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Ball milling for the formation of nanocrystalline intermetallic compounds from Ni-Ti elemental powders

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Abstract:

In the present research work nickel (Ni) and titanium (Ti) elemental powder with an ostensible composition of 50% of each by weight were mechanically alloyed in a planetary high energy ball mill in diverse milling circumstances (10, 20, 30 and 60 h). The inspection exposed that increasing milling time leads to a reduction in crystallite size, and after 60 h of milling, the Ti dissolved in the Ni lattice and the NiTi (B2) phase was obtained. The lattice strain of ball milled mixtures augmented from 0.15 to 0.45 at 60 h milling. With increase in milling time the morphology of pre-alloyed powder changed from lamella to globular. Annealing of as-milled powders at 1100 K for 800 s led to the formation of NiTi (B19′), grain growth and the release of internal strain. The result indicated that this technique is a powerful and highly productive process for preparing NiTi intermetallic compounds with a nano-crystalline structure and appropriate morphology.

Keywords: electron microscopy, intermetallic compound, phase transformation, X-ray techniques

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1 Introduction

Nikel-titanium (NiTi)-shape memory compounds are being used for a diversity of engineering and medical purposes owing to their unique shape memory effect and advanced superelasticity with excellent corrosion resistance, good biocompatibility and wear resistance. The shape memory behavior of this material is due to thermoelastic martensite transformation from high temperature B2 (austenite) to the low temperature B19′ (martensite) phase [1], [2], [3], [4], [5], [6], [7], [8], [9]. Shape memory materials are made by well-known methods related to melting and casting. These approaches are high energy consuming and imply difficulties in control of the processed alloy composition. In order to obtain inexpensive products, researchers have been focused on nearly synthetic processes in an effort to establish the optimum production route and to obtain material with improved properties.

Mechanical alloying (MA) is a versatile and widely used technique for the preparation of intermetallic compounds because of its advantages including high yield, low cost and ease of control of particle size and shape. Moreover in this process, the reduction of particle size to the nano-scale results in nanocrystalline materials with homogeneous structures. In this method high energy collisions of the balls and the vial lead to repeated cold welding and fracturing of powders and finally the preparation of alloyed powders [10], [11], [12], [13].

The present work reports the synthesis and investigation of the nanocrystalline intermetallic compound in the NiTi system by MA and further heat treatment. Meanwhile the effects of milling time and the annealing process on grain size, the morphological changes and the phase compositions were studied.

2 Material and methods

The elemental powders of Ni (99.5% purity) and Ti (99.9% purity) with a mean particle size of 20 μm and nominal composition Ni-50 at.% Ti were mechanically alloyed in a planetary high energy ball mill (Ball Mill-Fritch B6; Germany) using hardened stainless-steel balls (100 balls with diameter of 10 mm) and vials (300 ml) in different milling times. A ball to powder weight ratio of 12:1 was maintained and the milling speed was adjusted to 250 rpm. The milling was carried out under a protective argon atmosphere to avoid oxidation and atmospheric contamination of the powders.

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After certain milling conditions (10, 20, 30 and 60 h), a small amount of milled powder was collected for structural evaluation through X-ray diffraction (XRD) analysis with an Xpert Philips diffractometer (Bruker D8 Discover, X-ray source-Cu, 3 KW, Germany) using an Fe filler and CuKα radiation (λ = 0.15406 nm) at a voltage and electrical current of 40 kV and 30 mA, respectively. The evaluation of the morphology and a study of the particle size were performed via scanning electron microscopy (SEM) at a voltage of 15 kV (FESEM with an EDX model, Surpa 40 VP; Bruker EDS system, Germany). After 60 h milling, the as-milled powders were sealed in quartz tubes under high vacuum and annealed at 1100 K for 800 s followed by water quenching in order to study the phase transition occurring during annealing.

3 Results and discussion

3.1 XRD analysis

XRD diffraction profiles of milled powders at various milling times are shown in Figure 1. According to this figure, the peaks related to raw materials can be detected in the starting materials. With milling time, there is broadening of diffraction peaks and a significant decrease in the line intensity of all diffraction peaks. In addition, the position of diffraction lines corresponding to Ni moved to lower angles. The weakening of Ni and Ti peaks and the shift of Ni peaks toward lower angles indicated that Ti atoms diffused into the Ni matrix and the lattice parameter of Ni increased. The peaks related to Ti disappeared at 30 h of milling. On the other hand, there was no evidence for the existence of Ti in the mixture compound. In fact, Ti dissolved completely in the Ni lattice. After 60 h of milling, the Ni peaks vanished completely and only broadened NiTi (B2) peaks with a body centered cubic structure were identified. The broadening of NiTi peaks was considered to be due to the refinement effect of the crystallite size. The crystalline sizes of compound were calculated using XRD peak broadening and the Williamson-Hall formula as follows [10]:

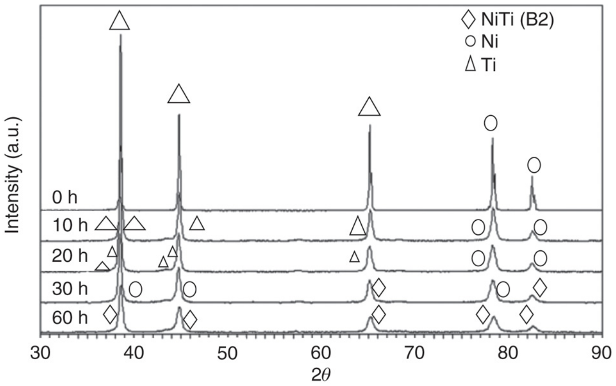

Figure 1: XRD pattern of mixtures powders at several milling times.

$$\sqrt{Bi^2 - Bo^2}\cos \quad = 0.89 \ /d + 2e\sin$$

where Bi is the peak full-width at the half of the maximum intensity of XRD patterns [full width at half minimum (FWHM)], Bo is the correction factor for instrument broadening, θ is the Bragg angle, λ is the wavelength of the X-ray used, d is the crystallite size and e is the lattice strain.

Figure 2 shows the variations of crystallite sizes versus milling time. Increasing the milling time led to the reduction in crystallite size that is mainly due to severe deformation of powders during ball milling. The other reason might be the increased probability of nucleation sites during crystallization, provided by higher defect densities [12]. At the onset of the milling process, the crystallite size decreased dramatically, and the lattice strain increased. However, for longer milling times, grain size almost did not change and became constant due to the balance between the rate of cold welding and the fracturing of powder particles [11]. After 60 h of milling, the crystallite size of milled powders reduced down to 40 nm.

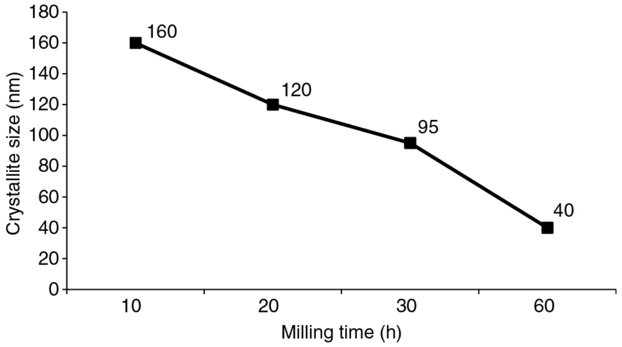

Figure 2: Crystallite size of mixture powders as a function of milling time.

3.2 SEM observation

Figure 3 shows the morphology of the milled powders after several milling times. At the beginning of milling, a lamellar structure was obtained because of continuous cold welding between the laminates during which ball milling of the ductile particle has occurred, but the spaces between these lamellae are high. Increasing the milling time to 20 h, the laminated structure becomes discontinuous and the thickness of the layers decreases. On further milling, the size of particles decreases because the increase in brittleness leads to the formation of smaller particles with a granular shape. Meanwhile the distribution of particle morphology and particle size becomes narrower. On the other hand, the repeated processes of cold welding and fracturing result in the generation of agglomeration consisting of nanosized Ni and Ti particles.

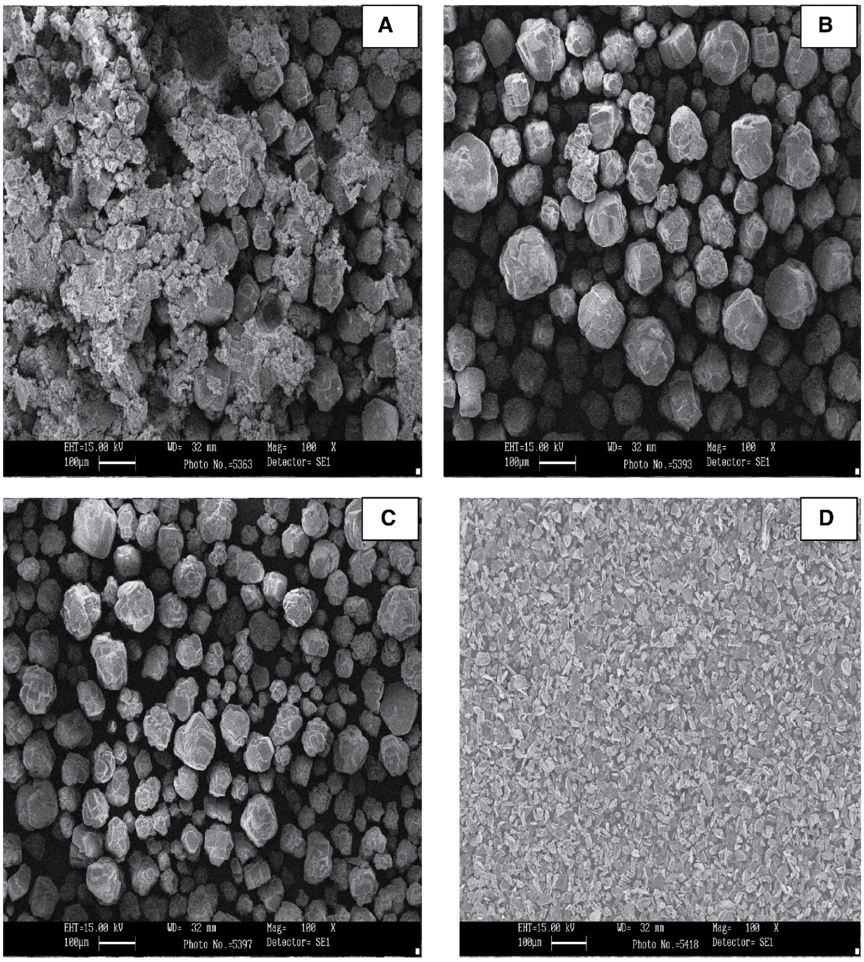

Figure 3: SEM micrographs of milled powders after different milling times (A) 10 h; (B) 20 h; (C) 30 h and (D) 60 h.

3.3 Lattice strain

Figure 4 shows the variation in the lattice strain of the ball milled powder with milling time. The relative lattice strain is increasing with increasing milling time. The lattice strain was increased from 0.15 to 0.45 for as-received ball milled powder after 60 h of ball milling. During ball milling intense mechanical deformation experienced by the ball milled mixtures leads to the generation of lattice strain and a balance between cold welding and fracturing among the powder particles is expected to effect the structural change in the powder.

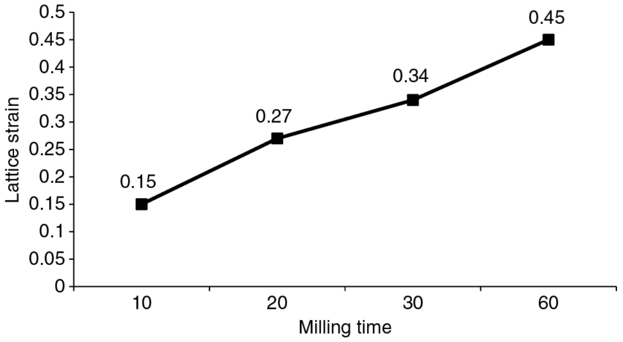

Figure 4: Lattice strain of mixture powders as a function of milling time.

3.4 Annealing

Figure 5 displays the XRD spectra of milled and annealed powders at 1100 K for 800 s, as can be seen the annealing process induced several diffraction peaks and led to the formation of NiTi (B19′ with a body monoclinic structure) and Ti_2Ni phases. As the annealing temperature was higher than 0.534 Tm (melting temperature of NiTi), recrystallization occurred and caused grain growth due to release of internal strains. The crystallite size increases from an initial value to 40 nm.

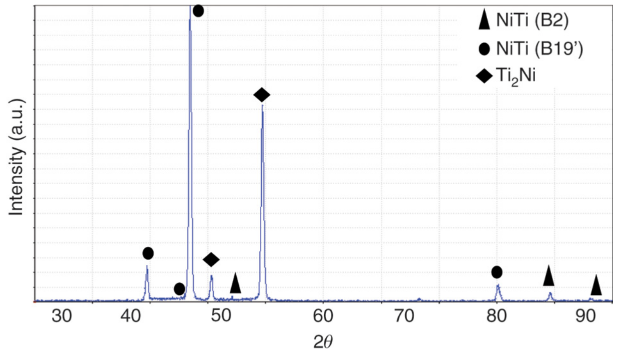

Figure 5: XRD patterns of powders after 60 h milling + 800 s annealing.

4 Conclusions

1. MA of pure elemental Ni and Ti powder mixtures in a planetary high-energy ball mill led to a solution of Ti into a B2 structure and the preparation of the NiTi intermetallic phase with a nanocrystalline structure and a homogeneous configuration.
2. The annealing of as-milled powders resulted in the production of the nanostructured NiTi (B19′) phase.
3. The XRD examinations and SEM observations revealed that the MA followed by annealing is a useful, cost effective and highly productive method for preparing NiTi intermetallic compounds with appropriate structure and suitable morphologies.

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