Effect of process control agent (PCA) on the characteristics of mechanically alloyed Ti-Mg powders

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Synopsis

This paper reports the 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%, whereas 6% PCA increases 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.

Keywords
Titanium–magnesium, mechanical alloying, PCA.

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 the potential to produce lightweight structures, with attendant improvements in fuel efficiency in automobiles, for example. A reduction in density is one of two options that can be used to produce lightweight structures. The other option is to use stronger or high modulus of elasticity materials, which make it possible to use thinner sections on components, leading to lightweight structures. However, studies have shown that density reductions 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, which is 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 optimized to ensure a high yield and short downtimes during milling. In this paper, the effect of varying amounts of PCA on yield and alloying of Ti-Mg powders will be investigated.

Methodology

Materials

The powder used in this exercise was Ti – 30 wt.% Mg. The powder mixture was made from elemental titanium powder (~99.5% purity) and elemental magnesium powder (~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, and those of Mg were plate-like in shape (Figure 1). Stearic acid was used as the process control agent, and the amount was varied between 0 and 6 wt.%.

Mechanical alloying

The powder mixtures were milled in a Simoloyer high energy ball mill using a rotation speed of 800 rpm, a ball-to-powder (mass) ratio (BPR) of 20:1 and a milling time of 8 hours. The mass of the Ti – 30 wt.% Mg powder mixture was kept constant at 100 g, while the PCA amounts used were 0, 2, 4 and 6 wt.%. At the end of each milling run, two

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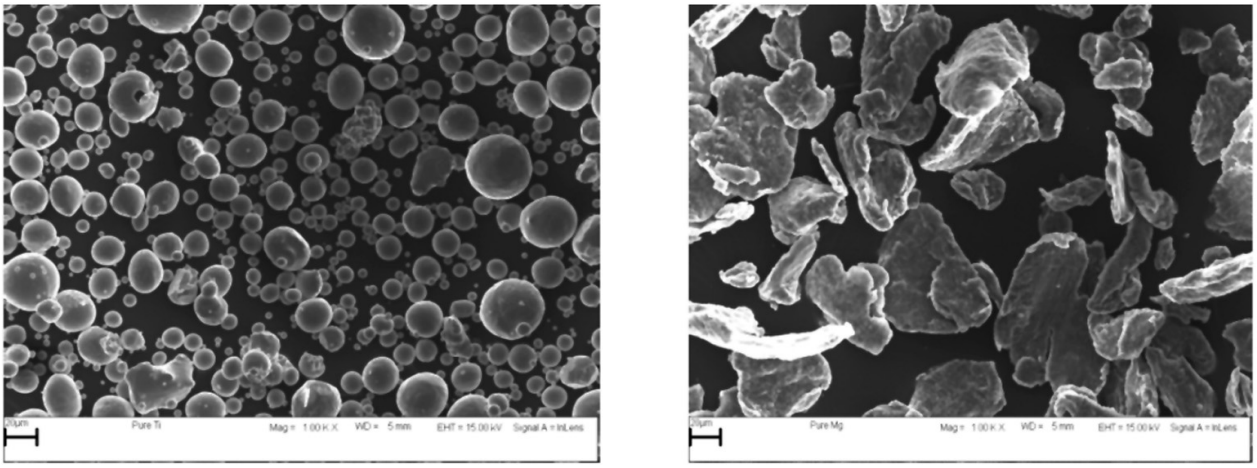

(a) (b)

Figure 1—Morphology of elemental (a) Ti and (b) Mg

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 6 wt.% 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 6 wt.% PCA, respectively.

Powder characterization

The yield powders were weighed and both yield and weld powders were analysed for phases using X-ray diffractometry (XRD) using 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.

Results and discussion

Particle size and particle morphology

The mean powder particle sizes and the particle size distribution for the various PCA contents are shown in Table I and Figure 2, respectively. The introduction of 2 wt.% PCA did not change the mean particle size significantly. However, the particle size distribution changed, with the appearance of a second peak in the normal powder distribution curve at higher sizes (Figure 2). Increasing the PCA content to 4 wt.% decreased the mean particle size by 34%, from 41 to 27 μm. Both the cumulative and normal particle size distributions curves shifted to smaller sizes. The normal particle size

Table I

Mean yield powder particle size for various PCA contents

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

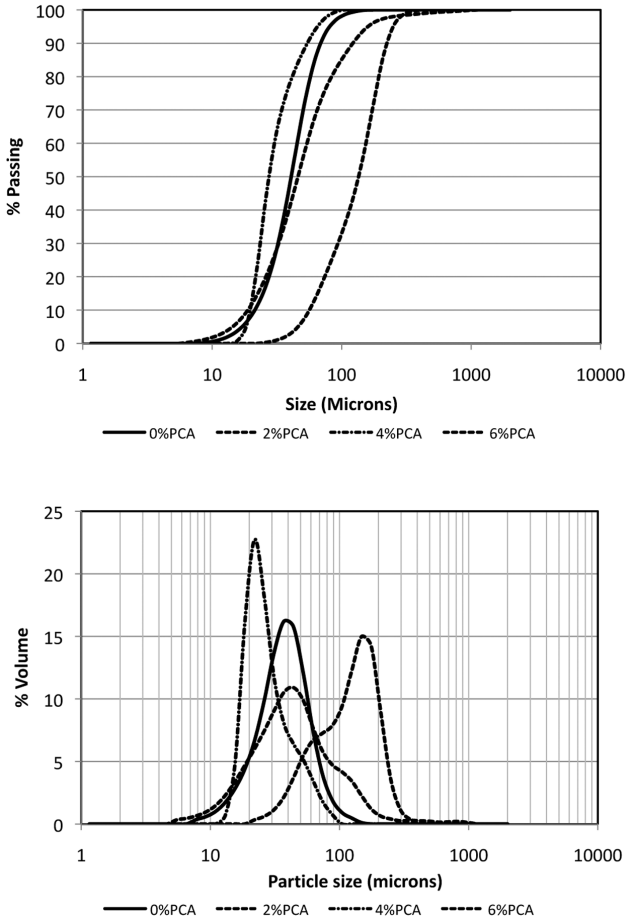

Figure 2—(a) Cumulative particle size distribution of milled powders as a function of PCA content (b) normal powder particle size distributions as a function of PCA content

distribution curve was narrower than for the powder mixture with 0 wt.% PCA. A further increase in the PCA content to 6 wt.% led to a bi-modal particle size distribution and to substantial powder particle coarsening, by up to 230%.

The evolution of particle size with varying PCA content observed here can be explained by the two processes occurring during milling, cold welding and fracture. Powder particles caught between colliding milling media are forced to aggregate, through cold-welding, into larger particles. However, further impacts by milling media lead to deformation and work-hardening, resulting in fracture, that produces finer particles. These finer particles, because of their large surface area, tend to agglomerate, through a process of cold-welding, to produce other larger particles. The interplay of these two processes is moderated by the amount of PCA available during milling; a higher PCA content reduces the tendency to cold weld, leading to the formation of finer powder particles[2]. The observation of a second peak in the powder size distribution curve for the powder with 2 wt.% PCA indicates that there had been a tendency for the powder to cold weld. The refinement of powder size moving from 2 wt.%PCA to 4 wt.%PCA shows that cold welding events significantly reduced and fracture became dominant. Particle size refinement increased with the increase in PCA to 6 wt.%. However, the resulting finer particles cold welded to produce the marked increase in particle size observed here.

The milling led to a change of particle shape from the spherical and plate-like morphology of the elemental powders to irregularly shaped particles (Figure 3). The shapes are typical of ball-milled powders[3].

Powder yield

Table II and Figure 4 show the variation of powder yield as a function of the amount of PCA used during milling. Up to 2 wt% PCA, the amount of yield powder was low (about 10%) and independent of PCA content. However, increasing the PCA content beyond 2 wt.% led to a clear increase in the powder yield. This indicates that up to 2 wt.%PCA, the PCA content was not sufficient to lubricate the powder particles and prevent cold welding. Apart from improving yield, the increase in PCA content also shortened the time between milling runs. This is because powder that welded on the milling chamber walls was progressively easier to remove with increasing PCA content. At 0 and 2 wt.% PCA, the powder on the wall of the milling chamber was much harder to remove, whereas a simple brushing off was sufficient at both 4 and 6 wt.% PCA. The powder milled with 6 wt.% PCA was readily flammable and required careful handling during

Table II

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

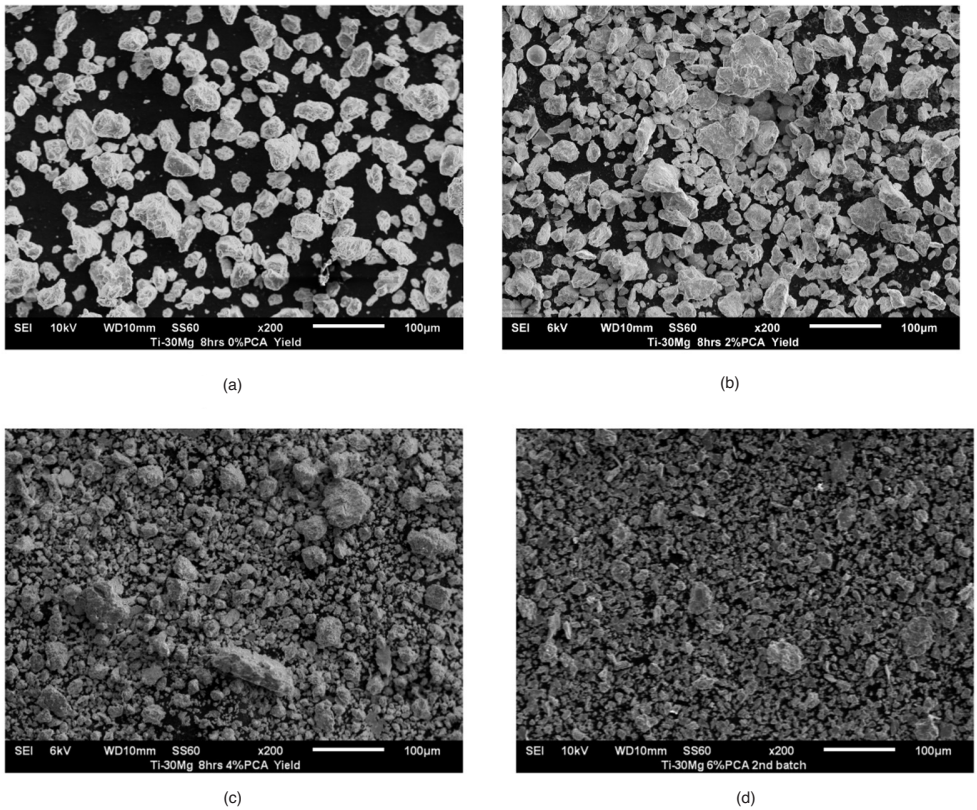

Figure 3—Morphologies of milled powders as a function of PCA content: (a) 0 wt.% PCA, (b) 2 wt.% PCA, (c) 4 wt.% PCA, and (d) 6 wt.% PCA

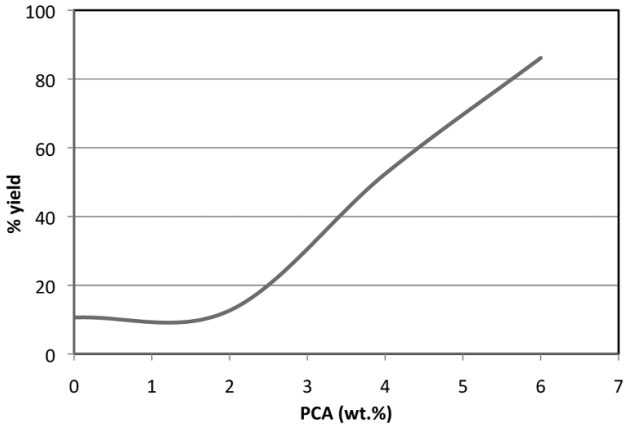

Figure 4—Variation of yield as a function of the amount of PCA

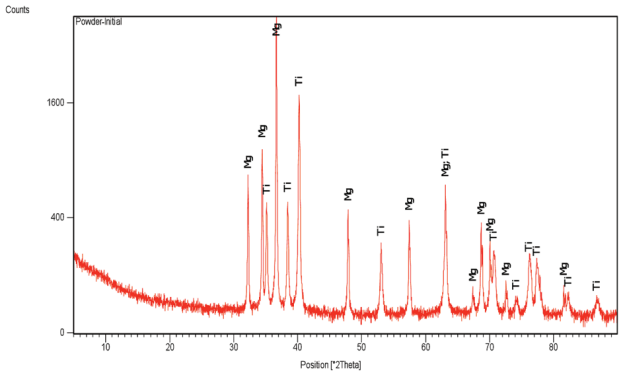

Figure 5—XRD pattern of initial powder showing distinct peaks for Ti and Mg

discharge from the milling chamber. It is possible the fine particles in the powder ignited because of their larger surface area. However, this reasoning does not appear tenable because the finer particles in the 6 wt.% PCA milled powder were larger than the particles of 4 wt.% PCA powder (Figure 2b) but the latter did not spontaneously ignite during discharge. The probable reason for the spontaneous ignition of the 6 wt.% PCA powder is that the powder mixture was still activated at the end of the milling cycle. As already noted, powder particles fracture during milling, an event that exposes oxide-free (clean) fracture surfaces. The particles are activated because they lack the oxide layer that would act as a barrier to oxygen. Even though the particles were covered by the PCA, the protection was not sufficient, leading to the reported spontaneous ignition.

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 comparison purposes. The higher prominence of the Mg peaks in Figure 5 could have been caused by the fact that Ti has a higher absorption of the Cu X-rays.

Figure 6 shows the XRD patterns of powders A1 and A2, the yield and weld powders of 0 wt.% PCA powder, respectively. Mg peaks were missing from the XRD pattern of powder A1 and the Mg and Ti peaks between 2θ = 34 and 39° were 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. Alloying occurs through atomic diffusion across the clean oxide-free surfaces that form after particle fracture. 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: longer milling does lead to the complete dissolution of Mg in Ti[4].

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

The difference of phases between the yield (A1) and weld (A2) powders emanates from, as already noted, the two, apparently opposing and yet complementary steps in

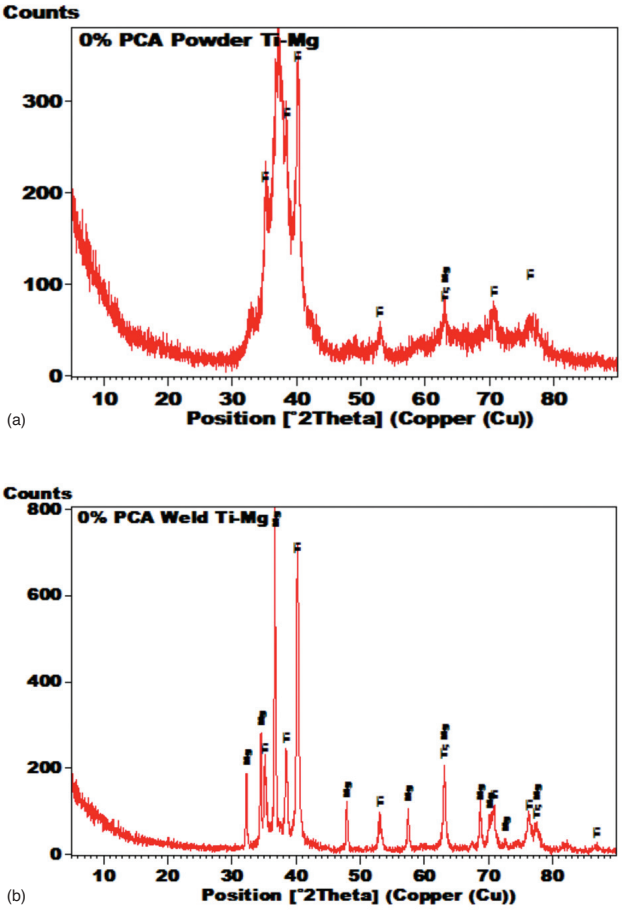

Figure 6—XRD patterns of powder milled with 0 wt.% PCA. Powder A1 is the yield while A2 is the weld. (a) XRD pattern of powder A1 showing that Mg peaks have disappeared, and a broad peak has emerged between 34 and 39° 2Theta. (b) XRD pattern of powder A2 showing the existence of both Mg and Ti. The relative intensity of the Mg peaks is still high

mechanical alloying, cold welding and fracture[2]. As already noted, reactions in mechanical milling occur via cold welding of the powders being milled and subsequent fracturing which 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.

Figure 7 shows a characteristic XRD pattern of yield powders obtained from 2 wt.% PCA powders, showing the presence of Mg peaks alongside Ti peaks. Similar patterns were obtained for the yield powders of 4 and 6 wt.% PCA. The presence of Mg peaks indicates that not all the Mg had dissolved in Ti, as had occurred for the 0 wt.% PCA yield powder (Figure 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 $2\theta = 34°$ (Figure 7) became increasingly more prominent with increasing PCA content, implying that the amount of Mg undissolved in Ti increased with PCA content.

The occurrence of Mg peaks in the 2–6 wt.% PCA yield powders makes them similar to the 0 wt.% PCA weld 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 2–6 wt.% PCA powders were similar to the XRD pattern of the 2 wt.% PCA yield powder (Figure 7). Unlike the 0 wt.% PCA weld 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, and encouraged fracture, that exposed fresh clean surfaces, and rewelding, which facilitated atomic diffusion both in the yield and in the weld powders[2]. Also, Mg powder undergoes a rapid decrease in size compared to the Ti during milling, and smaller particles diffract X-rays less than big particles[6].

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[2].

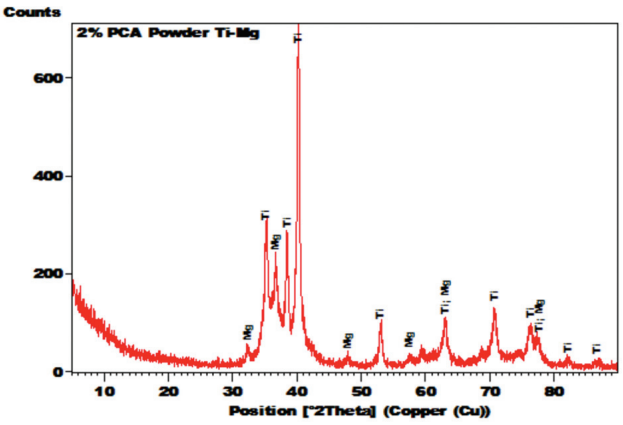

Figure 7—XRD pattern for powder B1 showing both Mg and Ti peaks. Similar patterns obtained from the yield powders of 4 and 6 wt.% PCA powders

Conclusions

This study has shown that the amount of PCA used in milling determines the yield of milled powder: a higher PCA content leads to a higher yield. However, a higher PCA content has also been found to retard the dissolution kinetics and leads 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 appears 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.

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