PRODUCTION OF SPHERICAL TITANIUM BASED POWDERS FROM POWDER METALLURGY BARS

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ABSTRACT

This article describes the results of preliminary investigations into the production of spherical titanium-based powders via powder metallurgy routes. The investigations seek to support the localization initiatives through the manufacture of high-quality low-cost spherical powders. Feedstocks for atomization (in billet and bar forms) were prepared using low capital-intensive equipment and atomized via the vacuum induction melting (VIM) or electrode induction gas atomization (EIGA) systems. The atomized powders were characterized for both properties and application using the Laser Engineered Net Shaping (LENS) Additive Manufacturing (AM) system. The investigated feedstock production routes were both found to be feasible and cost effective. The produced powders were spherical and suitable for application in the LENS AM system.

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1. INTRODUCTION

The Republic of South Africa is pursuing a strategy to beneficiate and add value to its bodies of ores [1]. One such body of ores is for titanium consisting of ilmenite and rutile, of which South Africa has the world's fourth largest deposits (behind Australia, China and India, in that order). Research efforts led by the Department of Science and Technology through the Titanium Centre of Competence (TiCoC), are currently underway to develop a suite of complementary technologies to help South Africa add value to its vast body of titanium ores. Key to these efforts is the development and commercialisation of a novel process for converting the titanium ores to titanium metal granules/powders, at low cost, and to further convert the metal powders to intermediate and finished products. Alongside the beneficiation strategy, the additive manufacturing (AM) strategy was recently commissioned [2]. The latter seeks to position and support AM as the next chapter in the industrial revolution [2]. Together these two strategies are complementary in that, the titanium from the beneficiation strategy is to be used as a raw material in the AM strategy.

AM has developed swiftly in the last decade. It has demonstrated significant potential and gained popularity; the reader is therefore referred elsewhere for discussions on its fundamentals and merits [2,3,4]. It is a generally accepted opinion that AM is a disruptive technology with the potential to significantly improve competitiveness and boost productivity across industry sectors. While the manufacture of Ti-based components by AM is currently well established [5], its uptake has been uncharacteristically underwhelming, caused by among other, things expensive feedstock powder materials [6,7]. In the case of South Africa, AM users bear exorbitantly high transport and import costs of the titanium-based feedstock powder due to the fact that such materials are considered hazardous under the OSHA Standard (in addition to the already high international commodity prices). There is a case therefore for localization of production of these AM powders.

Commercial Ti-based AM feedstock powder is currently produced via a number of processes, including atomization and spheroidization techniques. Atomization techniques, such as electrode inert gas atomization (EIGA), plasma rotating electrode process (PREP), plasma atomization (PA) [7] and lately vacuum induction melting (VIM) [8] have gained prominence. Currently, the most used methods of making AM Ti-based feedstock spherical powder start with Ti-based feedstock bars/billets or wires made by the ingot metallurgy technique of vacuum arc re-melting (VAR) [9] adding enormously on the cost of the powders. The cost of the feedstock bars can be reduced if they can be manufactured using powder metallurgy (PM), it is not surprising that methods such as plasma spheroidization have demonstrated cost-effective alternatives [10]. In this study, the feasibility of producing Ti6Al4V feedstocks for atomization (in the form of bars/billet) via powder metallurgy techniques was investigated. Powder metallurgy bars/billets were produced and atomized using the EIGA and VIM systems. Subsequently, the atomized powders have been characterised and components built using a LENS system.

2. METHODOLOGY AND RESULTS

2.1 Methodology

The starting titanium materials used in this preliminary study were the -325 mesh Ti HDH powder and the-100 mesh Ti HDH powder both supplied by Baoji Lihua Non-Ferrous Metals Co., Ltd of PR China. Two Ti6Al4V bars/billets used in this study were manufactured via blended elemental powder metallurgy starting with -325 and -100 mesh Ti HDH and a -325 mesh 60Al40V master alloy supplied by Reading Alloys, USA. The starting powders were weighed in the right proportion to make Ti6Al4V alloys by weight percent. The oxygen content of the powders was determined by an Eltra ONH 2000 gas analyser. The elemental powders were blended in a Jones double cone mixer for 30 minutes. Two sets of bars/billets, one from 100mesh TiHDH/325mesh 60Al40V (long bar) and another from 325mesh TiHDH/325mesh 60Al40V (short bar) were made via powder metallurgy. Long bars/billets were made by loose sintering, while the short bars/billets were made by the conventional press and sinter, where blended powders were introduced in a mould of diameter 72mm and pressed to a green density of 49% theoretical density (TD). Both the short and long bars were sintered at 1100°C for 4hrs in a furnace with controlled environment. The sintered long billets were atomized using EIGA, by a well-known atomization shop, TLS, Germany. The sintered short billets were atomized using VIM-gas atomization. The VIM process involved melting the billets under a vacuum of 9×10^{-3}mBar, followed by inert gas atomization of melt freely falling through an orifice.

For the purposes of comparison and benchmarking the processes, a commercial Ti6Al4V billet (meeting Ti Grade 5 AMS 4928R composition specifications) was also atomized via the EIGA system.

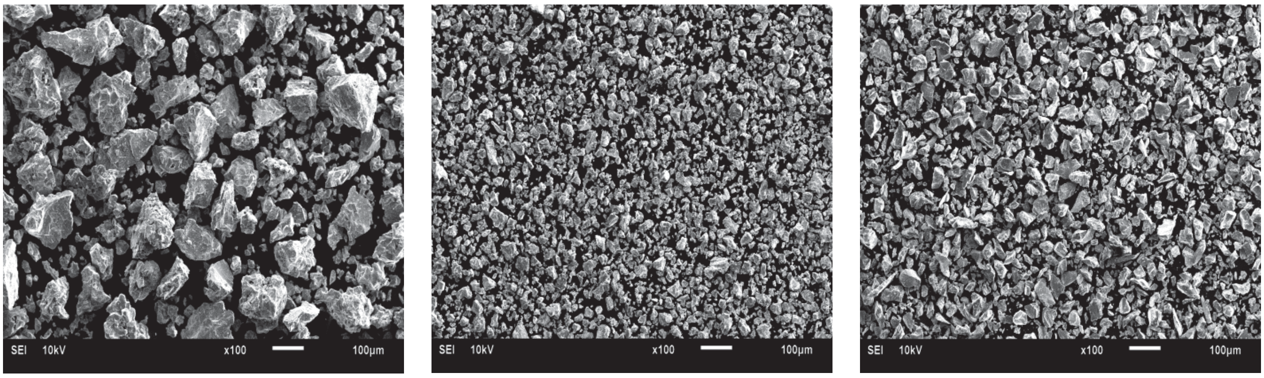
The atomized powder was classified for size distribution by sieving the as-received powder into different size fractions. Characterization powders were focused only on the size fraction suitable for the Laser Engineered Net Shaping (LENS) AM system. The LENS AM systems generally use powder within a 75 - 250µm particle size distribution range (Frazier, [4]). The atomized powders were characterised for particle size distribution, porosity and morphology, powder flowability, oxygen content, and microstructure.

In this study, powders with particle size in the 40 – 100 µm targeted range were used. The powder flowability was determined using a Hall Flowmeter, in accordance with standard ASTM B213-13. The oxygen content was determined as for the starting powders, by using an Eltra ONH 2000 gas analyser. The morphology was determined using a JEOL JSM-6510 Scanning Electron Microscope (SEM). The porosity and microstructures of the powders were both determined from polished cross-sections using the JEOL JSM-6510 SEM. The ultimate suitability of the powders for AM application was tested on a directed energy deposition (DED) technique Laser Engineered Net Shaping (LENS) 850-R system at the National Laser Centre, CSIR.

2.2 Results and Discussion

2.2.1 Starting materials

Figure 1 shows that the morphology of the starting powders was angular, and typical of the production process which includes crushing hydrogenated Ti powder to reduce particle size. Angular powder particles are easier to process using the conventional press and sinter powder metallurgy technique. The oxygen content of the powders was determined to be 0.18wt.% for 100 mesh TiHDH, 0.32 wt.% for the TiHDH 325 mesh and 0.03 wt.% for the 60Al40V master alloy. Oxygen content of titanium powders is a function of the particle size, being higher for finer than coarser particles, as in the current case.



(a) (b) (c)

Figure 1: Particle morphology of starting powders: (a) 100 mesh TiHDH, (b) 325 mesh TiHDH, (c) 325 mesh 60Al40V master alloy.

2.2.2 Sintered PM Bars/Billets

Figure 2 (a & c) show the Ti-6Al-4V long and short bars/billets produced via PM techniques at the CSIR; the bars/billets had theoretical sintered densities of 65 and 87 % respectively. The difference in the sintered density was caused by the difference in the green densities and powder particle size: the higher green density of the short billets, and also the fineness of the powders used to make them, accelerated densification. Oxygen analysis by ICP-OES found the sintered long bars/billets had 0.27wt.% oxygen while the short bars/billets contained 0.48wt.% oxygen. These oxygen contents were influenced by the oxygen contents in the starting powders, which were lower for the powders used to produce the long billets, and by the powder particle size, where finer particles, due to their higher surface area adsorb more gas at the surface which then diffuses into the powders during sintering. Apart from the powder characteristics, the environment in the furnace used to sinter the billets could also have been contaminated with air. Future work will endeavour to sinter billets in a vacuum furnace. An analysis of the microstructures of the bars/billets showed that they had the typical alpha-beta microstructure characteristic of the Ti-6Al-4V alloy, indicating that the sintering conditions had been sufficient to allow for elemental diffusion and chemical homogenization.

(a) (b)

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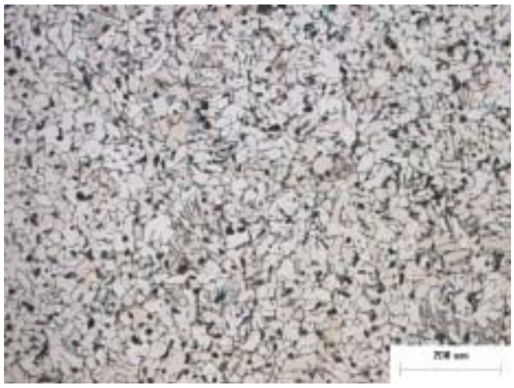
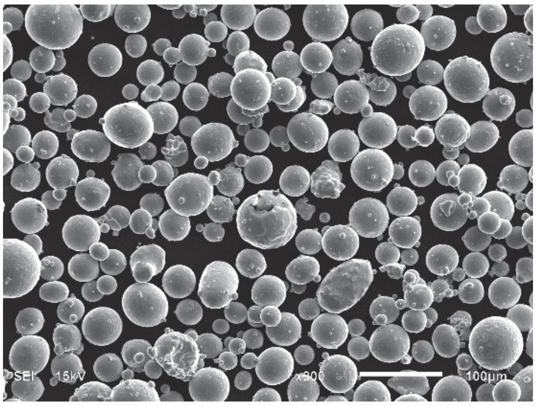


(d)

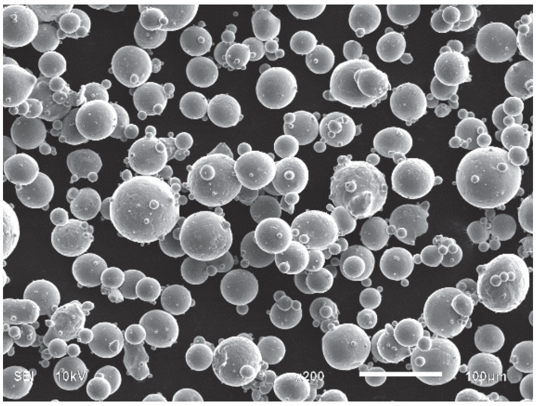
Figure 2: PM Ti-6Al-4V bars/billets and their characteristic alpha-beta two-phase microstructures.

2.2.3 Characteristics of atomized powders: Powder morphologies

Figure 3 shows the typical morphology of the powders atomized by EIGA and VIM. Regardless of the starting billets and the atomization route, all the atomized powders were mostly spherical with either smooth or textured surfaces. This was an important observation, and indicated that the atomization processes were not sensitive to the densities of the billet feedstock. Spherical powders, in theory, have an enhanced flowability, and a high flowability is required by all AM technologies (Anderson, [11]). By observation, the EIGA system produced powders from both the commercial and the long PM billets had a significant amount of 'satellite particles' compared to the VIM system produced powders. Satellites particles are the fine particles attached to larger particles and, according to Sun et al [7] form when fine particles are blown onto semi-solidified particles. The fact that more satellites appeared to have formed on the EIGA powders tallied with the known fact that the EIGA process produces more fine particles than the VIM process (Sun et al [7]). The presence of satellites introduces obstacles to smooth continuous flow.



(a)



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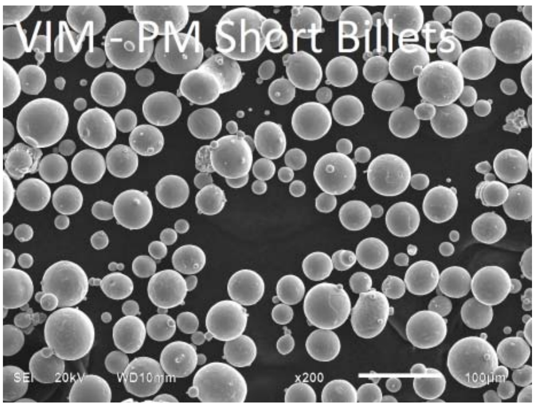


(c)

Figure 3: Morphology of EIGA (a) commercial (b) PM Long bar and VIM (c) PM short bar derived atomized powders.

2.2.4 Characteristics of atomized powders: Microstructures of powders

The polished cross-sections of all the powders indicated that some larger particles had pores in addition to the satellite particles attached to them (Figure 4). However, the volume of the porous particles constituted a small percentage overall. According to Rabin [12], pores in atomized powders are caused by the entrapment of the atomizing gas. Chen [8] recently determined that the amount of entrapped gas increased with powder particle size, which explains the reason why only larger particles in the current study had porosity. According to Anderson [11], the powder particle porosity is generally difficult to eliminate and is therefore detrimental to AM builds.

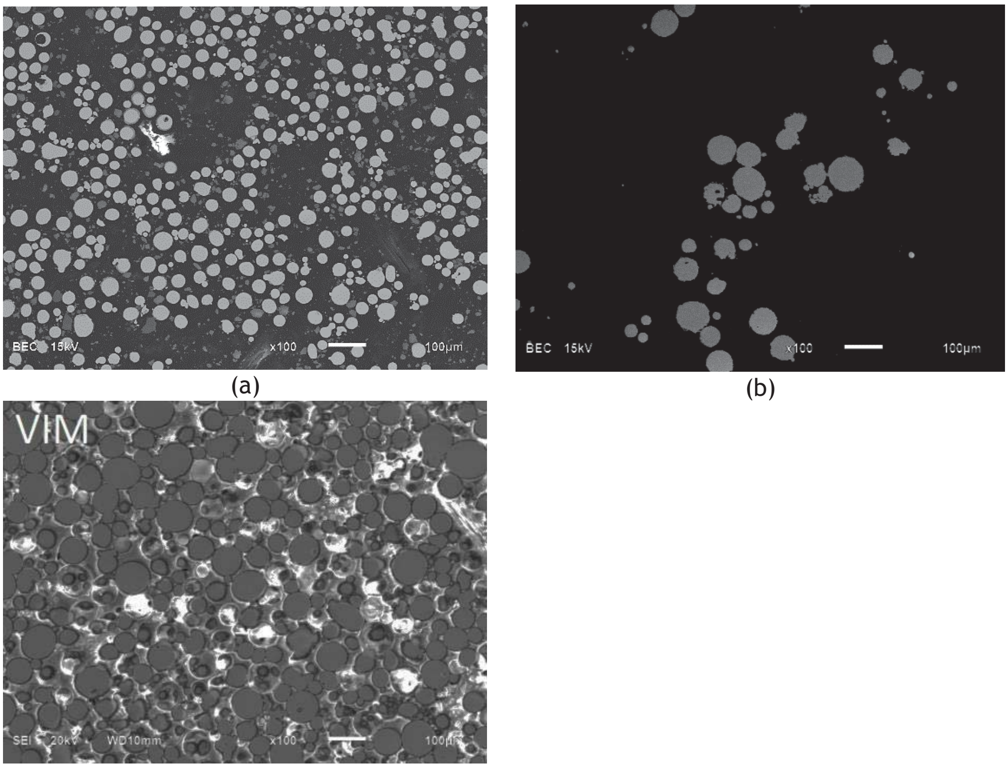
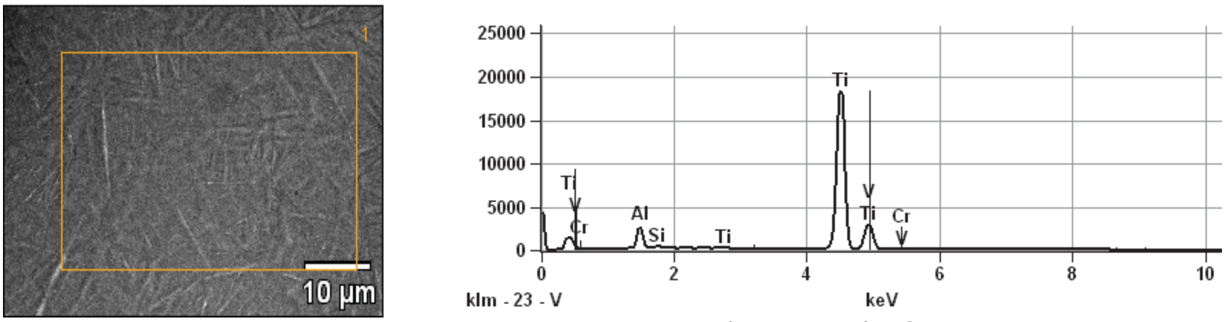


Figure 4: Polished sections of powders showing pores in some particles.

The microstructure of the polished cross-sections of the powder particles (Figure 5) showed the occurrence of martensitic laths, typical of fast cooled Ti6Al4V alloy for the EIGA- PM long bar derived powder. The corresponding energy dispersive spectroscopy (EDS) EDS analysis (Figure 5 and Table 1) indicated that the elemental composition of the powders was characteristic of Ti6Al4V alloy. Similar microstructures and compositions were observed for the VIM-PM short billet derived powder and the EIGA-commercial bar derived powder.



(a) EDS map (b) Measured EDS pattern

Figure 5: Microstructure and EDS analysis of powder particles.

Table 1: Characteristic chemical composition of powders from SEM-EDS.

Element (Line)	*Weight %*	*Atom %*
Al K	5.3 ± 0.06	9.03 ± 0.11
Si K	0.4 3 ± 0.03	0.71 ± 0.04
Ti K	90.77 ± 0.29	86.11 ± 0.27
V K	3.49 ± 0.09	3.15 ± 0.09
Cr K	0.00	0.00
Total	100.00	100.00

2.2.5 Characteristics of atomized powders: Powder oxygen content and flowability

Table 2 below shows the oxygen content and the flowabilities of the atomized powders. The oxygen contents, which ranged between 0.21 and 0.47 wt.% were generally a reflection of the oxygen contents of the billets. The oxygen content of the powder from the EIGA atomization of the commercial billet was only marginally higher than that of the starting billet. For the experimental PM billets, the EIGA atomization of the long billet also resulted in oxygen content pick-up compared to the starting powder. In contrast, the VIM atomization of the PM short billet did not cause a pick-up of oxygen: the oxygen content of the atomized powders marginally decreased. This observation for the VIM process was in line with literature: Takaki and Abiko [13] showed that the oxygen content of titanium bars did not change after VIM. The indication therefore is that the oxygen conent of the powders from the short PM billets could have been lower had the billet manufacture used lower oxygen content powders. In comparison to the ASTM F2924 [14], the standard for Ti Grade 5 powders for AM powder bed fusion, neither the EIGA nor VIM atomized experimental powder met the standard while the commercial billets also missed the standard for AM applications, but marginally. However, meeting the specifications of the ASTM F2924 standard was not the aim of the study, which was to investigate the feasibility of atomizing PM billets.

The flowability of the powders exhibited considerable dependence on the atomization technique. While all the EIGA atomized powders did not flow through the hall flowmeter, VIM atomized powders exhibited very good flowability through the hall flowmeter. The non-flowability of the EIGA powders was attributed to the occurrence of satellites on the powders. However, it is worth noting that the current standard for Ti Grade 5 powders for AM powder bed fusion fails to specify the minimum accepted flowability. While this flowaibility test was a comparative test, Spierings et al [15] advises that that the non-flowability of a powder in a Hall Flowmeter is not necessarily an indication of performance during use for building AM components.

Table 2: Oxygen content and flowability of atomized Ti-6Al-4V powders.

Specimen	Oxygen content (wt.%)		Flowability (s/50g)
	Starting powder or (billet)	atomized powders	
EIGA powder from commercial bars/billets	(0.18)	0.21	Did not flow
EIGA powder from PM bars/billets	0.20	0.27	Did not flow
VIM powder	0.32(0.48)	0.47	31
ASTM F2924 - Grade 5 Ti AM powders	--	0.20	Not specified

2.3 AM directed energy deposition - LENS Build

Figure 6 shows a component built by the LENS AM system using the atomized powders. In general, the relative ease of building varied between the powders. Generally, built components were difficult to grow using both the EIGA powders, and easier with the VIM powders- components from the EIGA powders suffered burns, and loss of defined fine edge and features, all caused tentatively by poor flowability. On the other hand, the good flowability of the VIM powders allowed for ease of material processing to the desired components.

Figure 6: AM product of VIM atomized powders.

3. CONCLUSION

The production of spherical Ti-based powders from powder metallurgy produced bars/billets was demonstrated. The density of the PM billets was shown to not affect the atomization process. The demonstration has the potential to lower the cost of Ti-based AM powders because PM billets are cheaper than wrought billets. Low cost AM powders have the potential to exponential increase the uptake of Ti AM by industry. The EIGA atomization of PM bars/billets produced powders with similar characteristics to those from commercial bars/billets. Atomization by EIGA and VIM produced powders that differed in flowability, and ease of processing using the LENS system. Both the powders from the PM bars/billets failed to meet oxygen contents stipulated in ASTM standards for powders for Ti Grade 5 AM applications: this was caused by the already high oxygen content of the starting Ti HDH powders. Work is continuing aimed at producing powders that meet the specifications of international standards. Emphasis will be placed on using Ti powder that has as low oxygen as possible.

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