Rheological assessment of titanium MIM feedstocks

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Transaction Paper

Synopsis

Titanium is an exciting structural material that can offer significant strength-to-weight advantages over currently used alloys. However, its Achilles' heel is its costly, energy-intensive production process that effectively eliminates it from competing with aluminium and high-strength steels, apart from critical applications where titanium forms only a small component of the total cost. Current attempts are being made to reduce the cost of titanium products and these recognize the importance of minimizing the costs over the total production chain. Powder metallurgy (PM) technologies play a crucial role within this, as the output of the existing and potential primary metal production methods is in the form of sponge or powder. By using PM, costly remelting and forming operations can then be avoided, except in the manufacture of large components.

Metal injection moulding (MIM) is an effective process for producing complex net-shape components in large volumes from metal powders. Nevertheless, the commercial use of titanium powders in this process is still in its infancy. The only major supplier of feedstock utilizes a polyacetal-based binder. This gives good green strength but requires a catalytic nitric acid process to remove most of the binder prior to thermal treatment. As this involves additional and expensive equipment and is a potentially hazardous process, there is interest in finding an alternative binder system that can be debound either purely thermally or that involves a less hazardous, more environmentally friendly solvent.

This paper describes the use of capillary rheometry to characterize the influence of temperature and shear rates on the flow behaviour of potential binder systems for titanium MIM feedstock.

Keywords

Titanium, metal injection moulding, capillary rheology, feedstocks, powder loading.

Introduction

The development of a feedstock involves an interplay of multiple activities, as indicated in Figure 1.

One of the factors is to optimize the powder loading, which should be at or slightly below the critical value. A schematic representation of this optimum level is shown in Figure 2.

Several problems are encountered when the powder loading is too low, the excess binder can separate from the powder during injection moulding leading to inhomogeneities in the final component, low brown strength (after debinding) that can result in fracture or even collapse of the shape and large shrinkage during sintering. If the loading is too high, the viscosity becomes too high, resulting in difficulties in moulding and the formation of voids that result in cracking of the shape during debinding.

Powder loading is usually measured in terms of the volumetric ratio of powder to binder, according to the following formula[1].

$$\frac{w_p / \rho_p}{w_p / \rho_p + w_b / \rho_b}$$

where w_p and w_b are the weight fraction of powder and binder and ρ_p and ρ_b are the densities of the powder and binder respectively.

The critical loading can be determined by measuring the density, melt flow and the mixing torque or viscosity of the powder binder mixture. For example, Figure 3 shows the typical increase in viscosity as the critical loading is approached. The exact value will vary depending on the powder particle size and morphology.

If the torque values of the mixer are monitored during feedstock compounding, the mixture shows an increase in mixing resistance as the critical loading is approached. Figure 4 shows the significant increase when changing the loading from 50 to 60 vol% (i.e. approaching the critical loading) as compared to the difference between 33 and 50 vol%.

Based on the above discussion, it is clear that the effect of powder loading is an important criterion in the development of feedstock. A balance needs to be struck between having sufficient loading to maximize the strength in the debound state as well as

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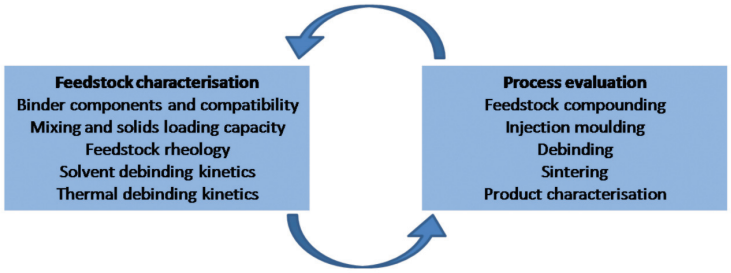

Figure 1—Activities involved in development of a MIM feedstock (adapted from[1])

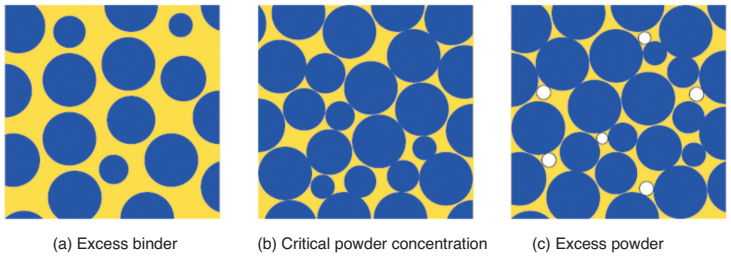

Figure 2—Schematic diagram showing the structures when the powder loading is too low (excess binder), optimal (critical), and too high (excess powder)[1]

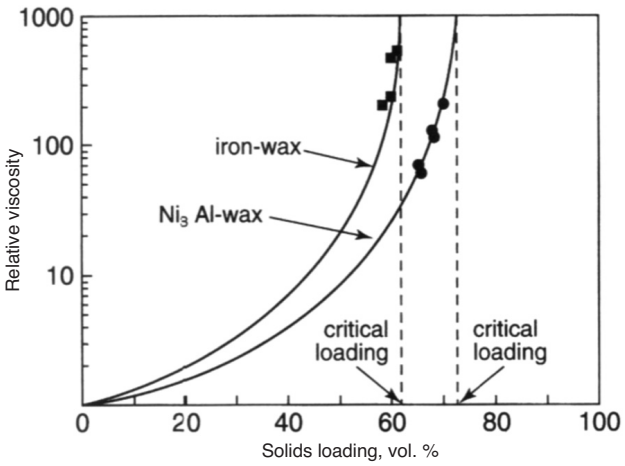

Figure 3—Effect of solids loading on feedstock viscosity for two powder types[2]

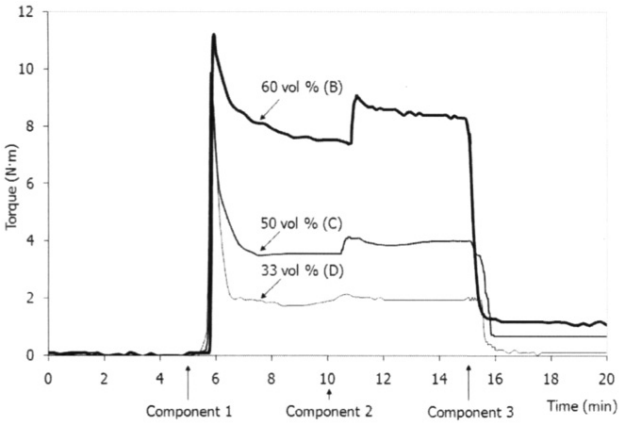

Figure 4—Torque evolution curves during compounding feedstock containing varying powder loadings at 180°C and 180 rpm[3]

the final density of the component, and maintaining adequate fluidity for good injectability. The first step in this process is to determine the critical loading value for the specific binder formulation and powder type.

Experimental procedure

A binder system, based on EVA (ethylene vinyl acetate) and a wax, was used to prepare feedstock with varying powder loadings. In all three cases the total mass of the binder components were kept constant, while only the mass of the metal powder was increased relative to the overall polymer content.

The various components that were used are the following:

Metal Powder: Ti 6Al 4V 25 µm atomized powder
Binder: EVA (Elvax 210), wax (Licomont TP EK), lubricant (stearic acid) and EVA plasticizer (Santicizer 261).

Three loadings were initially chosen i.e. 65 vol.%, 67 vol.% and 70 vol.%. The compositions that were used are given in Tables I–III

The rheological measurements were conducted with a Ceast SmartRheo® capillary rheometer (Figure 5) at shear rates between 900 and 7200 s^{-1}. The capillary diameter was 1 mm and the die length was 5 mm. The temperatures were varied according to the observed brittleness/fluidity of the resultant extrudate.

Experimental results

The results of the viscosity measurements of the various feedstocks are shown in Figures 6 to 8.

It was expected that the viscosity should decline with increase in temperature. However, this was not always observed. Figures 6 and 7 reveal an anomalous effect where the opposite can be seen to have occurred. This has been

Table I

Powder: Ti-6Al-4V 65 vol.%

	Density	Mass	m/o	Volume	v/o
Powder	4.42	210.00	89.04	47.51	65.0
LP	0.87	0.000	-	-	-
Elvax 210	0.93	8.966	3.80	9.64	13.19
Licomont	1.10	5.678	2.41	5.16	7.06
Stearic acid	0.94	2.241	0.95	2.38	3.26
Santi. 261	1.07	8.966	3.80	8.38	11.47
Total		235.85	100.00	73.08	100.00
Binder		25.85	10.96	25.57	

Table II

Powder: Ti-6Al-4V 67 vol.%

	Density	Mass	m/o	Volume	v/o
Powder	4.42	229.00	89.86	51.81	67.0
LP	0.87	0.000	-	-	-
Elvax 210	0.93	8.966	3.52	9.64	12.46
Licomont	1.10	5.678	2.23	5.16	6.67
Stearic acid	0.94	2.241	0.88	2.38	3.08
Santi. 261	1.07	8.966	3.52	8.38	10.83
Total		254.85	100.00	77.38	100.00
Binder		25.85	10.14	25.57	

Table III

Powder: Ti-6Al-4V 70 vol.%

	Density	Mass	m/o	Volume	v/o
Powder	4.42	250.00	91.08	56.56	70.0
LP	0.87	0.000	-	-	-
Elvax 210	0.93	8.490	3.09	9.13	11.30
Licomont	1.10	5.377	1.96	4.89	6.05
Stearic acid	0.94	2.123	0.77	2.26	2.80
Santi. 261	1.07	8.490	3.09	7.94	9.82
Total		274.48	100.00	80.77	100.00
Binder		24.48	5.40	24.21	

observed in some other binder systems and the reason for it is not clear. It may be due to a particular phase change in the binder constituents as it occurs below 100°C but is not observed at higher temperatures (Figure 8).

Nevertheless, a consistent effect of powder loading can be found when a shear rate of 900 s^{-1} is selected and the temperature to achieve a viscosity of 100 Pa.s is determined. Figure 9 shows the increasing temperature required as the loading is raised.

The 70 vol.% feedstock showed indications of having insufficient green strength and it was suggested that addition of more Elvax would be a possible solution. The powder loading was also reduced from 70 vol.% to 66 vol.%.

The composition of this new batch is given in Table IV.

Rheological tests results for this feedstock are given in Figure 10.

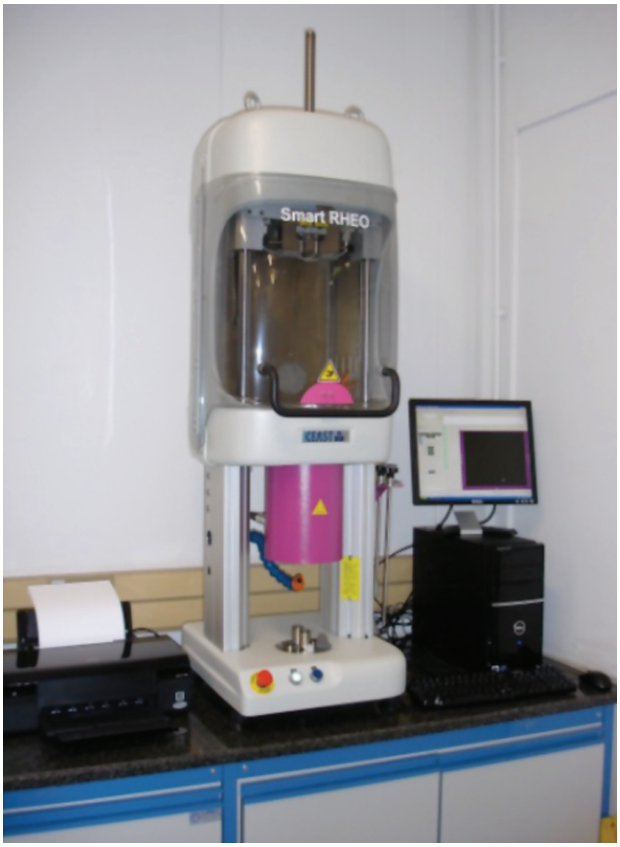

Figure 5—Ceast SmartRHEO® capillary rheometer

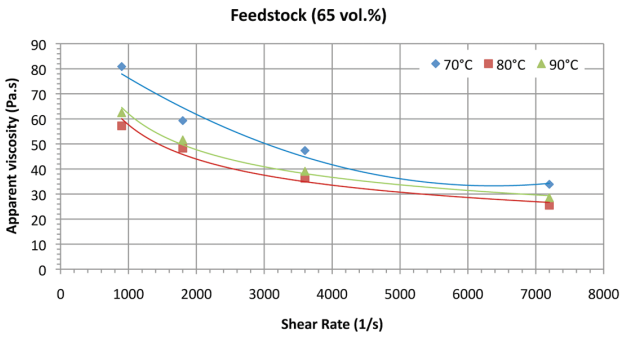

Figure 6—Capillary rheology results for feedstock containing 65 vol.% Ti-6Al-4V powder

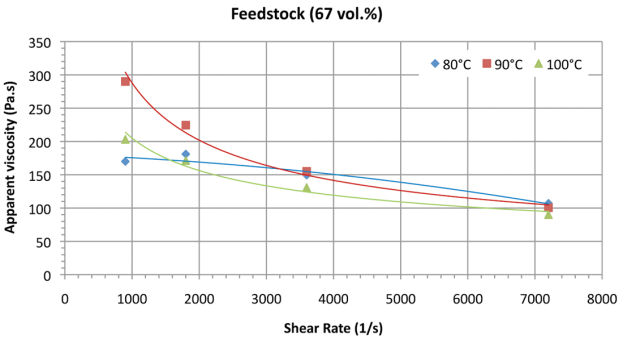

Figure 7—Capillary rheology results for feedstock containing 67 vol.% Ti-6Al-4V powder

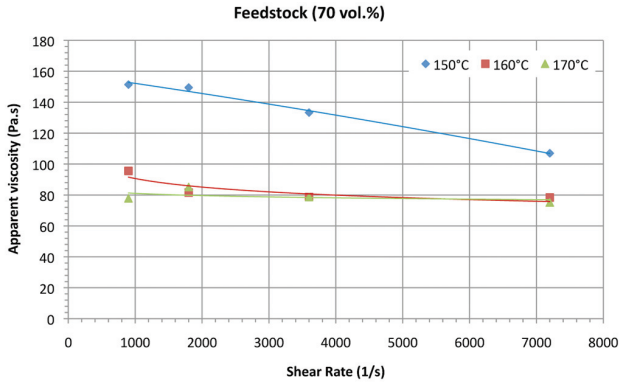

Figure 8—Capillary rheology results for feedstock containing 70 vol.% Ti-6Al-4V powder

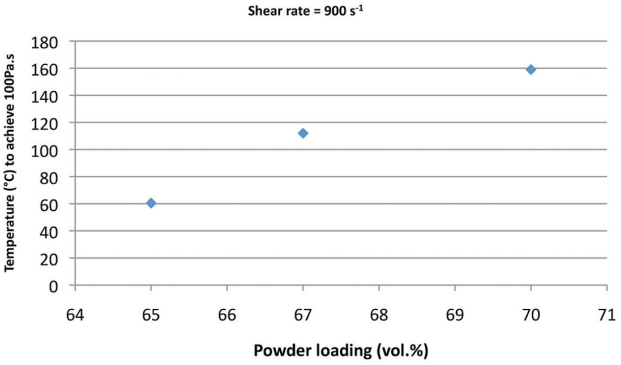

Figure 9—Effect of powder loading on the temperature required to obtain a viscosity of 100 Pa.s

Table IV

Powder: Ti-6Al-4V 66 vol.%

	Density	Mass	m/o	Volume	v/o
Powder	4.42	219.00	89.44	49.55	66.0
LP	0.87	0.000	-	-	-
Elvax 210	0.93	8.966	3.66	9.64	12.83
Licomont	1.10	5.678	2.32	5.16	6.87
Stearic acid	0.94	2.241	0.92	2.38	3.17
Santi. 261	1.07	8.966	3.66	8.38	11.16
Total		244.85	100.00	75.11	100.00
Binder		25.85	10.56	25.57	

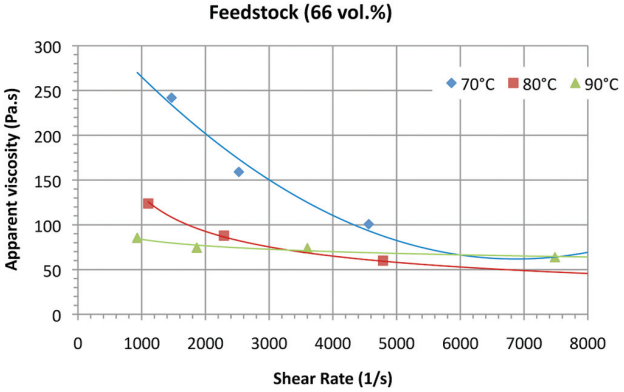

Figure 10—Capillary rheology results for feedstock containing 66 vol.% Ti-6Al-4V powder

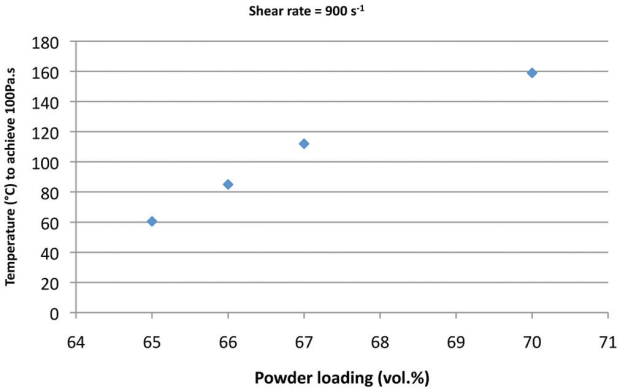

Figure 11—Effect of powder loading on the temperature required to obtain a viscosity of 100 Pa.s

The temperature required for the 66 vol.% feedstock to give a viscosity of 100 Pa.s was calculated and was seen to fit the previously determined powder loading vs. viscosity trend (Figure 11).

Conclusions

MIM feedstocks of Ti-6Al-4V powder and a wax-EVA binder were prepared with varying levels of powder loading.

An upper limit of approximately 70 vol.% powder loading has been suggested in literature. However, this is dependent on parameters such as powder particle size and shape as well as the binder system used. In the present work, the loadings chosen ranged from 65 to 70 vol.% and the viscosity measurements were determined in a capillary rheometer.

It was observed that at temperatures below 100°C the effect of temperature on fluidity was not predictable, as some stiffening was observed with increase in test temperature.

However, if a shear rate that is representative of the injection moulding process is selected (i.e. 900 s^{-1}), it was found that increasing the powder loading caused a significant rise in the temperature required to achieve the same viscosity (e.g. 100 Pa.s).

The optimum powder loading, based on the observed behaviour of this binder system, is considered to be between 66 and 67 vol.%.

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