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# Effect of ball milling on NiTi powder metallurgy alloy

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### ABSTRACT

Mechanical alloying, especially nickel-titanium alloys, has attracted great interest recently as researchers strive to enhance the properties of nanocomposites, expand their usefulness, and how produce them at minimal costs and produce homogeneous fine powders. In this paper, high-energy ball milling was used to produce Ti-Ni ultrafine powders. The effects of grinding parameters such as grinding tools and the initial state of powders, grinding conditions such as operating times, in addition to the effect of equal size balls and the speed of the grinding machine, were investigated while avoiding contamination of the powders in contact with air by applying silica. Milling process balls such as X-ray diffraction, SEM, EDS, plus particle size.

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## 1. Introduction

The complex nickel-titanium (NiTi) alloy has attracted the attention of many scientists due to its unique and distinctive properties such as elastic behavior and transition temperatures, in addition to its return to its original form after being deformed by up to 8%. [1,2,3]. The transformation temperatures (TTs) and shock resistance, in addition to the fact that they do not wear out easily and are inexpensive, have additional qualitative features that make them a mainstay in achieving the desired development. Other beneficial properties of this alloy include biocompatibility and low modulus of elasticity [4,5]. Due to the difficulty of creating these alloys by casting method, because their components are immiscible in the liquid state or difficult to melt, powder metallurgy has become popular in recent years [6,7]. net to create complex NiTi components that allow adaptation of the microstructure and thus important alloy features including valley elastic behavior and transition temperatures [8]. However, the inability to fabricate complex NiTi parts have limited the application potential, which can be linked to challenges in processing NiTi due to the strong alloy interaction, as well as martensite-induced stress, work hardening, and modification. As

a result, composite materials with specific features and properties are critical [9,10].

The highly flexible NiTi effect, combined with free-form design and production, creates a particularly attractive combination for engineering applications. Through reversible stress-induced conversion, NiTi can recover a large amount of strain (up to 8% strain) in superplastic [11]. In general, the highly elastic response of NiTi is highly dependent on the exact structural properties of the alloy. Sintering furnaces were produced in larger capacities with greater productivity, and vacuum sintering furnaces were used in some applications. The aviation sector, which includes aircraft, engines, and compressors, as well as the missile, ship, and vehicle engine industries, are the primary applications [12,13].

However, the high cost of manufacturing is a downside to these compounds. In this research, the mechanism of powder manufacturing in a ball mill will study the powder form after the mixing and blending process that takes place, knowing the percentage of change that occurs.

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## 2. Experimental setup

### 2.1 Material preparation

In this research, nickel-titanium powder was used with Nickel powder with 99.5% purity and titanium powder with 99.9% purity as shown in Table 1.

**Table 1. Powders, purity, and source supplied.**

Materials	Purity%	Average particle size( $\mu\text{m}$ )	Source
Titanium powder	99.75%	12.06	Galaxy International Trade CO., LTD.
Nickel Powder	99.65%	36.80	

### 2.2 Particle size analysis

The Ni and Ti nanoparticles produced were characterized in a particle size analyzer (Better size 2000 instrument, USA). About a gram of each nickel titanium was taken and placed in a glass beaker containing distilled water with a magnet to move the powder in the water. Then the powder with water is added to the particle size test analyzer as in Fig. 1.



**Figure 1. Particle size analysis**

### 2.3 X-ray diffraction (XRD analysis)

The sintered NiTi alloy sample as well as the produced NiTi nanoparticles were examined using an X-ray diffractometer (Model: LabX XRD-6000, Company: Shimadzu, US). The functional X-ray tube contained Cu (1.54060 Å), 40 kV, and 30 A. XRD data were gathered using a step size of 0.2 and an enumeration time of 1.20 seconds over a scan range of 10 to 90.

### 2.4 Energy Dispersive Spectroscopy (EDS)

This test is used to find out the components of the mixed powder and the proportion of materials forming the nickel-titanium alloy by placing the powder in an energy-dispersive spectroscopy test device and determining the average value of the chemical composition. The elemental structure of each of the NiTi nanoparticles was examined using EDS analysis (Company: Bruker, Germany)

**Table 2. shows the results of the EDS test for NiTi alloy after mixing**

Quantitative Results															
Elt	Line	Int	Error	K	Kr	W%	A%	ZAF	Formula	Ox%	Pk/Bg	Class	LConf	HConf	Cat#
Ti	Ka	863.7	1.7194	0.4641	0.4577	50.04	51.04	0.9959		0.00	43.58	A	45.53	46.40	0.00
Ni	Ka	328.8	0.7025	0.5359	0.5285	49.96	48.96	0.9780		0.00	29.19	A	53.22	54.86	0.00
				1.0000	0.9862	100.00	100.00			0.00					0.00

### 2.5 Scanning Electron Microscope (SEM)

SEM is one of the most common surface analysis techniques where a wide range of metrics and features can be observed. In the present work, SEM is used to identify the alloy components and the proportion of nickel and titanium produced by the powder metallurgy process. The test was carried out in the materials lab of Al-Razi Metallurgical Research Centre, Tehran/Iran. Micrographs are taken with a magnification of 5.00KX as shown in Fig. 2.



**Figure 2. Scanning electron microscope device**

### 2.6 Ball Milling

Spherical mills (N Q M - 0 4, made in China) were used. The mixing and homogenizing process takes place in different ways, but in ball mills, mixing and grinding take place at the same time as shown in Fig.3.



**Figure 3. The mixing mechanism in ball mills**

Initially, nickel powder and titanium powder were weighed in equal quantities utilizing a sensitive scale, with an accuracy of 0.001, and the two materials were placed together in the mixing pouches of the ball mill with nine balls (weighing 14 g, and a diameter of 16 mm made of stainless steel). These balls are used for grinding Powder more and more to get a fine powder. The process of mixing nickel and titanium powder was completed

for ten hours, and the speed of the mixing process was 35r.p.m, which is the maximum speed of the planetary ball mill. Fixed pauses were done at room temperature to control the porosity and bring it to the right amount.

### 3. Results and discussions

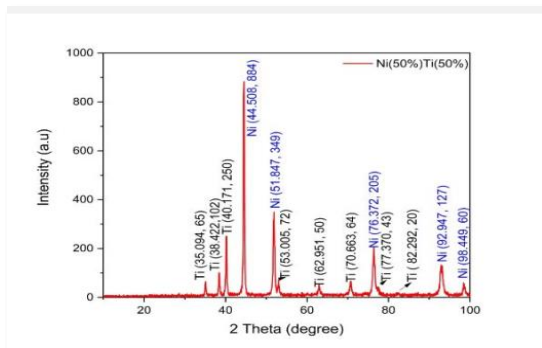
Through the experiments the particle size reduction, newly formed surface area, the changes in XRD diffraction pattern, and broadening of the diffraction lines and microstructural characters were measured and estimated. Some important features of the obtained results are given in the following sections.

#### 3.1 X-ray diffraction (XRD analysis)

The logarithmic graphic shows the NiTi particle size range (20–5000 nm) in terms of diameter. The chemical processes that occur between NiTi and the solution are mostly what determines how Ti and its alloys are treated. Five peaks can be observed at  $5.38 = 2\theta$ , 9.44, 65, 78, and 5.82 degrees. The observed Ni peaks are very close to the reference value of 40.0 nm. Compared to the starting powder style, the mills show an expanded density which means the powder size is effectively reduced after the grinding process.

Figure 4. X-ray test

The granular particles represent the Ni<sub>3</sub>Ti phase, whereas the matrix represents the NiTi phase. The XRD patterns of NiTi particles are displayed. The Ni<sub>3</sub>Ti phase is indicated by the greatest peak in the XRD pattern, and there are also high peaks for the phases between NiTi and TiO<sub>2</sub>. Ti is more reactive than Ni, so when it comes in touch with oxygen,



TiO<sub>2</sub> begins to develop on the powder right away. Since the free enthalpy of production of TiO<sub>2</sub> is negative and greater than that of nickel oxides, it consists primarily of TiO<sub>2</sub>. TiO<sub>2</sub> creates a physical and mechanical barrier to nickel oxidation and shields the alloy from corrosion. On the other hand, the NiTi nanoparticles' XRD pattern, Fig. 5.

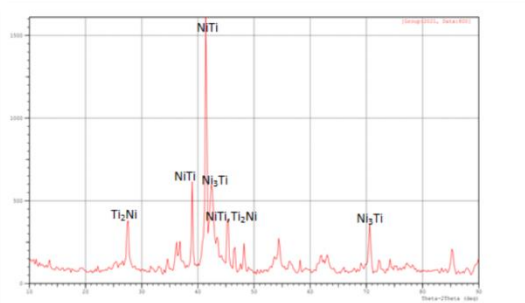
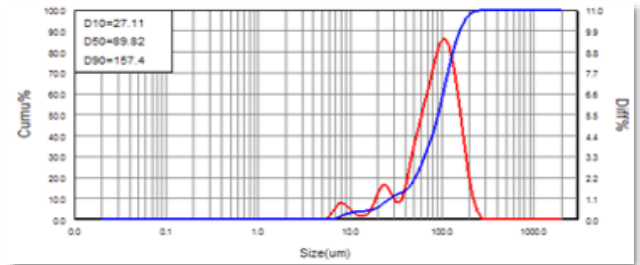
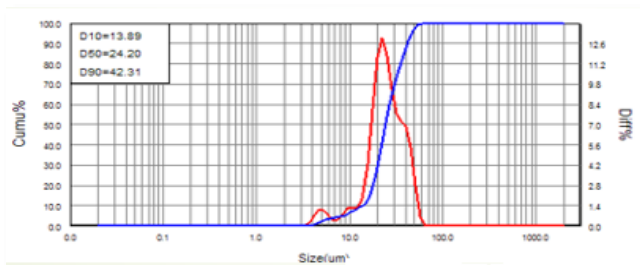


Figure 5. XRD pattern of NiTi particles

The main metallic phases are NiTi, Ni<sub>3</sub>Ti, and Ti<sub>2</sub>Ni, which are the stable phases of NiTi alloy. The particle size results are illustrated in Fig.3.



Particle size analysis (Ti)



Particle size analysis (Ni)

Figure 6. Particle size analysis diagram of Ni and Ti nanoparticles

#### 3.2 Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDS)

The outcome of NiTi nanoparticles is depicted in Fig. 6, based on SEM and optical micrographs and the objective of EDS investigation. Since Ti is more acid resistant than Ni, the observed variation in the elemental composition ratio of NiTi particles can be attributed to this differential in the acid resistance of each element. Additionally, compared to the Ti particles, the Ni particles used were smaller, Table 1.

Table 3 shows the chemical composition of the blended powder alloy. In this study, it was prepared in equal weight ratios to contain 50 wt% Ni + 50 wt% Ti, which corresponds to 49.9 wt% Ni and 50.04 wt% Ti. According to EDS results, Ni and Ti are the only elements present. These findings may be explained by the EDS analysis's characteristic of just analyzing the area where the electron struck. The findings of the EDS study were, as can be seen, rather near the required percentage.

#### 3.3 The ball milling

A little amount of powder is trapped between two colliding grinding balls. It typically held during this process, each impact produced around 1,000 particles with a combined weight of about 2.0 mg, allowing for adjustment of the powder's morphology. When dealing with tiny powders like nickel and titanium, overlapping Cold welds are created by the flat layers. As a result, layered composite powder particles are created. It is made up of many starting set compositions. It is possible for components that work to harden or composite powder particles to fail at the same time. These opposing actions—cold welding (plastic deformation and agglomeration) and crushing (size reduction)—occurred repeatedly during the milling operation. Finally, a finely tuned and homogenous microstructure is

achievable, and the powder particle composition is identical to that of the initial powder ratio.

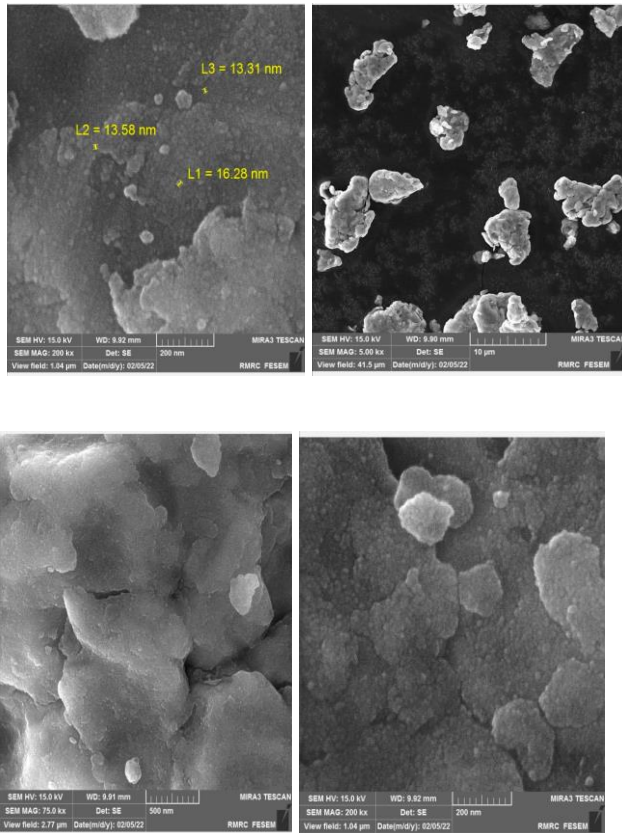


Figure 7. SEM micrograph of nickel-titanium powder milled for 10 h

#### 4. Conclusion

The results presented in this paper show that the techniques developed and used in this study are well suited to optimizing the grinding parameters of a nickel-titanium alloy to investigate the effect of grinding parameters on the structure and properties of ground materials as well as on the grinding energy values, revolutions per minute with a pause time and the powder remains in the grinding bowl of the ball mill for 24 hours after every 4 hours of operation time. The change in the powder composition was observed after the series of grinding and suspension, which generates high heat that leads to the cohesion of the powder, and then cracking this cohesion when the powder is confined between two balls, thus changing the shape, size and chemical composition of the powder, and this is what was observed in the test that we carried out in this research.

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