

THE CHALLENGES OF TITANIUM METAL INJECTION MOULDING

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ABSTRACT

Titanium has fired the imagination of engineers and designers for decades by its 'ideal' combination of high strength, low density and good corrosion resistance. However, its application has unfortunately been limited to those niche markets where performance is more important than cost, such as in the aerospace, military, medical and off-shore oil drilling fields. Extensive efforts have been and still are being expended on ways to make this metal cheaper. There are promising new processes but these have yet to be demonstrated commercially. Nevertheless, there has been a global surge in interest in titanium over the past decade, and in South Africa the government has recently made this a particular focus for research and development funding. With the increased availability of higher quality titanium powder, metal injection moulding offers an attractive method for producing small, intricate components at a reasonable cost. This paper will present an overview of the metal injection moulding process and discuss the particular challenges related to the use of titanium and titanium alloy powders. The state of the global and local industry and markets will also be reviewed.

Keywords: metal injection moulding, titanium, challenges, binders

1. OVERVIEW OF METAL INJECTION MOULDING

1.1. Introduction

Metal injection moulding (MIM) is essentially a specialised form of injection moulding using, instead of plastic, a metal powder mixed with some type of binder (usually polymeric) as the feedstock. The initiative to 'plasticise' powdered raw materials with the aid of thermoplastic additives and then to use injection moulding to form parts was first developed for ceramics in the 20th century. In the late 1970s, this process was adapted to metal powders in the USA [1] and subsequently MIM has developed into a well established and clearly defined manufacturing technology.

1.2. The MIM Process

Metal injection moulding (MIM) has emerged as a viable method of producing complex shaped parts at a competitive cost. The MIM process uses a combination of powder metallurgy and plastic injection moulding technologies to produce net-shape metal parts. The process is comprised of feedstock preparation, injection moulding, de-binding and sintering. The flow diagram of the process steps in MIM is presented in Figure 1.

The process involves preparation of the feedstock by mixing the metal powder (typically between 50-70vol%) with the binder, pre-heating to melt the latter and obtain sufficient fluidity for injection moulding. The MIM process relies a great deal on the production of a homogenous powder-binder feedstock mix. The binder is subsequently removed either by heating or chemical extraction and this is followed by sintering to produce the final component.

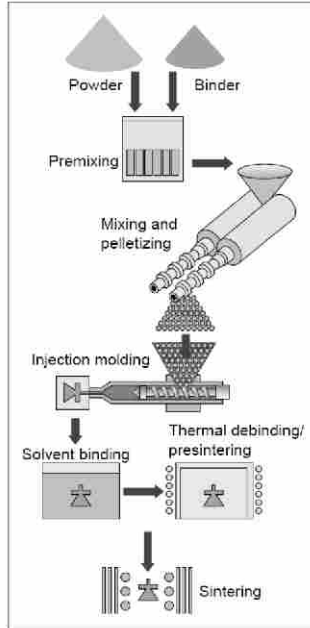


Figure 1: Schematic of the MIM process [2]

Some of the challenges of MIM compared with thermoplastic moulding are:

- the difference in flow and thermal behaviour of the MIM feedstock which has to be taken into account for uniform mould filling, and
- the need to accommodate the considerable shrinkage (up to 20%) of MIM parts after debinding and sintering in the design of the moulds.

1.3. Advantages of MIM process

Metal injection moulding is able to successfully compete with traditional machining and investment casting in the small parts arena. The economic benefit of MIM is mainly seen when a large number of small parts of complex configuration (> 25 measurable dimensions) is required (Figure 2).

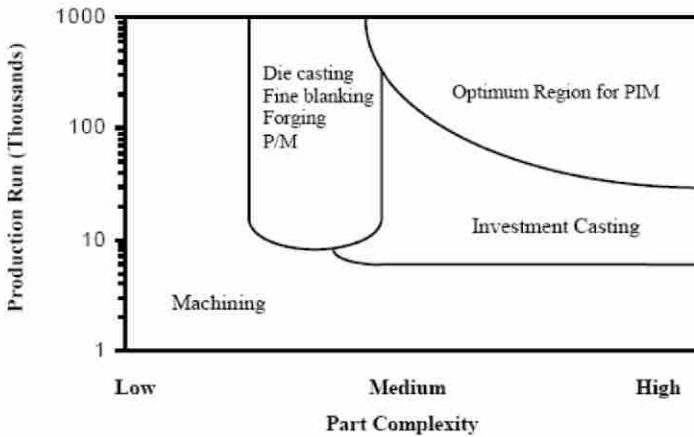


Figure 2: MIM (PIM) is suited to high volumes of complex shaped components [2]. (Low complexity = less than 10 measurable dimensions; high = more than 100 dimensions) Furthermore, MIM can achieve savings of up to 50% when compared with other manufacturing methods [3], especially those involving extensive machining, as indicated in Figure 3.

Relative cost vs shape complexity

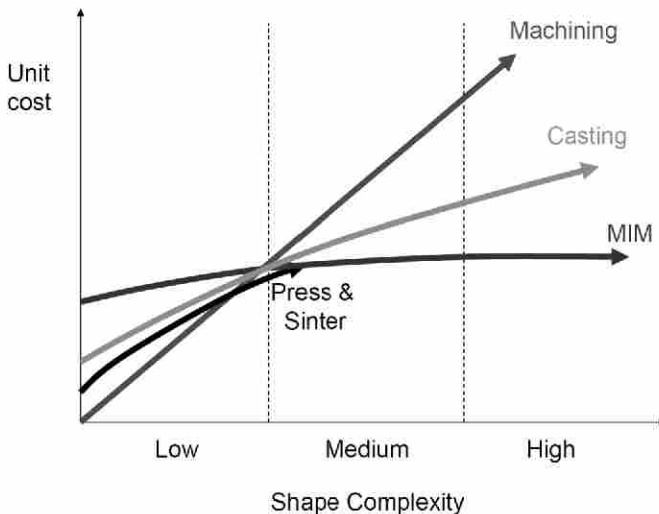


Figure 3: Cost comparison of MIM with other processing routes [4]

In addition to these advantages, MIM also confers several other benefits. These are listed in Table 1.

Table 1: Comparison of MIM with other production methods [5]

Method	Limitations	MIM Advantage
Investment casting	<ul style="list-style-type: none"> • Slow, labour intensive • Tolerances hard to control • Many secondary operations • Expensive 	<ul style="list-style-type: none"> • Lower cost • Short production cycles • High repeatability • Excellent surface finish • Minimal secondary operations
Die casting	<ul style="list-style-type: none"> • Poor mechanical properties • Rough finishes • Limited range of materials 	<ul style="list-style-type: none"> • Excellent mechanical properties • Wide range of materials
Machining	<ul style="list-style-type: none"> • High level of wastage • High tooling costs • Design limitations • Not good for intricate shapes 	<ul style="list-style-type: none"> • Virtually no material waste • Excellent for intricate parts
Conventional PM	<ul style="list-style-type: none"> • Lower densities • No complex shapes • Many secondary operations 	<ul style="list-style-type: none"> • Very high densities (93-99%)

Together with conventional powder metallurgy, MIM offers the versatility of blending different powders together to yield composite materials that offer special or added qualities.

The global MIM market (all metals) has grown annually at a consistent double-digit rate. The actual size in 2006 has been variably estimated by observers to be between \$435 million and close to \$1 billion. The top growth areas in 2007 are in the medical, automotive and electronic markets [6].

1.4. Materials for MIM process

MIM components are manufactured from a wide range of materials and more are being developed. At present, low alloy steel and stainless steel form the bulk of these and smaller quantities are made from tool steels, high-speed steels and non-ferrous alloys. A strong focus is now being placed on injection moulding of titanium, due to its unique property combination of high strength, light weight and good corrosion resistance. In addition, powder processing of titanium has the potential for significantly reducing the high cost of this metal. Currently MIM parts are being manufactured from alloys such as Ti6Al4V and Ti6Al7Nb.

2. TITANIUM MIM

As indicated above, the majority of MIM products are made from relatively easy-to-handle metal powders such as stainless steel, aluminium and copper. Titanium, however, is a much more difficult metal to work with, especially in the powder form. This entails a new learning curve with many additional challenges to be overcome. The major issues facing the industry are discussed below.

2.1. Cost

The usage of titanium in general has been severely limited by the availability of the metal in suitable form and quality and this is related to the high cost of its production. The total demand in 2007 is conservatively estimated to be approximately 80 000 tons (see Figure 4) and growth is envisaged to be slow unless there is a significant price change [3,7].

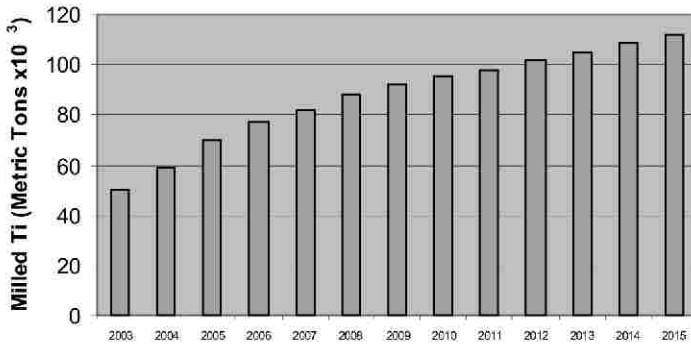


Figure 4: Projected growth (conservative) in global demand for milled titanium [3,7]

To put this into perspective, the cost of titanium at different stages of production is compared with that for steel, aluminium and magnesium (Table 2).

Table 2: Cost of titanium compared to other competing metals [8]¹

	Cost (\$ per pound contained)			
	Steel	Aluminium	Magnesium	Titanium
Ore	0.02	0.10	0.01	0.30
Metal	0.10	0.68	0.54	2.00
Ingot	0.15	0.70	0.60	4.5
Sheet	0.30-0.60	1.00-5.00	4.00-9.00*	8.00-50.00

¹ Mg sheet not commonly used. Castings are \$2.50-10.00 per pound.

The additional processing required to make titanium powder with conventional methods adds considerably to the cost, with prices reaching \$30/pound and even higher [3] (Figure 5).

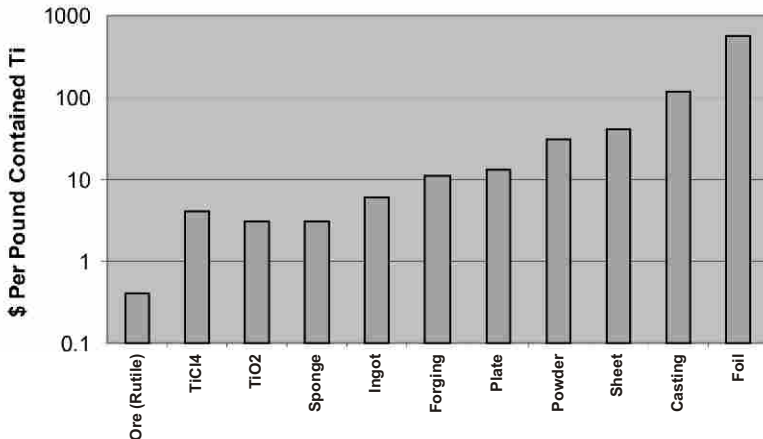


Figure 5: Current production costs of the different forms of titanium [3,9]

Currently, titanium parts produced by injection moulding are limited to less than 30cm in length and the world-wide production is between 3 and 5 tons per month. For expansion of this market, a powder cost of less than \$20/pound is needed [10].

However, there is some hope as several promising new low-cost titanium processes are being developed and, in several cases, the output will be in the form of powder. This has the added advantage of eliminating the costly step of atomisation or milling, resulting in the projected cost of the powder being reduced to as low as \$10/pound [3]. Nevertheless, the excitement around this has been diminished somewhat by the slow progress in bringing these alternative technologies to commercial reality. A variety of technical difficulties have been encountered and their resolution is taking longer than expected. Consequently, a significant decrease in the feedstock cost may only be realised in the next 5 years or even more. Despite the current high cost, titanium MIM is making inroads into high value areas, such as the medical field, where it now accounts for about 30% of the market for titanium surgical instruments [3].

2.2. Reactivity of titanium

The high reactivity of titanium, particularly towards interstitial elements (e.g. carbon, oxygen, hydrogen and nitrogen) is well known and this is exacerbated when the surface area is large, such as with small powder particles.

The major effect of this contamination is to reduce the mechanical properties of the consolidated component as indicated in Figure 6.

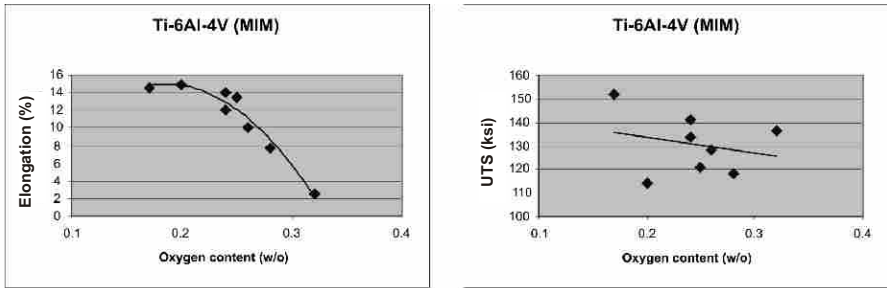


Figure 6: Effect of oxygen content on elongation and UTS of Ti-6Al-4V (adapted from [11])

This reactivity occurs even at low temperatures and so oxides, for example, are almost impossible to prevent from forming. Additional contamination will also happen during subsequent processing such as mixing with the binder, during debinding as well as on sintering. Significant absorption of impurities occurs above 260°C [10] and so any traces of the binder still present after debinding will result in a reaction with the metal, usually leading to additional carbon build-up.

Figure 7 shows an example of the increase in oxygen pick-up through the MIM process for Ti-6Al-4V. It can be seen that the initial content of the powder is key to being able to meet the ASTM specification of 2000ppm in the final component. The temptation to use lower quality and less expensive titanium powders is often counter-productive as these tend to have levels of oxygen that are too high [12].

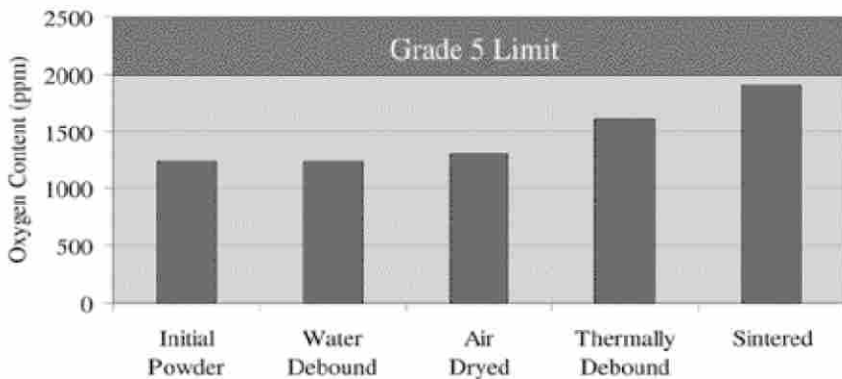


Figure 7: Increase in oxygen content during processing of Ti-6Al-4V powder system [13]

Carbon contamination can be reduced to a certain extent by using a higher debinding temperature but this has the drawback of raising the oxygen level as well (Figure 8).

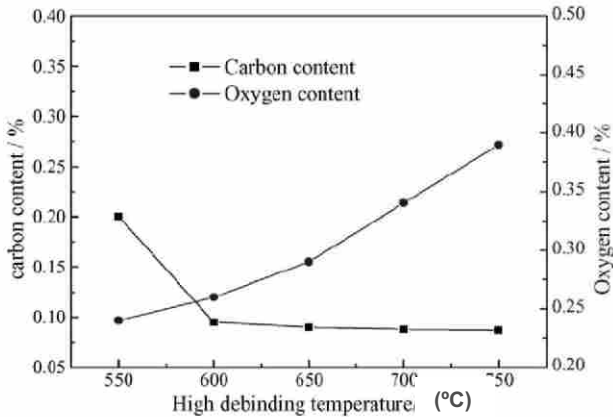


Figure 8: Effect of debinding temperature on carbon and oxygen levels in Ti-6Al-4V [14]

3. BINDERS

From the previous discussion, it is clear that the binder plays a critical part in ensuring the final quality of the component and failures/poor results can largely be attributed to the use of the wrong one.

The requirements of a binder are multiple:

- must 'wet' the powder particles
- must melt at a suitably low temperature for injection moulding;
- must be easily removed by using chemical or thermal means without leaving residues; if thermal, it must decompose at temperatures low enough to minimise chemical reaction with the metal powder particles (ideally <260°C);
- must contain no other deleterious elements that could degrade the titanium metal;
- must provide sufficient green strength;
- must provide lubricity;
- must be environmentally friendly.

In general, binders are composed of several constituents:

- Primary binder
- Powder surfactant/lubricant
- Additive to improve green strength

Examples of binder systems that are reported to be compatible with Ti-6Al-4V [2]:

- Polypropylene, ethyl vinyl acetate, paraffin wax, carnauba wax, dioctyl phthalate;
- Polyethylene, paraffin, stearic acid;
- Polypropylene, polymethyl methacrylate, paraffin wax, stearic acid;
- Naphthalene, stearic acid, ethylene vinyl acetate;
- Paraffin wax, polyethylglycol, polyethylene, stearic acid;
- Paraffin wax, copolymer, stearic acid;
- Atactic polypropylene, carnauba wax, paraffin wax, stearic acid;
- Atactic polypropylene, ethylene vinyl acetate, paraffin wax, carnauba wax, di-n-butyl phthalate.

The perfect binder has yet to be discovered and much research is being conducted in finding suitable combinations and as well as procedures for debinding. Unfortunately this is mostly of a proprietary nature and is not disclosed. Nevertheless there are some indications of what is being used and these are summarised in Table 3.

Table 3: Summary of binders and other details of commercial/research Ti MIM activities

Metal powder	Binder	Binder vol %	Mixing	Injection moulding	Debinding	Sintering	Ref.
γ -TiAl 20-45 μ m	Polyethylene Paraffin Stearic acid	32	Z-blade 120°C	Pressure: 420bar Feedstock: 90°C Mould: 45°C	1. chemical: hexane bath at 40°C 2. thermal: 250-400°C (vacuum)	1360°C for 3.5hr (300-900 mbar Ar)	15
TiH ₂ 8.6 μ m (ave)	Naphthalene (93 vol%) EVA* (6 vol%) Stearic acid (1 vol%)	33	85°C 10min, 50rpm Shear: Brabender blade	Binder melts at 81°C. Pressure: 20MPa Feedstock: 90°C? Mould: 20°C	1. 75°C for 48hrs at 2x10 ⁻² Torr 2. heat in Ar/2.75%H ₂ at 1°C/min to 375°C 3. hold at 375°C for 3hrs 4. heat at 1°C/min to 750°C, turn off gas flow and pull vacuum, hold at 750°C for 3hrs	Continue heating in 10 ⁻⁶ Torr vacuum at 5°C/min to 1100°C, hold for 4hrs. Cool in Ar to RT TiH ₂ decomposes to Ti at 350°C and higher	16 17
Ti-6Al-4V <20 μ m	Agar (1-3wt% of solids) Water (typically 45-55 vol%) Calcium/zinc borate (0.2-0.5 wt%)	39	Twin screw extruder / sigma blender	Feedstock: 80-95°C Pressure: 10-55bar Mould: 25°C	Air drying for about 1 hr	1150-1250°C argon atmosphere/vacuum	18 19 20
Ti-6Al-4V/HA [#] 90 μ m (ave)	PAN-250S (Adeka Fine Chemicals) Natural wax, fatty acid wax, stearic acid, poly-oxi-alkylene-ether and olefin-hydrocarbons	40	Sigma blade 90°C for 1.5hrs	Pressure: 80bar	1. heat to 120°C at 20°C/min, hold 60 minutes 2. heat to 380°C at 30°C/min, hold 60 minutes 3. heat to 450°C at 70°C/min, hold for 60 minutes 4. heat to 700°C at 250°C/min, hold for 90 minutes Ar gas flow: 250cm ³ /min	1150°C for 1 hr	21
Ti-6Al-4V	? (proprietary) water soluble				Water at 65-75°C, Air dried at 40-70°C Thermal: heat to 350°C at 4°C/min, hold for 30-60 minutes		9
Ti-6Al-7Nb 20-32 μ m	Polyethylene Paraffin Stearic acid	32	Z-blade mixer at 120°C	Pressure: 600bar Feedstock: 110°C Mould: 55°C	Chemical: hexane bath at 40°C Thermal: heat to between 250 and 400°C under vacuum (10 ⁻³ mbar)	1250°C for 2hrs under vacuum (10 ⁻⁵ mbar)	22

* gas atomised ** hydride dehydride # ethylene vinyl acetate ## hydroxyapatite

According to [14], the optimum powder loading is 72vol% as this gives the maximum green density. Shrinkage after sintering is dependent upon the powder loading as well as the particle size distribution. This can range from 20 to 10% for powder loadings of 50 to 70vol% respectively [24].

4. The MIM Market

The titanium MIM product needs to be tailored to the actual needs of a particular market. The previous discussions around minimising impurities are only of importance for components requiring mechanical integrity but there is a lower end of the market where aesthetic value is a more dominant need e.g. jewellery, non-load-bearing items etc. Here lower quality powders could potentially be used to help reduce costs. The market can be divided into four broad categories, depending on the level of quality required [25], as indicated in Table 4.

Table 4: Ti MIM market segmentation [25]

Application	Main property requirements	Typical standards used
Non-critical	shape complexity good surface finish good corrosion resistance	None
Engineering	static mechanical properties (strength, ductility etc)	ASTM B348
Aerospace	static mechanical properties (strength, ductility etc) dynamic properties (fatigue strength, fracture toughness)	SAE-AMS
Medical implants	Static properties Dynamic properties Biocompatibility	ASTM F67 ASTM F136

Although certain standards are mentioned, these are not specific to MIM as currently there are no specifications (apart from proprietary guidelines) relating to either the feedstock or final products. The need for internationally accredited standards is recognised as an urgent requirement and their absence is seen as a hindrance to more widespread acceptance of this technology [25].

5. CONCLUSIONS

Titanium has always appealed to engineers and designers as it has an almost ideal combination of properties: lower density, high strength and excellent corrosion and fatigue resistance. However the very high cost in producing titanium products has effectively limited its use to niche markets where function is of greater concern than cost, such as in the aerospace, military, medical and off-shore drilling fields.

Powder metallurgy however is seen as a possible saviour for titanium as many of the energy-intensive and time-consuming steps can be eliminated and thus the final cost can be substantially reduced. In addition, new primary titanium production technologies are being developed that promise to lower the costs even further, especially when the output is in the form of powder. This latter benefit would further eliminate the need for atomisation or milling and the price of the metal powder is estimated to fall from about \$30/pound to as low as \$10/pound.

MIM is a very attractive manufacturing method, akin to plastic injection moulding, and has been demonstrated to be particularly cost-effective when components are small (typically 30g), complex shaped and where high volumes are needed. Although the set-up costs are high, the large volumes and elimination of significant post-processing steps such as machining, means the component prices are reduced substantially. A well-established track record has been developed over the past 30 years using aluminium, stainless steel and copper powders.

However, titanium powders are a much more exacting proposition, due mainly to the very reactive nature of this metal, and significant challenges need to be overcome before the full potential of titanium PM, and in particular MIM, can be realised. The main issues are the following:

- the high metal price is unlikely to drop substantially within the next 5 years or so as the new primary metal technologies are still facing numerous technical difficulties;
- the need for higher quality powders, containing low levels of oxygen and other contaminants, to maximise mechanical performance for critical applications;
- better binder systems are required to prevent interstitial contamination;
- improvements in processing to fully remove the binder and minimise the pick-up of oxygen; and
- the development of international standards specific to MIM titanium products.

Although South Africa is a significant titanium pigment producer, there has been very little down-stream titanium metal processing/manufacturing activity. Titanium metal has to be imported, usually as the final product. At best there are some limited secondary processing (i.e. shaping, machining and joining etc.) activities being undertaken.

The Department of Science and Technology, together with several local institutions, has prepared a national strategy to develop a titanium metal industry and there are now plans to have a primary production facility in place by 2020.

Furthermore a Titanium Centre of Competence is currently being established to coordinate and expand existing research within South Africa institutions in regard to the above mentioned challenges and also to address other issues that will face the broader titanium industry in the future.

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