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Mechanical alloying of titanium

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ABSTRACT

The mechanical alloying of titanium has previously been performed using fugitive process control agents (PCAs). In this work, calcium, magnesium, MgY or CrY are used as process control agents to balance between the cold welding and fracture phenomena, significantly increasing the process yield. The milled powder was then sintered using Spark Plasma Sintering (SPS) at low and high temperatures to achieve the best mechanical properties and a high quality microstructure. To understand the behavior of the PCAs, the same samples were heat treated at high temperatures. The use of a small quantity of calcium, as low as 0.250 wt%, permitted a high process yield, low oxygen content, good powder morphology and thermal stability. Due to the refined microstructure, the samples sintered at 950 °C exhibited mechanical properties similar to commercially pure Grade 4 titanium but with a much lower oxygen content.

1. Introduction

Mechanical alloying is a flexible and powerful process that is used to produce advanced materials while in the solid state. It consists of deforming powder repetitively, continuously and plastically at room temperature to create new alloys and/or microstructures. Mechanical alloying can be performed using a number of different type of mills, such as planetary, ball, tumbler, roller, and drum mills. These are just a few examples and new systems are still in the process of being designed [1–3].

When using a planetary ball mill, the comminution and/or mechanical alloying of the material takes place primarily through the high-energy impact of grinding balls in rotating grinding bowls. The impact of the grinding balls on the charged powder and on the bowl walls causes plastic deformation in the particles and after sufficient time, alloying of the powder mixture.

From a microscopic point of view, during mechanical alloying, each powder particle undergoes two distinct processes: joining, or high-quality cold welding, and fracturing. Cold welding of different powder particles occurs when particles penetrate into themselves upon impact. Fracturing occurs when larger particles break into smaller pieces after continuous impacts. The equilibrium between these two processes provides a stable particle size and allows the balls to incessantly move over the powder without slowing down or freezing the process. Depending on the material, either the cold welding or the fracturing may overwhelm the other process to create too many small particles, or the powder may be completely cold welded on the balls and the bowl walls, thus compromising the process yield.

Many factors can alter the equilibrium between the cold welding and fracturing processes, such as the type of mill used, the nature of the materials, the initial particle size, the processing temperature and the milling energy. To properly setup the powder process, a Process Control Agent, or PCA, is needed. The PCA, which is usually a fatty acid, depresses the cold welding phenomena to restore proper equilibrium. Depending on the powder mixture, a different PCA is required, such as stearic acid, palmitic acid, ethylene bis (stearamide), ethanol, hexane, water and other surfactants [4]. The PCA can be either fugitive or non-fugitive depending on its ability to remain inside the alloys upto the finished product.

Even for cases where a PCA is needed, the PCA can also have negative effects. First, a PCA may introduce new and potentially undesirable elements into the mixture and therefore into the final alloy. Second the PCA must be removed at some point to achieve the final bulk alloy. When mechanically alloying aluminum, a PCA is needed: usually, the PCA is 2 wt% stearic acid to balance the cold welding and fracturing processes. At the end of the process, a portion of the stearic acid has been introduced into the aluminum matrix, and a portion still covers the powder particles. The portion alloyed to the matrix will transform in dispersoids (carbides, oxides and precipitates) during consolidation, sintering and plastic deformation. The other portion has to be removed, usually by hot vacuum degassing to avoid the formation of porosity or blistering during sintering or thermal treatments. Hot vacuum degassing is an expensive process because of technical and safety challenges. Additionally, handling mechanically alloyed powders can be risky. After milling, the surface powder may be very active, and if the particle size is very small, ($d < 10\ \mu m$), the powder may be

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pyrophoric. Therefore, contact with atmospheric oxygen must be avoided through the sintering step.

These issues are particularly problematic for advanced metals such as titanium, which requires strict control of the chemical composition and microstructure. Nevertheless, mechanical alloying of titanium is very interesting because it is possible to obtain new alloys without requiring a melting process, which is expensive due to the high reactivity of titanium [5]. Titanium is highly prone to cold welding, and standard fatty acid-based PCAs cannot be used because they introduce dangerous elements into the mixture, such as oxygen, carbon, hydrogen and nitrogen. For these reasons, it is difficult to find a solution to achieve a high process yield for mechanically alloyed titanium powder while balancing strict chemistry and ease of handling.

A partial solution has been devised by Watwe [6], who recommends using tin as a PCA. Tin is not harmful to titanium and can be introduced at levels of upto 5 wt% before issues arise. Unfortunately, tin does not completely avoid cold welding before the process to create the final alloy is finished, resulting in a low process yield.

It has been noted that magnesium, calcium and all the rare earth metals have a small solubility limit in titanium [7]. These data inspired the author to use these materials as possible PCAs for the mechanical alloying of titanium because the insolubility of these elements retards the cold welding phenomenon. Moreover, all these elements have a stronger affinity for oxygen than for titanium, providing a possible scavenging effect and protecting or limiting the presence of oxygen in the solid solution of the alloy [8–14]. Rare earth elements or their compounds not only have strong chemical affinity for oxygen but also have strong chemical affinity for chlorine, which is highly detrimental but unavoidable in most Ti powders [15–17]. Finally, recently a patent was filled in regarding the use of calcium for the production of titanium powder with low oxygen concentration [18],

This paper reports on the results of testing the mechanical alloying of pure titanium powder with different PCAs, such as pure magnesium, pure calcium, MgY and CrY master alloys. Different PCA quantities were used to provide high process yields, uniform microstructures and proper particle sizes. The milled powder was then sintered using Spark Plasma Sintering to create bulk samples for microstructural analysis and mechanical testing.

2. Experimental procedure

2.1. Mechanical alloying

The initial material used in this study was titanium powder with an average particle size of $<150\,\mu m$ (Toho Titanium Co., TS150). Magnesium powder (Alfa Aesar, -20+100 mesh, 99.8%), calcium powder (Alfa Aesar, -16 mesh, 99.5%), turning chips of MgY (50 wt% Mg–50 wt% Y, China Rare Metal Material Co.) and CrY (50 wt% Cr–50 wt% Y, China Rare Metal Material Co.) were used as the PCAs. The amount of PCA varied from 0.125 wt% to 0.500 wt% for the first three compositions, whereas PCA for the CrY master alloy varied between 1 wt% and 4 wt%.

Milling was performed with a Fritsch Pulverisette 6 using a jar with a volume of 500 ml and ∅10 mm hardened and tempered steel balls. The milling speed was set to 450 rpm with a ball-to-powder ratio of 10. To ensure that the powder temperature was less than 100 °C, after every 2 min milling, a 4 min pause was taken. The maximum number of cycles per powder batch was 60, and the mill was stopped after every 10 cycles to check the yield of the process. Milling tests were performed in an argon atmosphere or in a vacuum. A proper sealing plug was used to maintain the pressure for the vacuum at a few Pascals.

After ball milling, the powders cooled to room temperature in the jar in 2 h; the jar was always opened in air and the powders were simply stored for days in bottles with air.

The process batch yield was defined as the ratio of the weight of the recovered powder with a particle size less than 180 μm after the predetermined number of cycles and the weight of the starting powder. The oxygen and nitrogen content was measured at the start of the process and after 40 milling cycles using a LECO TC400 instrument.

Mechanically alloyed powder with a prescribed PCA quantity was extracted after 40 cycles and mounted in epoxy resin in vacuum, polished and analyzed with an optical microscopy. Etching was performed using either Kroll's or Weck's reagent.

2.2. Sintering, heat treatments and mechanical testing

The powders obtained with the most interesting features were sintered using Spark Plasma Sintering (Sumitomo Coal & Mining, Dr. Sinter® 1050). Three different sintering procedures were adopted for this work: maintaining a constant heating rate of 100 °C/min and then implementing a hold time of 1 min at a specified sintering temperature, which was set to 900 °C, 950 °C and 1250 °C. The temperature was recorded by a pyrometer placed in a hole drilled into the graphite dies. The load was applied after the temperature reached 800 °C to achieve a pressure of 30 MPa until the completion of the sintering process.

Two sample shapes were obtained for the sintering process: cylindrical samples with a diameter 20 mm and a thickness of 5 mm and dog-bone-shaped samples with a gage length of 20 mm, total length of 60 mm and a thickness of 5 mm.

Heat treatments were performed in a metallic furnace with a high vacuum (10^{-3} Pa) at 1300 °C for 1 h and then furnace cooled to evaluate the microstructure stability. Three-point bending tests were performed according to the ISO 3327 standard. The specimens for this test were machined from the cylindrical-shaped samples. The tensile tests were performed using a universal testing machine (MTS, Criterion C45) using the dog-bone specimens at a crosshead displacement rate of 0.5 mm/min.

3. Results

3.1. Process yield, microstructure and oxygen content

The first important dataset from the mechanical alloying tests is the process yield results. Fig. 1 shows the process yield characteristics from samples that were created using calcium as the PCA in an argon atmosphere. The milling was stopped every 10 cycles and the resulting powder was sieved to a particle size of $<180\,\mu m$. The data clearly show that the process yield is strongly dependent on the quantity of the PCA used and the milling time. However, even with a small amount of calcium, for example 0.25 wt%, and a milling time of 120 min, a 72% powder yield was achieved.

Fig. 2 shows data obtained under the same milling conditions but using a vacuum instead of an argon atmosphere. The presence of a vacuum appears to have advanced the cold welding and perhaps the degree of mechanical alloying as well. The same process yield was generated in a shorter milling time, between 20 and 40 min less than when using an argon atmosphere. However, the yield parameter is not the only indicator of a properly mechanically alloyed powder. An examination of the microstructure using light microscopy is usually performed, and the results are shown in Figs. 3 and 4. Fig. 3 shows the microstructure of the powder milled in an argon atmosphere for 80 min and Fig. 4 shows the microstructure of the powder milled in a vacuum for 80 min. The first picture shows a white zone, meaning that an

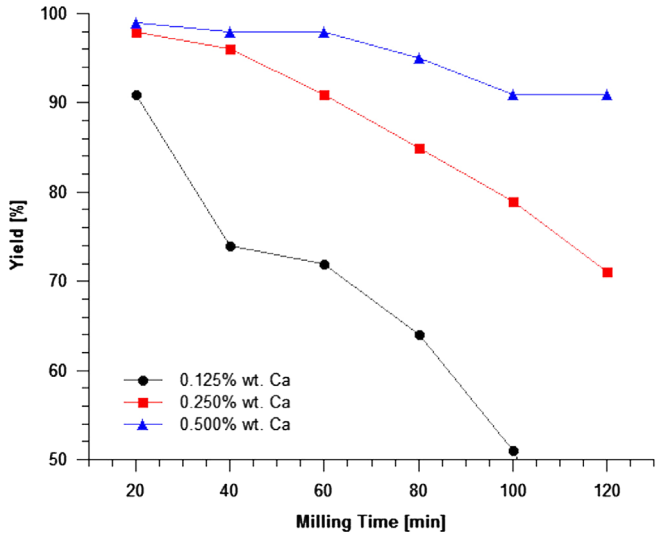

Fig. 1. Process yield using calcium as the PCA and argon as the milling atmosphere.

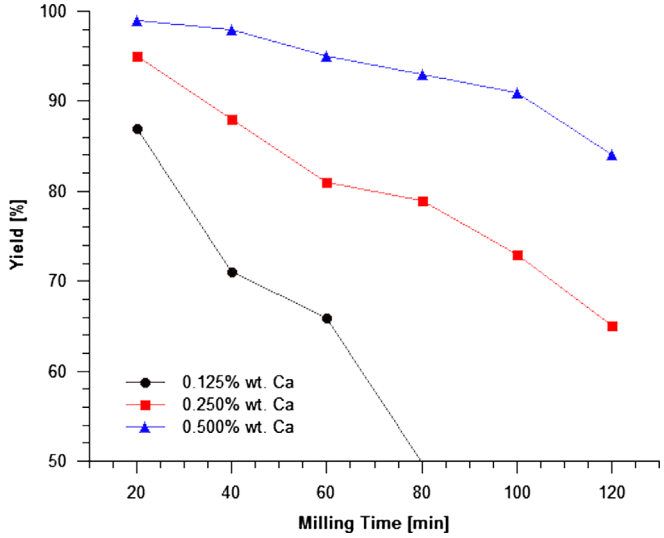

Fig. 2. Process yield using calcium as the PCA and a vacuum as the milling atmosphere.

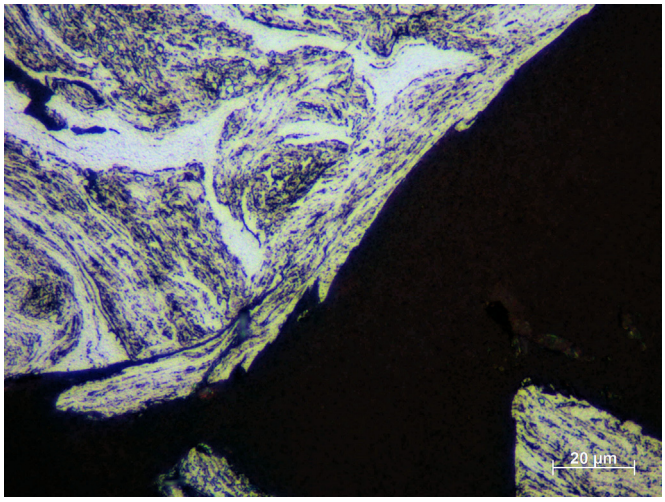

Fig. 3. Microstructure of the powder milled with 0.250 wt% calcium for 80 min in an argon atmosphere and etched with Kroll's reagent.

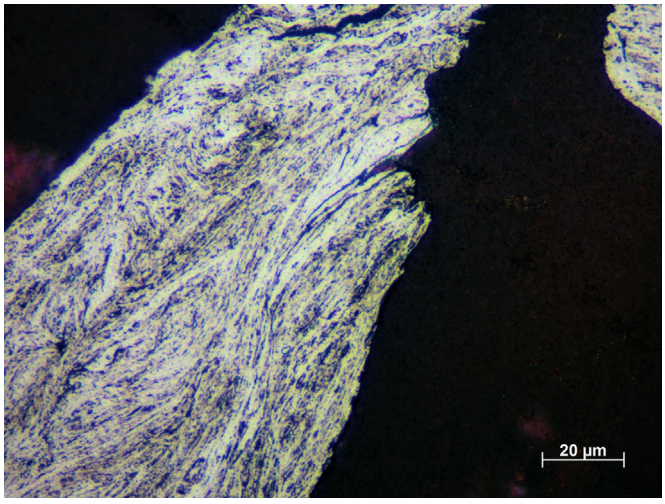

Fig. 4. Microstructure of the powder milled with 0.250 wt% calcium for 80 min in a vacuum and etched with Kroll's reagent.

incomplete milling/alloying process occurred. The second picture shows a uniform and sound microstructure showing that a better milling/alloying process occurred. This shows that the vacuum condition is better than the argon atmosphere at producing a high quality mechanically alloyed material. Moreover, for the vacuum condition, evidence of stronger cold welding was observed on the jar walls and ball surfaces after milling.

Based on this result, all the subsequent mechanical alloying experiments were conducted using a vacuum. This was also done to ensure that air did not contaminate the powder during milling because at the end of milling process, any air flow into the jar can be recognized.

Figs. 5 and 6 show the process yields using magnesium and the master alloy MgY as the PCAs. These trends are similar to those described in Fig. 2 but with lower values. Therefore, magnesium and the MgY master alloy were found to minimize the cold welding but did not produce process yields as high as those achieved by the calcium PCA.

The results for the CrY master alloy were substantially different. Fig. 7 shows that a much greater amount of PCA had to be used to avoid a completely adherent and tough coating of

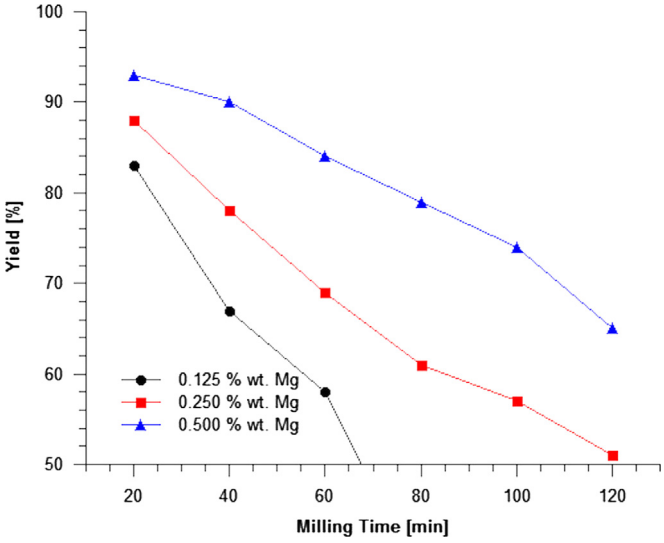

Fig. 5. Process yield using magnesium as the PCA.

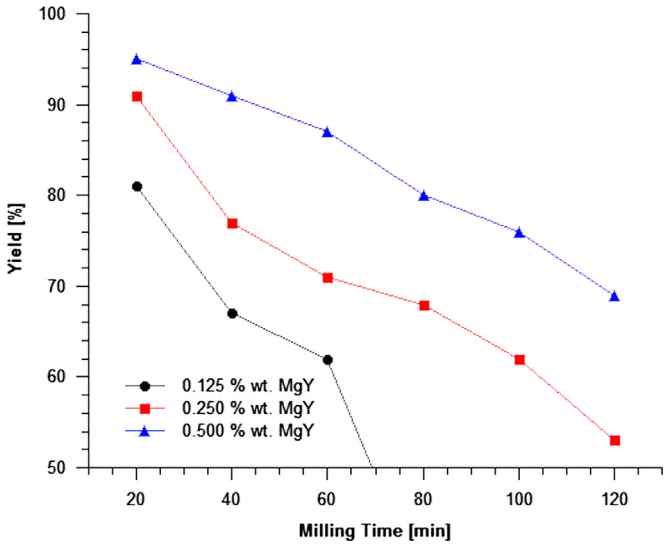

Fig. 6. Process yield using the MgY master alloy as the PCA.

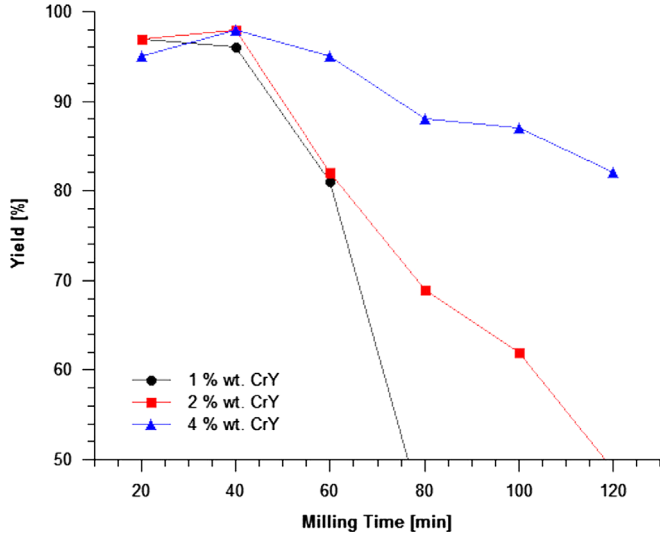

Fig. 7. Process yield using the CrY master alloy as the PCA.

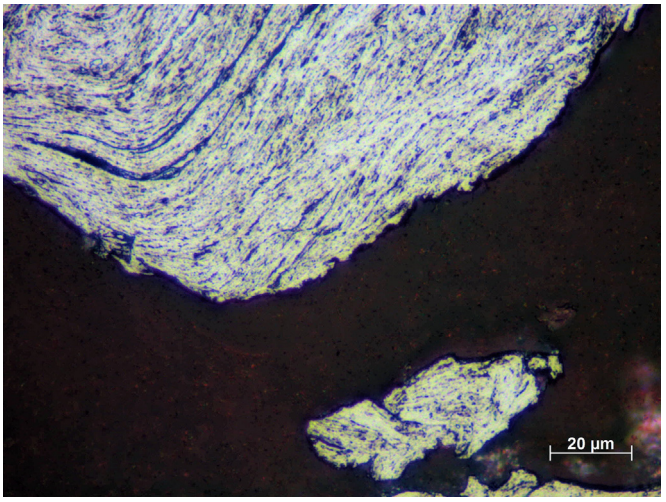

Fig. 8. Microstructure of the powder created using 0.250 wt% magnesium, milled for 80 min in a vacuum and etched with Kroll's reagent.

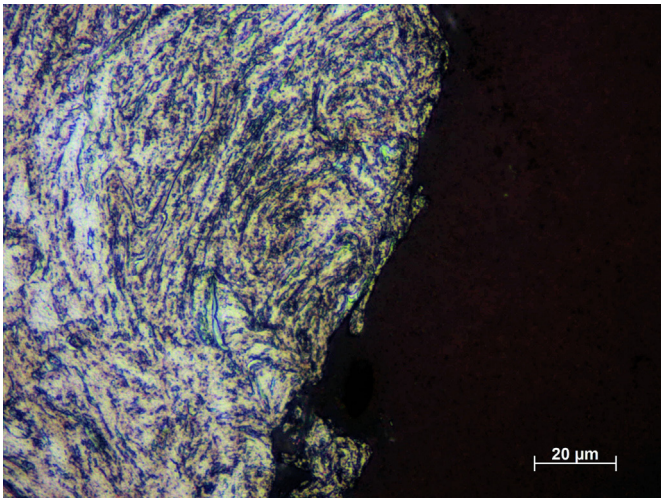

Fig. 9. Microstructure of the powder created using 0.250 wt% MgY master alloy, milled for 80 min in a vacuum and etched with Kroll's reagent.

titanium being welded onto the balls and jar walls. A quantity of approximately 4 wt% is needed to allow the mechanical alloying process to persist for at least 80 min.

To better compare the results from different mills, the powders obtained with a fixed PCA quantity of 0.250 wt% calcium, magnesium and MgY, 4 wt% CrY, a milling time of 80 min and 40 cycles were analyzed. This choice is based on metallurgic and economic points of view; the least possible amount of PCA is better so that a clean alloy comparable with commercially wrought titanium can be created with the highest possible yield in the shortest milling time.

Figs. 8–10 show the microstructures of the previously described mechanically alloyed powders and are compared with those shown in Fig. 4, which represent the alloys created with 0.250 wt% calcium. Additionally, independent of the different PCA used, all of the powder particles have the same characteristic onion-like shape due to the folding, cold welding and fracturing that occurs during mechanical alloying.

Figs. 11 and 12 show the surface of the powder particles milled with a 0.250 wt% calcium PCA for 80 min in vacuum. Some particles shape are round and likely have a high tap density,

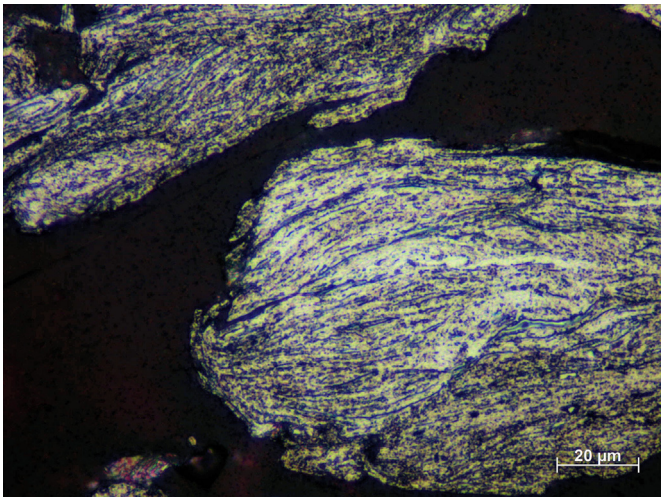

Fig. 10. Microstructure of the powder created using 4 wt% CrY master alloy, milled for 80 min in a vacuum and etched with Kroll's reagent.

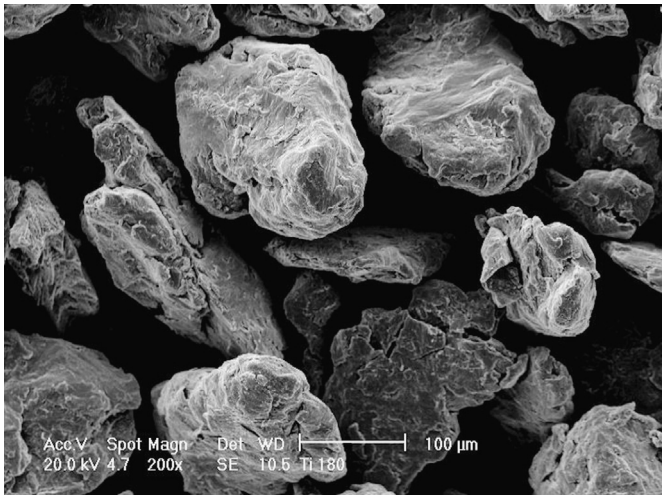

Fig. 11. Surface of the mechanically alloyed powder created using 0.250 wt% calcium as the PCA.

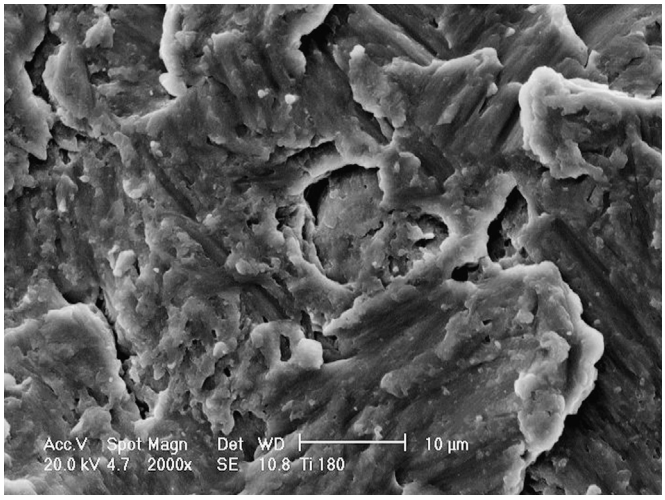

Fig. 12. Detailed surface of the mechanically alloyed powder created using 0.250 wt% calcium as the PCA.

which is a desirable feature. At high magnification, a type of river pattern is recognized as a result of the onion-like shape.

Table 1 presents the oxygen and nitrogen content of the powder before and after the completion of the mechanical alloying process. The oxygen and nitrogen content increase varies between ~0.05 wt% and ~0.01 wt% depending on the PCA used.

3.2. Sintering, heat treatments and mechanical testing

The four powders milled for 80 min in a vacuum with different PCAs were sintered at 1250 °C for 1 min. Figs. 13 and 14 show the behavior of the powder created using 0.25 wt% calcium as the PCA because there were no differences observed during sintering. Fig. 13 shows the process in terms of temperature, load and vacuum vs. time. For the first 6 min the temperature is not recorded because the pyrometer is used and so a fixed value of 570 °C is recorded.

There is no outgassing observed from the powder in a vacuum at temperatures upto 700 °C. At temperatures higher than this during SPS, vapor molecules release from the chamber wall. The two small pressure increases observed at 2 min and 5 min are

Table 1
oxygen and nitrogen content for initial and milled powder.

	Oxygen [wt%]	Nitrogen [wt%]
Ti powder TS150	0.18	0.018
Ti (0.250 wt% Ca)	0.23	0.022
Ti (0.250 wt% Mg)	0.24	0.031
Ti (0.250 wt% MgY)	0.23	0.029
Ti (4 wt% CrY)	0.22	0.023

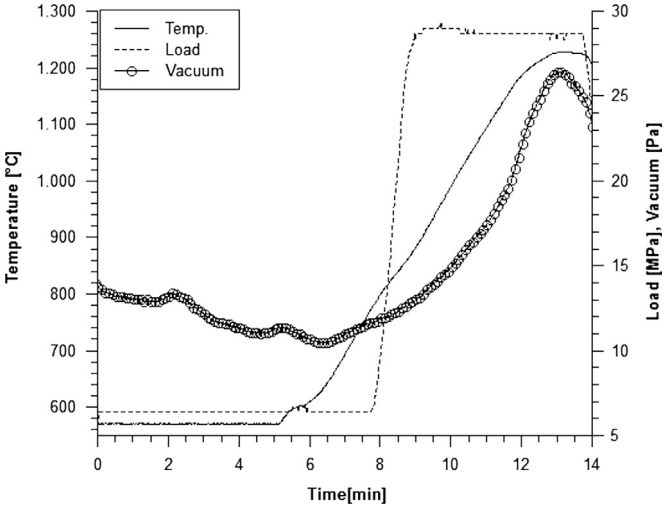

Fig. 13. Applied temperature, load and vacuum during the spark plasma sintering.

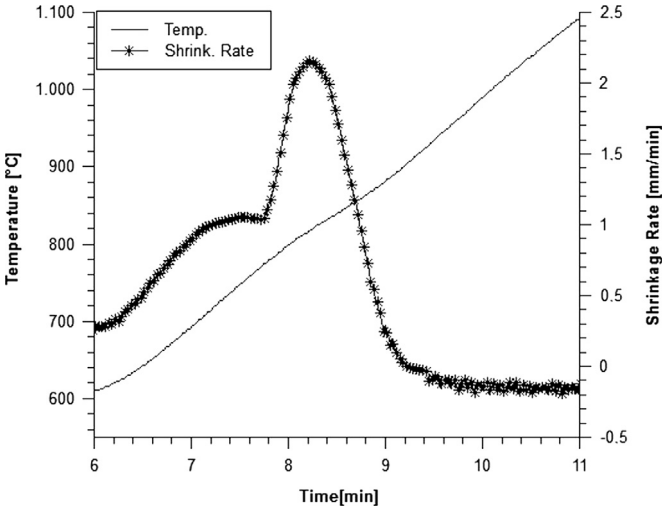

Fig. 14. Shrinkage rate during the spark plasma sintering.

meaningless because the values are too low. If a volatile PCA were present, a much higher increase in the pressure would have been recorded.

Fig. 14 shows the shrinkage rate with respect to time at 6 and 11 min. A positive derivative means that as the lower ram moves up, the powder placed into the die is compacted and/or sintered. After 6 min and at temperatures of approximately 600 °C, the powder in the die begins to contract. When the load is applied at 8 min, the derivative suddenly increases. This implies an increase in the densification rate. After 9 min and at temperatures less than 900 °C, the derivative returns to zero, meaning that there is no more shrinkage in the samples.

Fig. 15 shows the microstructure of the sintered mechanically alloyed powder created using a 0.25 wt% calcium PCA. The microstructure is uniform with a grain size of approximately 7 μm and with very small precipitates present in the matrix. The four sintered samples each respond very differently to the etchant and it is therefore difficult to directly compare them. It can, however, be seen that the sample obtained from the mechanically alloyed powder created using a 4 wt% CrY PCA shows a high quantity of precipitate with a particle size of approximately 1 μm, as shown in Fig. 16.

Four other samples were sintered at 1250 °C and heat treated at 1300 °C for 1 h to evaluate the microstructure stability. After cooling, the samples created using magnesium and MgY as the PCAs exhibited poor quality surfaces containing bubbles and pores.

Fig. 17 shows a microstructure with the same composition and created with the same milling condition as the sample in Fig. 15 after the heat treatment. Larger grains are observed, with an average size of 13 μm, but there is not a significant explosion of the grains. Fig. 18 shows the microstructure of the heat treated sample created using a 0.25 wt% magnesium PCA. It is clear that part of the magnesium precipitates out of the matrix, causing blistering to occur inside the sample. Therefore, using magnesium or magnesium master alloys as PCAs is dangerous because not all of the magnesium will remain in the matrix, yielding a final material with unstable behavior.

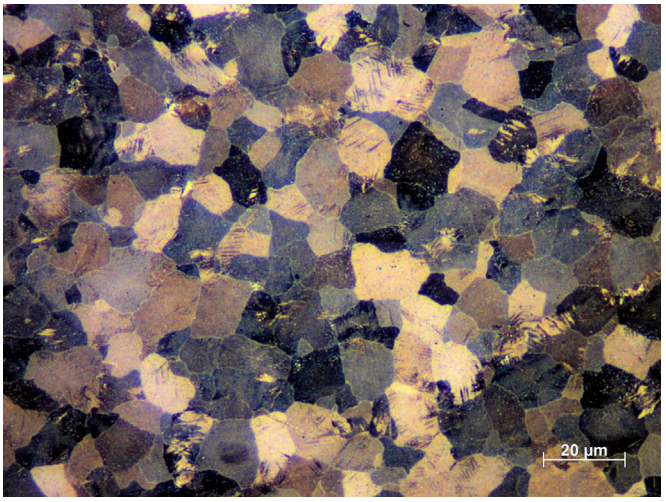

Fig. 17. Microstructure of the sintered and heat treated sample from the mechanical alloyed powder created using 0.25 wt% calcium as the PCA and etched with Weck's reagent.

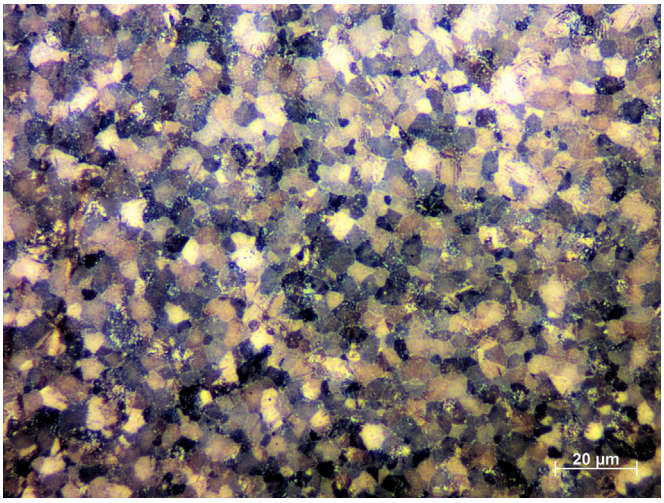

Fig. 15. Microstructure of the sintered sample from the mechanical alloyed powder created using 0.25 wt% calcium as the PCA and etched with Weck's reagent.

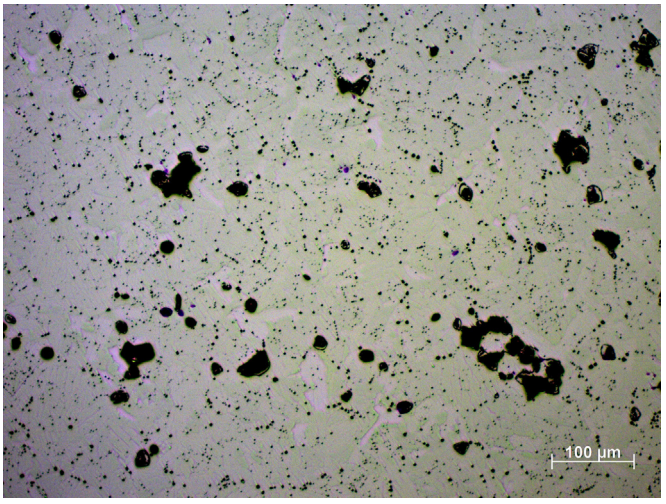

Fig. 18. Pores in the sintered and heat treated sample of the mechanical alloyed powder created using 0.25 wt% magnesium as the PCA.

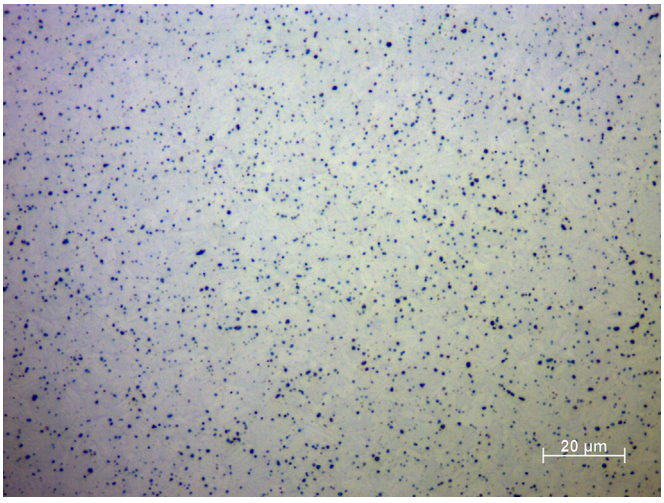

Fig. 16. Slightly etched microstructure of the sintered sample from the mechanical alloyed powder created using 4 wt% CrY as the PCA.

Figs. 19 and 20 show the results of the three point bending tests. Fig. 19 shows the results for the mechanical alloyed powder sintered at 1250 °C, and Fig. 20 shows the results for the mechanically alloyed powder sintered at 1250 °C and then heat treated at 1300 °C for 1 h. The best results are observed with the sample created using a 0.25 wt% calcium PCA. In the as-sintered condition this sample can withstand a plastic deformation of upto 37% without fracturing. For the powder mechanically alloyed with magnesium or the MgY master alloy, the results are unsatisfactory. These samples exhibit brittle fracture characteristics, denoting a poor material sinterability. The milled powder created using a 4 wt % CrY master alloy PCA behaves in the middle of these two extremes.

A similar trend is noted when the heat-treated samples are examined (see Fig. 20). Only the sample created using a 0.25 wt% calcium PCA exhibits positive deformation characteristics and fractures at approximately 36% plastic deformation.

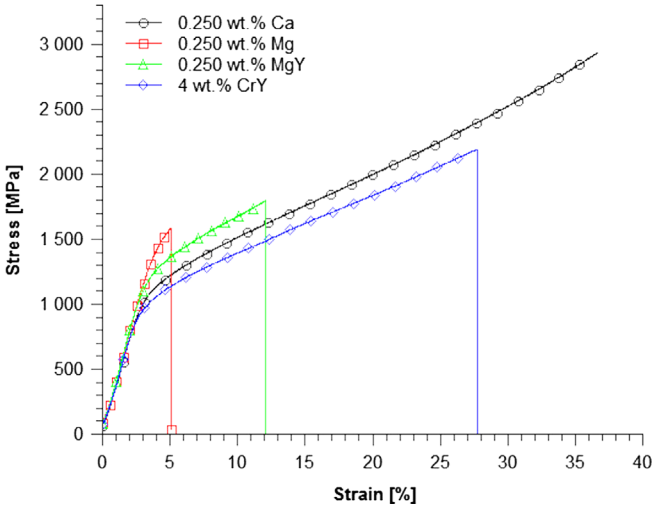

Fig. 19. Three-point bending stress vs. strain for the samples sintered at 1250 °C for 1 min.

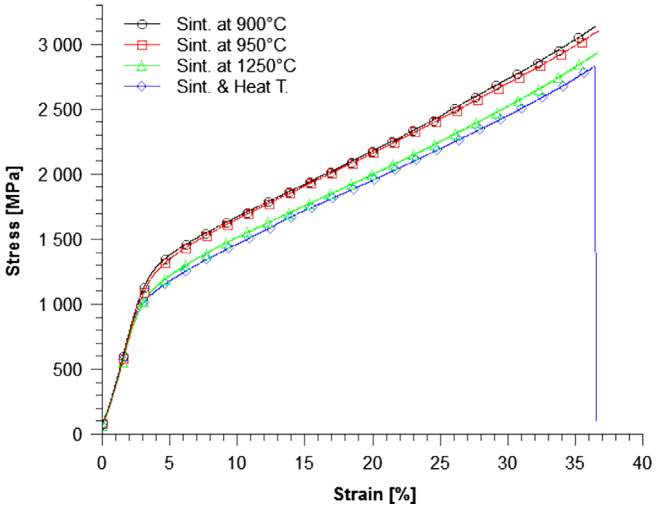

Fig. 21. Three point bending stress vs. strain for the mechanically alloyed powder created using a 0.250 wt% Ca PSA and sintered and thermally treated.

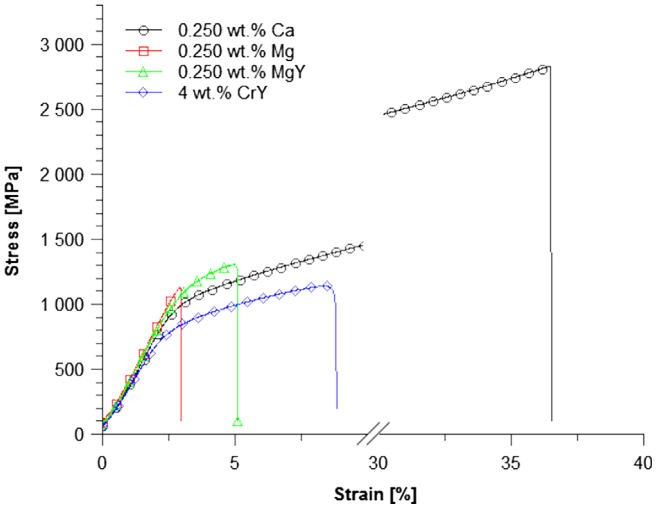

Fig. 20. Three-point bending stress vs. strain for the samples sintered at 1250 °C for 1 min and heat treated at 1300 °C for 1 h.

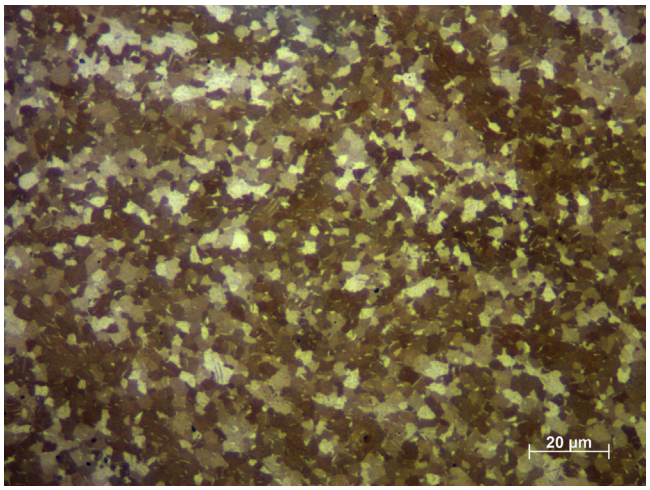

Fig. 22. Microstructure of the mechanical alloyed powder created using a 0.25 wt% calcium PCA sintered at 950 °C for 1 min and then etched with Weck's reagent.

The most interesting powder is the one created using a 0.25 wt % of calcium PCA. Samples of this powder were sintered at 900 °C and 950 °C based on the data from Fig. 14 in order to fabricate full density samples using 30 MPa of pressure.

Fig. 21 presents the three point bending results for all the samples sintered at 900 °C, 950 °C, 1250 °C and 1250 °C plus heat treatment for the powder Ti–0.250 wt% Ca. All of the as-sintered samples deform plastically with greater than 37%, whereas the sample that was heat treated at 1300 °C for 1 h fractured at 36%. There is also an increase in the mechanical properties as the sintering temperature is reduced from 1250 °C to 950 °C and again when the sintering temperature was reduced to 900 °C.

Fig. 22 shows the microstructure of the mechanically alloyed powder created using a 0.25 wt% calcium PCA that was sintered at 950 °C for 1 min. The average measured grain size was 5 µm for this sample.

The Ti–0.250 wt% Ca mechanically alloyed powder was sintered at 950 °C for 1 min in a dog-bone shape. Fig. 23 shows the tensile test results including yield strength (YS)=627 MPa, ultimate tensile strength (UTS)=722 MPa and an Ef=17%. The fractography shown in Fig. 24 highlights the presence of dimples and very small precipitates in the microstructure.

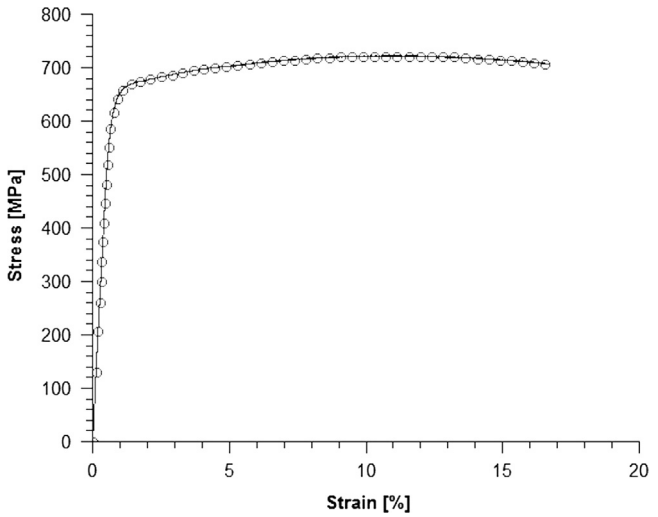

Fig. 23. Tensile stress vs. strain for mechanically alloyed powder Ti–0.250 wt% Ca sintered at 950 °C for 1 min.

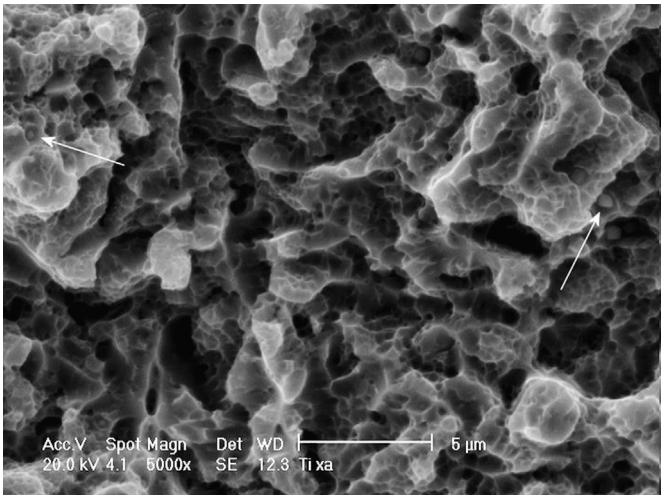

Fig. 24. Fractography of the Ti–0.250 wt% Ca tensile sample sintered at 950 °C for 1 min.

4. Discussion

The milling results clearly show that calcium, magnesium, MgY and CrY are very effective PCAs for mechanical alloying of titanium. It should be stressed that without these PCAs, the cold welding phenomenon is so strong that just a few milling cycles are enough to completely smear the titanium on all the impacting surfaces.

As previously mentioned, the use of olefinic PCAs has been avoided to reduce the possibility of introducing potentially dangerous interstitial elements such as oxygen, carbon, nitrogen and hydrogen into the mixture. Hydrogen can be easily removed during vacuum annealing, and a few authors have previously adopted this trick to mechanically alloy Ti [19,20]. Hydrogen is highly soluble in titanium and causes the formation of a brittle powder, which can be easily milled to a very fine size without causing cold welding to occur. However, the use of hydrogen or better PCAs based on carbon, silicon or oxygen gives rise to other problems. Foremost amongst these is that the resulting powder is highly pyrophoric, which is unacceptable from an industrial point of view.

In this work, the mechanical alloying process performed independently of the choice of PCA produced very little powder below a size of 25 μm. Additionally, the particles have a clean and smooth surface, as shown in Fig. 11. This implies two important things: the particles have a high tap density and possess a low risk for operators to handle. A smooth and medium sized powder is also exposed to a small amount of oxygen from the atmosphere when the milling jar is opened. An increase of ~0.05 wt% of oxygen after milling is due to the oxidation of the new and fresh surfaces produced during mechanical alloying process [21].

Nevertheless, the powder is fully alloyed with just a small amount of PCA.

This is not completely true for the CrY master alloy. At least 4 wt% is needed to create a fully alloyed powder. However, chromium and yttrium have a significantly higher density than do calcium or magnesium. Therefore, if a comparison is performed in term of at%, there is not much difference between the materials. Moreover, CrY is very tough and strong, and the lower efficiency that it exhibits is likely due to its low ability to break up and disperse during the first stage of milling. A brittle (i.e., MgY) or tough but soft (i.e., Ca and Mg) material experiences fewer problems.

The use of calcium, magnesium, MgY or CrY as the PCA also has the positive effect of ensuring a fast and therefore cheap process to create the alloyed powder. A time of 80 min is sufficient to obtain a high-quality microstructure, implying that the milling conditions examined here are highly efficient.

All the powders were sintered using SPS at a pressure of 30 MPa. The data show that a temperature of approximately 900 °C is needed to ensure full density samples.

For samples sintered at 1250 °C, the resulting grain sizes were as low as 13 μm. Comparable data obtained under similar conditions with atomized or HDH powder exhibited much larger microstructural dimensions [22], showing that the mechanical alloying process and the following sintering process introduce small precipitates, possibly CaO, which pin the grain boundaries in place. Further analysis is needed to examine the microstructure from a nanoscale point of view.

The three-point bending test and the microstructures before and after the heat treatment show that calcium and CrY are the only PCAs that produce positive results. Magnesium or magnesium-based master alloys may function well as PCAs but should be avoided because they tend to melt or sublimate when heated. This was visually evident after the SPS because the dies used for producing the magnesium based samples were slightly coated with a shiny metal at certain points.

Calcium and magnesium act similarly as PCAs but behave differently because of their physical properties. For example, calcium has a higher melting point, boiling point, vapor pressure and a lower solubility in titanium than does magnesium. These properties are important during sintering, and because calcium is generally more stable with respect to temperature, it does not precipitate out of the matrix in the form of an oxide or as a solid solution. The instability of magnesium is responsible of the blistering and formation of pores observed in the microstructures.

It is interesting to compare the microstructure of the samples sintered at 950 °C and 1250 °C with those heat treated at 1300 °C for 1 h. The grain size remains very small, almost independent of the applied heat. This fact is important because a high creep resistance is expected.

The small grain size and the very small particles are responsible for the mechanical properties depicted in Fig. 23. From these data, it appears that this mechanically alloyed titanium has mechanical properties similar to those of commercially pure Grade 4 Ti but with a much lower oxygen content that is comparable to commercially pure Grade 2 Ti.

5. Summary

In this work, the effect of using calcium, magnesium, MgY or CrY as process control agents for mechanically alloying titanium was studied. Maximizing the process yield and minimizing the process time were treated as the most important goals and obtaining a sound microstructure and ensuring ease of handling were secondary objectives. The addition of 0.25 wt% calcium proved to be the best solution in terms of maximizing the powder yield and ensuring high quality microstructural stability and mechanical properties.

Consolidation was performed by means of spark plasma sintering. Full density samples with good mechanical properties were obtained at a sintering temperature of 900 °C. Due to a small grain size and the presence of small oxides, a yield stress of upto 627 MPa and an elongation of 17% was obtained for a titanium alloys with a low oxygen content of 0.25 wt%.

The use of calcium as a PCA for mechanically alloying titanium allows the production of virtually any type of titanium alloy for a low price without requiring expensive melting and hot plastic deformation.

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