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Review: Fatigue Properties of Ti-6Al-4V Alloys Fabricated by Metal Injection Moulding

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Abstract: Metal injection moulded Ti-6Al-4V alloy exhibits sufficient tensile properties, however, the fatigue properties are not comparable to the wrought alloy. This is due to the residual porosity and coarse microstructure, which stems from the sintering process. This review presents a comprehensive assessment of the physical properties, tensile properties, fatigue properties, related processing parameters and potential methods to improve the fatigue properties of the Ti-6Al-4V alloy fabricated by metal injection moulding (MIM). The potential methods to improve the fatigue properties of the MIMed Ti-6Al-4V are the grain refinement methods by the addition of alloying elements or by sintering in the alpha and beta phase temperature region, the surface treatment method by shot peening process, and the higher binder content method. All these methods can improve the fatigue properties of the MIMed alloy to different extents. The most effective method, which can provide fatigue properties comparable to the wrought alloy, is when both grain refinement and shot peening are simultaneously applied. This paper should be useful for people who are interested in the fatigue properties of the Ti-6Al-4V fabricated by MIM.

Keywords: Metal injection molding, Fatigue, Sintering, Grain refinement, Shot peening

1. Introduction

Metal injection moulding (MIM) is a cost-effective process that is suitable for mass-producing small net-shape parts with high complexity and accuracy [1]. MIM can process various types of sinterable materials such as carbon steel, stainless steel, nickel and titanium alloys. The mechanical properties of the MIMed parts are outstanding when comparing to the conventional compaction due to its low residual porosity after sintering [2].

MIM combines characteristics of two processes, which are (1) injection moulding process that using to form polymer with complex shape and (2) the powder metallurgy method based on the sintering process to consolidate metallic powders. The general steps in MIM are presented in Figure 1. Initially, the metal powders, which can be used as elemental, master-alloyed or pre-alloyed powders, are mixed with a polymeric binder to produce granulated feedstock [3,4]. The feedstock is then heated and injected into the mould cavity by an injection machine to obtain the "green" part. The binder is removed from green parts by the solvent (optional) and/or thermal debinding process before sintering in a controlled environment, e.g. Ar or vacuum atmosphere to densify the part. During sintering, the metal atoms diffuse and form necks and subsequently, the voids are closed. This causes the shrinkage of the specimens, which is theoretically uniform. Nevertheless, in practice, the uniformity of shrinkage, which lies in the range of 12-18%

depends on several factors, e.g. the uniform distribution of powder within the green parts, geometry, gravity and friction between specimens and setters [2,5]. Hence, the mould is needed to be oversized to compensate for the shrinkage. After the sintering process, the density of the specimen can reach up to 99% theoretical density if the optimised parameters are used. The secondary processes are normally unnecessary. However, some secondary processes, e.g. machining, hot isostatic pressing (HIP) and shot-peening can be applied to enhance properties, e.g. the dimensional accuracy and the mechanical properties by reducing the effects of remaining porosity at both inside and surface regions for the critical parts. Another advantage of MIM is the green parts, e.g. runner, uncompleted and failed injected specimens, can be almost 100% recycled by re-granulation and then re-injection.

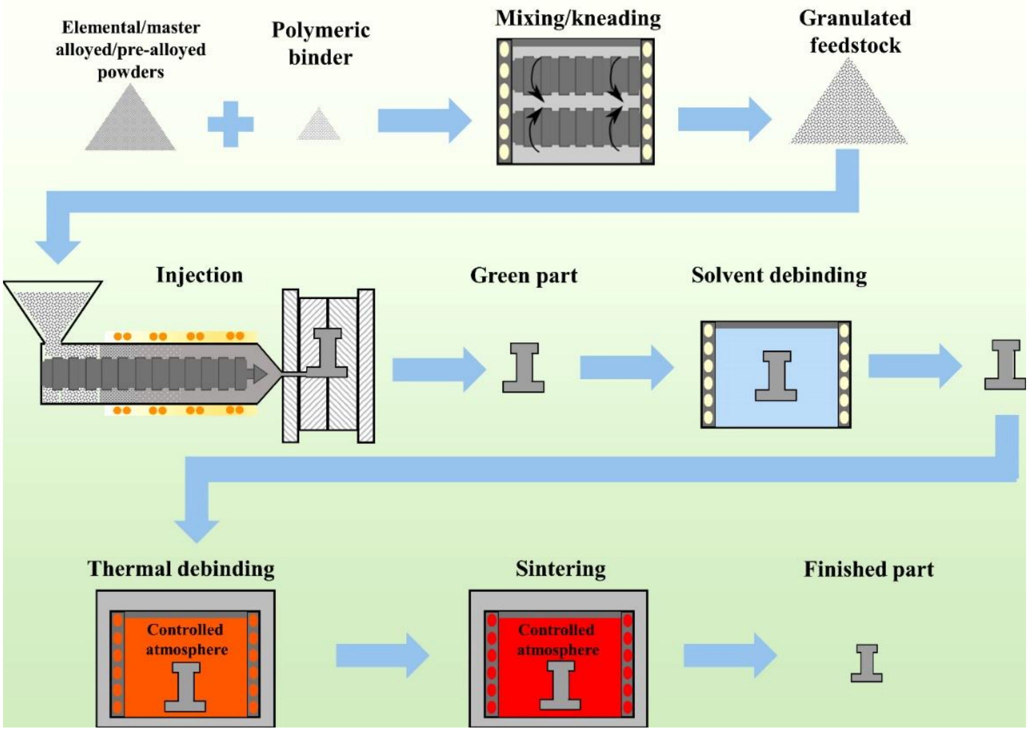

Figure 1 Overall process of metal injection moulding.

Titanium (Ti) and its alloys exhibit excellent mechanical properties with high strength to weight ratio. Nonetheless, Ti alloys are very reactive materials that are hard to be conventionally cast and hot forged. Moreover, the finishing process of the Ti alloys significantly increases the production cost. Therefore, MIM, which provides the net-shape product, is advisable for the fabrication of Ti and its alloy. From the literature, many types of Ti alloys have been successfully fabricated by MIM, i.e. alpha-type CP-Ti [6,7], alpha-beta-type Ti-6Al-4V [8-15], near-beta [16], beta [16-23] and gamma types [24,25], which can apply to various industries such as biomedical, chemical and aerospace applications [2,26]. Among all Ti alloys, Ti-6Al-4V is the most widely used. Although the tensile properties of Ti-6Al-4V fabricated by MIM are sufficient, the fatigue properties of the MIMed alloy are still lower than those of the wrought alloy. This is due to the nature of pressureless sintering with a slow cooling rate that results in having residual porosity and the coarse lamellar structure [27]. The aims of this review are to systematically summarise and present the process parameters that affect the mechanical properties focusing on the fatigue testing, properties and the potential methods to improve the fatigue properties of MIMed Ti-6Al-4V.

2. Process parameters for MIMed Ti-6Al-4V

The overall process parameters and the mechanical properties of Ti-6Al-4V based alloys fabricated by MIM are tabulated in Table 1. It is well known that oxygen is one of the most critical impurities that directly affects the mechanical properties of Ti alloys [2]. An increase in oxygen content results in increasing tensile strength but decreasing elongation. From Table 1, the oxygen content of the MIMed alloys is controlled at the limit of 0.35 wt.%, which is consistent with the report of Ebel [5] that the elongation of MIMed Ti-6Al-4V rapidly decreases when the oxygen content exceeds 0.35 wt.%. Therefore, the contamination from all MIM steps, e.g. raw powder, debinding and sintering, is needed to be carefully controlled as low as possible. Two techniques are proposed and successfully utilised to reduce and control those contaminations are: placing sacrificial Ti-sponge having high specific surface area together with the specimens during sintering to minimise the carburisation and oxidation [2,28]; and the minor addition of Y to the alloys that acts as oxygen scavenger from the matrix [12]. In addition, the other impurities such as C and N, which commonly contaminate from the polymeric binders during debinding and sintering, also influence the mechanical properties of the MIMed Ti alloys. The overall effects of the interstitial elements can be summarised and expressed in form of oxygen equivalent (O_{eq}) as presented in Equation 1 [29].

$$O_{eq} = O + 2N + 0.75C \qquad (1)$$

where O, N and C are the contents in wt.% of oxygen, nitrogen and carbon respectively. As reported by Ebel [27], the oxygen equivalent should not exceed 0.4 wt.%. Although the effect of C content on mechanical properties is less detrimental than the effect of O content, the carbon content is still required to be controlled. Excessively high carbon content can contribute to the reduction in elongation, since brittle Ti carbides can potentially precipitate at the grain boundaries and within grains and potentially become the initiation of cracks [30].

Regarding the processing parameters, the sintering temperature and time are the most important and commonly adjusted parameters of the MIM process. These parameters need to be optimised to yield the combination of small grain size with sufficient relative density. The sintering temperature and time for Ti-6Al-4V sintering from the literature can be divided into two groups: (i) the higher temperature with shorter sintering time (less than 8 h) and (ii) the lower temperature with a longer sintering time (more than 24 h). For the first group, which provides up to 97% of the relative density, the sintering temperature is in the range of 1250 to 1400 °C and the dwell time is in the range of 2 to 8 h. The high relative density contributes to somewhat higher tensile properties, especially when the porosity is found at the surface due to an increase in a load-bearing area [27,31]. Although the high sintering temperature and longer sintering time can yield a higher relative density, it should be carefully chosen to avoid the abnormal grain growth and the excessive increase in oxygen content, which causes the reduction in some mechanical properties, e.g. elongation. The second group was proposed by Kudo *et al.* in 2018 [15] with the objective to refine the grain size, leading to an improvement of the fatigue properties by sintering at 980 °C for 24 to 96 h in the alpha and beta phase region below the beta transus temperature. It is noted that Ti alloys are conventionally sintered in the beta phase region. Sintering in the alpha and beta phase temperature region will reduce grain growth but also required significantly longer sintering time which might not be preferable for mass production. It provides the relative density that is comparable to the high sintering temperature group (up to 98%). The average grain sizes for MIMed Ti-6Al-4V that were sintered at the low temperature with long dwell time were between 18-25 μm, while the typical high temperature with short dwell time produced significantly larger grain sizes ranging between 63-177 μm. The tensile strength of both sintering groups is between 800 to 900 MPa with different elongations from 0.5 to 15% depending on the sintering conditions as shown in Table 1. The effects of using low sintering temperature with long sintering time on the fatigue properties will be discussed in more detail next.

Table 1 Available data related to tensile and fatigue properties of Ti-6Al-4V fabricated by MIM

Alloys	**O content (wt.%)**	**Tensile strength (MPa)**	**Solid loading (Vol.%)**	**Elongation (%)**	**Testing method**	**Defined endurance limit at 10^6 or 10^7 cycles (MPa)**	**Sintering condition (°C, h)**	**Relative density (%)**	**Grain size (μm)**	**Ref.**
Ti-6Al-4V	<0.3	850 850	65	ND ND	Rotating bending	200 230	1350, 2 1350, 4	97.5	130 170	[2]
Ti-6Al-4V	<0.3	910	65	15	Rotating bending	280	1350, 4	>96	170	[32]
Ti-6Al-4V	ND	840	65	15	Rotating bending	290	1350, 4	97.4	177	[15]
Ti-6Al-4V	0.29	854	ND	8.5	Rotating bending	200	1350, 1	94.02	63	[33]
Ti-6Al-4V	0.32	878	ND	0.5	Rotating bending	250	1350, 5	96.33	170	[33]
Ti-6Al-4V	ND	830	65	5	Rotating bending	375	980, 24	96.25	18	[15]
Ti-6Al-4V	ND	900	65	12	Rotating bending	475	980, 48	98.3	23	[15]
Ti-6Al-4V	ND	925	65	18	Rotating bending	480	980, 96	98.8	25	[15]
Ti-6Al-4V	0.23	850	ND	10.3	Uniaxial	178	1315, 1.7	~96	100	[34]
Ti-6Al-4V	0.19	800	68	15	Bending	350	1250, 2	96.5	-	[10]
Ti-6Al-4V	0.21	806	69	13.7	Bending	350	1250, 2	95.74	97	[9]
Ti-6Al-4V	0.23	824	65	13.4	Bending	400	1250, 2	95.75	148	[9]
Ti-6Al-4V	0.21	784	65	10.8	Bending	400	1350, 2	95.84	93	[9]
Ti-6Al-4V-0.03B	0.37	900	65	ND	Rotating bending	360	1350, 4	>97	100	[2]
Ti-6Al-4V-0.03B	<0.3	880	65	20	Rotating bending	350	1350, 4	>96	115	[32]
Ti-6Al-4V-0.06B	<0.3	900	65	18	Rotating bending	390	1350, 4	>96	55	[32]
Ti-6Al-4V-0.12B	<0.3	920	65	13	Rotating bending	400	1350, 4	>96	60	[32]
Ti-6Al-4V-0.24B	<0.3	900	65	13	Rotating bending	420	1350, 4	>96	35	[32]
Ti-6Al-4V-0.40B	<0.3	880	65	13	Rotating bending	450	1350, 4	>96	40	[32]
Ti-6Al-4V-4Cr	<0.3	1030 1030 1000	65	ND ND ND	Rotating bending	~230 ~240 ~260	1150, 8 1200, 8 1250, 8	93 >96 >96	60 110 160	[2,35]
Ti-6Al-4V-SP	0.24	884	65	15.3	Bending	450	1400	96.5	125	[36]
Ti-6Al-4V-SP	0.23	824	65	14.3	Bending	450	1350, 2	96.4	148	[8]
Ti-6Al-4V-SP	0.19	>800	68	ND	Bending	450	1250, 2	96.5	ND	[10]
Ti-6Al-4V-SP1	0.23	870	ND	9.4	Uniaxial	306	1315, 1.7	~96	100	[34]
Ti-6Al-4V-SP2	0.23	950	ND	7.6	Uniaxial	289	1315, 1.7	~96	100	[34]
Ti-6Al-4V-SP3	0.23	940	ND	8.5	Uniaxial	323	1315, 1.7	~96	100	[34]
Ti-6Al-4V-0.5Y-SP	0.25	794	65	12.5	Bending	470	1400	95	90	[36]
Ti-6Al-4V-0.5B-SP	0.20	902	65	11.8	Bending	640	1400, 2	97.7	18	[8,37]

Note: ND means no detail and SP# means shot peening with different peening variables

3. Fatigue testing for MIMed Ti-6Al-4V

From the literature, there have been three fatigue testing methods for the MIMed specimen, i.e. uniaxial testing [34] with the frequency of 150 Hz and stress ratio of 0.02 (R = 0.02); four-point bending [8-10,36,37] with the frequency of 95 Hz and stress ratio of -0.2 (R = -0.2) and; rotating bending [2,15,32,33] with the frequency of 150 Hz and stress ration of -1 (R = -1). The dimension in mm of the specimens is shown in Figure 2(a) to 2(d) respectively. However, with our experience, the rotating bending specimen with circular cross-section will be difficult to debind and sinter to maintain the circularity. In addition, the cantilever gauge length part can possibly collapse during depending and sintering. This could be prevented by specially designed supports to prevent distortion or over-sized MIM cylindrical parts were machined to size after sintering. However, MIMed parts are commonly used with minimal machining. Hence, the fatigue testing of as-sintered parts is more applicable.

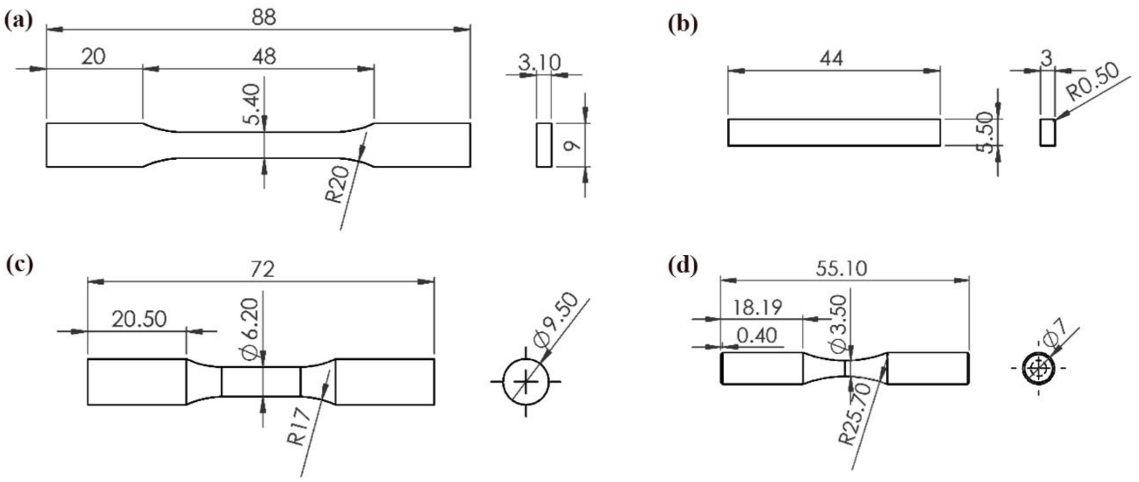

Figure 2 Dimension in mm of MIMed specimens utilized for fatigue testing of (a) uniaxial fatigue, (b) four-point bending fatigue, (c) and (d) rotating bending fatigue.

4. Grain refinement to improve fatigue properties in MIMed Ti-6Al-4V

Grain refinement (GF) is known as the mechanism which significantly improves the fatigue properties of Ti-6Al-4V since it decreases slip distance for the dislocation motion [38]. Because the structure of MIMed Ti-6Al-4V alloy is coarse lamellar, the fatigue properties are in the shadow of the corresponding wrought alloy, especially when parts were sintered using either too high sintering temperature or too long sintering time [13,39]. One of the alternative methods in which the grain size can be controlled is using finer raw powders [5,40]. It can limit and control the as-sintered grain size. Furthermore, the lower sintering temperature and shorter sintering time can be applied to minimise grain growth.

Two potential methods successfully used for refining the structure of MIMed Ti-6Al-4V alloy are the additions of the alloying elements and the sintering in the alpha and beta phase temperature region [2,15,37]. The effect of both grain refinement methods on the mechanical properties of MIMed Ti-6Al-4V is illustrated by the rotating bending fatigue strength vs tensile strength map in Figure 3. The tensile (<925 MPa) and fatigue strength (<300 MPa) of Ti-6Al-4V without grain refinement (the pink area [2,15,32,33]) are significantly lower than those of the wrought alloy (the grey area [2,41]). However, the tensile and fatigue properties of the grain-refined alloys (the orange area [2,15,32]) are significantly improved and some are comparable to wrought Ti-6Al-4V alloy. The grain refinement by sintering in the alpha and beta phase temperature region seems to provide slightly higher fatigue properties than the alloying element addition method, whilst still maintaining its tensile strength comparable to the element-added alloys. Nonetheless, it is noted that even for the same alloy with the same fabrication method, the mechanical properties exhibit a wide window of both tensile and fatigue properties. This is due to the differences in powder size, powder distribution, composition, relative density, binder, impurities and sintering conditions.

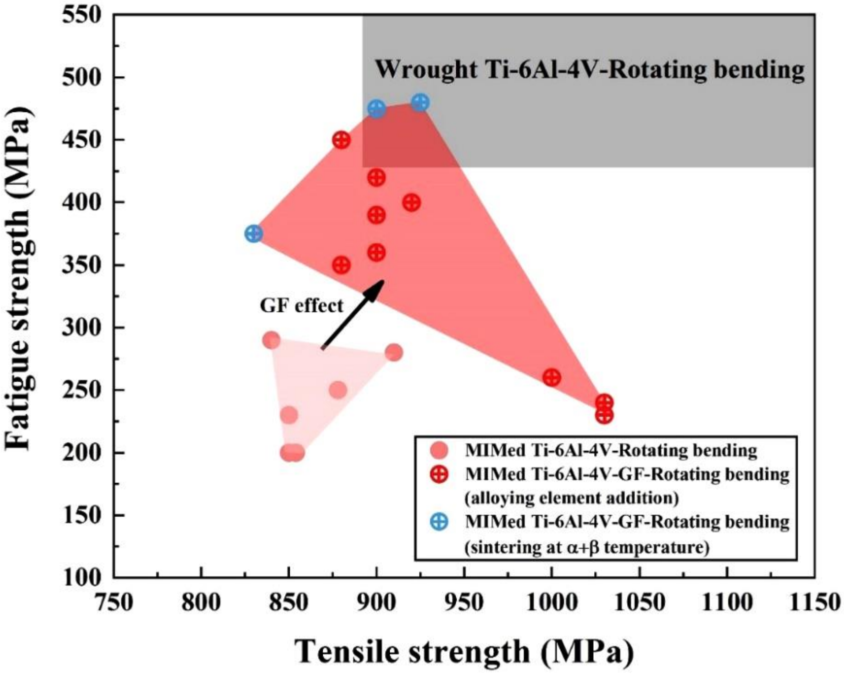

Figure 3 Rotating bending fatigue vs tensile strength map of Ti-6Al-4V with and without grain refinement (GF) that fabricated by MIM process [2,15,31-33,42].

Gd, Y, Cr and B were proposed as grain refiners for MIMed Ti-6Al-4V alloys [2,11,12,32,35]. For the Gd and Y additions, which are rare earth elements, they reacted with oxygen in the Ti matrix, then transformed into oxide particles. The element additions acted as oxygen scavenging, which consumes oxygen from the Ti matrix, thus, improving ductility [36]. These oxide particles also refined the grain size by acting as nucleation site for Gd [11] and hindering the grain growth for Y [36]. Figure 4 shows the effects of grain refinements by alloying element addition, i.e. Cr and B, and its concentration and sintering time when sintered in the alpha and beta phase temperature region on the fatigue strength and the average grain size. For Cr, it can be seen that 4 wt.% provides limited grain refinement with ~30 MPa improvement in fatigue strength. The grain refinement by Cr addition originated from its beta-stabilising effect, which results in the refinement of the acicular alpha structure [35]. The most effective alloying element in terms of grain refinement for MIMed Ti-6Al-4V is B, which by only 0.03 wt.% addition provides a stronger effect to refine the microstructure than the 4 wt.% Cr addition. From Figure 4, an increase in the B content results in decreasing average grain size. The optimum content of the B addition is 0.4 wt.%, providing the highest fatigue strength [32]. The grain refinement by B addition originates from the formation of TiB particle, which simultaneously acts as heterogeneous nucleation of alpha phase and pinning sites to restrict grain growth [32,37]. Importantly, it is suggested that the sintering temperature should be increased when the B content increases [32].

The other possible method of the grain refinement in MIMed Ti-6Al-4V is sintering in the alpha and beta phase temperature region (~980 °C) [15]. This is much lower than the conventional sintering temperature (1250 to 1400 °C), where Ti-6Al-4V is in the beta phase. This low sintering temperature results in the fine grain since the prior beta grains growth was restricted by the pinning effects of alpha grains. Because the sintering temperature is around 250 °C lower than the conventional temperature, the sinterability is poor. Therefore, the sintering time needs to be extensively extended (the lowest is 24 h, which is more than 6 to 12 times the conventional sintering time). The grain size of the Ti-6Al-4V sintered at the low temperature is seven times smaller (from ~175 to ~25 μm in Figure 4), resulting in two to three times improvement in fatigue properties. Comparing to the B-modified Ti-6Al-4V, the grain size of Ti-6Al-4V sintered in the alpha and beta phase temperature region is slightly smaller and results in better fatigue properties than the alloying element addition method. The sintering temperature of 980 °C for 48 h seems to provide the best combination of the tensile and fatigue properties and is recommended.

From the fatigue crack propagation analysis [43] by comparing to wrought Ti-6Al-4V alloy, fatigue crack propagation starts from slipping inside the grain and then grows to the grain boundary [43], however, in MIMed alloys, both trans- and intergranular fracture occur. It reflects that crack initiation starts at the pores both inside and at the grain boundary and then connects to each other, thus limiting the fatigue properties of MIMed alloys. This is consistent with prior reports [44,45] that the fatigue properties decrease with increasing the pore size. Hence, porosity is another important factor influencing fatigue behaviour [46]. Considering the grain-refined alloys, if the grain size is smaller than the porosity, the remaining porosity, especially at the surface will dominate the fatigue properties in MIMed alloys [15]. Therefore, the porosity, especially at the surface should be minimised to improve the fatigue limit of the MIMed and grain-refined MIMed alloys.

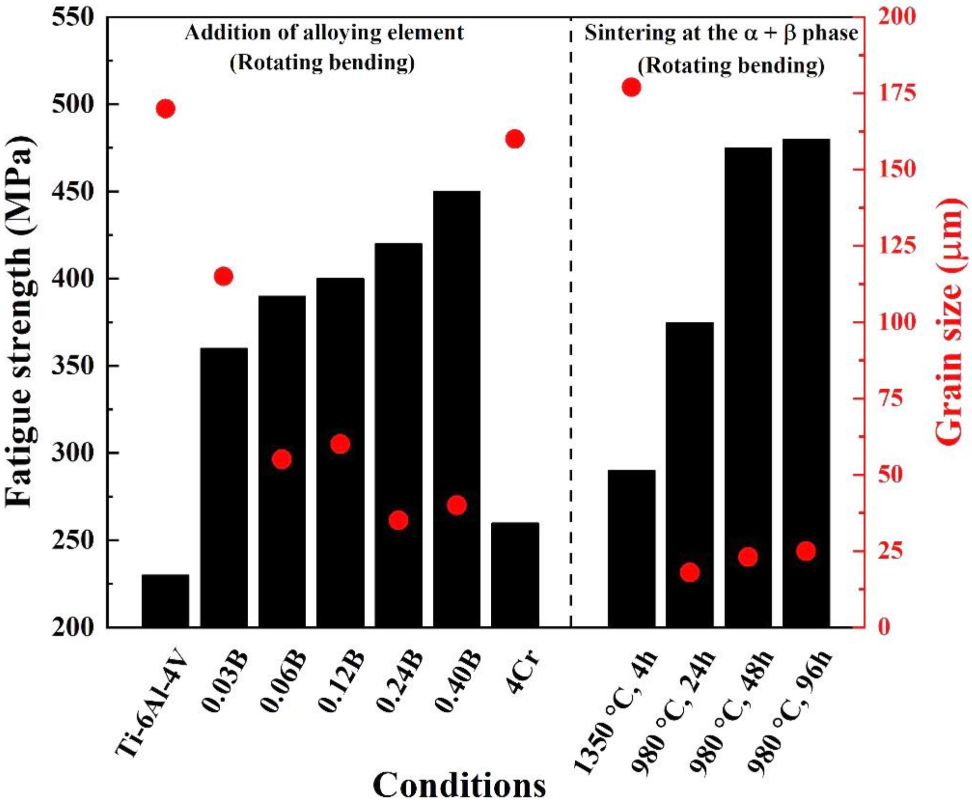

Figure 4 Effects of grain refinement by alloying element addition and sintering at low temperature on endurance limit and its relationship to grain size [2,15,32,33].

5. Surface treatment to improve fatigue properties in MIMed Ti-6Al-4V

Shot peening (SP) is one of the mechanical surface treatments that have been the most widely used to originate the compressive residual stress near the surface by the impaction of ceramic or metal shots (the media size larger than 200 μm) to the specimen. This residual stress can significantly enhance bending strength and bending fatigue strength [47,48]. The shot peening technique also successfully improves both the uniaxial and bending fatigue properties of MIMed alloys, shown in Figure 5. For the MIMed alloys with the shot peening treatment, it exhibits improved fatigue properties up to 450 MPa (the purple area), which is higher than the highest fatigue properties of MIMed alloy without the shot peening treatment, even though it is still lower than the wrought alloy (the grey area) without any grain refinement. For the uniaxial fatigue properties, it exhibits the same trend as the bending fatigue properties that the shot peening treatment (the blue area) significantly improves the fatigue properties up to ~100 MPa. However, without any grain refinement, it is still significantly lower than the wrought alloys (the green area).

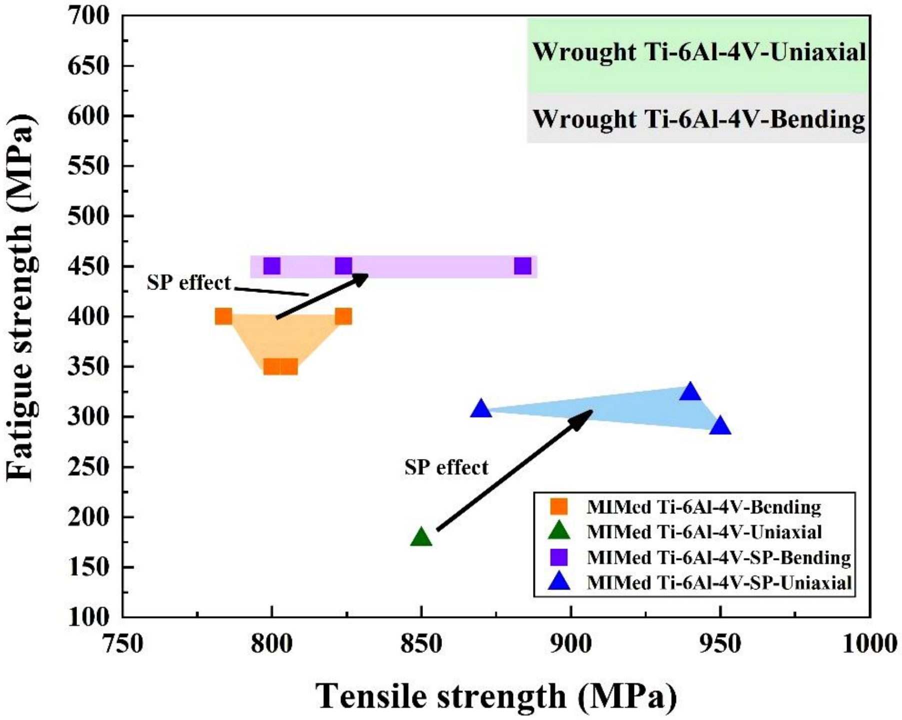

Figure 5 Effect of shot peening treatment (SP) on bending and tensile properties of Ti-6Al-4V fabricated by MIM process [9,10,31,34,49,50].

Surface quality is an important variable that significantly affects fatigue properties [9]. The relationship between surface roughness and uniaxial fatigue properties is shown in Figure 6. The shot peening process significantly improves the uniaxial fatigue properties, although the surface roughness slightly increases. It is suggested that the effects of residual compressive stress generated by the shot peening process provide an overwhelming impact than the slightly different surface roughness. In addition, the shot peening parameters also influence the improved fatigue properties of MIMed alloys. The parameters using for each shot peening conditions are SP1: steel ASH230, diameter = 600 µm, 14 Almen, SP2: steel ASH230, diameter = 600 µm, 18 Almen and SP3: Zirshot Z210, diameter=200-300 µm, 12 Almen [34]. The results show that the SP3 condition provides the highest fatigue properties among all conditions. It can be interpreted that although the shot size of SP3 is smaller than SP1 and SP2, it can generate sufficient residual compressive stress with better surface quality. With respect to surface quality, the fine shot peening technique (shot size less than 200 µm), which is applied after the conventional shot peening process to improve the surface quality, is the promising method to improve fatigue strength after conventional shot peening was applied [51].

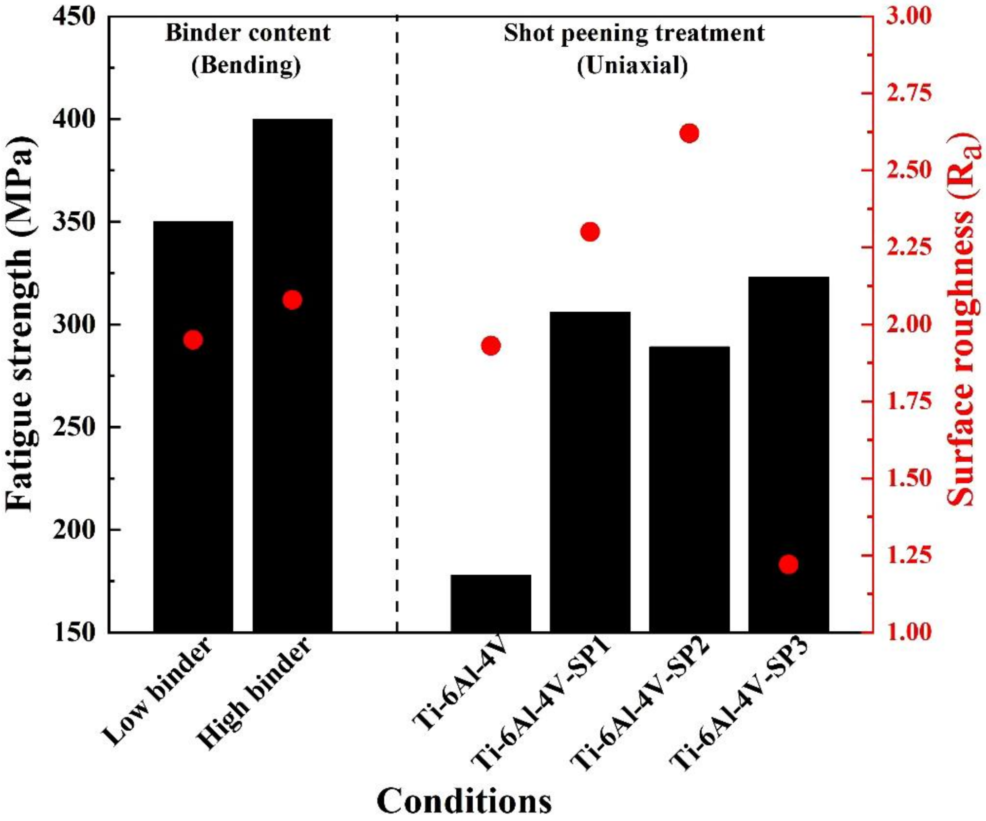

Figure 6 Effects of surface quality and shot peening on the fatigue strength of Ti-6Al-4V fabricated by MIM process [9,34].

Interestingly, except for the surface treatment by shot peening process, there is a study reporting that the binder content also affects the surface quality [9]. From Table 1, the solid loading from many studies is around 65 vol.%. However, it is found that the use of higher binder content can significantly improve fatigue properties as presented in Figure 6. It is expected to be the results of the better surface quality due to its better flowability of high binder content condition, although the surface roughness (R_a value) is insignificantly different.

6. Simultaneous effects of grain refinement and surface treatment on fatigue properties

The fatigue properties vs tensile strength of MIMed Ti-6Al-4V with and without improvement is presented in Figure 7. The simultaneous improvement by grain refinement (GF) and shot peening treatment (SP) (the orange diamond symbol with a purple edge) can significantly improve the bending fatigue properties of more than 200 MPa and tensile strength at least 50 MPa (the orange area). Although the effects of simultaneous improvement by grain refinement and alloying element addition on the uniaxial and rotating bending have not been reported, it can be anticipated that the simultaneous improvement will be significantly beneficial for both fatigue and tensile properties. Therefore, the combination of grain refinement and surface quality is the most potent method to improve the fatigue properties of the MIMed Ti-6Al-4V.

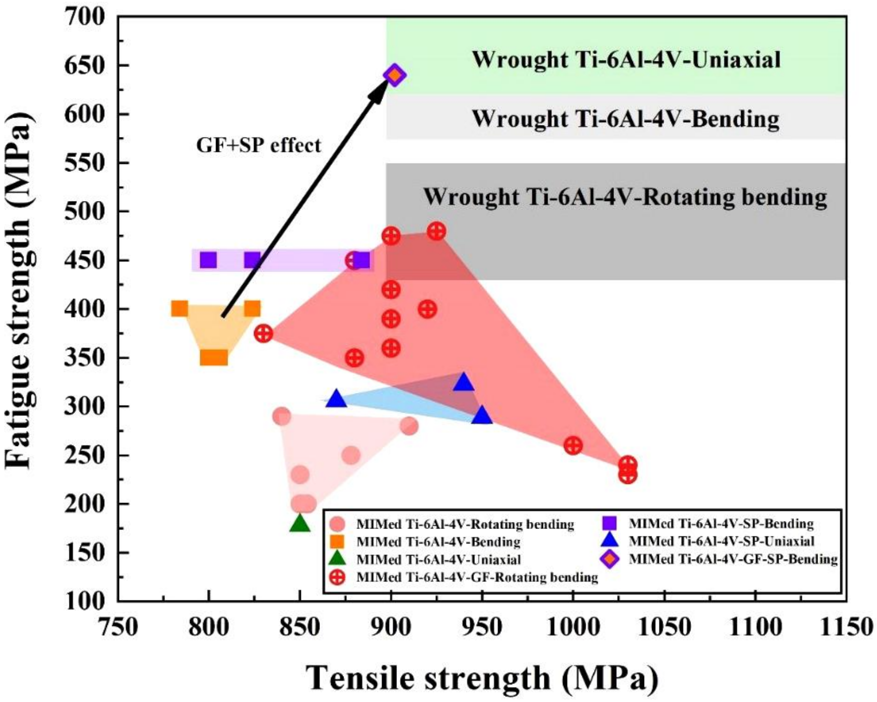

Figure 7 Fatigue and tensile properties map of Ti-6Al-4V fabricated by MIM process both with and without the improvements by grain refinement (GF) and surface treatment process (SP) [2,9,10,15,31-34,36,42,49,50].

7. Conclusions and future directions

Based on the comprehensive assessment of the physical properties, tensile properties, fatigue properties, related processing parameters and potential methods to improve the fatigue properties of the Ti-6Al-4V alloy fabricated by metal injection moulding (MIM), the following conclusions can be drawn:

- Impurities contents in terms of oxygen equivalent need to be controlled as low as possible and should not excess 0.4 wt.% to achieve sufficient elongation. Two possible methods to control the oxygen content during processing are (i) the addition of rare earth elements that will consume the oxygen from the matrix and (ii) placing oxygen getter materials such as sacrificial Ti sponge to trapping oxygen during the sintering process.
- The sintering temperature and time of MIMed Ti-6Al-4V can be divided in two groups, which are sintering at (i) 1250 to 1400 °C for 2 to 8 h and (ii) 980 °C for 24 to 96 h. Both groups provide comparable relative density up to 98% with sufficient tensile properties.
- The improvement in fatigue properties of MIMed Ti-6Al-4V can be divided into two potential methods, which are (i) the grain refinement by adding alloying element and (ii) the grain refinement by sintering in the alpha and beta phase temperature region. The effectiveness of alloying elements can be ranked as B, Y and Cr respectively. Sintering in the alpha and beta phase temperature region can refine the microstructure more effectively than the addition of alloying element method.
- Although the shot peening treatment process may result in a rough surface, the residual compressive stress generated from the mechanical treatment process can significantly improve the fatigue properties.
- The binder content of the MIMed alloy and shot peening can affect the surface quality and fatigue properties. Moreover, the fine shot peening after the conventional shot peening process is a promising method to enhance and the effect of the conventional shot peening process and improve the surface quality.

- The most successful method to improve the fatigue properties of MIMed Ti-6Al-4V is the simultaneous use of grain refinement and shot peening methods, which significantly improve the fatigue properties comparable to that of wrought materials without the reduction in the tensile properties.

Future works to improve the fatigue properties of MIMed Ti-6Al-4V are the combination of all fatigue improvement methods: B addition, sintering in the alpha and beta phase temperature region and surface treatment should provide improved fatigue properties due to the simultaneous effects of grain refinement, generating residual stress and improve surface quality. The trend of MIMed Ti-6Al-4V in aerospace applications is still regarding reducing contamination during processing, improving their mechanical properties to eliminate expensive secondary process, e.g. hot isostatic pressing. However, for biomedical applications, there is a trend moving toward beta Ti alloys, which can provide lower elastic modulus closer to the human bone. However, MIM of beta titanium is still under developing to control the readily formed titanium carbide.

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