Effects of high energy ball milling on synthesis and characteristics of Ti-Mg alloys.

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Abstract

The synthesis of Ti-Mg alloys using mechanical alloying method has been investigated. Effects of the mechanical alloying parameters on the resultant microstructural features have been studied. This work presents the effects of milling speed and milling time on green-compacts density of mechanically alloyed Ti-Mg alloy powders. Ti and Mg have been mechanically alloyed and an average green density of 75% was achieved after compaction.

Introduction

Ball milling of elemental powders is a well known process for the production of new materials, nanostructured materials and amorphous materials [1]. Mechanical alloying (MA), using a high energy ball mill, is an alternative alloy synthesis route used to produce super-saturated solid-solutions and ultra-fine grained microstructures. Ball milling of single component powders and mechanical alloying of multi-component powders involve the repeated fracturing, welding and re-welding of powder particles. The final particulate material should be suitable for consolidation and further processing to achieve desired properties. The MA technique allows for production of alloys that cannot be produced by the conventional casting methods. Ball milling is thus used as a non-equilibrium synthesis technique. MA by ball milling has advantages

of being relatively inexpensive, low processing temperatures and has potential for easy scaling-up, compared to other techniques.

MA has been found to be an effective way of producing meta-stable solid solutions in systems such as T-Mg [2]. Alloying Ti with Mg using the conventional way is very challenging as the boiling temperature of Mg is below the melting temperature of Ti. Mg therefore, vaporises before Ti is melted. However, MA has the potential to produce solid solubility extension [3].

Although the most important milling parameter is the ratio of ball weight to powder sample weight, this work focuses on the effects of two adjustable milling parameters on mechanically alloyed powders. These were: i) rotation speed and ii) total milling time.

Experimental Procedures:

The starting materials for this study were high-purity Titanium (from TLS Technik 45μ) and Magnesium (from Ecka Granules > 75μ) powders. Although several mixtures of these powders are under investigation, this paper reports on the mixture consisting of 50 at% Ti and 50 at% Mg (66.2 wt % Ti and 33.8 wt% Mg). A predetermined powder to ball ratio of 1:20 was used. Powder charge size was 100 g.

Samples were prepared in a high energy ball mill (Simoloyer) using a 1 litre stainless steel vial and 5mm ∅ stainless steel balls. Before charging the Ti-Mg mixture, Ti powder was milled in order to produce a Ti- based protective coating on both the vial walls and ball surfaces. Samples were milled in an argon atmosphere. A small amount of stearic acid was used as a process control agent to reduce the tendency of the powders to stick. Two rotational speeds of 400 rpm and 800rpm were used for

milling times up to 24 hours. Milled powders were compacted at varying pressures ranging from 82 to 408 MPa.

Phases and microstructures of the original and the mechanically alloyed powder samples were examined using a Philips PW 1830 diffractometre with a monochromatic CuKα radiation (λ = 0.15405nm) over a 2θ angle from 5º to 90º. Microstructural features of the powder particles were studied using a LEO 1525 FE-SEM with Oxford Link Pentafet EDS detector, operating at 10kV.

Results and Discussion:

Green Compacts Densities

The effects of milling speed and time on green compacts are presented in figures 1 and 2. The achieved green densities were in the range 69 -83%. Highest densities could be achieved by increasing the compaction pressure. Increasing compaction pressure by 2.5 times reduced the range of achieved densities by halve. However, increasing the milling speed did not always achieve a higher density. Densities in the of 2.20 - 2.41 g/cm^3 could be achieved by doubling the milling speed and reducing the milling time by 58 - 75% while using pressures ranging from 240 – 330MPa. .

Increasing milling speed and milling time had the initial tendency to reduce the green density to some minimum before increasing it. For 400 rpm the increase in density started after 16 hours of milling while at 800 rpm it started only after 4 hours (figure 2). Figures 3F and 4F show samples compacted to achieve a green density of 79%. An increased green density assures achievement of close-to- full density after sintering.

Metallographic Analyses (SEM)

Photomicrographs showing the changes that occur when the milling speed and milling times are varied are presented in figures 3 and 4. There is evidence of particle

fracturing and Ti powder particles are changed from their initial spherical shape to some flat irregularly shaped discs. MA is thus reliant on heavy plastic deformation of the powder particles [4].

A lamellar structure was observed in some particles suggesting a build up of alternate layers of different phases (figure 5A). Other areas showed featureless microstructures, signifying single phase formation (figure 5B). Considerable amounts of fine single phase regions will ensure a fine grained structure after sintering and improved strength properties.

Chemical constituents' analyses of isolated clusters of particles showed three distinct compositions: Ti rich, Mg-rich and differing proportions of both elements (figures 3 G and 4G), confirming the intended mix of Ti and Mg.

XRD analyses

The XRD peaks of the original Ti and Mg structures change intensity during milling, as can be seen in Figure 6. Some are almost disappearing after 8 and 16 hours of milling at 800 rpm and 400 rpm respectively. There is evidence of gradual reduction in the intensity of some Mg peaks. The reduction can be due to Mg dissolving in Ti. These results show that there is a significant extension of the solid solubility of Mg in Ti from 4 hours at 400 rpm and 1 hour at 800rpm [3].

The Titanium structure seems to be more resistant to milling, and this is consistent with the observations in SEM micrographs that show severe plastic deformation of these particles. However, in the interval between 39 and 42° (2 θ) and again around 75º, new sets of low-intensity peaks appear. The intensities of these new peaks increase with milling time indicating the appearance of new phases.

Average crystallite sizes have so far been evaluated using the Scherrer method from broadening of the diffraction lines.

$$t = \frac{0.9\lambda}{B\cos\theta}$$ Where,

t is the time average grain size (nm); λ is the Cu Kα wave length (nm), B is the diffraction peak width a half maximum intensity (radian) and θ the Bragg diffraction angle. A correction for instrument broadening was accounted for in the measurement of peak broadening: ($t^2 = t_m^2 - t_s^2$) where, t is half of maximum breadth with no broadening and t_m^2 and t_s^2 are breadths from milled powders and standard, respectively [2]. At 800 rpm, crystallite sizes of 21 nm were achieved after I hr, reducing further to 10 nm during 4 and 8 hours of milling and finally increasing to 13 after 16 to 24 hours. The initial reduction in size is due to heavy deformation and fracturing of the powder particles. During this process the reacting surfaces are increased but with longer milling times, particles will tend to cluster and have increased sizes.

Conclusions

- The research confirms that Ti – Mg solid solution was formed by high energy ball milling.
- The mechanically alloyed Ti-Mg can be compacted to a green density of at least 75% by carefully controlling MA parameters.

Further work is underway to determine the solubility limits of Mg in Ti that are obtained under the optimized milling parameters. However, a major challenge is to develop a sintering programme that minimizes the decomposition of the formed TiMg.

References

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2. Fusheng Sun and F H (Sam) Froes, Synthesis and characterisation of mechanical – alloyed Ti-xMg alloys, Journal of Alloys and Compounds,vol 340 (2002) pp220-225.
3. Gardiner R. W., Goodwin P. S., et al, Non-Equilibrium Synthesis of New Materials, Advanced Performance Materials, 3, (1996) 343-364.
4. El-Eskandarany M. S. Mechanical alloying for fabrication of advanced engineering materials, (2001) William Andrew Publishing, Norwich, New York, U.S.A.

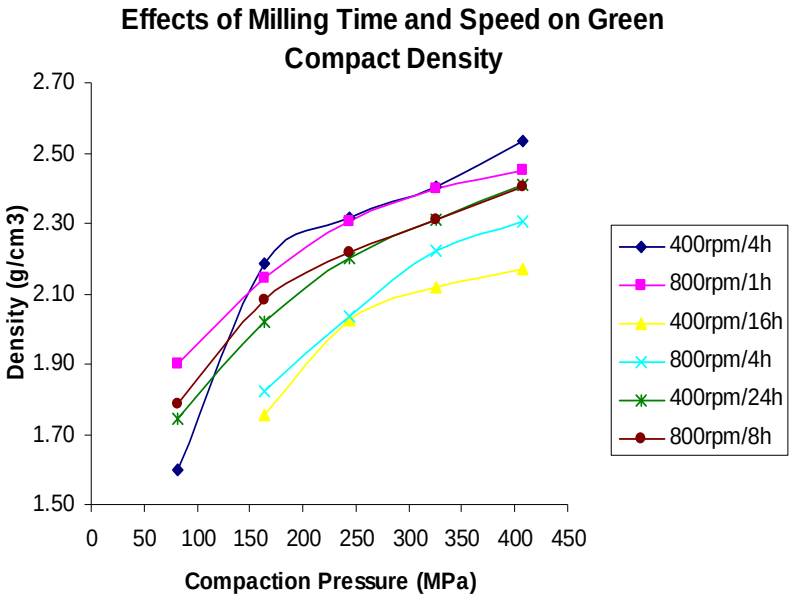


Figure 1:

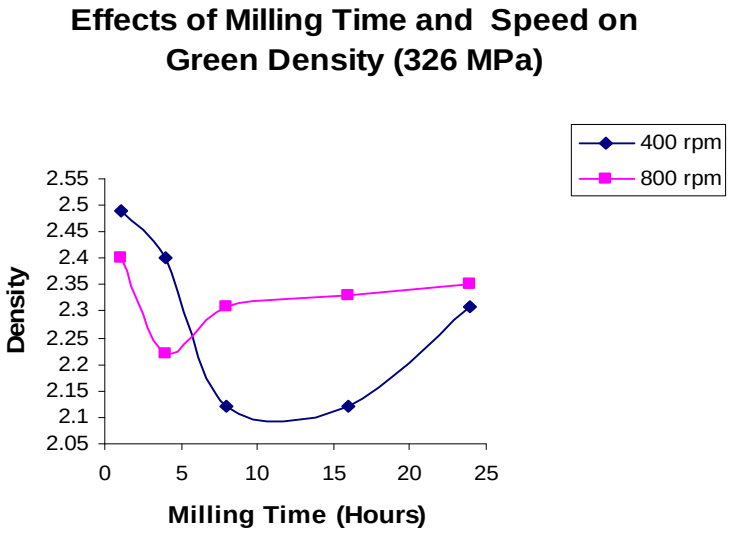
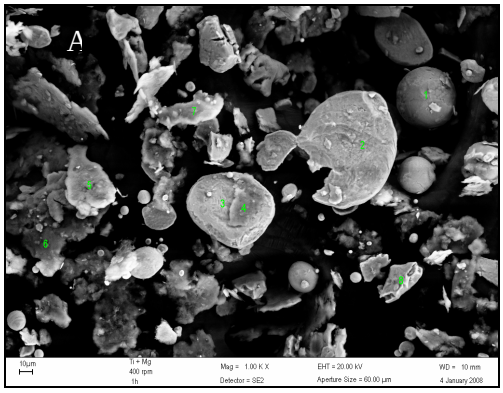
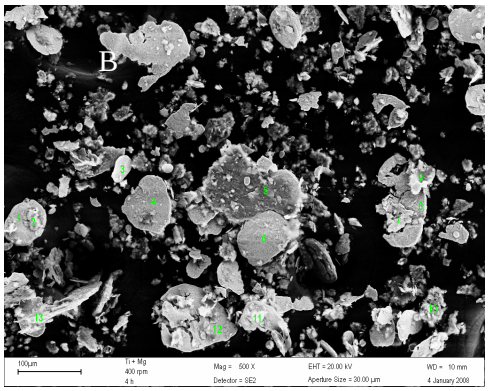


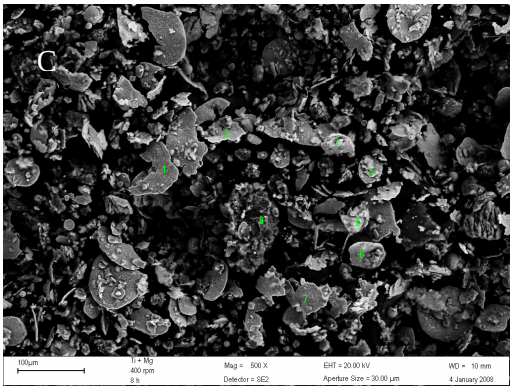
Figure 2:



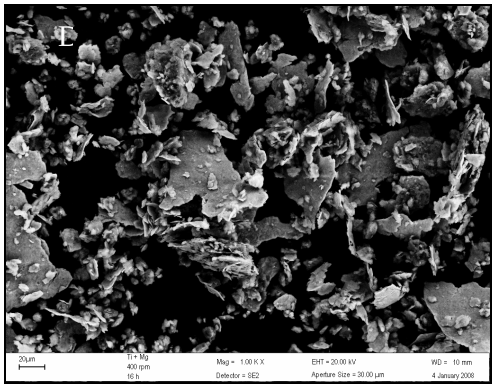
1 hour X1000



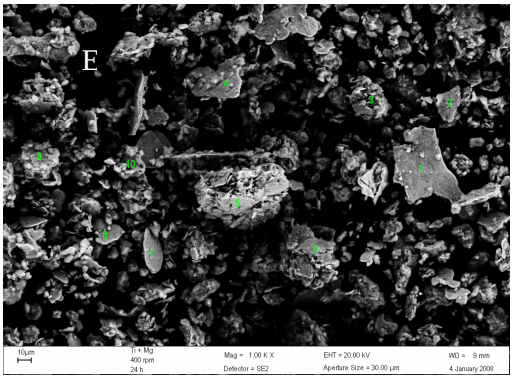
4 hours X500



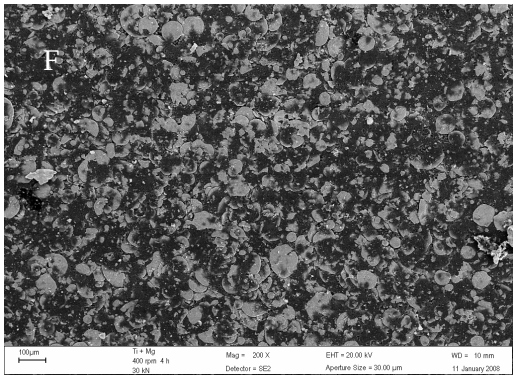
8 hours x500



16 hours X1000



24 hours X1000



4 hours 245 MPa

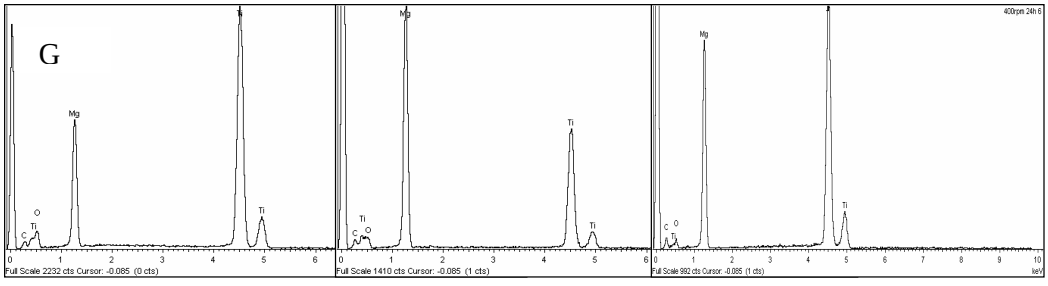
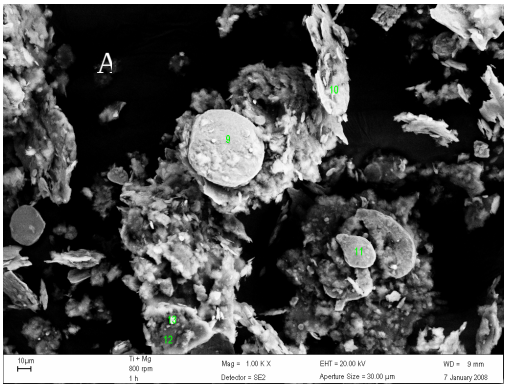
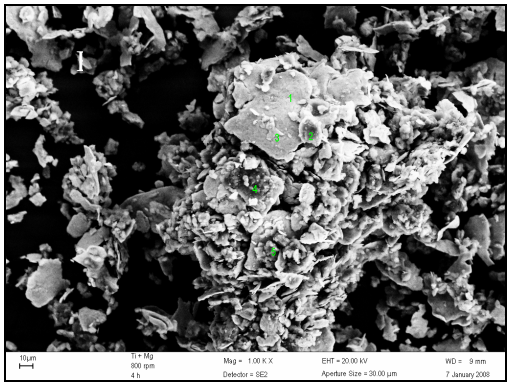


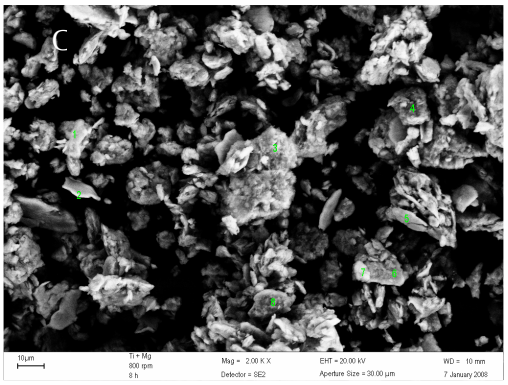
Figure 3: Photomicrographs of powders and compacts milled at 400 rpm for different times (a –f) and EDX spectra



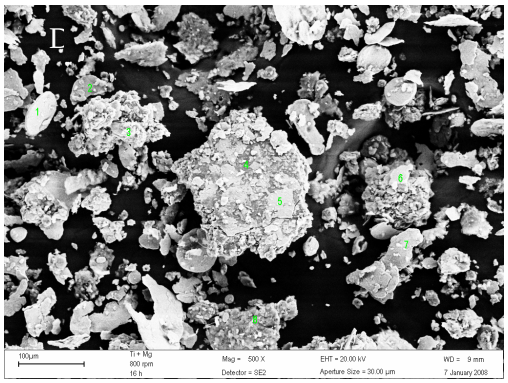
1hour X1000



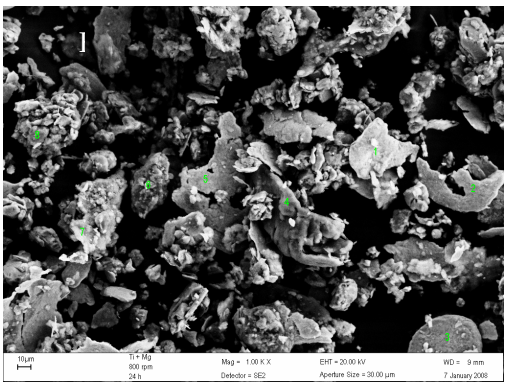
4 hours X1000



8 Hours



16 hours X500



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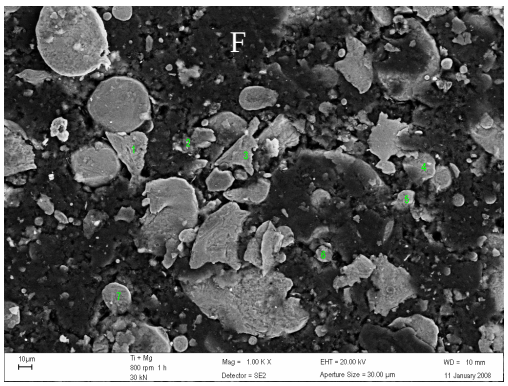


1 Hour 245 MPa

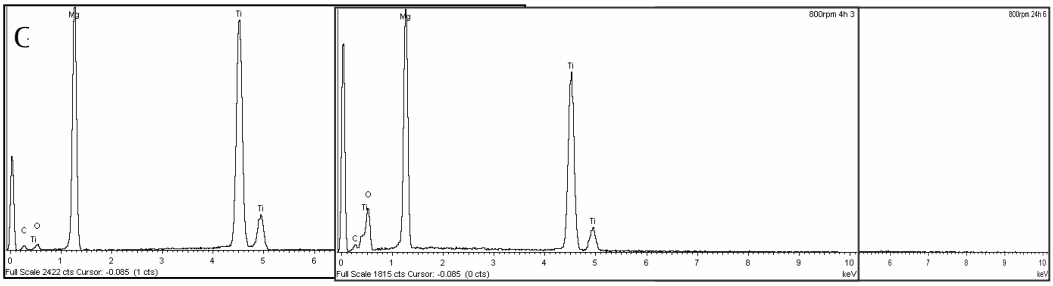


Figure 4: Photomicrographs of powders and compacts milled at 800 rpm for different times (a –f) and EDX spectra

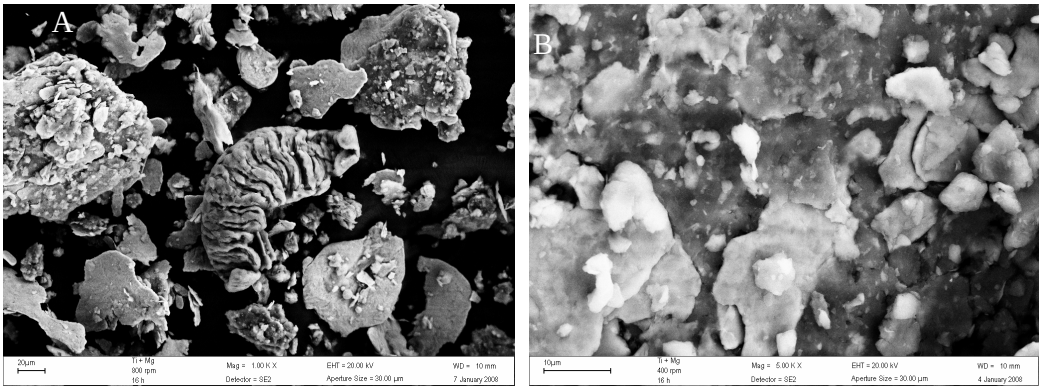
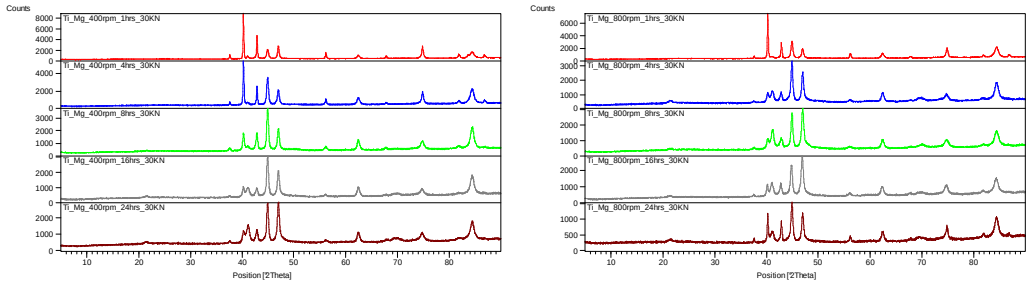
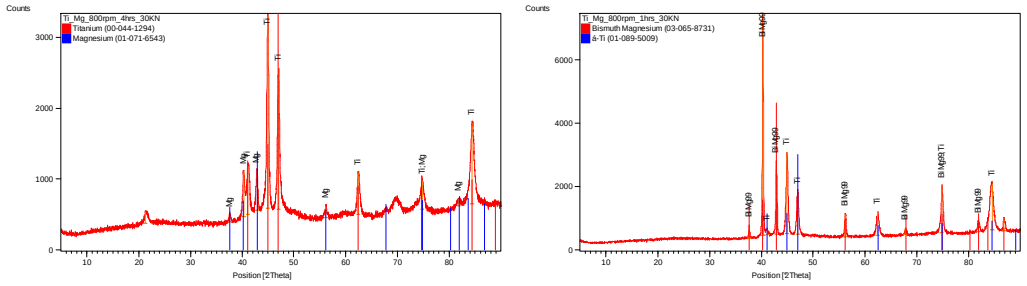


Figure 5: Photomicrographs showing lamellar Structure (A) and single phase particles (B).



A B



C D

Figure 6: X-ray diffraction spectra of the Ti Mg powders mechanically alloyed at 400 rpm (A) and 800 rpm (B) for different times.