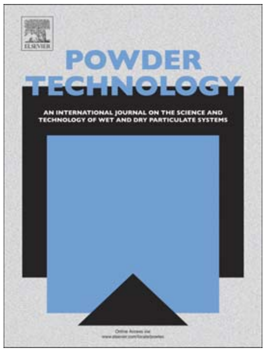

Analysis of rheological behaviour of titanium feedstocks formulated with a water-soluble binder system for powder injection moulding

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Analysis of rheological behaviour of titanium feedstocks formulated with a water-soluble binder system for powder injection moulding

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Abstract

Binder selection and formulation are critical in powder injection moulding. Binders play a key role in controlling the rheological properties of a feedstock and influence whether the resulting feedstock can be successfully injection moulded, debound and sintered without defects. A four-step process was used to mix hydride-dehydride titanium alloy (processed) powder (Ti-6Al-4V) with a polyethylene glycol (PEG) based water soluble binder system. The rheological properties, including flow behaviour index, flow activation energy, fluidity and melt flow index of the homogeneous feedstock, were determined with a capillary rheometer. All feedstock formulations exhibited shear thinning flow behaviour. The optimum feedstock consisting of 60 vol.% powder content, 32 vol.% PEG, 6 vol.% polyvinyl butyryl and 2 vol.% stearic acid was suitable for titanium injection moulding.

Keywords: Powder injection moulding; Rheological properties; Polyethylene glycol; Titanium; Binder; Feedstock.

1. Introduction

In spite of many unique properties such as high specific strengths and high resistance to corrosion, titanium alloys are restricted to high value products such as the aerospace and biomedical sectors where their high manufacturing costs can be fully justified. Powder injection moulding (PIM) offers a solution to reducing the cost and hence produces affordable titanium parts [1]. This innovative moulding technique is used to produce complex shaped components from feedstock of metal powders with thermosetting or thermoplastic binders, which provide the required fluidity for successful injection moulding [2].

Having a suitable binder system is a major factor in obtaining feedstock with good rheological properties and good mechanical properties in the final products. In general, a binder system is composed of at least three components: a backbone polymer that retains the shape of a moulded part during debinding and sintering, a low viscosity polymer that gives the feedstock suitable viscosity and which can be easily extracted during solvent debinding, and an additive that improves wettability of powder particles and binder [3]. A good binder system must have good flow characteristics, be comparatively low cost, have good interactions with metal powders, enable easy debinding, easily disposed of, and environmentally safe [4]. Binder removal in a multi-component binder system is carried out sequentially; otherwise the debound parts would collapse during debinding and sintering. In general, the low-melting-point polymer is removed either via solvent debinding or low temperature thermal debinding. Upon complete removal of most of the low-melting-point polymer, the high-melting-point polymer can still retain the shape and geometry of the moulded part. Also, the open pores formed during the first stage of debinding (either solvent debinding or low temperature thermal debinding) provide the extraction pathways for removing the backbone (i.e. high-melting-point polymer) [5]. The decomposition mechanisms of the polymers depend on the nature of the polymers used and the temperature or medium they are exposed to.

To produce a homogeneous feedstock for PIM, the powder and binder must be miscible and have desired rheological properties. Therefore, the appropriate mixing conditions need to be identified. A typical feedstock [6] was formulated using polyethylene glycol (PEG), high density polyethylene (HDPE), polyvinyl butyryl (PVB) and stearic acid (SA). These ingredients were kneaded for 5 minutes at 180 °C and then 10 minutes at 160 °C. In another report [7] the PIM feedstock was made by dry mixing both zirconia powder and a binder system made of PEG, PVB, HDPE, SA and an anti-oxidant in a Type R02 intensive mixer at room temperature for 15 minutes, then extruded using a twin-screw extruder at 150 °C – 181 °C. The powder contents were in the range 40 to 50 vol. % [7]. Although these authors claimed two extrusion passes were sufficient for feedstock homogeneity, there was no direct evidence supporting the homogeneity claim [7].

While a substantial amount of research has only resulted in low and moderate powder contents, some research reports higher powder contents. For instance, Krauss *et al.* [8] obtained a homogeneous feedstock with 55 vol.% powder content and enhanced rheological properties by mixing alumina powder and a binder system made of PEG, PVB and SA in a sigma-type blade mixer for 30 minutes at 180 °C, followed by further mixing for 30 minutes at 160 °C. Weil *et al.*

[9] obtained a 65% vol.% homogeneous feedstock for reactive metal based PIM by mixing Ti-6Al-4V powder with an aromatic-based binder system of naphthalene, ethylene vinyl acetate and stearic acid at 90 °C for an unspecified time in a Hake Record 90 mixer. Amin *et al*. [10] obtained a 64 vol.% homogeneous feedstock with good rheological properties by mixing stainless steel powder with a binder system made of PEG, polymethyl methacrylate (PMMA) and SA in a Z-blade type mixer for 30 minutes at room temperature followed by 60 min of mixing at 70 ºC [10]. There have been other reports on formulating feedstock using different binders, various mixing conditions and powder contents. Recently, Wen *et al.* [11] published a comprehensive overview on the development of binder systems specifically used for titanium injection moulding.

A direct indicator of feedstock quality is its rheological properties. A feedstock with low viscosity, low activation energy and low flow behaviour index has better rheological properties for effective injection moulding [12]. During PIM, the temperature of a feedstock changes throughout the moulding process, starting from the feed section (medium temperature, 60 °C – 100 °C), intermediate section (high temperature, 120 °C – 200 °C) to the end mould (low temperature, 25 °C – 50 °C). Viscosity can significantly change with temperature and is a critical factor in analysing rheological properties of a feedstock. Flow activation energy is widely used to analyse the temperature dependency of viscosity and depends on the composition of a binder and a feedstock [2]. A high flow activation energy indicates a strong effect of temperature on viscosity. Therefore, a small change in temperature during PIM may cause a significant change in feedstock viscosity.

In this research, feedstocks were formulated by mixing hydride-dehydride (HDH) titanium Ti-6Al-4V alloy powder with a PEG based water soluble binder system. The hydride-dehydride

(HDH) process involves hydrogenating titanium sponge, crushing the TiH_2 and then dehydrogenating the TiH_2. In general, spherical gas-atomised Ti-6Al-4V powder is preferred for formulating titanium feedstock because this powder has higher purity and the preferred spherical particles. Unfortunately, such a powder with required small particle size is expensive and usually unavailable commercially. On the other hand, HDH Ti-6Al-4V powder is more affordable and commercially available with many options of particle sizes and purity levels.

2. Experimental procedures

2.1. Materials

Feedstocks of various compositions were prepared using HDH Ti-6Al-4V alloy powder and a water soluble binder system consisting of PEG, PVB and SA. The following materials were used for this research: HDH Ti-6Al-4V powder (Xi’an Baode Powder Metallurgy Co. Ltd., Xi’an, China), PEG with a molecular weight of 8000 g/mol and a melting point of 65 °C (Union Chemical Ltd, Albany, New Zealand), PVB with a molecular weight of 50,000 and a melting point of 185 °C (Sigma Aldrich, Australia), and SA with a molecular weight of 285 g/mol and melting point of 74 ºC (Sigma Aldrich, Australia). Scanning electron micrographs showed the HDH Ti-6Al-4V alloy powder had irregular shaped particles (Figure 1). The average particle size (Figure 2) was 70 microns.

[Figure 1 here]

[Figure 2 here]

2.2. Methods

2.2.1. Mixing and blending

The homogeneous feedstock was manufactured using four successive mixing stages. A mix of titanium alloy powder and binder constituents (Table 1) were dry mixed at room temperature (25 °C) for 30 minutes at 20 rpm in a planetary mixer (Kenwood). The mixture was then transferred to a roller mixer and mixed for 16 hours at room temperature at 100 rpm. The mixture was then fed into the mixing chamber of a compounder, which had been preheated at 130 °C and mixed for 40 minutes at 55 rpm. The feedstock was then mixed in a twin screw extruder at a screw speed of 150 rpm and with a barrel temperature profile of 110 °C – 120 °C – 130 °C – 130 °C – 140 °C (feed to nozzle) to enhance feedstock homogeneity. Feedstock homogeneity was tested using burn-out tests (via TGA/DTA) and density analysis (with a pycnometer) on samples collected from the four mixing stages.

2.2.2. Rheological analysis

A Shimadzu CFT 500D capillary rheometer with a 1-mm diameter, 10-mm long die was used to investigate rheological properties of triplicate 3-g samples of each feedstock formulation. The rheometer was operated at 125, 140 and 165 ºC and the Bagley correction was performed automatically. To obtain a wide range of shear stresses and shear rates, the rheometer was loaded at 20 N, 40 N, 60 N, 100 N and 160 N. The lower loads (20N, 40N and 60N) were selected to obtain rheological properties under lower shear stress and shear rates, and the higher loads (100 N and 160 N) for obtaining the rheological properties at higher shear stress and shear rates. To measure the rheological properties, a random sample was fed into the capillary barrel, which had been preheated for 5 minutes. Data were reported as the average of three random samples. Flow activation energy from the different temperatures was used to determine the effect of temperature on feedstock viscosity.

[Table 1 here]

3. Results and discussion

3.1. Flow behaviour index

The viscosity of all feedstock formulations decreased with increase in shear rate (Figure 3) indicating time-independent, non-Newtonian fluid behaviour known as shear thinning (pseudoplastic). Shear thinning behaviour indicates that increased shear promotes uniform particle distribution and allows small particles to fit within the gap between large particles [13]. The stable particle network structure within the polymer melt at lower shear rates is broken at higher shear rates. Metallic particles and polymeric binders in the feedstock rotate and re-arrange themselves along the flow direction to allow inter-particle motion, resulting in shear thinning. Viscosity is dominated by hydrodynamic interactions [14]. In addition, the metal powder particle agglomerates tend to break apart, which improves packing of particles and homogeneity of the feedstock.

[Figure 3 here]

The data fitting showed a good fit (R^2 of between 0.98 and 1.00) to the power law or Ostwald de Waele model, $\tau = m\gamma^n$. Viscosity for a power law fluid is given by:

$$\eta = \frac{\tau}{\gamma} = m\gamma^{n-1}$$

The value of *n* is obtained by re-arranging the equation:

$$\log \eta = \log m + (n-1) \log \gamma$$

Flow behaviours of a power-law fluid depend on the value of n [15]:

For $n < 1$, the fluid shows shear thinning flow behaviour

$n = 1$, the fluid shows Newtonian flow properties n > 1, the fluid shows shear thickening flow behaviour

Feedstocks that exhibit shear thinning (n < 1) are suitable for PIM but feedstock with dilatant flow behaviour (n > 1) is not suitable because powder and binder separation can occur under high shear rate. Some feedstocks that show shear thinning may also exhibit dilatant flow behaviour under high shear rates, so rheological properties should be assessed over a wide range of shear rates. The loads used in the capillary rheometer (20 N to 160 N) allowed a wide range of shear rates to be investigated. Flow behaviour indices (*n*) of all the feedstocks, obtained from gradients of the flow curves (Figure 4), are given in Table 2. Flow behaviour indices of all the feedstocks were less than unity indicating they have shear thinning behaviour. Feedstock F4 had the lowest flow behaviour index (n = 0.63) and would therefore have the highest shear thinning behaviour.

[Figure 4 here]

[Table 2 here]

3.2. Fluidity

For the shear rate range used, the highest feedstock viscosity at the moulding temperature (i.e. worst case) was 1000 Pa·s [16]. Viscosities and shear rates of all feedstocks produced in this study were within the range of 26 Pa·s to 214 Pa·s at 230 s^{-1} and 14800 s^{-1} respectively. Fluidity of the feedstocks at 140 ˚C increased with PEG content (Table 3). Feedstocks F3 and F4 had similar fluidity, which was higher than fluidity of F1 and F2.

[Table 3 here]

3.3. Melt flow index (MFI)

Melt flow index (MFI) indicates the ease that molten feedstock will flow through a capillary die as pressure is applied at a given temperature. The MFI of feedstocks, obtained under a wide range of shear stresses, decreased as powder content in the feedstock increased from 55 vol.% to 60 vol.% at a constant load for a particular binder ratio (Table 4). On the other hand, MFI values increased significantly when PEG vol.% increased from 75 vol.% to 85 vol.% at a constant powder content and load or when applied load was increased. Feedstocks F3 and F4 had similar values, which were higher than for F1 and F2 (Figure 5). These results have the same trend as shown for fluidity (section 3.2).

[Table 4 here]

[Figure 5 here]

3.4. Flow activation energy (E_a)

Mobility of liquid molecules is a temperature-activated process so temperature dependency of viscosity is important when analysing rheological properties of a feedstock. Temperature dependency of feedstock viscosity can be expressed using the Arrhenius equation

$$\eta = \eta_o \exp\left(\frac{Ea}{\mathrm{RT}}\right)$$

which can be rewritten as: $\ln \eta = \ln \eta_o + \left(\frac{Ea}{\mathrm{R}}\right)\frac{1}{\mathrm{T}}$

where η_o is viscosity at a reference temperature, E_a is flow activation energy, R is the universal gas constant and T is absolute temperature [12]. The value for E_a can be obtained from the gradient of ln η versus 1/T. Figure 6 shows the relationship between viscosity and temperature of feedstock F2 under various shear stresses (σ = 49.03 to 392.26 kPa). Data were an excellent fit to the Arrhenius equation (R^2 of between 0.99 and 1.00). The derived activation energy data of feedstocks F2 and F4 were summarized in Table 5.

Feedstocks F2 and F4 were selected because of their better flow properties and higher powder content than F1 and F3. Flow activation energies of F2 and F4 were in the range of 35.7 to 42.5 kJ/mol and 14.8 to 29.7 kJ/mol respectively. Furthermore, the flow activation energies of F4 are consistently lower than for F2. This might be because F4 had a higher PEG content. A high flow activation energy indicates viscosity is strongly affected by temperature so a small change in process temperature during injection moulding will cause a substantial change in feedstock viscosity, which may lead to mould defects [10]. Therefore, F4 was identified as being the most suitable for PIM.

[Figure 6 here]

[Table 5 here]

4. Conclusion

All PIM feedstock formulations developed in this study exhibited shear thinning, the desired flow behaviour for successful PIM. Viscosity decreased with increasing shear rate, which indicates non-Newtonian pseudoplastic flow. Based on the results, an optimised feedstock consisting of 60 vol.% hydride-dehydride Ti-6l-4V alloy powder, 32 vol.% PEG, 6 vol.% PVB and 2 vol.% SA had the best rheological properties including a low activation energy, low viscosity, high shear thinning, high fluidity, and high melt flow rate. This feedstock also contained a high powder content, making it the most suitable feedstock for PIM.

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Figure and Table Captions

Figures

Tables

Brief biography of the authors:

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School of Engineering, University of Waikato, Hamilton, New Zealand.

Qualifications

- Bachelor of Science of Engineering [B. Sc. Eng.(Hon)]
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- Sri Lanka Association for the Advancement of Science (Life member)
- Association for Iron & Steel Technology (Student member)

Research Interests

Metal injection moulding, developing new thermoplastic binders, materials processing, rheological investigation of plastic and metal/plastic slurry, powder metallurgy, optimization, process control, engineering planning and supply chain management.

2. Kim Pickering

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- Member of the Institute of Materials, Minerals and Mining

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Expertise

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- Member of Society of Chemical Engineers New Zealand
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Academic and Professional Leadership Roles

- Associate Dean of Engineering, University of Waikato, 2005 - ongoing
- Chair, New Zealand Council of Engineering Deans, 2009 - ongoing
- Council member, Marsden Fund, 2010
- Chairperson, Department of Engineering, 2006-2008
- Chairperson, Department of Materials and Process Engineering , 1997-2003
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- Member of Materials Research Society

Research interests

Light alloys, powder metallurgy, metallic biomaterials and casting.

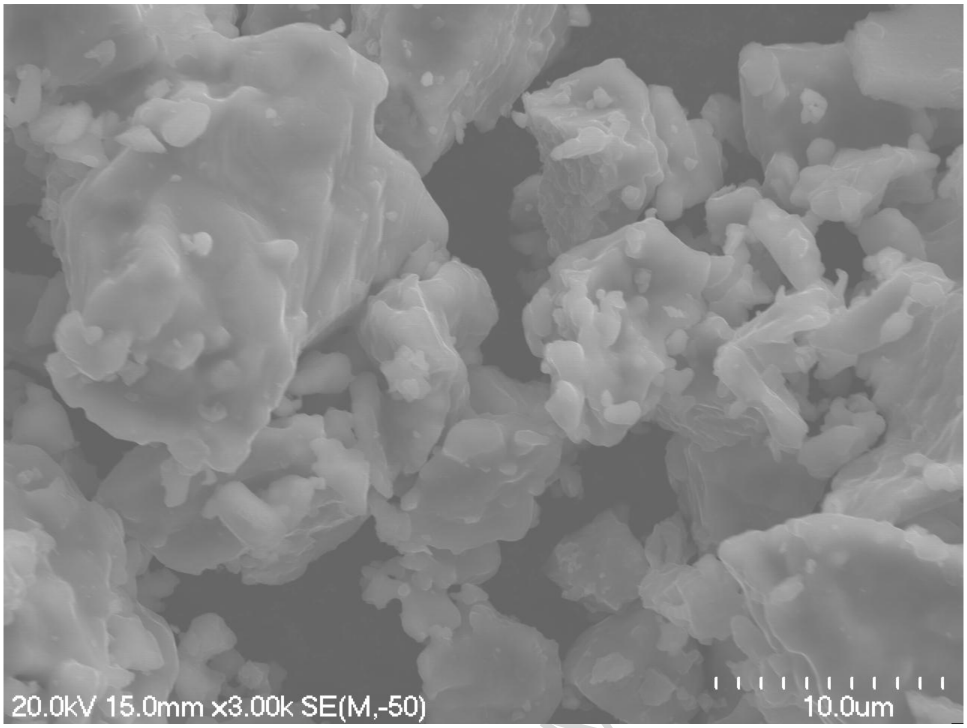


Figure 1

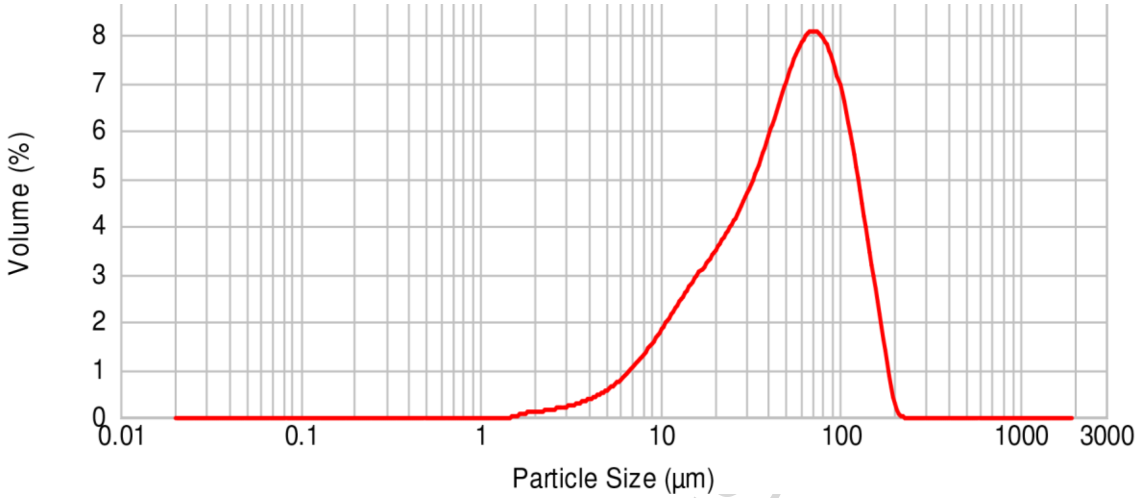
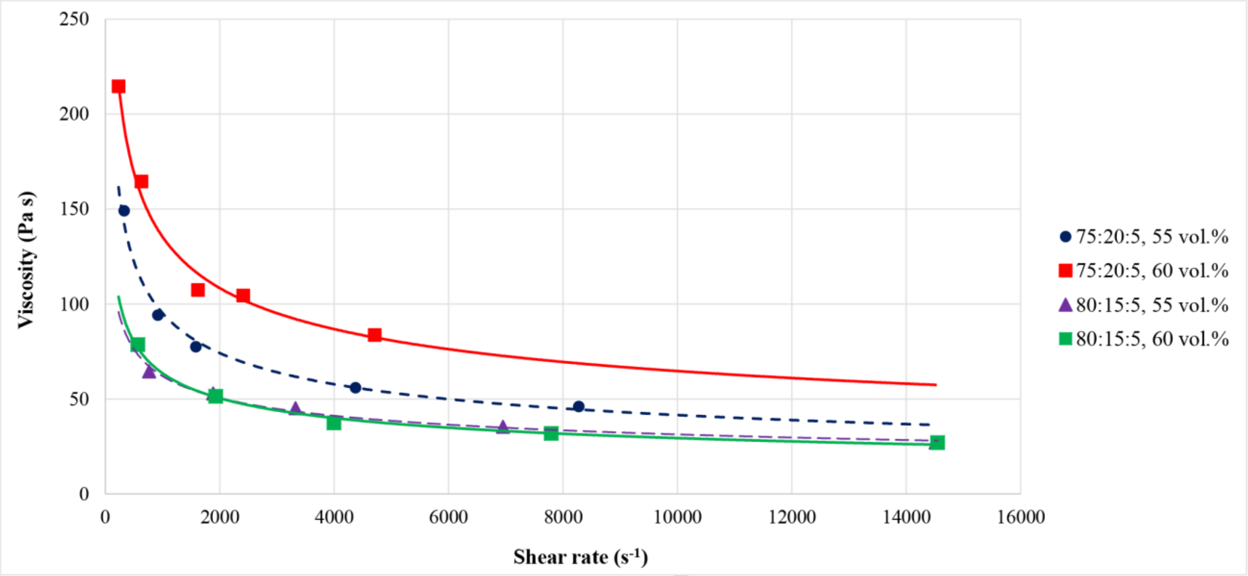


Figure 2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250
200
150
100
50
0
0
2000
4000
6000
8000
10000
12000
14000
16000
Shear rate (s^{-1})
● 75:20:5, 55 vol.%
■ 75:20:5, 60 vol.%
▲ 80:15:5, 55 vol.%
■ 80:15:5, 60 vol.%

Figure 3

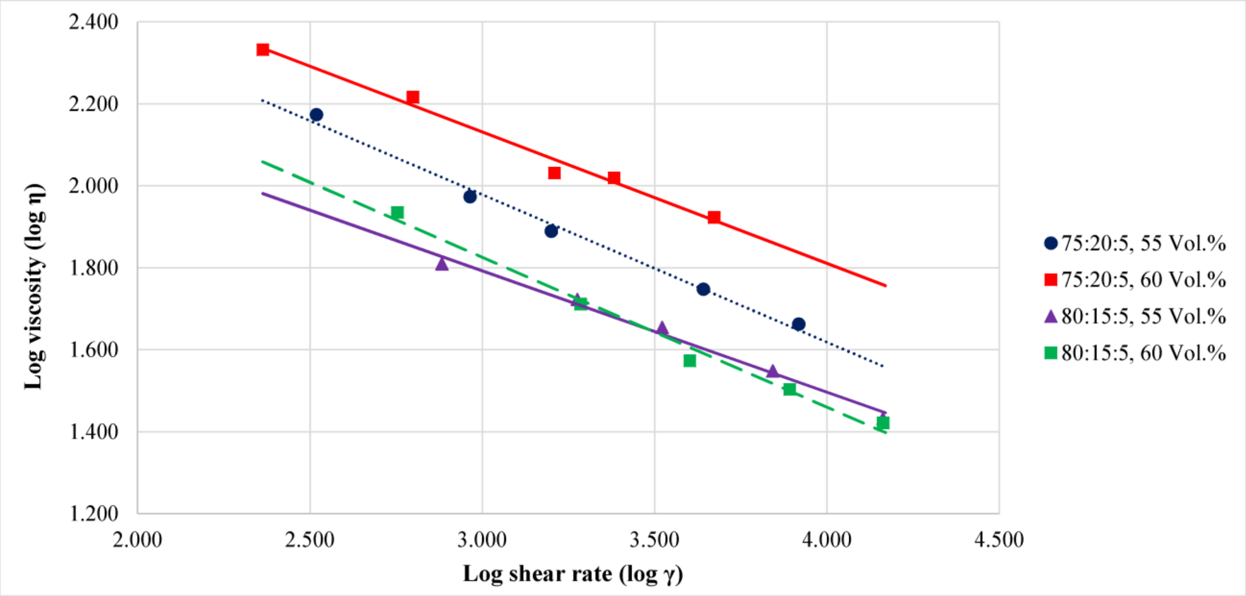

2.400
2.200
2.000
1.800
1.600
1.400
1.200
Log viscosity (log η)
2.000
2.500
3.000
3.500
4.000
4.500
Log shear rate (log γ)
● 75:20:5, 55 Vol.%
■ 75:20:5, 60 Vol.%
▲ 80:15:5, 55 Vol.%
■ 80:15:5, 60 Vol.%

Figure 4

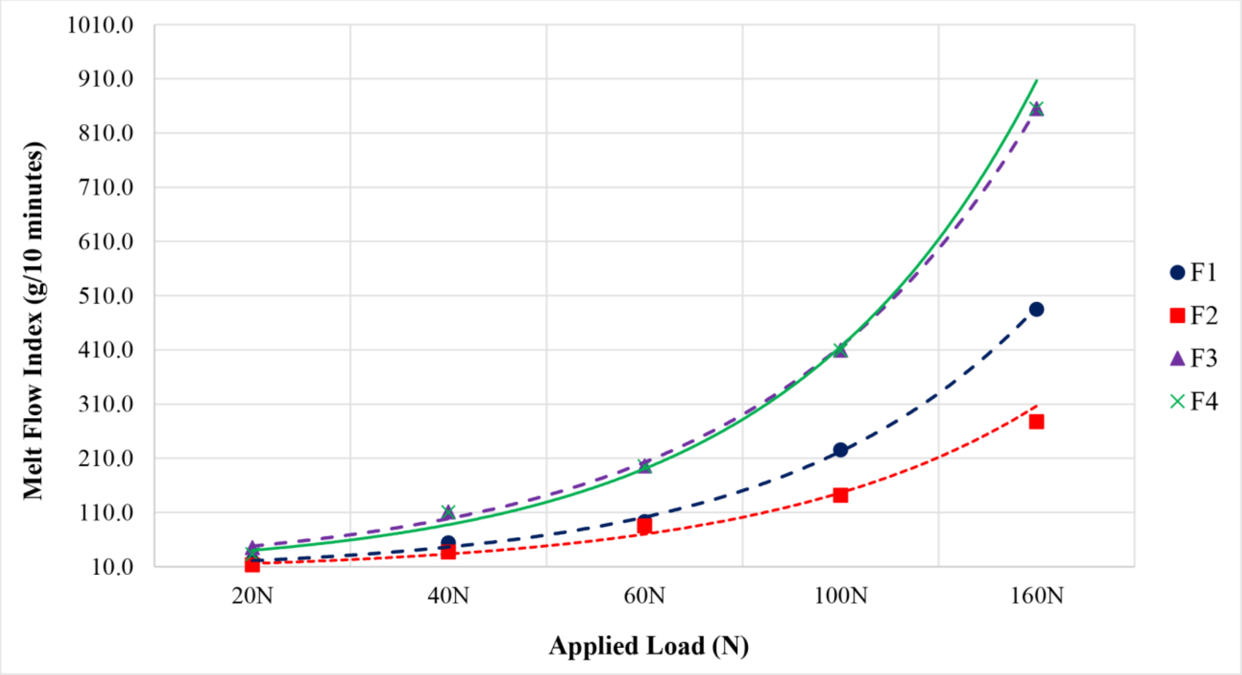

Melt Flow Index (g/10 minutes)
1010.0
910.0
810.0
710.0
610.0
510.0
410.0
310.0
210.0
110.0
10.0
20N
40N
60N
100N
160N
Applied Load (N)
● F1
■ F2
▲ F3
× F4

Figure 5

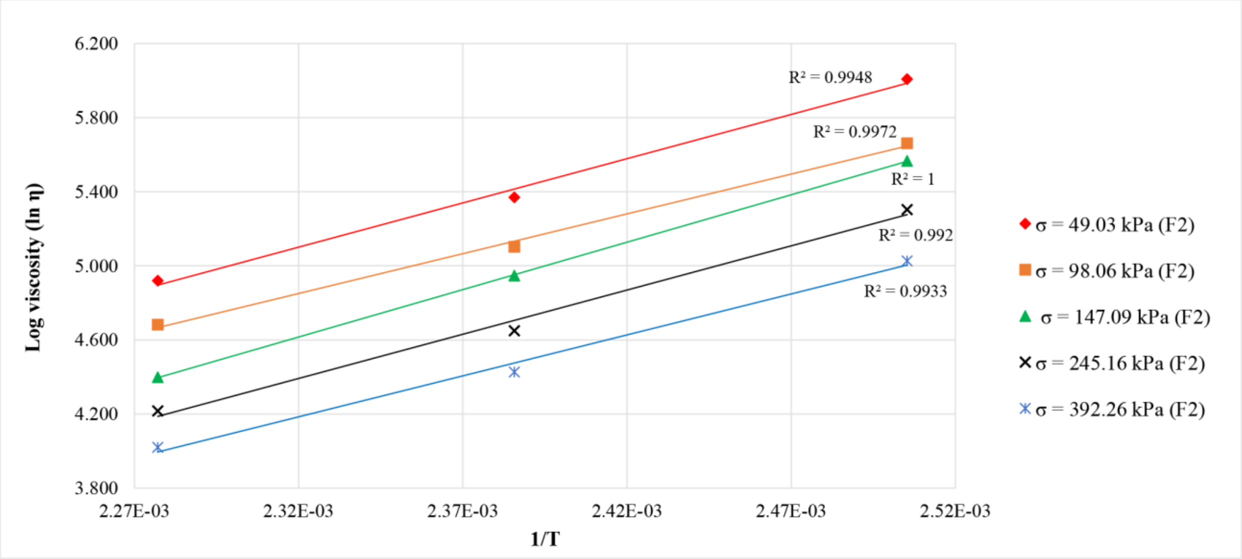

6.200
5.800
5.400
5.000
4.600
4.200
3.800
Log viscosity (ln η)
2.27E-03
2.32E-03
2.37E-03
2.42E-03
2.47E-03
2.52E-03
1/T
R² = 0.9948
R² = 0.9972
R² = 1
R² = 0.992
R² = 0.9933
σ = 49.03 kPa (F2)
σ = 98.06 kPa (F2)
σ = 147.09 kPa (F2)
σ = 245.16 kPa (F2)
σ = 392.26 kPa (F2)

Figure 6

Tables

Table 1: Feedstock formulations

Feedstock no.	Powder content (vol.%)	PEG (vol.%)	PVB (vol.%)	SA (vol.%)	Binder ratio (vol.%) (PEG:PVB:SA)
F1	55	33.8	9.0	2.3	75:20:5
F2	60	30.0	8.0	2.0	75:20:5
F3	55	36.0	6.8	2.3	80:15:5
F4	60	32.0	6.0	2.0	80:15:5

Table 2: Flow behaviour index of feedstock formulations

PEG: PVB: SA (vol.%)	Powder content 55 vol. %	Powder content 60 vol. %
75:20:5	0.64	0.68
80:15:5	0.70	0.63

Table 3: Fluidity of feedstock formulations at shear rate of 800 s^{-1}

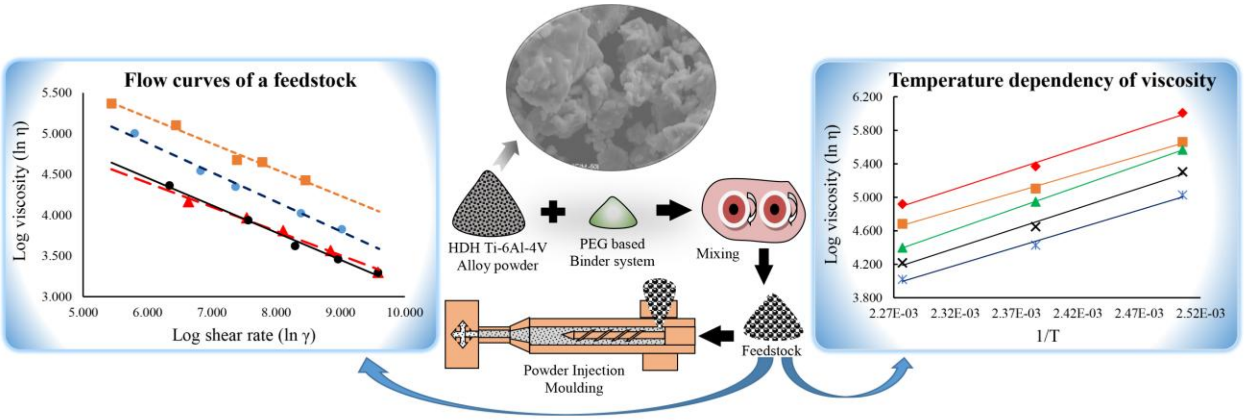
Feedstock	Fluidity (x 10^{-3} Pa^{-1}s^{-1})
F1	9.7
F2	6.9
F3	15.1
F4	14.6

Table 4: Melt flow index of various feedstock formulations at 140 ºC

Feedstock Formulation	Powder content (vol.%)	Binder ratio PEG:PVB:SA (vol.%)	MFI (g/ 10 minutes)				
			20 N	40 N	60 N	100 N	160 N
F1	55	75:20:5	19.5	54.2	93.3	225.6	484.9
F2	60	75:20:5	13.6	37.0	85.3	141.9	277.4
F3	55	80:15:5	45.0	111.1	195.8	409.5	855.1
F4	60	80:15:5	33.4	110.6	195.6	409.1	855.0

Table 5: Flow activation energy of feedstocks (kJ/mol)

Feedstock Formulations	Shear stress (Pa)				
	49030	98060	147090	245160	392260
F2	39.8	35.7	42.5	39.7	36.8
F4	27.8	29.7	27.2	26.8	14.8



Graphical abstract

Highlights

- A water soluble binder system for titanium injection moulding was developed.
- The rheological properties of the newly developed feedstock were reported.
- The feedstock formulation was optimised.