EFFECT OF PROCESS CONTROL AGENT (PCA) ON THE CHARACTERISTICS OF MECHANICALLY ALLOYED TI-MG POWDERS

C Machio, H K Chikwanda and S Chikosha
[a]Council for Scientific and Industrial Research (CSIR)

Abstract

This article reports results of a study to determine the effect of process control agent (PCA) on the characteristics of Ti-Mg powders during milling. It has been shown that a 2% increase in PCA content leads to up to a 40% increase in yield of the milled powder but reduces the kinetics of the mechanical alloying process. The introduction of 4% PCA decreases the mean powder particle size by up to 30%, while 6% PCA increased the mean particle size by up to 230%. The characteristics of the milled powders will be discussed in relation to the role of PCA in the milling process.

1.0   Introduction:

The production of alloy powders through mechanical milling has advantages such as increasing solid solubility limits. Alloying occurs through repeated cold welding and fracturing resulting from impacts from the milling media. The fracturing process exposes new surfaces over which atomic diffusion occurs leading to alloying. Mechanical alloying has been applied to titanium (Ti) and magnesium (Mg) powders in order to produce Ti-Mg alloy powders. Ti-Mg alloys are of particular interest because they have potential to produce lightweight structures, with attendant improvements of e.g. fuel efficiency. Studies have shown that density reductions, especially in the aerospace industry, are more effective in reducing component weight than increases in strength or modulus of elasticity [1].

The mechanical alloying process is sensitive to the milling parameters. Throughput or yield, the amount of powder obtained from a milling run, is determined by the milling speed and time and the amount of process control agent (PCA) used. The PCA has a lubricating effect that minimizes the cold welding effect. Without the PCA, the powder being milled welds on the milling chamber vessel walls resulting in only a fraction of the charge load being recovered. The welding also increases downtime between milling runs. On the other hand, too much PCA affects the kinetics of alloying. It is therefore important that the amount of PCA is optimised to ensure a high yield and short downtimes during milling. In this article, the effect of varying amounts of PCA on yield and alloying of Ti-Mg powders will be investigated.

2.0   Methodology

2.1   Materials

The powder used in this exercise was Ti – 30wt.%Mg. The powder mixture was made from elemental titanium powder (~99.5% purity) and elemental magnesium powder

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(~99.5% purity). The sizes of the powder particles were less than 45 µm for Ti and less than 75 µm for Mg. The particles of the Ti powder were spherical while those of Mg were platy (Figure 1). Stearic acid was used as the process control agent, and the amount was varied between 0 and 6 wt.%.
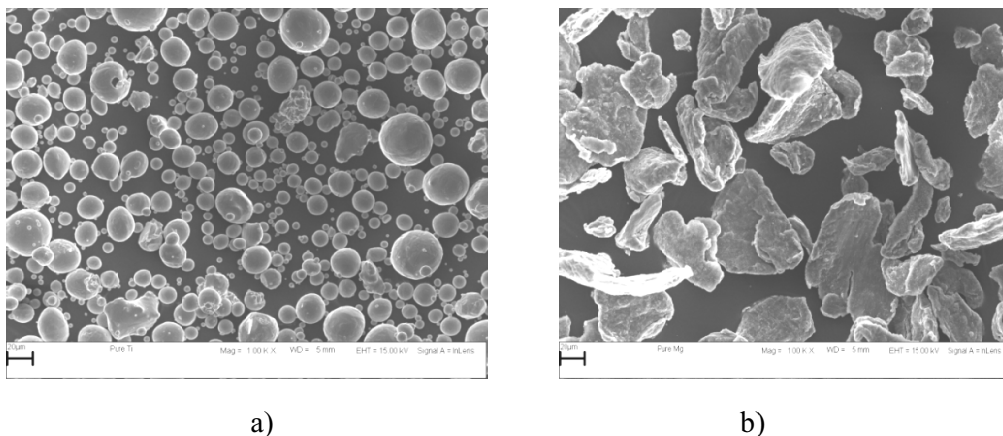
a)	b)

Figure 1 Morphology of elemental a) Ti and b) Mg.

2.2   Mechanical alloying

The powder mixtures were milled in a Simoloyer high energy ball mill using a rotation speed of 800rpm, a ball-to-powder (mass) ratio (BPR) of 20:1 and a milling time of 8hrs. The mass of the Ti – 30wt.% Mg powder mixture was kept constant at 100g, while the PCA amounts used were 0, 2, 4 and 6wt.%. At the end of each milling run, two types of powders were collected: yield powder and weld powder. Yield was defined as the free flowing powder obtained by inverting the milling chamber to the discharge position and was labelled A1, B1, C1, and D1 for 0, 2, 4, and 6wt.% PCA respectively. Weld powder was powder that stuck on the chamber walls and was labelled A2, B2, C2, and D2 for 0, 2, 4, and 6wt.% PCA respectively.

2.3   Powder characterization

The yield powders were weighed and both yield and weld powders were analysed for phases using X-ray diffraction (XRD) studies on a Phillips PW 1710 X-Ray Diffractometer. Powder particle morphologies were determined using a JEOL JSM-6510 Scanning Electron Microscope (SEM), and the particle size and size distributions were determined using a Microtrac® Bluewave particle size analyser that uses laser diffraction to determine particle size.

3.0   Results and Discussion

3.1   Particle Size and particle morphology

The mean powder particle sizes and the particle size distribution for the various PCA contents are shown in Table 1 and Figure 2 respectively. The introduction of 2wt.% PCA did not change the particle mean size significantly. However, the particle size distribution changed, with the appearance of a second peak in the normal powder

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distribution curve (Figure 2) at higher sizes. This shows that there was a tendency for the powder to cold weld. Increasing the PCA content to 4wt.% decreased the mean particle size by 34%, from 41 to 27 μm. Both the cumulative and normal particle size distributions curves were moved to smaller sizes. The normal particle size distribution curve was narrower than for the powder mixture with 0wt.% PCA. The tendency towards cold welding was less than when 2wt.% PCA was used. The evolution of the particle size distribution for this PCA content suggests that the process of powder particle fracture had dominated cold welding during milling. A further increase in the PCA content to 6wt.% led to a bi-modular particle size distribution and to substantial powder particle coarsening, by up to 230%, that can be accounted for by cold welding. The particle refinement observed when 4wt.% PCA was used continued even at 6wt.% PCA, but the fine particles generated had a high tendency to cold weld.

Table 1 Mean yield powder particle size for various PCA contents

PCA content (wt.%)	Mean particle size (μm)
0	41
2	46
4	27
6	135

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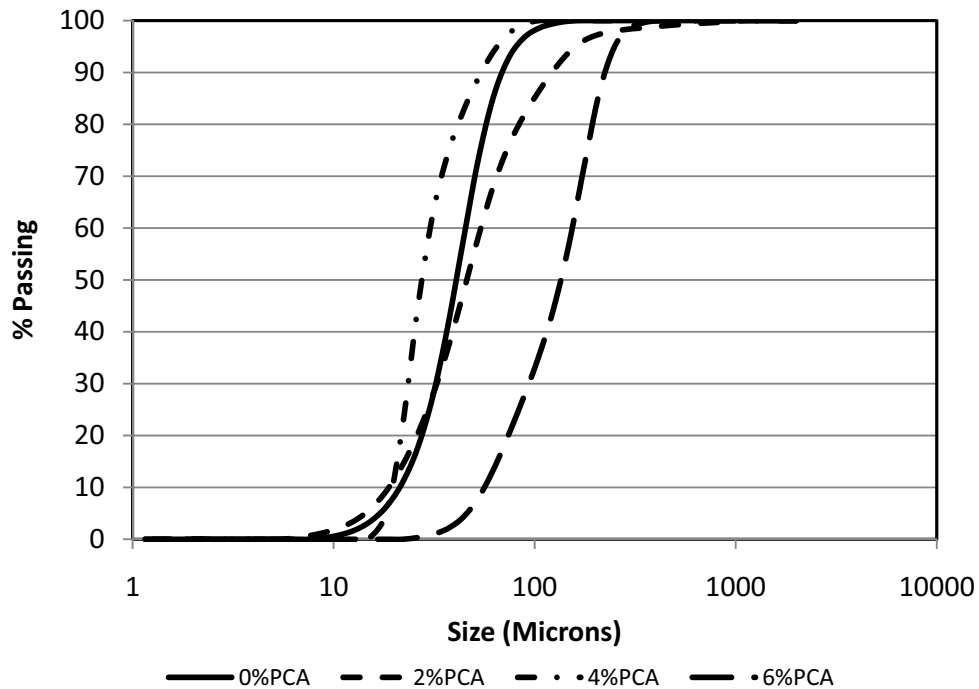
a) Cumulative particle size distribution of milled powders as a function of PCA content
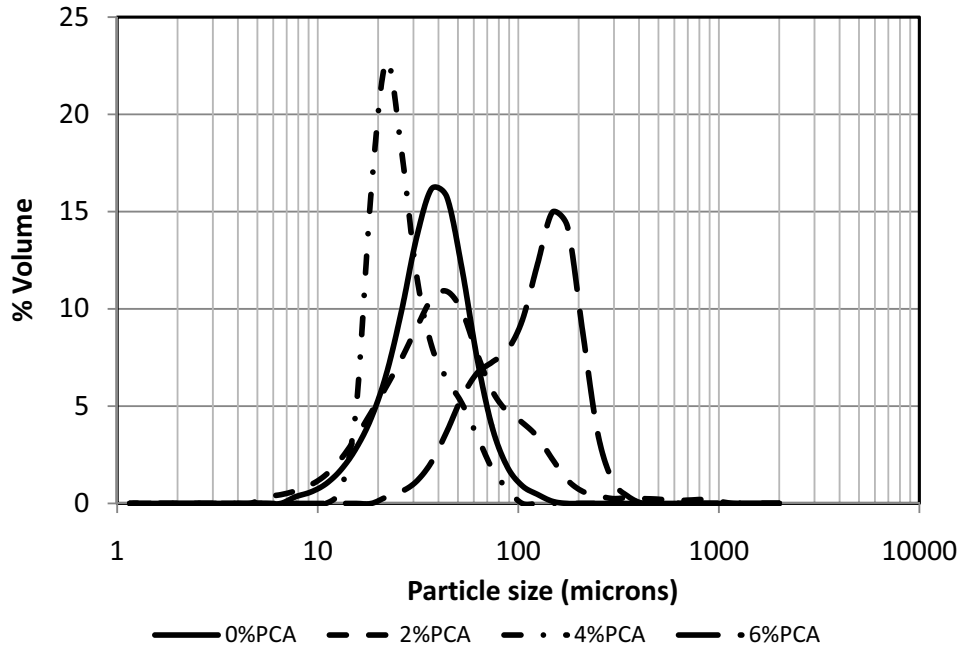
b) Normal particle size distribution curves of powders as a function of PCA content

Figure 2 Cumulative and normal powder particle size distributions as a function of PCA content

The milling led to a change of particle shape from the spherical and platy morphology of the elemental powders to irregularly shaped particles (Figure 3). The shapes are typical of balled milled powders [2].
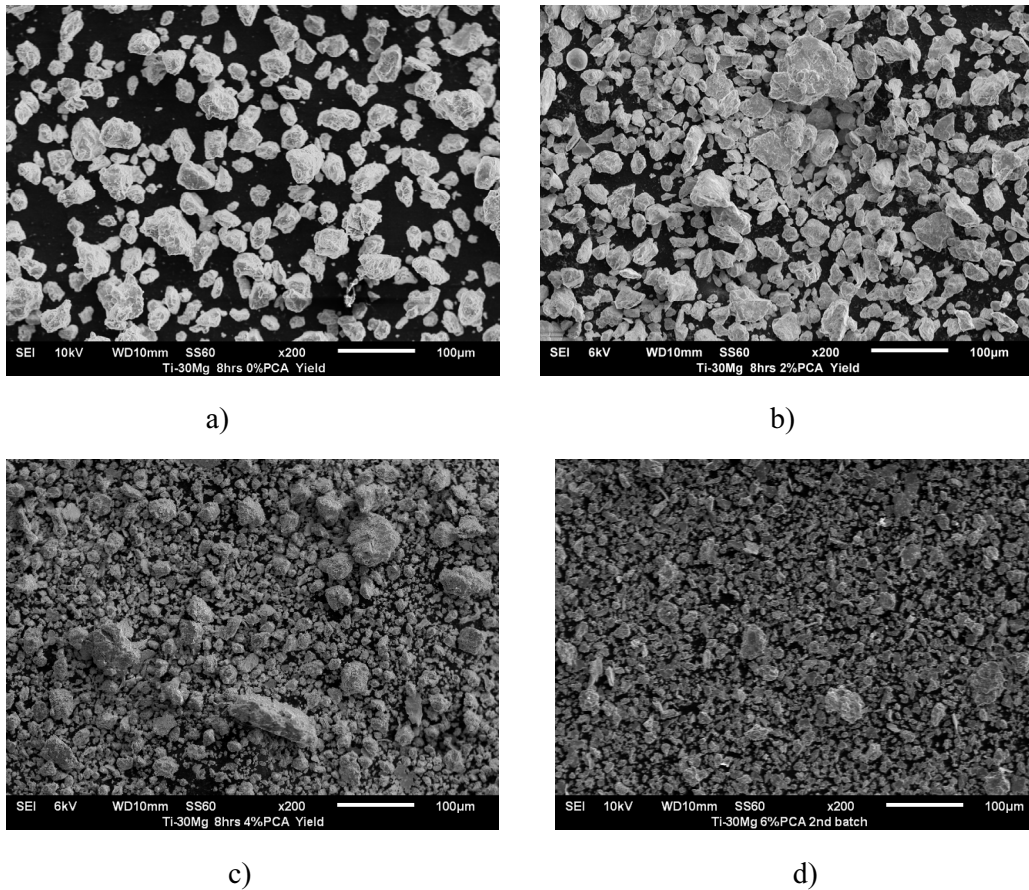
Figure 3 Morphologies of milled powders as a function of PCA content: a) 0wt.% PCA, b) 2wt.% PCA, c) 4wt.% PCA, and d) 6wt.% PCA

3.2   Powder yield

Table 2 and Figure 4 show the variation of powder yield as a function of the amount of PCA used during milling. The introduction of 2wt.% PCA did not change the amount of yield powder but increasing the PCA content further led to an increasing powder yield. The time between milling runs also got shorter with increasing PCA content because powder that had welded on the milling chamber walls was progressively easier to remove with increasing PCA content. At 0 and 2wt.% PCA, powder on the wall of the milling chamber was much harder to remove, while a simple brushing off was sufficient at both 4 and 6wt.%PCA. The powder milled with 6wt.% PCA was readily flammable and required extra handling during discharge, implying that the powder mixture was still activated at the end of the milling cycle. This high reactivity was caused by the existence of fresh fracture surfaces and indicates that the high PCA content slowed down the kinetics of alloying.

Table 2: Powder yield as a function of amount of PCA

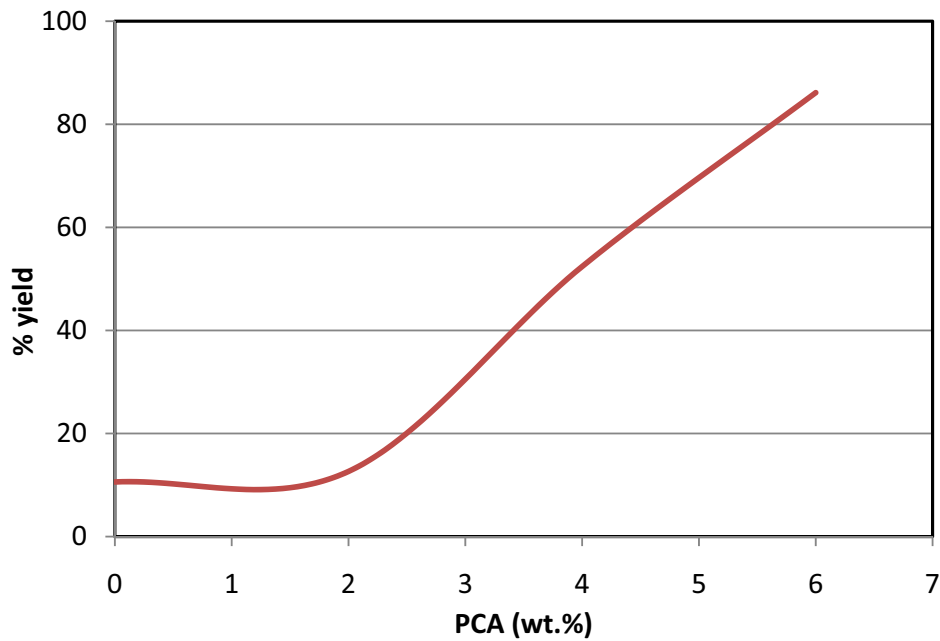| Powder	PCA content (wt.%)	Yield (g)	Yield (%)
A1	0	11	11
B1	2	13	13
C1	4	52	52
D1	6	91	86

Figure 4 Variation of yield as a function of the amount of PCA

3.3   Phases of milled powders

The XRD pattern of the unmilled powder mixture showed the presence of Ti and Mg (Figure 5), and is given here for purposes of comparison. The higher prominence of the Mg peaks in Figure 5 could have been caused by the fact Ti has higher absorption of the Cu X-rays.
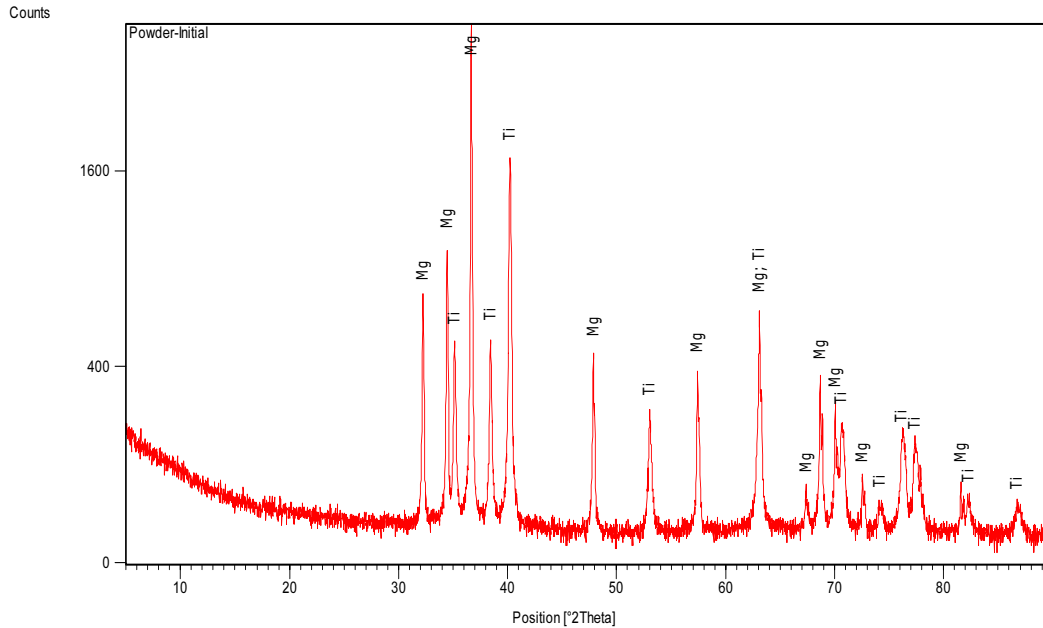
**Figure 5** XRD pattern of initial powder showing distinct peaks for Ti and Mg

Figure 6 shows the XRD patterns of powders A1 and B1, the yield and weld powders of 0wt.%PCA powder, respectively. Mg peaks were missing from the XRD pattern of powder A1 and the Mg and Ti peaks between 34 and 39° 2Theta had been replaced by one broad peak (Figure 6 a)). On the other hand, the pattern for the weld powder had both Mg and Ti peaks. The missing Mg peaks on the XRD patterns of the yield powder indicates the formation of a Ti-Mg solid solution. However, the solid-solution was not fully formed because there were no noticeable shifts of the peaks of Ti. This observation indicates that the milling time should have been longer than what was used in the study if complete dissolution had been desired, as subsequent work on other Ti-Mg powders has shown [3].

The occurrence of Mg peaks on the XRD pattern of powder A2, the weld powder of the powder mixture milled with 0wt.% PCA, showed that there had been minimal, if any, dissolution of Mg in Ti. The high prominence of the Mg peaks indicates the Mg particles were still large. Both of these observations imply that the weld powder was not exposed to the same impact energy like the yield powder.

The difference of phases between the yield (A1) and weld (A2) powders emanates from two, apparently opposing and yet facilitating steps in mechanical alloying, which are cold welding and fracture [4]. As already noted, reactions in mechanical milling occur via cold welding of the powders being milled and subsequent fracturing. Fracturing makes available fresh surfaces over which atomic diffusion occurs when the surfaces are cold welded. Unchecked cold welding effectively stops any reaction from occurring. Powder A2, as noted previously, was strongly bonded to the milling chamber, suggesting that cold welding was very advanced and that new surfaces required for diffusion were not formed.
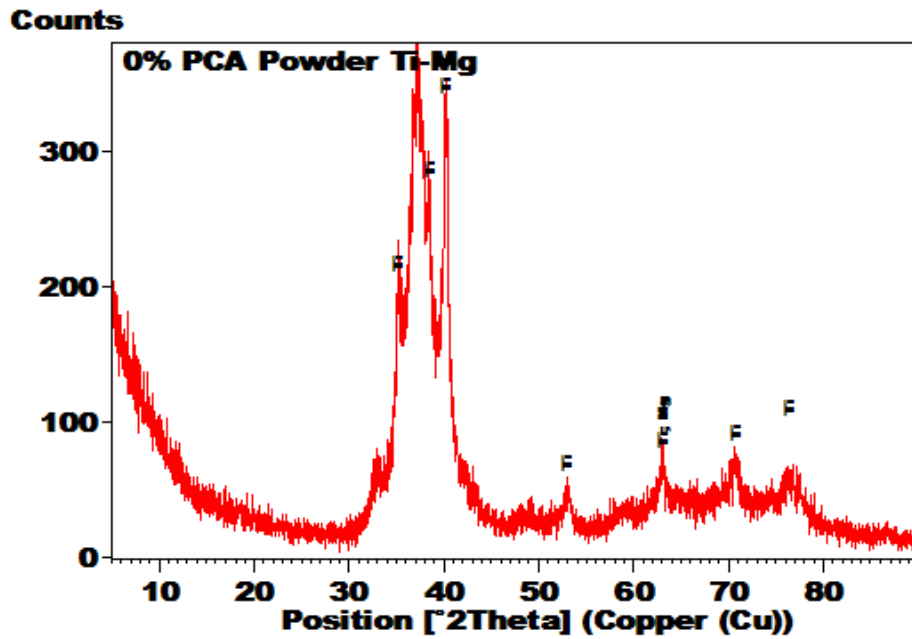
a) XRD pattern of powder A1 showing that Mg peaks have disappeared, and a broad peak has emerged between 34 and 39° 2Theta.
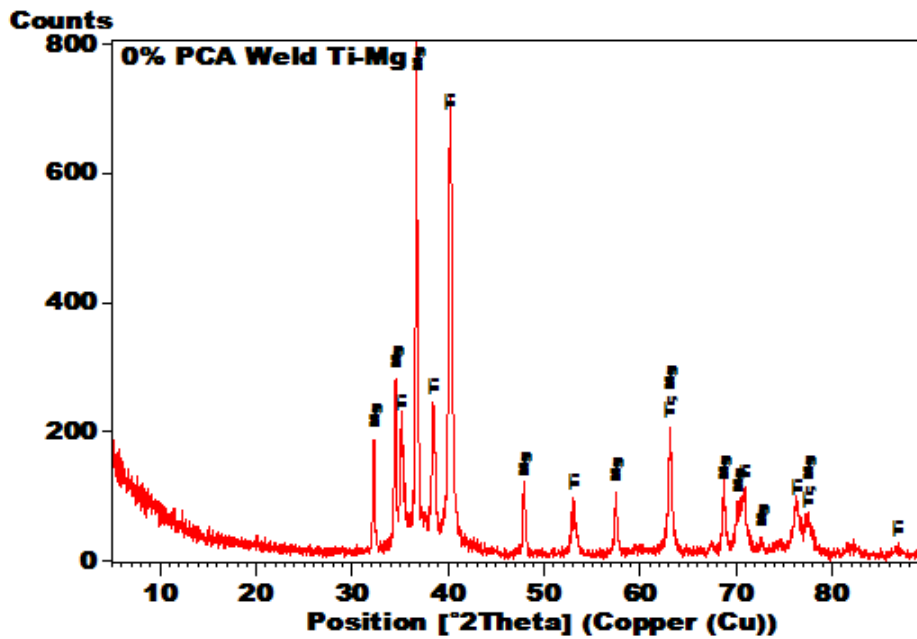
b) XRD pattern of powder A2 showing the existence of both Mg and Ti. The relative intensity of the Mg peaks is still high.

Figure 6 XRD patterns of powder milled with 0wt.% PCA. Powder A1 is the yield while A2 is the weld.

Figure 7 shows a characteristic XRD pattern of yield powders obtained from 2 PCA powders, showing the presence of Mg peaks alongside Ti peaks. Similar patterns were obtained from the yield powders of 4 – 6wt.% PCA. The presence of Mg peaks indicates that not all the Mg had dissolved in Ti, as had occurred for yield powder from 0wt.% PCA powder (Fig 6 a)). The lack of dissolution was caused by the higher PCA contents that retarded diffusion of Mg into Ti, and is in line with literature on other powders [5]. The Mg peak at about 34° 2Theta (Figure 7) became increasingly more prominent with increasing PCA content, implying that the amount of Mg undissolved in Ti increased with PCA content.
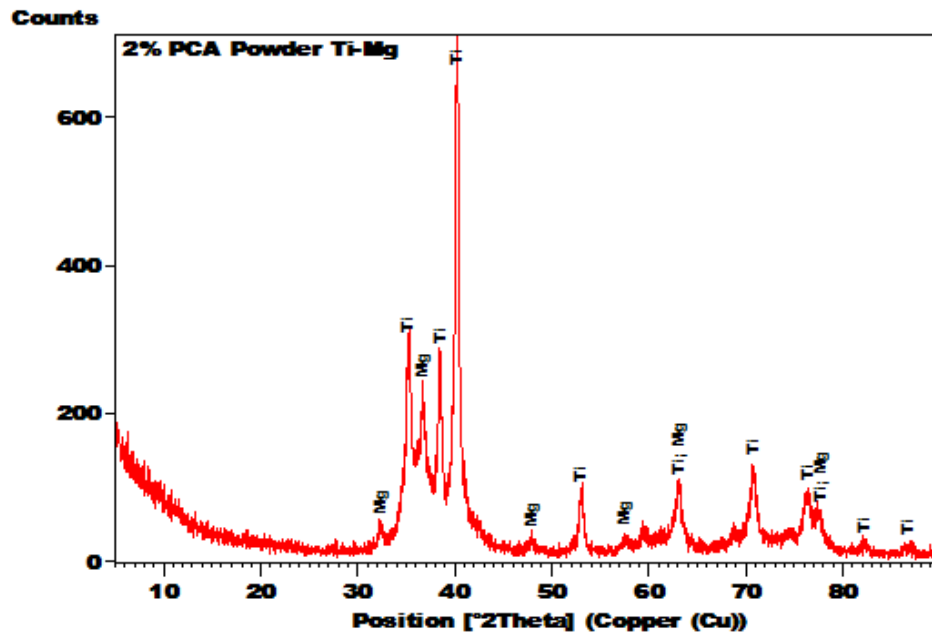

Figure 7 XRD pattern for powder B1 showing both Mg and Ti peaks. Similar patterns obtained from the yield powders of 4 and 6wt.% PCA powders.

The occurrence of Mg peaks in the 2 – 6wt.% PCA yield powders makes them similar to the weld powder of the 0wt.% PCA powder. These powders did not experience the necessary impact energy required to force Mg into solution in the lattice of Ti. The XRD patterns of the weld powders from 2 – 6wt.% PCA powders were similar to the XRD pattern of the yield powder from 2wt.% PCA (Figure 7.). Unlike the weld powder from 0wt.% PCA powder (Figure 6 b)), the Mg peaks had a lower relative intensity. This could have been caused by a combination of dissolution and particle size reduction. The presence of PCA reduced the intensity of cold welding during the milling process, as it is supposed to [4], and encouraged fracture, that exposed fresh clean surfaces, and re-welding, which facilitated atomic diffusion both in the yield and in the weld powders. Also, Mg undergoes a rapid decrease of size compared to the Ti during milling [6], and smaller particles diffract X-rays less than big particles.

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The peaks of phases in the milled powders were broader than the same phases in the unmilled powder. Peak broadening occurs when the particles diffracting X-rays become smaller and/or when strain is introduced in the lattice of the diffracting particles [7, 8]. These two factors are in play in the current study because particle size reduction through mechanical deformation is inherent in milling [4].

4.0   Conclusions

This study has shown that the amount of PCA used in milling determines the yield of milled powder: a higher PCA leads to a higher yield. However, a higher PCA content has also been found to retard the dissolution kinetics and to lead to a situation where the phases in the yield powders appear more and more like the phases in the initial powder mixture. The inverse variation of yield and phase composition observed in this system appear to suggest that each PCA content has a characteristic milling duration below which initial phases are retained in the yield powder. Future work can start by determining what this characteristic milling duration is for the PCA contents investigated in the current study. The current work also suggests that this characteristic milling duration will have to be determined each time a new PCA content is chosen for milling.

Acknowledgement

References

1. J. C. Ekvall, J. E. Rhodes, G. G. Wald (Eds.), Design of fatigue and fracture resistant structures, ASTM Special Publication 761, ASTM, Philadelphia, 1982, pp. 328 – 336

2. Sun, F., Froes, F. H. (Sam), Synthesis and characterization of mechanical-alloyed Ti-*x*Mg alloys, *Journal of Alloys and Compounds,* 340 (2002) 220 – 225

3. C Machio, D Nyabadza, H K Chikwanda, M Phasha, V M Sibanda, *Mechanical alloying and sintering of Ti-10Mg powders*, Material Science Forum 618-619 (2009) 105 – 108

4. Suryanarayana, C., Mechanical alloying and milling, Progress in Materials Science 46 (2001) 1 – 184

5. Lu, L., Zhang, Y. F., Influence of process control agent on interdiffusion between Al and Mg during mechanical alloying, J. Alloys and Compounds 290 (1999) 279 – 283

6. Hwang, S., Nishimura, C., McCormick, P. G., Mechanical milling of magnesium powder, Materials Science and Engineering A318 (2001) 22 – 33

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7. Gittoum, A., Layadi, A., Bourzami, A., Tafat, H., Souami, N., Boutarfaia, S., Lacour, D., X-ray diffraction, microstructure, Mőssbauer and magnetization studies of nanostructured $Fe_{50}Ni_{50}$ alloy prepared by mechanical alloying, J. Magnetism and Magnetic Materials 320 (2008) 1385 – 1392

8. Lu, L., Lai, M. O., Froyen, L., Effects of mechanical milling on the properties of Mg-10.3%Ti and Mg-5%Al-10.3%Ti metal-metal composite, J. Alloys and Compounds 387 (2005) 260 – 264

The Author

Christopher Machio, *CSIR*, Senior Researcher

May 2008 to date: Senior Researcher, CSIR
Feb 2006 – April 2008: Lecturer, School of Chemical and metallurgical Engineering, University of the Witwatersrand
June 2005 – January 2006: Deputy Head, Department of Mechanical and Production Engineering, Moi University, Kenya
1994 – 2001: Lecturer, Department of Mechanical and Production Engineering, Moi University, Kenya