Nanostructural study of Ti-Ni binary alloy prepared by mechanical alloying

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Abstract

In the present study, a powder mixture of elements Ti and Ni was mechanically alloyed in a high energy ball mill. Microstructure of the nanosized amorphous milled product in different stages of milling has been characterized by X- ray diffraction, scanning electron microscopy and differential thermal analysis. We found that time of mechanical alloying is more significant to convert all crystalline structure to the amorphous phase. Nanocrystalline phase was achieved as a result of the mechanical alloying process. The results also indicates that the phase transformation and the grain size occurs in these alloys are controlled by ball milling time.

Key words
Mechanical alloying , amorphousnization Ni-Ti system, Nanocomposite , Biomaterials.

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دراسة البناء النانوي لسبيكة النظام الثنائي Ti – Ni المحضرة بطريقة التسبيك الميكانيكي

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الخلاصة

في الدراسة الحالية، خلطت عناصر من التيتانيوم مع النيكل بأستخدام طريقة التسبيك الميكانيكي من خلال الطحن بكرات ذات طاقة عالية. تم تشخيص البناء النانوي بمديات زمنية مختلفة من الطحن بواسطة تقنيات حيود الاشعة السينية، المجهر الالكتروني الماسح والتحاليل الحرارية التفاضلية. لقد لوحظ بان زمن الطحن يلعب دورا رئيسياً لتحويل البناء البلوري المنتظم الى الحالة العشوائية. كما اكدت النتائج التجريبية بان التحولات الطورية مع الحجم الحبيبي يمكن السيطرة عليها من خلال زمن الطحن بواسطة الكرات .

Introduction

Pure Titanium and some of its alloys are widely used as promising industry materials due to their excellent corrosion resistance, high degree of strength to weight ratio [1, 2]. Titanium exhibits on allotropic changing from a body- centered cubic crystal structure at high temperatures (β phase) to a hexagonal close packed crystal structure (α phase) at lower temperatures [1, 3].

Nickel has properties which make it useful in such applications such as low thermal expansion, shape memory and electrical resistance alloys [4]. It is also known for its good corrosion resistance and excellent strengthening properties when compared with other elements [5]. The growing development of nanoscience and nano technology in recent years provides opportunities for the design of new composite materials and to acheive superior physical properties [6,7]. The technology of producing nanomaterials with small particle

size such as Ti- Ni-Cu, Ti-Zr, Ti-Sn and Ti-ceramics systems are of great importance as brazing filler alloys or biomaterials [6,8]. Mechanical alloying (MA) technique is preferred as a practical method of producing small particle size materials successfully compared to the conventional powder metallurgy, or casting techniques[8]. MA is a high energy ball milling process by which constituent powders are repeatedly deformed, fractural and welded by grinding media to form a homogeneous alloyed microstructure. After repeated collisions the powder particles become brittle, interdiffuse and alloy under frequent mechanical impacts [9, 10]. The most important process variable of MA is the milling time which appears to have a great effect on the final constitution of powders as on the micro and nano scales.
In the present study, the binary system with composition of (Ti_{50} – Ni_{50}wt %) alloys were prepared from elemental powders by using new techniques of mechanical alloying. Some structural and thermal properties are studied to characterize the amorphous and crystalline phases.

Experimental Methods

Powders of elements Ti and Ni each of purity of ≥ 99.7% and particle size less than ≤ 45 (- 325 mesh)were mixed and blended to obtain the binary alloy of (Ti_{50} – Ni_{50}) composition. The powder blends were subjected to high energy ball milling in the stainless steel grinding media at mill equipment. Total 300 grams of powder was used and steel balls of 6 mm diameter were taken. The powder to ball weight ratio was maintained at 10:1. Milling was done for different times and then powders were taken out from mill for analysis. The powder was characterized by X- ray diffraction (XRD) to investigate amorphous and crystalline peaks of milled samples. The refined values of lattice parameter (a) were calculated from the peak positions in the XRD pattern by Nilson – Riley extrapolation of (a) against ($\cos^2\theta/\sin\theta$) [9].
The average crystallite size parameter was determined and estimated directly from the broadening of reflection by using Scherrer formula [10]. The thermal stability of the mechanically alloyed powder products were determined by using Differential Thermal Analysis is (DTA) type (STA 409 NETZSCH, Germany). The temperature range was from room temperature to1100 °C and the heating rate was 10°C M^{-1}.

Results and Discussion

In Figs.1 and 2 the XRD patterns of the investigated powder mixture in the intial state and after various milling times are shown. These figures show the modulation of XRD patterns of Ti_{50} Ni_{50} composition with the progress of MA up to 25 hours of milling. It is interesting to note that Ti and Ni peaks were clearly visible during early stage of milling at (0-5) hours and no intermetallic compound was found during this range of milling. It is to be noted that the peak intensity gradually decreased and broaden with progress of milling.

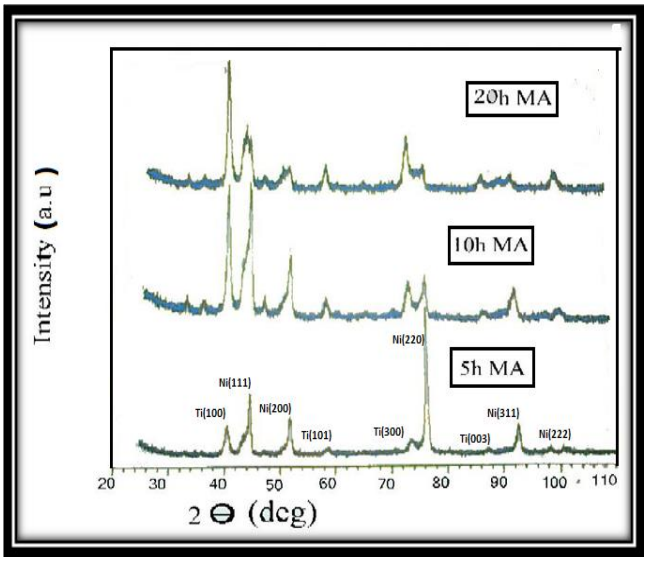

Fig.1: XRD patterns of Ti-Ni powder mixture at different milling times

The peaks are corresponding to Ti and Ni. Intermetallic phase TiNi3 was found and the structure appeared to be amorphous as evidenced by the presence of a broad amorphous bump in the XRD pattern Fig.2. Analysis of the XRD in the pattern during milling time revealed that the crystallite size could be range of 15-35 nm as shown in Fig.3. It shows that crystallite size decreased very rapidly during milling in the intermediate stage, but finally after (20 – 25) hr, it tends to attain almost constant value.

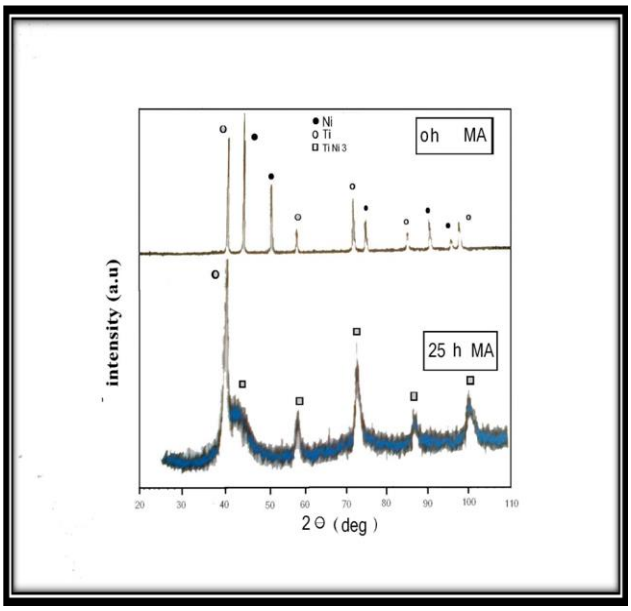

Fig.2: XRD patterns of Ti-Ni un- milling (0 h) and milled (25 h)

This is in reasonable agreement with the XRD patterns in Fig.1 and 2 respectively, which revealed that phase transformation occurred in the Ti_{50} – Ni_{50} alloy during milling time stages. This change appeared after 10 hr of milling, from crystalline phase to amorphous phase.

As mentioned above, the intermetalic phase TiN_3 co – existed in the amorphous phase with the crystalline phase as revealed that by abroad hump at 25 hr.

Similar kind of behavior has been reported by Krasowski et a1. [12] for Ti – Al system and Nagarajzn et al. [13] in their studies on $Al_{20}Ni_{25}Ti_{50}$ alloy.

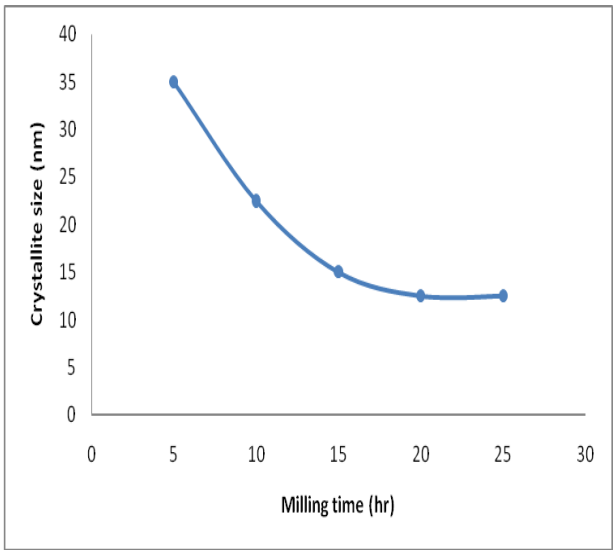

Fig.3: Variation of crystallite size of $Ti_{50}Ni_{50}$ with milling time

The SEM micrograph for 25 hours revealed an alloyed lamellar microstructure of Ti and Ni layers as seen in Fig.4. It shows a diffused uniform lamellar structure Ni (light) and Ti (ark). This Indicates the presence of amorphous phase generated in the Ti_{50}– Ni_{50} alloy.

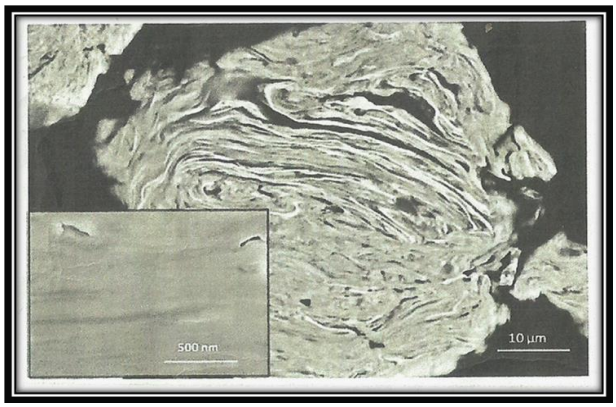

Fig.4: SEM micrograph of 25 hours milling $Ti_{50}Ni_{50}$ powder (uniform amellar structure of Ti (dark) and Ni (light) regions is observed at the micron scale and at the nano scale)

By DAT investigations this it can provide more information regarding the different stages after 15 hr and 25 hr of mechanical alloying as shown in Fig.5, which depicts the DTA curves. In both cases two overlapping broad peaks are visible. The first one is associated with endothermic effect and the second is an exothermic effect that occurs at higher temperature. However, there is a difference between both DTA scans for 15 hr and 25 hr respectively. The endothermic peak which recorded for 15 hr milled sample is higher temperature than for the sample after 25 hr of the MA process.

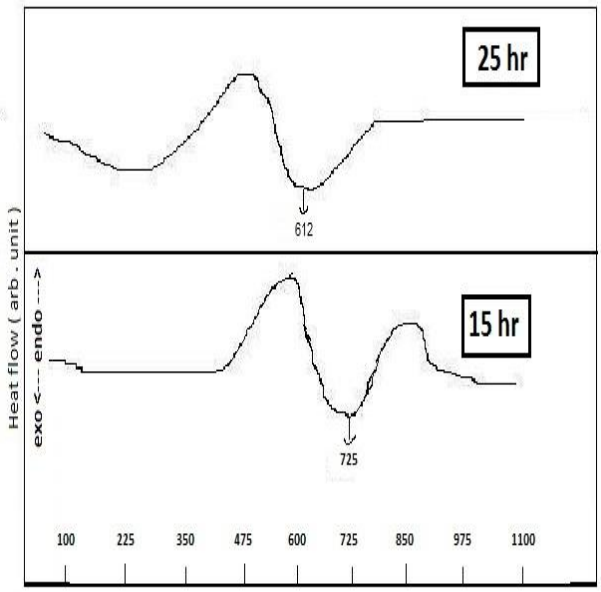

Fig.5: Thermal curves of the amorphous powders after 15 and 25 hours of mechanical alloying

The exothermic effect is certainly related to amorphous structure. In the case of 15 milled samples, the temperature of exothermic peak is equal to ~ 725 °C, while for the 25 hr is 612°C. The exothermic effect is a result of the formation of the solid phase, and we suggest in our case , this is due to thermal formation of intermettalics within the product .

Conclusions

This work is attempt to examine, how structures controlled by mechanical ball milling in a binary $Ti_{50} - Ni_{50}$ alloy from 5 to 25 hrs. Characterization of samples was carried out by XRD, SEM and DTA. The following conclusions of this work are summarized as follow:

1-This technique of MA were successfully to obtain nanocrystalline and amorphous structures as evidenced by the results. The particle size after (20 – 25) hr of milling was contained within the range of (15– 35) nm.

2-The SEM micrograph results also supported the presence of the amorphous plus crystalline phases formed at some intermediate stages of milling.

3-Exothermic and endothermic reactions with some dissimilarity between thermal curves after 15 and 25 hrs of milling were observed by differential thermal analysis (DTA).

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