Magnetic methods for phase control in titanium-base alloy synthesized by ball milling

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Research Article

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Abstract

The nanocrystalline TiAlV alloys have been synthesized from pure titanium, aluminum and vanadium powders by mechanical alloying technique within a high energy planetary ball-mill. Magnetic behavior, Morphology and micro-structural properties were examined by a Vibrating sample magnetometer (VSM), Scanning Electron Microscope (SEM), and X-ray diffraction (XRD), respectively. Crystallite size reduced from 48.73nm to 9.38 nm and lattice strain increased from 0.15% to about 0.81% for 60 hours of grinding. X-ray diffraction testing confirms the apparition of new phases during grinding time. The Magnetic non-destructive testing showed that the nanocrystalline TiAlV contain a magnetic particle which is different from one period to another period, the change of magnetic properties is due by the reduction in crystallite size of magnetic particle which is due to the collision between the balls and the magnetic particles of the powder. The NDT by magnetic measurement confirms that we can control the state of the nanocrystalline alloy using the vibrating magnetometer samples.

1. Introduction

Nanocrystalline alloy has been synthesized with diverse methods; powder metallurgy is one of these procedures which have many uses in various sectors. The ability to master the control of nanocrystalline materials is a key factor that opens up significant perspectives in engineering. The control of nanocrystalline alloy by non-destructive techniques occupies a prominent place in different industrial applications, because of the increased knowledge of the benefits that can be obtained from its techniques for evaluating the performance of various equipment.A lot of research has been done on non-destructive testing of different physical properties in order to replace destructive methods. Titanium alloys have been used extensively in the aerospace manufacturing for engines and chassis due to their excellent compromise of properties [1–2], excellent mechanical properties, corrosion resistant, high strength to weight ratio. Despite all the mentioned advantages, titanium alloys are non-magnetic materials and poor tribological behaviour. The most frequently applied Ti alloy which has aluminum and vanadium as the main alloy components. TiAlV is defined by α and β phases, which is thermodynamically stable at low temperature [3–4],In addition, little data have been notified on the production of nanocrystalline titanium alloy via mechanical milling [5].Mechanical milling is a technique which is able to manufacture nanometric structures for different type of materials [6], In this study, the nanocrystalline TiAlV alloy was synthesized by a mechanical milling process, which is based on the phenomena of welding and fracture due by mechanical impact between the powder particles, grinding balls and the inner jar wall. The aim of this research is to evaluate the obtained samples by the magnetic non-destructive testing techniquein order to find different impurities generate during grinding and their effect on the different properties obtained during a production cycle. The approach used in this work is to use the magnetic measurement by vibrating samples magnetometer to control the progression of the nanocrystalline alloy and the different defects generate during mechanical grinding.

2. Materials And Experimental Procedure

The starting materials were the Ti (99.9%), Al (99,7%) and V (99,9%) powders according to suppliers. The elemental Ti, Al and V powders are grinded in a high energy planetary ball millPM400 at room temperature in a controlled atmosphere with a speed of 300rpm using Hardened Cr steel balls with 20 mm of diameter. The Ball to the powder weight ratio used in this experiment was 12:1. The grinding time was varied from 0h to 60 h. The ball grinding process is used to make TiAlV alloys and to reduce the grain sizes via severe plastic deformation. The magnetic non-destructive testing method was realized by vibrating samples magnometers (EV9).Morphology of the powders was analyzed by scanning electron microscopy using Gemini SEM 300 attached to the EDS unit (Energy Dispersive X-ray Analyses). Structural properties were examined by X-ray diffraction using a PAN analytical X-Pert X-Ray diffract meter with Co Kα radiation (=1,7889 Å), with a scanning step size of 2θ = 0.02 from10° to90°.

3. Results And Discussion

3.1 Evaluation by magnetic measurements

Figure 1 illustrates the vibrating sample magnetometer(VSM) a scientific instrument, which works on the basis of Faraday's Law of Induction, which tells us that a varying magnetic field generate an electric field. This electric field can be monitored and gives us information about the magnetic field changing. The total magnetic moment induction is generally measured as a function of an external magnetic field, which is referred to as magnetic hysteresis analysis [7–8], which gives information about magnetic characteristics, all of which play a vital role in the investigation of magnetic materials [9–11].

The variation of magnetization with the magnetic field at room temperature of the nanocrystalline TiAlV alloy in function of effect of collision of the balls is shown in Fig. 2. The elementary powder before milling is non-magnetic material. However, the samples after ball milling exhibit much larger value of Hc and also the saturation magnetization decrease during mechanical grinding.

Table 1 shows the variation of different magnetic parameters during mechanical alloying, The coercivity vary during milling, this a variation is due to reduction of crystallite size of iron worn off from the milling balls after milling due of ball collision. Saturation magnetization (Ms) is a characteristic of magnetic materials, M_S depends on the variation of chemical composition of the local environment of atoms and their electronic structures, the apparition of iron worn off from the milling balls with Ti(Al) and Ti(Al,V) leads to a non-saturating magnetic behavior.

Table 1
Different magnetic parameters as function of milling time

Millig time (h)	Mr (emu/g)	HcOe (Oe)	Hs (Oe)	Ms (emu/g)	Area (Oe * emu/g)	S
10	0,02518	130,42	2728,5	0,065	40,9404	0,16
20	0,01834	166,49	3061,39	0,184	67,2069	0,16
40	0,00629	49,39	4075,65	0,121	22,0921	0,02
60	0,00696	136,72	3972,09	0,0325	36,5094	0,1

3.2. Morphology

Figure 3 shows the morphology of nanocrystalline TiAlV at the different grinding time of 0, 20, and 40 hours, which is indicates an important variation in size, shapes and nature of powder particle with changing of grinding time. This change is due to the grinding process, which leads to repeated welding and fracturing phenomenon during mechanical alloying.

Before grinding, Ti, Al and V particles appears in irregular shapes with different particle sizes. After 20h of grinding, the powder particles are spherical and irregularly shaped with an average size in the range (5–10) µm, while continuously grinding reduces the particle size at longer grinding times, this decrease is attributed to the fracturing phenomenon. After 40 h of grinding, the powder particles became more homogeneous while maintaining the granular morphology, and the two processes of fracturing and cold welding are in equilibrium and the powder particles appeared more homogeneous compared to the earlier grinding.

Figure 4a depicts the EDS micrograph of the nanocrystalline TiAlV alloy before grinding, the Ti, Al and V element composition was not homogeneously dispersed and we have no contamination caused by collisions of the balls and the vial during fabrication.

Figure 4a. EDS spectrum and EDS elemental distribution maps of the nanocrystalline TiAlV alloy before grinding

Figure 4b demonstrates the EDS micrograph of nanocrystalline TiAlV alloy during mechanical grinding at 40h, the repartition of Ti, Al and V elements is uniform and there is some contamination of iron worn off from the grinding balls after milling due to ball collision, the appearance of TiAlV alloy powder was previously formed in 40 hours

Figure 4c represents the EDS micrograph of the nanocrystalline TiAlV alloy through mechanical grinding at 80h, we observe that we have some particles of the iron residue dropped as a result of the collision between the grinding balls and that these elements are homogeneously dispersed.

An EDS study was performed on the powder particles during the grinding times in order to verify the formation of the nanocrystalline TiAlV alloy. It is found in the EDS results that there was no considerable change in the chemical composition and mass ratio of the powder and the TiAlV alloy is formed with some particle of iron after grinding.

3.3. Structural analysis

X-ray diffraction (XRD) patterns were performed in an XPERT PRO X-ray diffractometer utilizing CoKa radiation. XRD diagrams were taken in 2h with a step size of 0.10°. The size of crystallites (D) was calculated from the broadening (ß) by use of the Scherrer formula as described below [12]:

$$D = \frac{0.9\lambda}{\beta cos\theta}(3.1)$$

The lattice strain (ε) was determined for the similar diffraction lines from the formula below [13]:

$$= \frac{\beta}{4tan\theta}(3.2)$$

While λ is Co intensity (1.7889 Å),θ is the angle in radians, ß is the full width at half maximum and ε is the lattice strain

The X-ray diffraction (XRD) pattern of the grinded nanocrystalline TiAlV alloy is depicted in Fig. 5. XRD diagrams before mechanical grinding of TiAlV powder. It can be seen the presence of all Ti, Al and V peaks and the absence of any new phase. Following 10 h of grinding, certain Ti and Al peaks have been disappeared, some other Ti and Al peaks are enlarged, and all the peaks of V have been disappeared. This broadening of the principal Ti peaks extends to 20 h. This could be due to a progressive diffusion of Al and V atoms inside the Ti lattice, which suggests a continuing reduction in crystallite size resulting from the formation of new phases. After 40 h of grinding time the XRD pattern shows the formation of another phase as a result of crystallization of the amorphous phase. The increase in grinding time to 60 h resulted in a reduction in intensity and a broadening of the XDR peaks, which is signified the decrease in the crystallite size, which is a result of the chemical segregation produced by the high energy grinding. The repeated cold welding and fracturing of the particles and the increasing density of crystal defects and dislocations result in the dispersion of Al and V, which also resulted in the broadening of the XRD peaks [14].
Table 2 illustrates the impact of grinding time on the crystallite size and lattice strain of the nanocrystalline TiAlV alloy. Through 20 h, the crystallite size reduces from 48.73 to 13.93 nm, at this period; fracture process is dominant, which is due to severe plastic deformation of the powder particles

that occurs during mechanical grinding. After 20h, a gradual reduction in crystallite size from 13.93 to 9.38 nm, which is caused by the equilibrium between the fracturing and the welding process. The lattice strain increases from 0.15–0.81%. This may be due with the introduction of dislocations, impurities, and other lattice defects during grinding [15].

Table 2
Crystallite size and lattice strain of TiAlV at different milling time.

Milling Time (h)	crystallite size (nm)	Latice strain (%)
0	48.73	0,15
10	25,48	0,3
20	13,93	0,45
40	10,96	0,71
60	9,38	0,81

4. Conclusion

Nanocrystalline TiAlV were successively synthesized by high energy ball grinding for different durations, the formation of the nanocrystalline TiAlV alloy involved several stages.The elementary powders before milling is a non-magnetic material. The coercivity vary during milling, this variation is due to reduction of crystallite size of iron worn off from the milling balls after milling due to ball collision. EDS analysis of nanocrystalline TiAlV alloy after mechanical grinding show the presence of some contamination of iron worn off from the grinding balls after milling due to ball collision, which confirm the magnetic behavior of TiAlV nanocrystalline find by magnetic measurement results. The results of X-Ray diffraction show the apparition of nanocrystalline alloy with the reduction of crystallite size from 48.73 nm to about 9.38 nm and augmentation of lattice strain from 0.15% to about 0.81% for 60 hours of grinding.

Declarations

Ethical statement

The manuscript has not been submitted to more than one publication for simultaneous consideration. The submitted work is original and has not been published elsewhere in any form or language.

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Figures

Figure 1

Vibrating sample magnetometer instrument

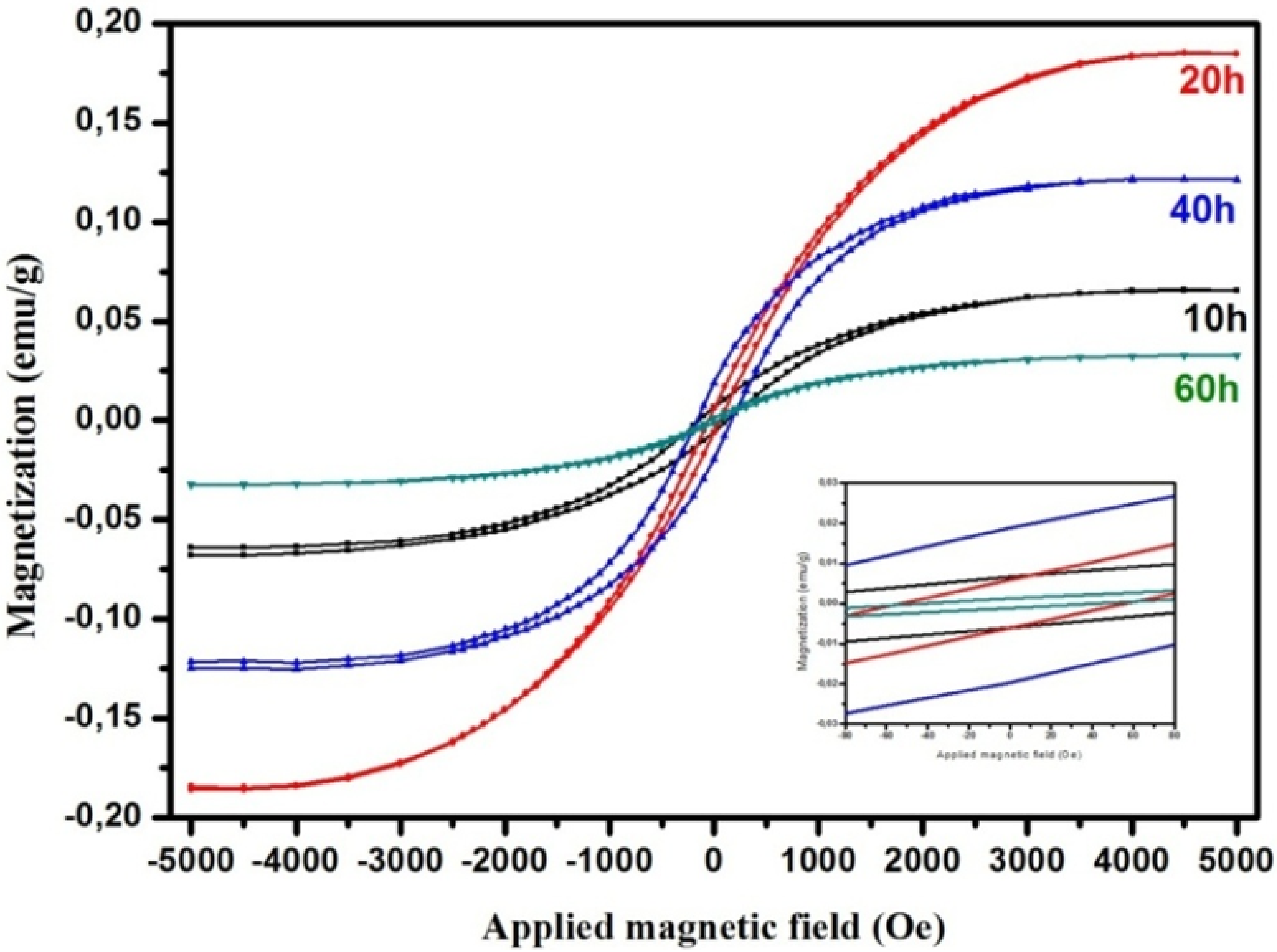

Figure 2

Hysteresis loops of nanocrystalline TiAlV alloy grinded for different time

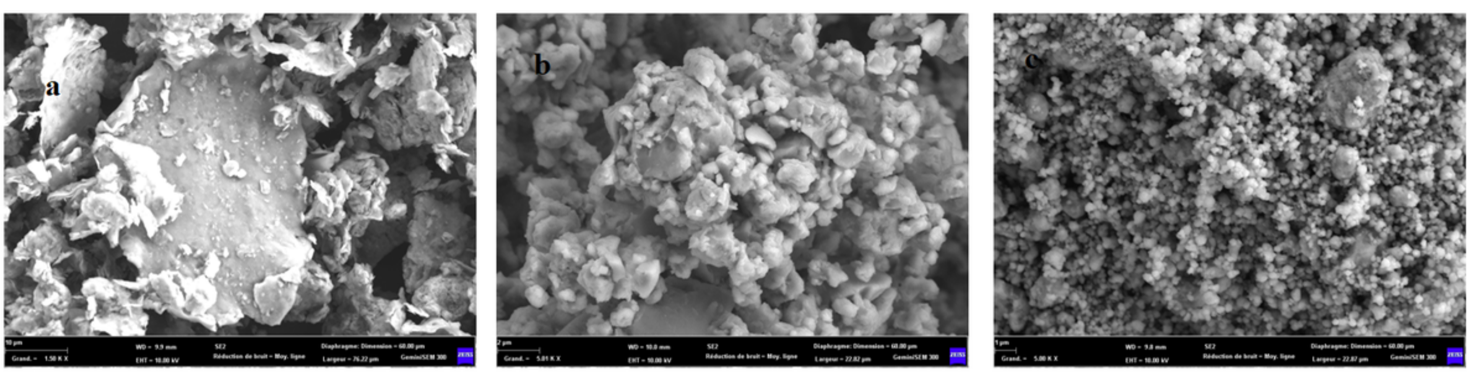

Figure 3

SEM micrographs of nanocrystalline TiAlV alloys grinded for different times: (a) 0 h, (b) 20 h, and (f) 40 h.

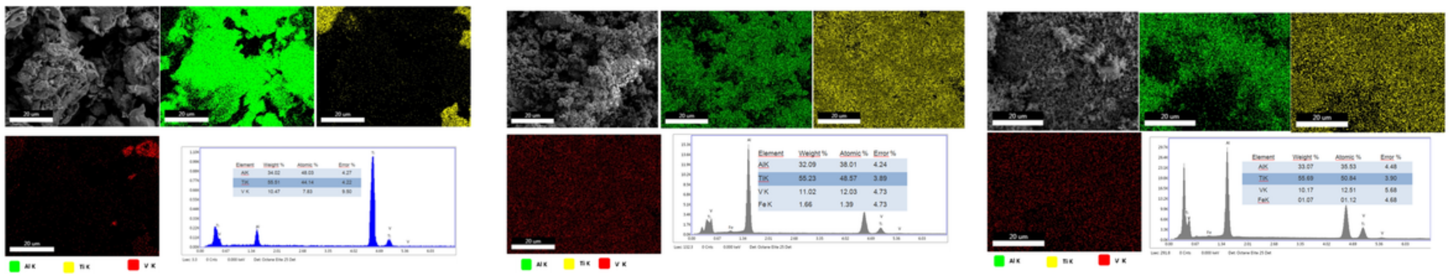

Figure 4

4a. EDS spectrum and EDS elemental distribution maps of the nanocrystalline TiAlV alloy before grinding

4b. EDS spectrum and the mapping of the nanocrystalline TiAlVgrinded at 20 h

4c. EDS spectrum and EDS elemental distribution maps of the nanocrystalline TiAlV grinded at 40 hours

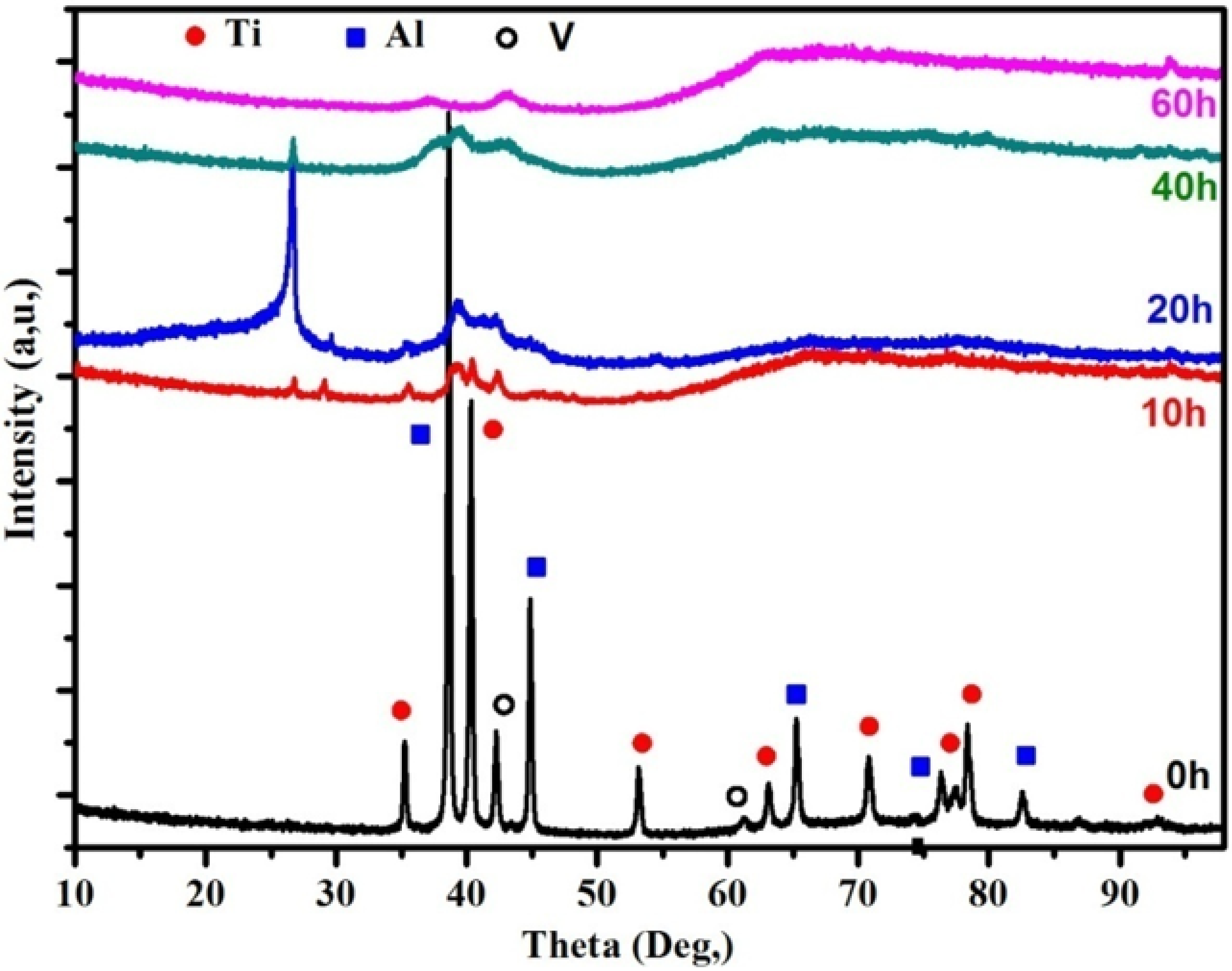

Figure 5

XRD of nanocrystalline TiAlV alloy during different grinding times.