

During the heating of studied $Ni_{52}Ti_{41}Nb_7$ alloys the exothermic peaks were formed. Figure 17 shows DSC curves of the tested alloys.

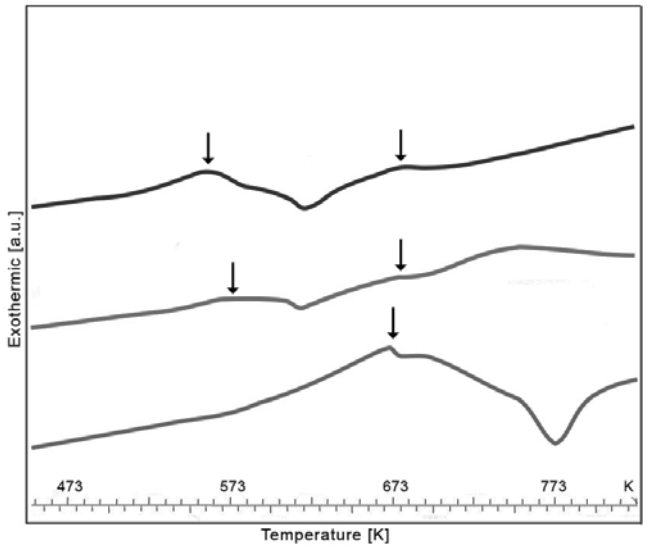

Fig. 17. DSC curve of $Ni_{52}Ti_{41}Nb_7$ powder after 5 h, 25 h and 40 h of mechanical alloying process

The DSC analysis proved enlarged changes occurring in the heating to temperature of 700ºC. The amorphous phase became the most visible in the last sample after 40h of mechanical alloying. The volume fraction of the amorphous phase has been increased progressively.

4. Conclusions

Results of research show, that the application of mechanical alloying technique to obtain $Ni_{50}Ti_{50}$ and $Ni_{52}Ti_{41}Nb_7$ alloys result in obtaining crystalline and amorphous structure.

The tests carried out on the scanning electron microscope show that the particles' size of the $Ni_{50}Ti_{50}$ and $Ni_{52}Ti_{41}Nb_7$ powders decrease together with the increased time of grinding. The pictures show irregular shape of Ni-Ti and Ni-Ti-Nb powder particles consisting of smaller particles after longer time of alloying and particle agglomerates. The extension of mechanical alloying process will cause a little increase of homogeneity and refinement of powder particles. Probably, the agglomerate structure will be broken as the process is continued, and a homogeneous structure will be formed.

Based on the X-ray examinations it may be concluded that all tested alloys have crystalline structure with small amount of amorphous phase. It is expected that solid solution appears. The new NiTi phase after 25h of mechanical alloying was detected.

The exothermic reaction were conducted by differential scanning method. The crystalline structure at 553 K and 753 K (Ni-Ti alloy) as well 553 K, 573 K and 673 K (Ni-Ti-Nb alloy) was achieved.

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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