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# MECHANICAL ALLOYING AND SINTERING OF TI – 10WT.% MG POWDERS

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## Abstract

A Ti-10wt.%Mg powder alloy has been produced by mechanical alloying. Elemental powders of Ti and Mg were ball milled in a Zoz-Simoloyer CM01 for 16 and 20 hours under argon. Mechanical alloying was followed by XRD, SEM and particle size analysis. Test specimens of the milled powders were produced by cold compaction and sintering (under argon). The milling resulted into a fcc TiMg solid solution for both milling durations. Sintering leads to a partial decomposition of the fcc TiMg to hcp TiMg and Ti.

## Introduction

Advanced titanium (Ti) alloys are finding increasing use in aerospace and automotive industries where only stronger and lighter materials can lead to improved performance. Widespread use of the alloys requires that both cost and density be reduced. It has been recognized, for weight sensitive industries, that a density reduction is 3-5 times more effective in reducing the structural weight than increases in tensile strength and elastic modulus [1].

Low density Ti alloys are obtained by using low density alloying elements such as aluminum (Al) and silicon (Si) for which there are commercial alloys [2]. Attempts have been made to produce Ti - magnesium (Mg) based alloys using mechanical alloying, with varying degrees of success [3 – 6]. Mechanical alloying has been used to increase the solubility of Mg in Ti, which under equilibrium conditions, is almost zero even at high temperatures. This study aims to produce and sinter Ti-10wt.%Mg alloy powders by mechanical alloying.

## Experimental Procedures

Elemental powders of commercially pure Ti (< 45µm), and Mg (<150µm) were mixed to obtain Ti-10Mg nominal compositions. Milling was carried out in a high energy ball mill using a stainless steel vial under argon. The milling speed was kept at 800rpm for uninterrupted durations of 16 and 20 hours. Steel balls of 5mm diameter were used as the grinding media and a ball-to-powder mass ratio of 20:1 was used. To avoid powder agglomeration, a carefully calculated process control agent (PCA) was added to the powders before milling.

An X-ray Diffractometer (Phillips – Type PW 1710) fitted with a Cu anode was used to determine the different phases in the milled powders. The Scherrer formula was used to calculate the average grain sizes of the powders. Powder particle morphologies and other structural features were studied using a scanning electron microscope (SEM) while the powder particle sizes and size distributions were measured using a particle size analyser.

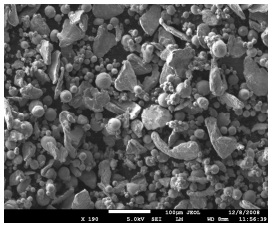
Milled powders were subsequently compacted and sintered. Compaction was achieved by pressing the mechanically alloyed (MA) powders using a uniaxial single action press at compaction pressures of 10, 20, 30 and 40MPa. Sintering was done at $630^0$C for 12hours under argon followed by furnace cooling. Metallographic analyses of the sintered specimens

were carried out using both SEM and optical microscopy. Hardness testing was done using a Vickers hardness tester.

## Results and discussion

### Powder characterization

The different morphologies of the blended and milled powders are shown in Figure 1. Ti powder particles were spherical while Mg powder particles were plate-like while the milled alloy powders were characterized by globular agglomerates (Figure 1 (b), (c)).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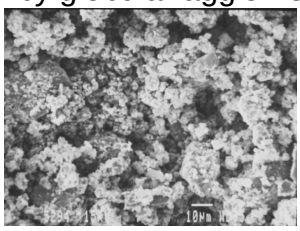
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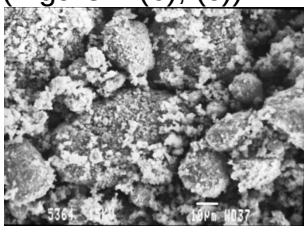

(c)

**Figure 1** Morphology of blended powder (a) and powder milled for 16hrs (b) and 20hrs (c)

Figure 2 shows the particle size distribution of blended and milled powders. Milling led to a decrease in particle size and narrowed the particle size distribution. Increasing milling time changes the mean particle size and but does not have an effect on size distribution.

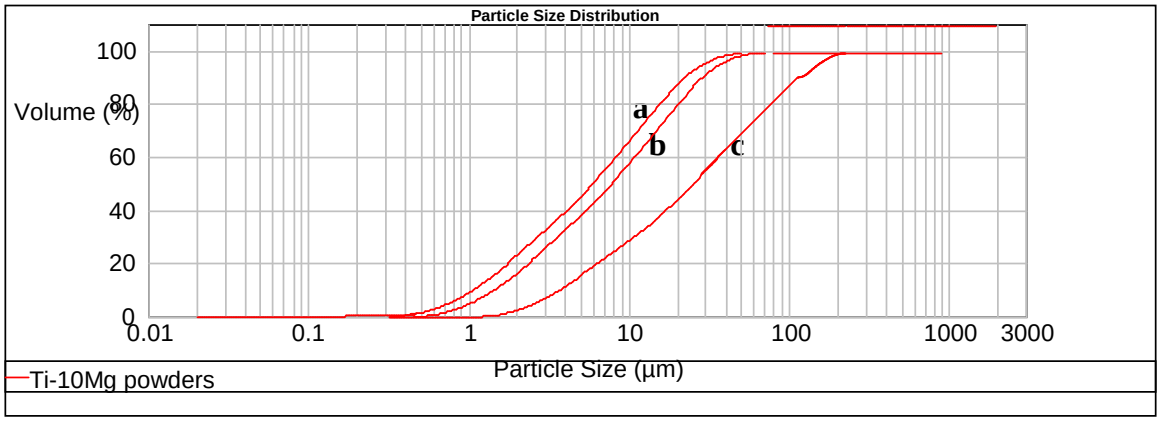
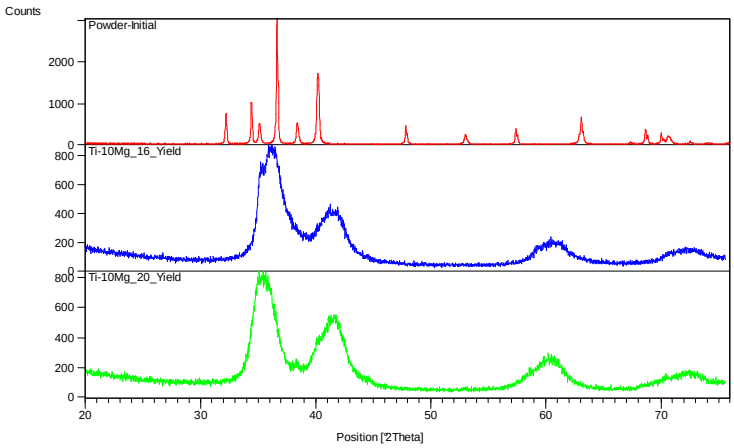



**Figure 2** Powder particle size distributions a) Powder milled for 16hrs, b) Powder milled for 20 hrs, and c) Blended powder

Figure 3 shows the XRD patterns of the blended and milled powders. Mg and Ti peaks were observed in the blended powders. Upon milling, the Mg peaks disappeared and peaks for a new face centred cubic (fcc) phase identified as Ti-Mg alloy phase emerged. The observation of the fcc phase is in agreement with literature [3]. Increasing the milling time did not have an effect on this fcc end phase. This observation has also been made for milled Ti metal [7]. The Ti-Mg alloy peaks were broadened, suggesting a decrease in crystallite size [2]: the powders had an average grain size of 3.5nm according to the Scherrer formula.

The green density of compacts increased with compaction pressure (Figure 4), in line with literature [8]. The increase is attributed to an increase in inter-particle contact and the increasing plastic deformation. It was also noticeable that milling for longer times led to a slight decrease in green density. It is possible that this could be due to the different mean particle sizes (Figure 2), where the powder with the coarser particles has the lower green density and is in agreement with literature [9].



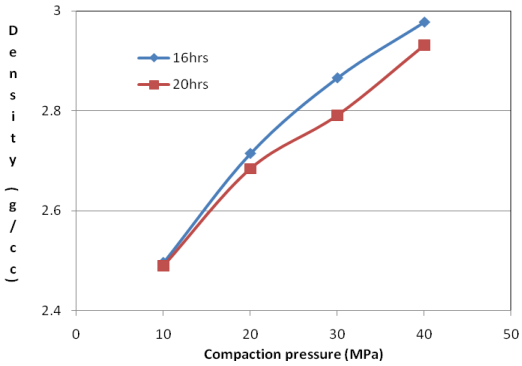




**Figure 3** XRD patterns of blended and milled powders **Figure 4** Green density of compacts

## Characteristics of sintered bodies

### Microstructures

The SEM images of the sintered specimens obtained in back scattered mode, were characterized by varying grey level contrast. This was determined by EDS to have been caused by the varying amounts of Mg in solution in Ti. Areas that had more Mg in solution were darker (Figure 5). Dark spots were found to have been Mg rich. However, their very fine sizes precluded extensive analysis.

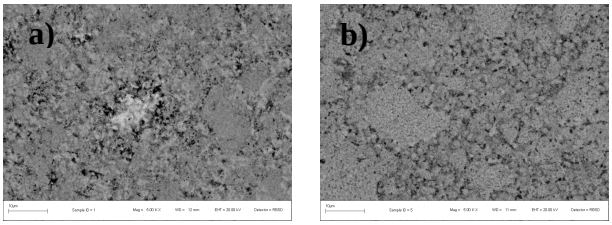



**Figure 5** Typical microstructure of sintered bodies. Microstructure for powder milled for 16hrs and compacted with a load of **a)** 10MPa, **b)** 30MPa.

### Phase composition

Analysis of the phase compositions revealed that while milled powders had a single-fcc Ti-Mg alloy phase, the sintered bodies had multi-phases consisting of fcc TiMg and hcp TiMg solid solutions, Ti metal, and $Ti_xO_y$-type and MgO oxides. This was the case for all components regardless of the milling time. The three common phases were fcc TiMg, hcp TiMg, and Ti with the former two being the major phases. Generally, increasing the compaction pressure increases the intensity of the strongest peak of the hcp TiMg phase (Figure 6). The intensity of the free Ti peaks increases with the compaction pressure. The occurrence of free Ti suggests that there has been some decomposition of fcc Ti-Mg solid solution in the powders during sintering. However, the decomposition was minimal because no Mg peaks were observed and only a few sintered specimens showed MgO peaks. It is possible that some of the free Mg evaporated. Testing is still underway to determine how much Mg was left in solution in Ti. The occurrence of type $Ti_xO_y$ phases in some specimens showed that some Ti oxidation had occurred. Both the oxidation of Mg and Ti might have been caused by air entrained in the argon used during sintering.

The occurrence of both fcc TiMg and hcp TiMg solid solutions in sintered MA Ti-Mg has not been reported before. FCC Ti-Mg solid solution has been reported in sintered Ti-Mg MA powders [3], while an hcp Ti-Mg solution has been observed in annealed vapour quenched Ti-Mg alloys [10]. The mixture of TiMg solid solutions obtained here might have been due to solid phase sintering as opposed to liquid phase sintering.

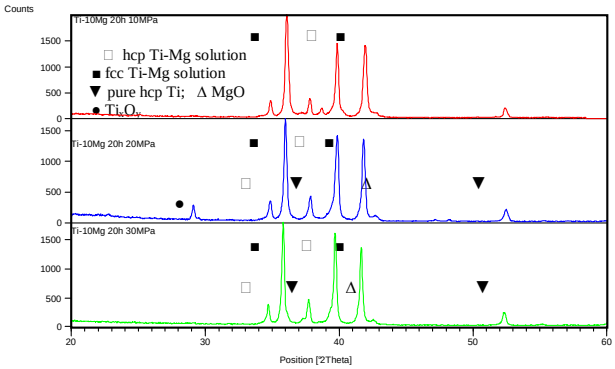



**Figure 6** General XRD patterns for sintered specimens showing a more phases than in the milled powders.

## Hardness values

The hardness values had a small scatter, indicating a homogeneous chemical composition on the macro level. They increased with increasing compaction pressure (Figure 7). This is in line with the green density (Figure 4). The hardness values are yet to be linked to the fcc and hcp TiMg phases and will be addressed in future work.

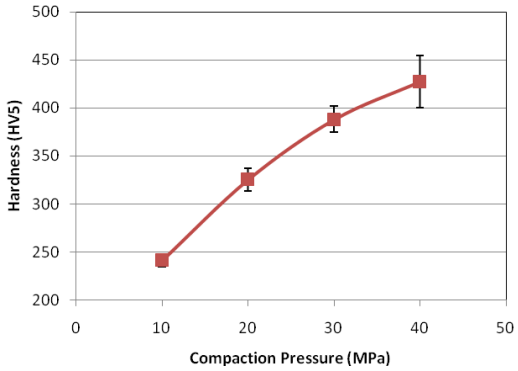



**Figure 7** Variation of hardness with compaction pressure for specimens sintered from powder milled for 20 hours.

The hardness values obtained in this study are higher than those obtained from comparable vapour quenched Ti-Mg alloys [10]. The higher hardness observed here could have been caused by the presence of MgO in the alloys.

## Conclusions

The present results have indicated that high Mg content fcc Ti-Mg alloy powders can be produced by mechanical alloying. The end phase in the milled alloy powders (16 – 20hrs) is independent of the milling time. Sintering of the milled alloy powders resulted in the partial decomposition of the fcc phase to produce some hcp Ti-Mg alloy and some pure Ti metal. Work on the relation between hardness and phase composition is underway.

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