Influence of binder composition on powder injection moulding process

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Doctoral Thesis Summary



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Influence of binder composition on powder injection moulding process

Vliv kompozice polymerního pojiva na proces vstřikování práškových materiálů

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ABSTRACT

Powder injection moulding (PIM) is a modern technological process combining powder metallurgy and plastic injection moulding. PIM is suitable for mass production of small metal or ceramic items, with complex geometry and tight tolerances. Polymer binders for PIM technology are still in the developing area due to the complexity of their requirements. One of the main challenges in PIM technology is optimization of PIM process and detection/elimination of powder binder separation resulting in inhomogeneous final products.

The research team at Tomas Bata University in Zlín under prof. Hausnerová has been involved in investigating the interactions and chemical mechanisms occurring within a binder system. The aim of this thesis is devoted to polymer binder composition based on previous results, including binder components characterization and their influence on PIM process. Particular emphasis was also placed on an eco-friendly approach and maximisation of solid loading.

Major component/s of binder systems provide low viscosity and easy removal in the first stage of debinding. Polyethylene glycol (PEG) is a suitable candidate due its non-toxicity, solubility in water and commercial availability. The first part was dedicated to the effect of PEG molecular weight on the overall PIM process. Results showed that PEG molecular weight affected processing parameters, but without any final impact on mechanical properties of sintered Inconel 718. Feedstock viscosity can be tailored via PEG molecular weight.

The second part was devoted to backbone components, especially to carnauba wax (CW) and acrawax (AW) as possible substituents of polyolefin based binder systems. Previous research showed twice stronger binding for AW/PEG than for CW/PEG, suggesting strong interactions between polymers. Feedstocks based on CW and AW showed overall lower viscosity than polyolefin-based ones and commercial feedstocks, and additionally a binder based on CW, could be used for reactive powders due to low processing temperatures.

Concentration of stearic acid (SA) as a surface active agent was investigated in third part in order to increase powder loading and improve flow properties of feedstocks. Surfactant SA positively affected processing parameters and its appropriate concentration is strongly dependent on binder composition. Optimal concentration of SA can increase maximal powder loading.

Binders are designed as multi-component polymer systems, in which each component performs a specific task. Better understanding of each component in this process can help eliminate some drawbacks, improve processing and produce less defective parts.

ABSTRACT IN CZECH

Vstřikování práškových materiálů (PIM) je moderní technologický proces kombinující práškovou metalurgií s vstřikováním plastů. PIM technologie je vhodná zejména pro hromadnou výrobu malých kovových nebo keramických dílů komplexního tvaru a s úzkou tolerancí. Polymerní pojiva pro tuto technologii jsou stále ve vývoji z důvodu komplexních požadavků. Jednou z výzev v této technologii je optimalizace procesu detekce a/nebo eliminace fázové separace, t má za následek nehomogenní finální produkty.

Vědecký tým na Univerzitě Tomáše Bati ve Zlíně pod vedením prof. Hausnerové se zabýval studiem interakcí a chemických mechanismů, ke kterým dochází v polymerním pojivu. Cíl této práce zaměřený na kompozici polymerního pojiva na základě předchozích výsledků, charakterizaci jednotlivých komponentů a jejích vliv na PIM proces. Klade důraz ekologické hledisko a zvýšení objemového plnění, které je taktéž důležitým aspektem této technologie.

Hlavní komponent polymerního pojiva zaručuje nízkou viskozitu a snadné odstranění pojiva v první fázi. Polyetylen glykol (PEG) je vhodným kandidátem, protože není toxický, je rozpustný ve vodě a je komerčně dostupný. První část této práce se zabývá vlivem molekulou hmotností PEG na celkový PIM proces. Výsledky ukázaly, že molekulová hmotnost PEG ovlivní procesní parametry, ale nebude mít vliv na finální mechanické vlastnosti sintrovaného Inconelu 718. Teplota při odstraňování polyetylen glykolu měla zásadní vliv na vznik defektů a na hustotu sintrovaných dílů. Viskozita PIM směsí může být nastavena na míru pomocí molekulové hmotnosti PEG.

Druhá část se zaměřuje na tzv. páteřní polymery, konkrétně se jedná o karnaubský vosk (CW) a acrawax (AW), jako možné substituenty pro polymerní systémy na bázi polyolefinů. Předchozí výsledky ukázaly dvojnásobně silnější vazby pro AW/PEG než pro CW/PEG, což naznačuje silnou interakci mezi polymery. Viskozita směsí na bázi CW a AW se snížila oproti viskozitě směsí na bázi polyolefinů a komerčního pojiva. Pojiva na bázi CW mohou být použitelná pro zpracování reaktivních prášků z důvodů nízké zpracovatelské teploty.

Koncentrace kyseliny stearové (SA) povrchově aktivní látky byla zkoumána v třetí části za účelem možnosti zvýšení práškového plnění a zlepšení tokových vlastností plněných směsí. SA pozitivně ovlivnila zpracovatelské parametry, ale její vhodná koncentrace bude silně záviset na složení polymerního pojiva.

Pojiva jsou navržené jako více komponentní systémy, ve kterých má každý komponent specifikou roli. Lepší porozumění jednotlivých složek v procesu pomůže eliminovat některé nedostatky, zlepšit proces a snížit výrobu defektů.

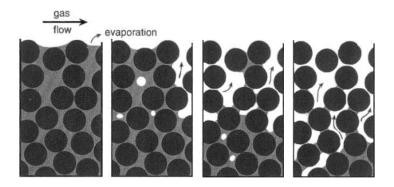
GLOSSARY

Basic terminology used in PIM technology:

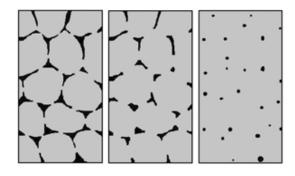
- **Powder** fine ceramic or metal particles
- **Binder** an individual component or a composition of polymeric materials
- **Feedstock** a mixture of powder and binder



- **Mixing** a process of compounding powder and binder into feedstock
- **Injection moulding** a process in which the feedstock is injection moulded into mould cavity
- **Debinding** a process in which the binder components are removed from moulded component



• **Sintering** – a thermal process during which powder particles are bonded and densified at a high temperature



1 Introduction to Powder Injection Moulding

Powder Injection Moulding (PIM) is an expanding technology for small ceramic or metal items of complex shapes with tight tolerances, which are expensive and/or difficult to produce by conventional methods [1-6]. PIM technology consists of four basic steps (Fig. 1).

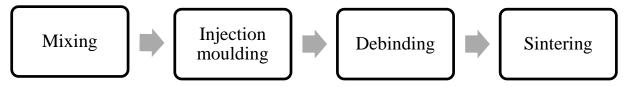


Fig. 1 Basic steps in PIM technology.

1.1 The role of binder in PIM process

A binder plays a crucial role during the entire PIM process. It has to fulfil several tasks and requirements [7-15]. Especially, a binder must allow flow of the powder into mould cavity, shape retention of moulded components and step by step removal. A typical binder is usually composed of: (i) major component/s, (ii) backbone, (iii) and additive/s. Each component performs a specific task.

1.2 PIM feedstock formulation

Fig. 2 shows the main important aspects which have to be considered for formulation of PIM feedstocks. The most important powder characteristics are particle size, shape and particle size distribution, as well as particle dispersion or agglomeration. Tab. 1 summarises typical binder components used in PIM technology.

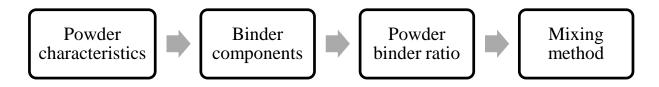


Fig. 2 The main aspects of PIM feedstocks.

Tab. 1 Most commonly used binder components used for PIM.

Major part	PW, CW, AW, BW, PEG, POM
Backbone	PP, PE, LDPE, HDPE, EVA
Additives/surfactants	SA, OA

Fig. 3 demonstrates three possible situations of powder binder ratio. According to German and Bose [3], optimal solid loading is proposed to be approximately 2 - 5 vol. % less than critical loading, while Dihoru *et al.* [16] recommends 7 - 8 vol. % less than critical. In both cases, they refer to a critical solid loading as a point prior to void formation.

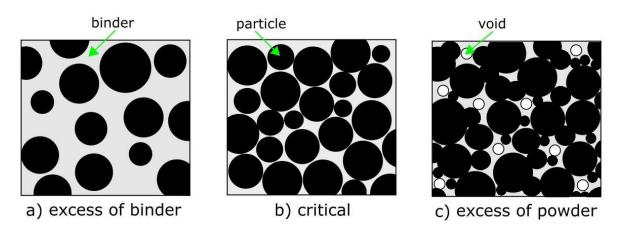


Fig. 3 Powder loading (a) excess, (b) critical, (c) insufficient.

1.3 Determination of critical powder loading

Measurement of mixing torque is a common method used for the experimental determination of critical solid loading. Fig. 4 demonstrates the evolution of the torque for different solid loading.

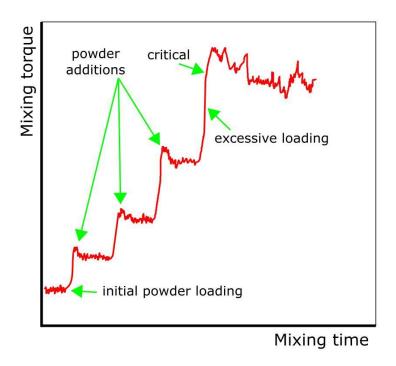


Fig. 4 Torque evolution by continuously increasing powder loading.

Critical powder loading from mixing torque can be obtained by two different ways. It can be carried out by continuously increasing powder loading in the binder system [17] or by increasing powder loading for each batch separately [18].

1.4 Rheological characterization of PIM feedstocks

Successful injection moulding of PIM feedstocks is dictated by its rheological behaviour. PIM feedstocks exhibit complex flow behaviour that depends on several factors [19][20]. PIM compounds are strongly shear rate dependent despite the fact that low molecular weight components in a binder behave like Newtonian fluids [21]. Rheological properties of PIM feedstocks can be obtained from both capillary and/or torque rheometers. Capillary rheometers offer a shear rate range usually from 100 to 10 000 1/s, which corresponds more to real conditions occurring during injection moulding. Fig. 5 shows the main factors affecting flow properties and consequently injection moulding process.

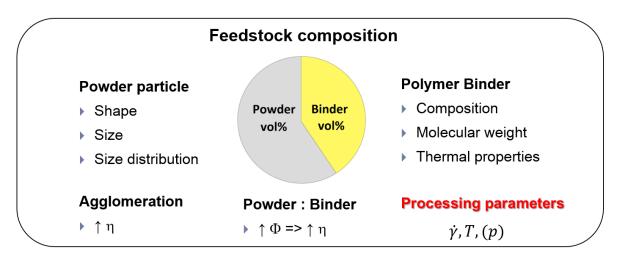


Fig. 5 Main factors affecting flow properties of PIM feedstocks.

The most appropriate method of studying the effect of a polymeric binder is by understanding the steady shear viscosity changes using different polymer systems, but with the same filler and at a fixed level of loading [22]. Feedstock viscosity can be possibly enhanced by a high molecular weight of the binder involved.

One of important requirements on PIM feedstocks is their low temperature sensitivity [23]. Smaller activation energy is desirable in order to diminish the problems, such as process fluctuation, and further stress concentration, cracks and shape distortion. Hayat *et al.* [24] found out that activation energy of feedstock decreased as PEG molecular weight increased.

2 AIM OF THE THESIS

The aim of this doctoral thesis characterization and optimization of binder systems used for PIM technology with emphasis on binder composition, rheological properties and an eco-friendly approach in order to achieve high quality final products. The main framework focuses on accomplishing the following partial aims:

Effect of PEG molecular weight on the PIM process of Inconel 718 based feedstocks.

- Rheological characterization of PIM feedstocks
- Effect of PEG molecular weight on debinding rate at different removal temperatures
- Effect of PEG molecular weight on mechanical properties after sintering and post-heat treatment

Investigation of newly developed binder systems for PIM based on AW and CW as possible substitutes of polyolefin backbone polymers.

- Rheological characterization
- Thermal analysis

Impact of SA on powder loading and flow properties of PIM feedstocks

- Evaluation of powder loading
- Effect of powder loading on flow behaviour
- Influence of SA concentration on feedstock powder loading
- Influence of SA concentration on flow behaviour

A summary of feedstock compositions used in this work (detailed formulation of the feedstocks is described in each section):

• Effect of [major part] → various PEG molecular weight

Powder	Binder
Inc718 (59 vol. %)	PEG/PMMA/SA

• Effect of [backbone] binder → LDPE, CW, AW vs. commercial binder

Powder	Binder
Al ₂ O ₃ (50 vol. %)	LDPE PW PEG OA SA
	Licomont SA

• Effect of [surfactant] \rightarrow various concentration of SA (0 - 5 wt. %)

Powder	Binders					
Torque: Al ₂ O ₃ (50 - 60 vol. %) Rheo: Al ₂ O ₃ (52 vol. %)	LDPE	EVA	PW	SA		
	CW	PEG	PVV	SA		
	Licomo	ont		SA		
Rheo: ZRO ₂ (52 vol. %)	Licomont		SA			

• Evaluation of [**powder loading**] → Al₂O₃, 17-4PH on critical volume and flow properties

Powder	Binder
Al_2O_3 (50 – 60 vol. %)	Licement
17-4PH (60 - 70 vol. %)	Licomont SA

3 RESULTS AND DISCUSSION

In order to investigate the overall area of PIM and explore the influence of binder components as well as powder loading, the presented chapter on the results and discussion within the experimental part was divided according to individual objectives of the thesis into the following sections:

- Effect of [major component]
- Effect of [backbone]
- Effect of [surfactant] including evaluation of powder loading

Each section consists of a brief introduction, results and discussion.

The presented results have been also published in the following papers:

I. Rheological Investigation of Highly Filled Polymers: Effect of Molecular Weight

Hnatkova E., Hausnerova B., Hales A., Jiranek L., Alcon J. M. *Novel Trends in Rheology VI*, 1662 (2015) 040003-1-04040003-8.

II. Processing of MIM Feedstocks based on Inconel 718 Powder and Partially Water-Soluble Binder Varying in PEG Molecular Weight Hnatkova E., Hausnerova B., Hales A., Jiranek L., Derguti F., Todd, I. *Powder Technology*, 322 (2017) 439-446.

III. Effect of Backbone Binders on Rheological Performance of Ceramic Injection Molding Feedstocks

Hausnerova B., Kasparkova V., Hnatkova E. *Polymer Engineering and Science*, 57 (2017) 739-745.

IV. Impact of Stearic Acid on Powder Loading and Flow Properties of Ceramic Injection Molding Feedstocks

Hnatkova E., Hausnerova B.

Submitted to Ceramics International in 2019

3.1 Effect of major component

The main objective of this section was to investigate the effect of PEG molecular weight on overall the PIM process chain including its eventual impact on final mechanical properties of Inconel 718 (see paper I, II). This part of research was based on joint work with the Department of Materials Science and Engineering at Sheffield University. For this purpose, 7 feedstocks with a fixed amount of powder loading (59 vol. %) and the same binder composition differing in PEG molecular weight (in a range from 1,500 to 20,000 g/mol) were investigated. Fig. 6 shows a schematic view of feedstock composition.

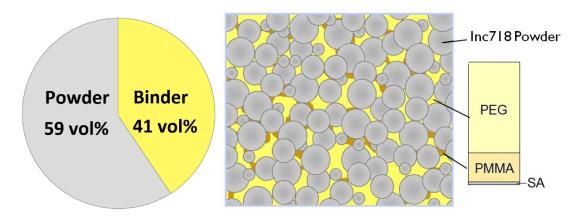


Fig. 6 Illustration of PIM feedstock composition based on Inconel 718.

The binder system was designed for a two-stage debinding process. The major component (PEG) should provide the feedstocks with low viscosity simultaneously enabling its easy removal in water, the backbone polymer (PMMA) ensures the strength of the moulded part, and the surfactant (SA) facilitates lubrication. The formulation of feedstock compositions is summarized in Tab. 2.

Tab. 2 Composition	of feedstocks	based on different PEG mo	lecular weights.
	5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	- $ -$	

Feedstock	Powder (59 vol. %)	Binder system (41 vol. %)				
		83 wt. %	15 wt. %	2 wt. %		
P1.5K	Inconel 718	PEG 1 500	PMMA	SA		
P4K	Inconel 718	PEG 4 000	PMMA	SA		
P6K	Inconel 718	PEG 6 000	PMMA	SA		
P8K	Inconel 718	PEG 8 000	PMMA	SA		
P10K	Inconel 718	PEG 10 000	PMMA	SA		
P12K	Inconel 718	PEG 12 000	PMMA	SA		
P20K	Inconel 718	PEG 20 000	PMMA	SA		

Rheological characterization

❖ Flow and viscosity curves

Fig. 7 shows shear stress and viscosity dependence on shear rate at three temperatures. All feedstocks demonstrate pseudoplastic behaviour in the measured shear rate range suitable for injection moulding. What can also be noticed is that the shear rate sensitivity of most of the feedstocks tends to diminish at higher shear rates; especially at 120 °C, it seems that viscosity curves even approach the 2nd Newtonian plateau.

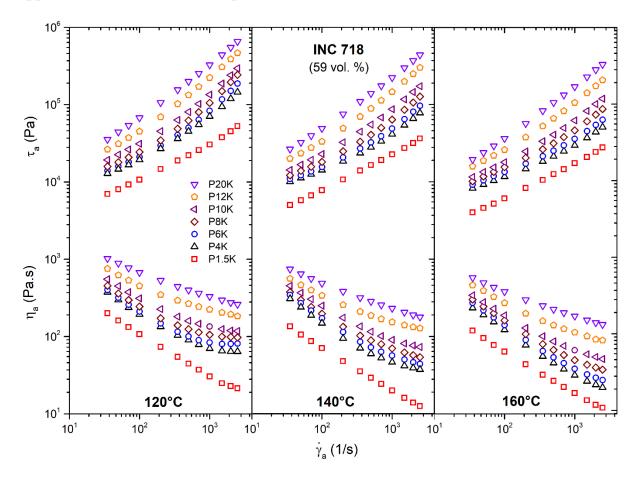


Fig. 7 Effect of PEG molecular weight on flow behaviour of Inc718 feedstocks.

At a higher temperature, the shear stress decreases due to a greater free volume available for molecular motion and easier disentanglement of the polymeric chain in the flow direction [25]. Moreover, it reduces friction among particles because the thermal expansion of the polymeric binder is higher than that of the powder resulting in slightly lower powder loading [26]. Nevertheless, a higher temperature is not always favourable; *e.g.* Yang *et al.* [27] reported that feedstocks based on PEG 1 000 and 1 500 demonstrate dilatant flow above 80 °C as a result of possible powder binder separation.

In addition, the viscosity of feedstocks also decreases with lower molecular weight of PEG due to shorter polymeric chains allowing more mobility between particles. These results are in accordance with the previous findings reported by Hayat *et al.* [24]. The viscosity of their feedstocks based on PEG 1500 and 4000 was also extremely low and demonstrated dilatant flow from certain shear rates at elevated temperatures, which could indicate powder binder separation.

❖ Viscosity-temperature dependence

Fig. 8 illustrates the obtained activation energies as a function of both, apparent shear rate $(E_{a,\dot{v}})$ and apparent shear stress $(E_{a,\tau})$.

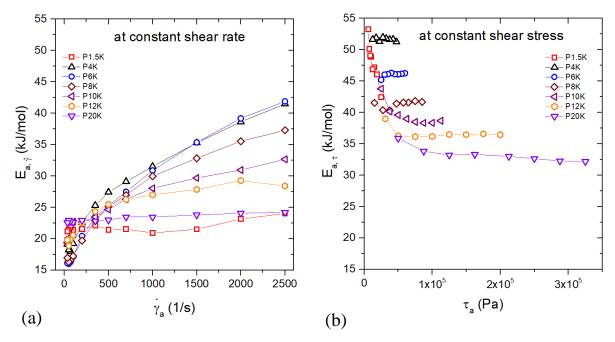


Fig. 8 Activation energy of feedstocks at constant: shear rate (a) and constant shear stress (b).

In both cases, the feedstock viscosity temperature dependence decrease with higher molecular weight of PEG, except feedstock P1.5K. As can be seen, the flow activation energy at a constant shear rate (Fig. 8a) is shear dependent. Oppositely, flow activation energy at constant shear stress (Fig. 8b) demonstrated constant values from a certain shear stress.

The shear dependence of activation energy at a constant shear rate diminishes with higher PEG molecular weight and in case of feedstock P20K its value is almost constant. Different trends for activation energy obtained at a constant shear rate or constant shear stress have been already reported by some authors [22] [28]. Similar results of activation energy at a constant shear rate were obtained by Yan *et al.* [27] and more recently by Hayat *et al.* [24], who tested feedstocks based on

PEGs (1 500 - 20,000 g/mol). Based on the results in this study as well as on studies performed by other authors investigating the impact of PEG molecular weight it can be concluded that PEGs with higher molecular weight will be more suitable for injection moulding from the processing stability point of view.

***** Entrance and exit effects

Fig. 9 shows feedstock dependence of pressure drops on apparent shear rate obtained with 3 capillaries of the same diameter (1 mm) and different lengths of 5, 10 and 20 mm.

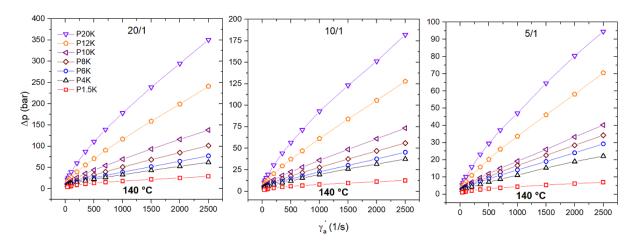


Fig. 9 Measured pressure drop of all feedstocks using three capillaries.

Fig. 10 shows an example of measured pressure drops for individual feedstock plotted in the "Bagley plots" as a function of L/D ratio at a given shear rate.

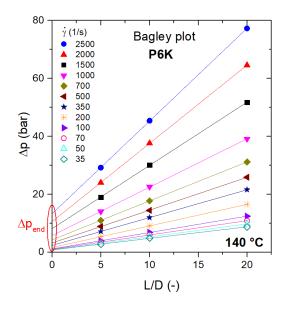


Fig. 10 Extrapolation of end effects from Linear Bagley plots.

Direct measurements of end effects were carried out using a zero "orifice" die of the same diameter and with a nominal length of 0.2 mm. However, the nominal length was neglected for further calculation. Fig. 11 shows a comparison of results between direct measurement (zero die) and indirect extrapolation (Bagley).

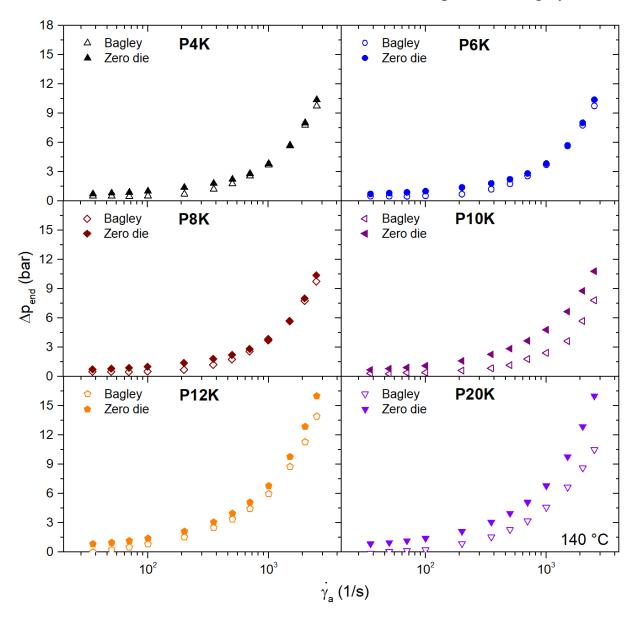


Fig. 11 Comparison of end effects obtained from direct and indirect measurements.

As can be seen, both methods bring almost the same values of end effects with a slight deviation in case of feedstocks P10K and P20K. For further comparison or corrections, the end effects obtained from the zero die were considered.

Tab. 3 summaries measured pressure drops of feedstocks for selected capillary dies at a shear rate of 2500 1/s and highlights influence of end effects pressure drops on measured pressure.

Tab. 3 Measured pressure drops Δp in bar at shear rate of 2500 1/s using various capillary lengths and impact of end effects on it in %.

L/D	0/1	20/1		10/1		5/1	
Feedstock	Δp_{end} (bar)	Δp (bar)	∆p (%)	Δp (bar)	∆p (%)	Δp (bar)	∆p (%)
P4K	10.4	62.2	17	37.5	28	22.1	47
P6K	13.9	77.3	18	45.4	31	29.2	48
P8K	14.3	101.5	14	56.1	26	34.2	42
P10K	10.8	138.2	8	73.4	15	40.2	27
P12K	16.0	241.0	7	127.8	13	70.5	23
P20K	16.7	350.2	5	182.1	9	94.6	18

The values in the highlighted columns show impact of end effects ($\Delta p_{\rm end}$) on measured pressure drop (Δp) in %. End effects of feedstocks constituted with lower molecular weight of PEG will have greater impact on Bagley correction influencing corrected shear stress. This means, that comparison of apparent values of viscosity curves can bring incorrect results.

❖ Non-Newtonian index

The Non-Newtonian index (n) can vary from point-to-point, or it can be constant at all data points in the shear rate range measured. Therefore, the main issue is how to properly determine it from discontinuous data set of τ_c and $\dot{\gamma}_a$ measured experimentally. Fig. 12 shows for comparison an example of fitted data based on different approximate functions (linear and non-linear approximation).

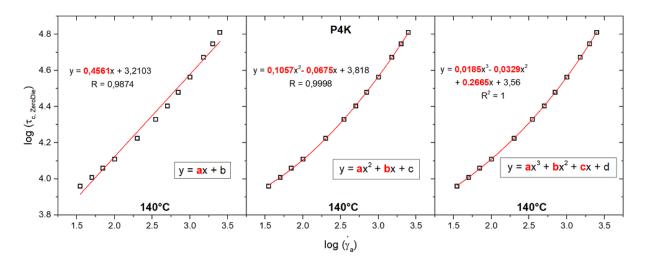


Fig. 12 Linear and non-linear approximate functions of non-Newtonian index.

Tab. 4 lists non-Newtonian indexes derived from linear and non-linear functions.

Tab. 4 Effect of approximate function (linear or polynomial regression) on the value of Non-Newtonian index as an example for the P4K at 140 °C.

$ au_c$	Ϋ́a	$\log au_c$	Log γ _a	n - (1 st)*	$n - (2^{nd})^*$	n (3 rd)*
(Pa)	(1/s)	(-)	(-)	(-)	(-)	(-)
64738	2500	4.81	3.40	0.46	0.65	0.69
55915	2000	4.75	3.30	0.46	0.63	0.66
47073	1500	4.67	3.18	0.46	0.60	0.62
36548	1000	4.56	3.00	0.46	0.57	0.57
30166	700	4.48	2.85	0.46	0.53	0.53
25288	500	4.40	2.70	0.46	0.50	0.49
21331	350	4.33	2.54	0.46	0.47	0.46
16756	200	4.22	2.30	0.46	0.42	0.41
12875	100	4.11	2.00	0.46	0.63	0.36
11453	70	4.06	1.85	0.46	0.32	0.33
10191	50	4.01	1.70	0.46	0.29	0.32
9128	35	3.96	1.54	0.46	0.26	0.30

^{*} Non-Newtonian index obtained from (1st), (2nd) and (3rd) polynomial fitting.

As can be seen, for this type of feedstock there is a huge difference of values of non-Newtonian indexes obtained depending on the selective approximate function. Fig. 13 shows a graphical comparison of non-Newtonian indexes obtained for selected feedstocks at 140 °C from the linear regression with those obtained from 2nd polynomial fitting as the function of shear rate.

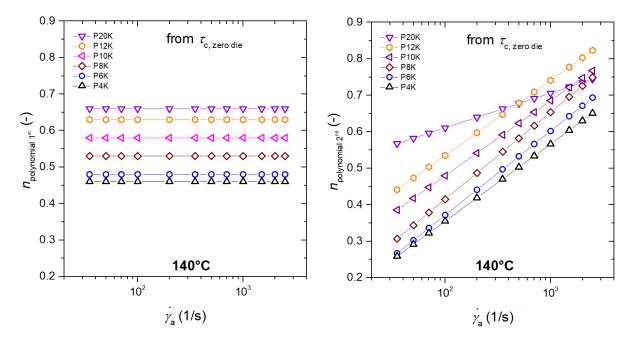


Fig. 13 Non-Newtonian indexes obtained from the (a) linear and, (b) 2^{nd} polynomial fitting in dependence on apparent shear rate.

***** Corrections of capillary flow

Fig. 14 demonstrates differences between apparent and corrected viscosity curves. As can be noticed, the corrected viscosity curves are more affected with a higher molecular weight of PEG. This is due to higher influence of end effects considering the results from Tab. 3. One of them is slip wall, which was not taken into consideration in this study, but is often present in processing of compounds with high solid loading [29].

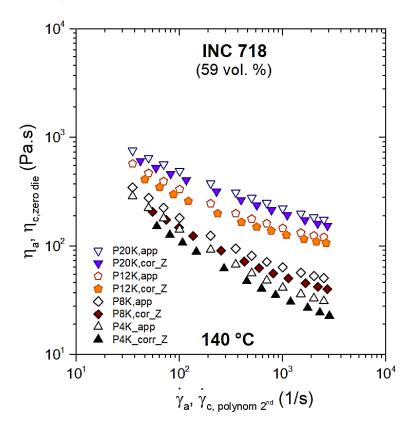


Fig. 14 Apparent and corrected viscosity curves for selected Inc718 feedstocks.

Debinding at different temperatures

Fig. 15 displays the PEG removal (weight loss in % due to PEGs leaching from moulded tensile specimens) in a water bath at three temperatures as functions of time. There was a rapid removal rate in the first hours; afterwards the PEG leaching gradually slowed down. Moulded specimens from feedstocks containing PEG with higher molecular weights required a prolonged time to achieve corresponding weight loss. Higher temperatures of water (50 and 60 °C) accelerated the debinding rate, however resulting in a few part defects. In case of tensile samples prepared from feedstock P1.5K particular defects were found inside the tensile specimens indicating a probability of void formation during

solidification in the previous processing step. Similar internal defects were also found by other researchers [30][31].

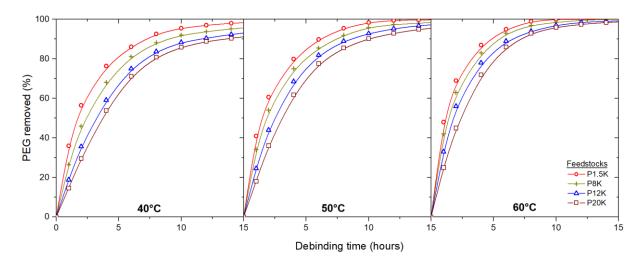


Fig. 15 PEG removal rate during water debinding at three temperatures.

Introductions of other defects at higher debinding temperatures can have a correlation with a rapid enlargement creating internal stresses due to thermal expansion, and then consequently structural damage. This finding is also reported by German [3] demonstrating that a too high debinding rate results in a strength loss, formation of cracks and blisters, distortion or even collapse of the compact.

In previous research, Liu *et al.* [25] examined the effect of PEG 600 and PEG 4,000 in binder composition and found that during water debinding at 40 °C the PIM parts formulated with PEG 4,000 resulted in surface defects originated from swelling, such as blistering and cracking, while using PEG 600 brought difficulties to maintain the shape of the component.

Park *et al.* [32] investigated water-soluble binder systems consisting of cellulose and PEG; moulded components from feedstocks containing low molecular weight PEGs exhibited excellent shape retention during extraction in water at room temperature, while the components containing PEG with molecular weights higher than 8,000 exhibited cracks at the edge of the moulded parts because of swelling during PEG removal. Similar results were obtained by Hayat *et al.* [24], their samples containing PEG 20,000 significantly swelled and cracked during water debinding at 50 °C. Based on these studies and confirmed by our results, components based on PEGs with a higher molecular weight are more prone to cracks and blisters during the debinding process.

Tensile testing of sintered and post-heated treatment samples

Fig. 16 shows a variation of Inconel 718 sintered densities based on debinding temperatures.

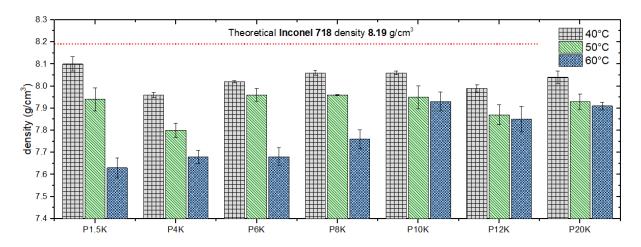


Fig. 16 Densities of sintered Inc718 based on water debinding temperatures.

As can be observed, there is a huge impact of water debinding temperatures on Inconel 718 component density after sintering. Particularly, the components from feedstocks with lower molecular weight of PEG reached lower densities at water debinding at 60 °C in comparison with those debinded at 40 °C. This phenomenon might be explained by larger thermal expansion and swelling, which probably increased the pore size among powder particles and caused more defects. Due to the formation of defects during water debinding at higher temperatures, tensile properties were measured on sintered and heat treated tensile bars previously debinded at 40 °C.

The results from tensile testing, are shown in Fig. 17. As can be seen, PEG molecular weight did not affect the final mechanical properties.

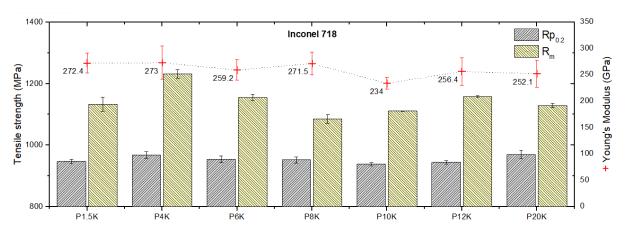


Fig. 17 Tensile properties of sintered Inc718 previously debinded at 40 °C.

3.2 Effect of backbone

In this section newly developed binder systems contacting carnauba wax (CW) or acrawax (AW) were investigated as possible substituents of polyolefin based binders for PIM feedstocks (see paper III). Ceramic powders are about one order of magnitude smaller than metallic ones and they are more prone to form agglomerates, as can be seen in Fig. 18. Moreover, small size and irregular particle shape enhance the resistance to flow. This leads to increased demands on the selection of binder composition which will guarantee low viscosity, and simultaneously good adhesion to powder.

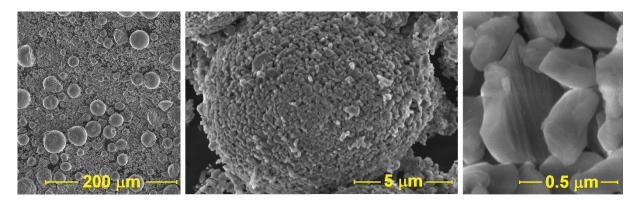


Fig. 18 SEM image of Al_2O_3 – tendency to agglomeration.

In previous research, AW and CW exhibited higher interactions of AW/PEG blend and superior debinding properties with gradual thermal extraction up to late debinding [33][34]. Based on this ground, these two candidates were selected as backbone components for further investigation in terms of rheological and thermal behaviour. Ceramic feedstocks based on novel proposed binders were evaluated and compared with feedstocks containing LDPE or a commercial binder with respect to their suitability for injection moulding.

All analysed feedstocks were formulated with a fixed amount of Al₂O₃ (50 vol. %) and various binder compositions, as listed in Tab. 5. Initial polyolefin based feedstocks were designed with LDPE, PW and PEG of the same or different molecular weights. In the newly developed feedstocks, the LDPE was substituted with CW or AW. Additionally, all feedstocks adopted in this study contained 1 wt. % of surfactant stearic acid (SA) or oleic acid (OA).

Tab. 5 Binder composition (content of each component is given in wt. %).

In-house	LDPE	CW	AW	PW		PEG		SA	OA
Binder					6000	4000	1000		
LDPE 1	40	-	-	10	49	-	-	1	-
LDPE 2	40	-	-	10	25	14	10	1	-
LDPE 3	40	-	-	10	25	14	10	-	1
Carnauba 1	-	40	-	10	49	-	-	1	-
Carnauba 2	-	40	-	10	25	14	10	1	-
Carnauba 3	-	40	-	10	25	14	10	-	1
Acra 1	-	-	40	10	49	-	-	1	-
Acra 2	-	-	40	10	25	14	10	1	-
Acra 3	_	-	40	10	25	14	10	_	1
Commercial	Licomont								OA
Binder	99							1	-

Rheological characterization

The flow behaviour of feedstocks based on in-house binders with various compositions is shown in Fig. 19. In addition, feedstock based on a commercial binder is also considered for comparison (Fig. 20). As can be seen, viscosity dependence on a shear rate of investigated ceramic feedstocks is rather complex. Pseudoplastic behaviour was observed only in a specific shear rate range. All viscosities were bellow1 000 Pa.s, which is, according to German [1], suitable for injection moulding from processing point of view. Moreover, feedstocks based on CW and AW showed a significant reduction in feedstock viscosity, which is about one magnitude lower in comparison with polyolefin and Licomont based feedstocks.

In case of LDPE based feedstocks, viscosity dependence on a shear rate shows pseudoplastic behaviour up to a shear rate of about 500 1/s then the flow turns into dilatant in the shear rate region approaching real processing conditions. On the other hand, both novel feedstocks based on CW, AW including a commercial Licomont exhibit pseudoplastic behaviour in this range. The pseudoplastic regions indicate that polymeric chains and powder particles are orienting in the same direction with flow. The abrupt change of the flow course of LDPE based feedstock was satisfactorily explained by Hoffman as early as 1972 [35] as the consequence of inability of particles to form layers and slide over each other at a certain shear rate. However, the flow of wax (CW and AW) and Lincomont based

feedstocks is even more complex. Their viscosity curves show overshoots at certain shear rates. The onset of overshoots was postponed to higher shear rates at higher processing temperatures.

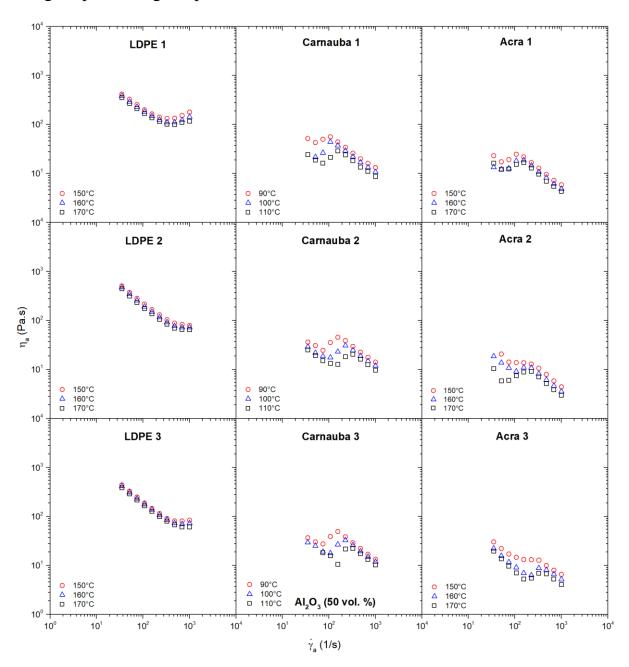


Fig. 19 Flow properties of Al_2O_3 feedstocks (50 vol. %) based on binders with LDPE, CW or AW as backbone component.

The overshot is caused by particle structure reorganization, and a mathematical model has been proposed recently, which was employed successfully for fitting of rheological data of Al_2O_3 with a commercial binder Licomont [36]. Additionally, CW as a backbone component allows processing of feedstock

around 100 °C, which is beneficial in comparison to polyolefin or AW based binder systems.

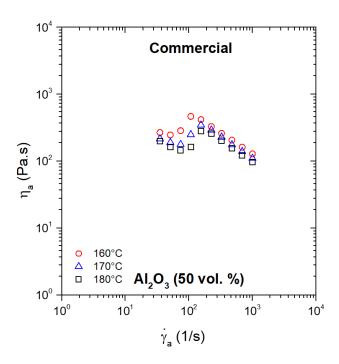


Fig. 20 Flow properties feedstock based on Al_2O_3 and commercial binder.

Considering the effect of various PEG molecular weights, the flow of traditional polyolefin (LDPE2) based feedstock is only affected in the dilatant region, which is in case of various molecular weights of PEG postponed towards higher shear rates. In case of CW (Carnauba2) based feedstocks, combination of various PEGs shifted the transition from pseudoplastic to dilatant and back to pseudoplastic flow towards higher shear rates (about 100 1/s). From processing point of view, a high molecular weight PEG (PEG 6,000 in our case) in combination with CW shows better performance. However, in case of AW (Acra2), various PEG combinations bring viscosity decrease in the overall measured shear rate range.

Finally, role of surfactant (OA and SA) was considered. These two surfactants are often used with a presumption that their effect on the flow properties will be the same. In case of binder components studied within this work, it can be confirmed. The overall performance of the investigated novel feedstocks is in accordance with previous research [33][37] as well as with findings obtained from contact angle measurement of wettability of the respective binders [34].

Thermal properties

Thermal characterization in terms of melting temperatures ($T_{\rm m}$) was conducted using different scanning calorimetry (DSC), which is a suitable technique for assessing polymer miscibility. Fig. 21 shows DSC curves of analysed samples obtained from the $2^{\rm nd}$ heating scan to avoid thermal history of the samples. Any change of the peaks, such as a shift of melt temperatures may disclose occurrence of interactions between individual polymers, their mixtures and the powder.

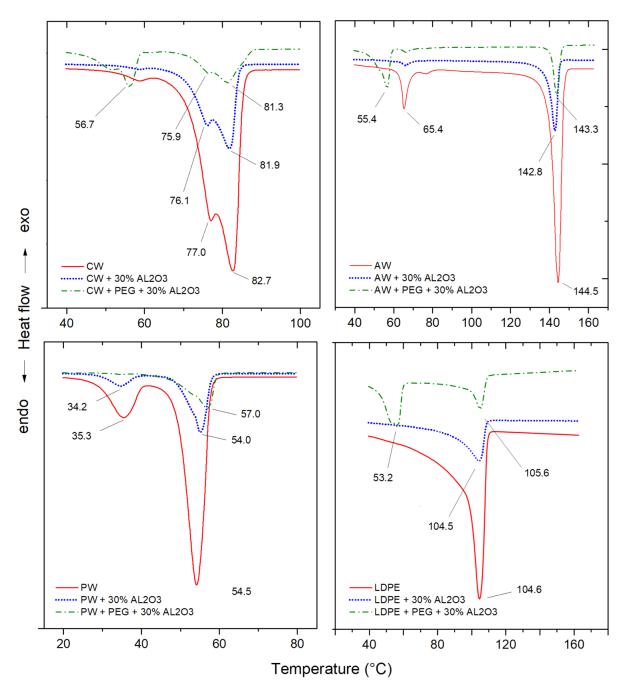


Fig. 21 DSC curves of each analysed samples from the 2nd heating scan.

DSC curves show different thermal behavior of studied components. They differ both in melting temperatures ($T_{\rm m}$) and the course of melting. Adding Al₂O₃ into a binder, it lowered $T_{\rm m}$ of mixtures (binder/Al₂O₃) compared to $T_{\rm m}$ of individual binders. The shift was most pronounced for PEG/Al₂O₃ with a decrease of about 3 °C. No change was observed for LDPE/Al₂O₃. The presence of ceramic particles within a binder can hinder mobility of polymeric chains, which could initiate a reduction of their crystalline domains and consequently causing a $T_{\rm m}$ depression.

The three-component mixtures (binder/Al₂O₃/PEG) behaved in different ways. All samples (apart from that containing PW) showed two clearly separated endothermic peaks, one originating from PEG and the other from the respective binder (AW, CW or LDPE). The uniform thermal behaviour might be favourable when using these two components in more complex samples. The melting peaks originating from PEG in mixtures were displaced to lower temperatures a $T_{\rm m}$ depression of PEG was the greatest observed in LDPE/Al₂O₃/PEG.

Fig. 22 clearly shows differences in behaviour of both wax-based feedstocks (CW, AW) compared to those containing hydrocarbons, *i.e.* PW and LDPE. Taking into account that Al₂O₃ powder is of a polar character, as reported by Bleyan *et al.* [37], it seems credible that both CW and AW containing polar groups enable better miscibility with PEG and powder compared to PW and LDPE.

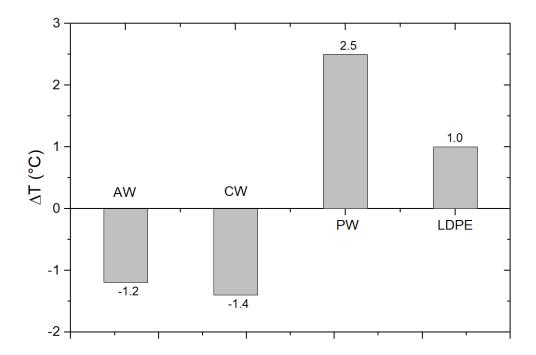


Fig. 22 Change in peak melting temperature ΔT of three-component mixture (binder/Al₂O₃/PEG) relatively to corresponding individual binder.

3.3 Effect of surfactant

In this section, an impact of SA concentration on powder loading and flow properties of PIM feedstocks was investigated (see paper IV). Feedstocks were formulated with various amount of Al_2O_3 (50 - 60 vol. %) and three types of binder compositions A, B and C, in which concentrations of SA varied from 0 to 5 wt. %, as listed in Tab. 6.

Ceramic powders with submicrometric size and irregular shape represent a very high specific surface area [5]. This behaviour tends to reduce powder loading, while it is desirable to mix as much powder as possible in order to avoid distortion, as well as to lower volumetric shrinkage.

Additionally, this part also includes an evaluation of powder loading and effect of powder loading on flow behaviour. In this case mixing torque measurement was carried out via continuously and separately increasing powder loading in order to compare both methods. Two different types of powder were used: a highly irregular ceramic Al₂O₃ and a spherical 17-4 PH stainless steel powder. The composition of binder in this case consists of the commercial Licomont (98 wh. %) and SA (2 wt. %), labelled as binder type B.

A	PEG	CW	PW	SA
	45-50	40	10	0-5
В	Licomont			SA
	95-100			0-5
C	LDPE	EVA	PW	SA
	48-53	26	21	0-5

Tab. 6 Composition of binder components used (wt. %).

Binder A is designed with all components having a melting temperature below 100 °C, which facilitates the processing of Al₂O₃ powder and prevents from polymer degradation [38]. Polar CW was selected due to its enhanced interaction with PEG and Al₂O₃ powder [39]. This should prevent phase separation during injection moulding, which is one of the most important issue in PIM technology.

Binder B is based on the commercially available binder Licomont. This binder was selected to test a possible improvement with addition of processing aids and to compare it with other in-house binder compositions.

Binder C is a polyolefin based binder system previously investigated by Bleyan *et al.* [39] and providing stearic stabilization with copolymer EVA.

Evaluation of powder loading

❖ Mixing torque - continuously increasing powder loading

Both Fig. 23 and Fig. 24 demonstrate a mixing torque obtained by continuously increasing solid volume loadings. When the powder is added to the melted binder, there is a certain resistance to the rotation of the blades resulting in an increase of torque. Then, once the homogeneity of the mixture becomes uniform, the mixing torque achieves a steady state value [40].

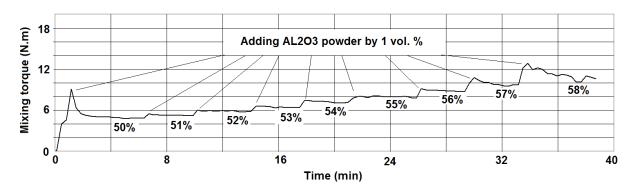


Fig. 23 Mixing torque obtained by continuous increasing volume of Al_2O_3 .

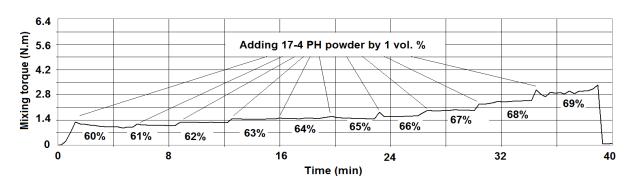


Fig. 24 Mixing torque obtained by continuous increasing volume of 17-4 PH.

As can be observed, the torque of mixture based on ceramic A_2O_3 reached stable values of up to 56 vol. %. Exceeding this loading, the torque starts to be unstable, and at 58 vol. % becomes completely erratic. A similar situation could be observed for stainless steel powder 17-4 PH. However, the unstable torque was shifted to a higher value of solid loading and became unstable with a tendency to increase after 66 vol. %. Then, reaching powder loading of 69 vol. %, the mixing torque became completely erratic.

* Mixing torque - separately increasing powder loading

Fig. 25 illustrates an example of mixing torque as a function of time for batch mixing, where powder loading was increased in the whole volume for each time separately.

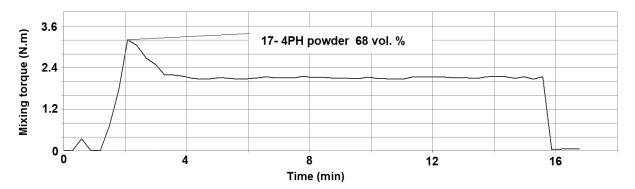


Fig. 25 Mixing torque obtained by separately increasing 17-4 PH volume.

* Comparison of both methods

Fig. 26 shows a comparison between mixing torque evolution obtained by separately and continuously adding powder. Both methods demonstrated same starting points from which, the rapid increase mixing torque occurred. This can indicate closer particles being in contact during processing, therefore increasing resistance and thus the mixing torque more rapidly.

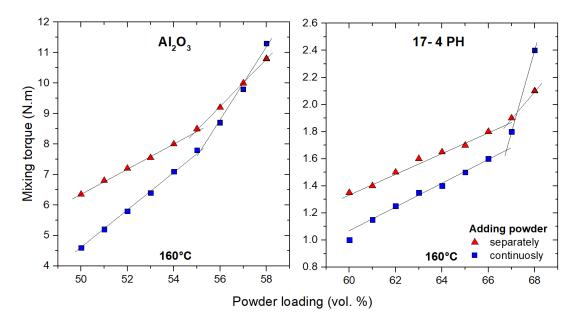


Fig. 26 Comparison of mixing torques obtained by two methods.

In both cases there is almost a linear increase in torque with higher solid loading. However, for all feedstocks, there is an abrupt in the slope at certain

powder loading. Then, the torque progression raises up to a certain limit, where it starts to decay or becomes erratic. The decay can be explained by introducing voids among powder particles as the solid loading reaches the critical solid loading value.

Fig. 27 demonstrates a SEM picture of a highly filled mixture based on both Al₂O₃ (58 vol. %) and 17-4PH (68 vol. %) loading. As can be seen, all powder particles are covered with the binder. Nevertheless, it cannot be observed if there are already voids in the binder or not.

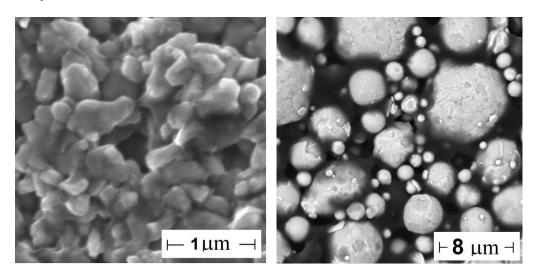


Fig. 27 SEM of feedstocks: Al_2O_3 , 58 vol. % (a) and 17-4PH, 68 vol. % (b).

Volumetric solid loading of the powder in feedstock formulations strongly varies for ceramic and metals. These results are in accordance with previous findings. Usually, powder loading of ceramic particles varies between 50 and 55 vol. %, but it is strongly dependent on powder characteristics and binder composition [3].

Effect of powder loading on flow behaviour

Fig. 28 shows flow properties of feedstocks based on A₂O₃ and17-4PH with different powder loading. The viscosity of feedstock based on A₂O₃ increases with an enhanced amount of powder, and overshot was postponed to a lower shear rate range. However, from certain solid loading (57 vol. %) it was not possible to measure the pressure generated, as can be seen in Fig. 29. These instabilities can be caused probably due to slip on the wall and/or as a consequence to exceeding optimal solid loading. In the case of 17-4PH, the viscosity was stable for all powder loadings measured.

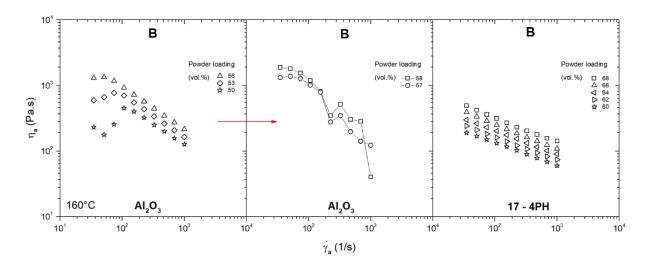


Fig. 28 Flow behaviour of feedstocks based on Al_2O_3 and 17-4PH with different powder loading.

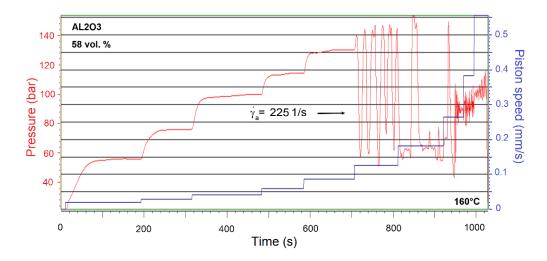


Fig. 29 Raw (online) data on rheometer of mixture with 58 vol. % of Al₂O₃.

Influence of SA concentration on feedstock powder loading

Fig. 30 demonstrates the evolution of mixing torques for all three binder systems as a function of both powder loading and concentration of SA. As can be seen, the critical solid loading corresponding to the decay in torque is clearly visible for all three binder systems without SA: 56 vol. %, 57 vol. % and 58 vol. % for feedstock A, B and C, respectively.

In this work, critical powder loading was considered the point, where the mixing torque drops down off the linear increasing function due to formation of voids or become erratic. To ensure an appropriate mould filling during injection moulding, the optimal solid loading was considered lower to this critical solid loading.

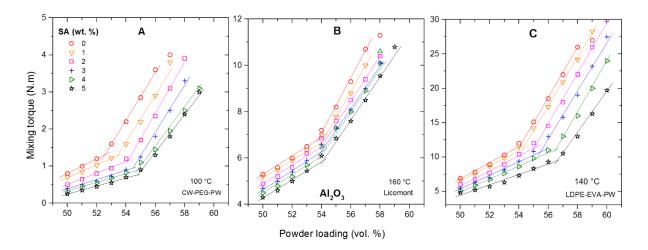
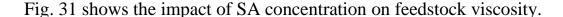


Fig. 30 Obtained torques based on powder loading and different concentration of SA for binder A, B and C.

Moreover, it can be seen that in case of binders A and C, a rapid increase in mixing torque (which could be considered as optimal solid loading) depends on SA concentration. For the binder B, no change in the rapid increase in mixing torque was found. In case of binder A, obtained results showed that addition of 3 wt. % SA increased optimal solid loading in the tested feedstocks from 52 to 55 vol. %, in case of binder C 3 and 5 wt. % of SA raised this value from 54 to 55 and 56 vol. %, respectively. As shown recently [18], the role of SA surfactant might be, to some extent, substituted with CW, because it contains esters of stearic acid with high fatty alcohols, and exhibits very similar surface properties.

Influence of SA concentration on flow behaviour



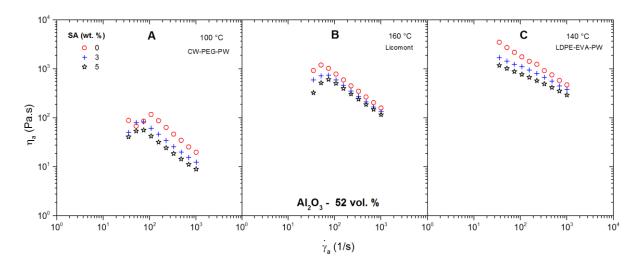


Fig. 31 Effect of SA on viscosity curves of feedstocks with 52 vol. % of Al₂O₃.

All measured feedstocks exhibited pseudoplastic behaviour at higher shear rates. Nevertheless, the feedstocks based on binder A and B demonstrated an overshoots in the viscosity at a certain shear rate, which were shifted to higher shear rates for SA treated compounds. In this respect, SA has a similar effect on flow behavior of alumina feedstocks as the temperature [41].

Feedstocks based on binder A (CW/PW/PEG) can be processed at lower temperatures (*i.e.* around 100 °C) and provides suitable viscosity, which is more than one order magnitude lower than that of the feedstock based on commercial binder. Similar values of viscosities in the corresponding shear rate range were obtained by Kong *et al.* [18] for stainless steel powder in CW/PW/PEG binder.

The viscosity of feedstocks based on commercial binder B can also be, to some extent, modified by SA. For fitting rheological data of this commercial binder, the recently proposed mathematical model [36] may also reflect the effect of SA. In addition, flow properties of the feedstock based on ZRO₂ (52 vol. %) with binder B were investigated as well. As can be seen in Fig. 32, viscosity of zirconia feedstocks was even higher and flow properties demonstrated more instabilities in the flow course. Difficulties in measuring can be caused by high tendency for agglomeration, which made almost impossible their measurement.

Viscosity of feedstock based on binder C (LDPE/EVA/PW) exceeded the value suggested by German and Bose [3] as suitable for PIM.

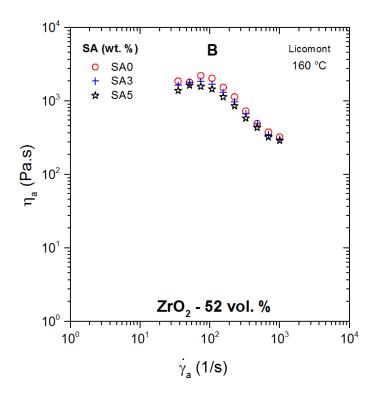


Fig. 32 Effect of SA on viscosity curves of feedstocks with 52 vol. % of ZRO₂.

CONCLUSION

The aim of this thesis is devoted to polymer binder composition, including individual binder components characterization and their influence on powder injection moulding (PIM) process. Particular emphasis was also placed on an eco-friendly approach and maximisation of solid loading as one of the most important aspects in PIM technology. The overall framework was divided into three sections according to typical composition of a polymeric binder in which each component performs a specific task: effect of major component (i), backbone (ii) and additive (iii). Binders used in this work were designed as multi-component polymer systems with respect to previous results of a research team at Tomas Bata University in Zlín, Czech Republic and in collaboration with the Department of Materials Science and Engineering at Sheffield University, United Kingdom.

A comprehensive investigation of Inconel 718 based feedstocks demonstrated the important role of polyethylene glycol (PEG) molecular weight during PIM process. Feedstocks with various molecular weights of PEG demonstrated suitable viscosity for injection moulding due to pseudoplastic behaviour in the shear rate range measured. Higher molecular weights of PEG increased viscosity of feedstocks. On the other hand, these feedstocks were less viscosity temperature sensitive. The flow activation energy at a constant shear rate was shear dependent and increased with a higher shear rate and lower PEG molecular weight. Oppositely, the values of flow activation energy at a constant shear stress tended to be constant approaching a higher shear stress. No significant differences were found between indirect and direct measurements of end effects. Flow and viscosity curves are often based on apparent values, i.e. without corrections to capillary flow due to comparative character. However, it was found that the impact on a corrected shear stress is strongly dependent on PEG molecular weight used. Attention should also be paid to adequate approximate function for derivation of non-Newtonian indexes.

PEG as a major component soluble in water can be removed in the first stage of debinding by water leaching. This will create a network of interconnected pores, through which residual binders would be later thermally removed. Higher temperatures of water bath accelerated water debinding of PEG from moulded components. On the other hand, a negative aspect of acceleration was introduction of defects into debinded components. Internal voids were found, especially in case of feedstock composed from PEG 1 500, while other types of defects, such us cracking and blistering during water debinding appeared in samples composed with higher molecular weights of PEG. Sintered density of Inconel 718 tensile

samples was also affected by water debinding temperatures resulting in lower sintered density for samples debinded in water at higher temperatures. No significant differences were found in mechanical properties of sintered and heat treated Inconel 718 tensile samples, previously debinded in a water bath at 40 °C.

After the first stage of debinding, when the major part of the binder is removed, an important role is played by the backbone component/s, which has to assure shape stabilization of the component prior to sintering. In a novel binder system, carnauba wax (CW) and acrawax (AW) were considered to replace synthetic polyolefin backbone polymers, such as low density polyethylene (LDPE), enhancing the environmental sustainability of PIM technology.

Flow behaviour of investigated ceramic feedstocks was rather complex. Polyolefin based feedstocks demonstrated pseudoplastic flow up to a certain limit, then at higher shear region (approaching the real processing conditions) the flow turned into dilatant. On the other hand, both novel feedstocks based on CW, AW as well as feedstock based on the commercial binder exhibited pseudoplastic behaviour in this range. Their viscosity curves showed a sudden change in rheological behaviour (overshoots) at lower shear rates. With higher temperature, the onset of these overshoots was postponed to higher shear rates. An overshot is probably caused by internal particle structure reorganization. Both novel multicomponent binder systems, based on CW and AW, showed lower viscosity in comparison with feedstocks based on LDPE (as backbone component) or based on the commercial binder. Additionally, CW based feedstocks allow processing temperatures around 100 °C, which can be essential for injection moulding of reactive powders.

Thermal analysis was carried out via different scanning calorimetry (DSC) (from the 2nd second heating scans to eliminate thermal history of samples) in order to observe interaction between individual components (individual binder components, their mixtures and their combination with ceramic powders) within feedstocks. The presence of Al₂O₃ powder lowered melting temperatures of all tested binders except from LDPE. This may be related to changes in size of the crystalline domains of binders, caused by the presence of other components in the blends. The results obtained clearly showed differences in thermal behaviour of both wax-based feedstocks containing CW and AW in comparison to those containing hydrocarbons paraffin wax (PW) a LDPE, which are strictly non polar. Taking into account that Al₂O₃ powder has a polar character, it may indicate that both CW and AW containing polar groups provide better miscibility with PEG and the powder compared to paraffin PW and LDPE. Stronger cohesion can be beneficial for avoiding phase separation between the powder and the binder.

The powder binder separation can also be minimized by addition of small amounts of surface active agents, which lower the contact angle between the powder and binder resulting in better wettability of the powder. Surfactants create a thin layer composed of chemical absorption bonds on the surface of the powder allowing easier particle sliding that reduces viscosity of feedstocks and facilitate the processing during mixing and injection moulding. Moreover, surfactants can also enhance packing density resulting in higher powder loading. It is one of the options of how to modify critical powder loading instead of changing powder characteristics, because critical powder loading also depends on binder composition. Obtained results showed that incorporation of surfactant SA in binder composition positively affected processing parameters by lowering feedstock viscosity and consequently mixing torque as well. In order to receive higher critical solid loading, the amount of sufficient concentration of SA was strongly dependent on the type of the binder. In case of the commercially available binder Licomont, the influence of concentration of SA on the onset of a rapid increase in the mixing torque was negligible, but it reduced the viscosity of feedstock. In feedstock A, based on CW/PEG/PW, there was a notable shift of a rapid increase of mixing torque between concentrations of 0 and 3 wt. % of SA, while in using binder C based on LDPE/ethylene vinyl acetate (EVA)/PW there was a huge difference in all concentrations of SA resulting in a better interparticle arrangement with a higher amount of surfactant.

The powder loading of a feedstock plays a key role in the PIM process. Stability of feedstocks is highly sensitive to solid loading and a minimum amount of binder concentration leads to better shape retention, reduced shrinkage after solidification and a tight dimensional tolerance control. On the other hand, if the solid content is higher than critical powder loading, this may result in increased wear and process instability during the injection or debinding stage. For an evaluation of critical powder loading it is suitable to use a combination of both methods, mixing torque and rheology measurements. The onset of a rapid increase in powder loading could also be a good indicator for optimal solid loading.

In summary, this work contributed to deeper understanding of each component in PIM process. PEG molecular weight affected all processing parameters, but without final impact on mechanical properties of sintered Inconel 718. Water debinding temperature influenced sintered density and creation of defects. Feedstocks based on CW and AW showed overall lower viscosity than the polyolefin-based LDPE and the commercial feedstocks. Surfactant SA affected positively processing parameters, but its effect on critical loading strongly depends on binder composition.

CONTRIBUTION TO SCIENCE AND PRACTICE

Binder systems for PIM technology are still in a developing phase due to their requirement complexity. There are still challenges to eliminate drawbacks, improve the PIM process and produce less defective parts. The results of this study can serve for setting up suitable compositions of polymeric binders, debinding parameters and/or designing binder formulations with relevant PEG molecular weights. However, proper selection has to be always related to powder loading, powder properties, component size as well as to overall part shape complexity. PIM is an interdisciplinary approach combining polymer processing with powder technology, and not always all specifications for a given field are taken into account. For numerical simulations the values obtained from rheological measurements have to be corrected, and the present thesis offers guidelines on how to proceed with rheological evaluation (apart from phase separation).

The following findings of the presented thesis can be considered as important contributions for PIM process and science:

- Feedstock viscosity can be tailored via PEG molecular weight. The water debinding temperature for PEG removal influenced creation of defects and sintered density.
- Backbone components AW and CW are suitable, especially for micro injection moulding due to low feedstock viscosity. Feedstocks based on CW can find utilization in processing reactive powders due to low processing temperatures.
- Higher concentrations of SA can increase maximal powder loading.
- Eco-friendly approach in designed binders.

The results presented in the present thesis were also published in international scientific journals and at conferences.

Ongoing research and future perspectives

Ongoing research is carried out with mathematical modelling of PEG based feedstocks of different molecular weights. This means that viscosity of feedstocks could be tailored via PEG molecular weight. In all instances of newly developed binders it would be worth to test their tendency to phase separation. In case of CW and AW as backbone binders it is necessary to test their shape stability during late stages of debinding at higher temperatures.

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LIST ABBREVIATIONS AND ACRONYMS

Abbreviations

Al₂O₃ Aluminum oxide (alumina)

AW Acrawax BW Beeswax

CIM Ceramic injection moulding

CW Carnauba wax

DSC Different scanning calorimeter/y

EVA Ethylene vinyl acetate

HDPE High density polyethylene

Inc718 Inconel 718

LDPE Low density polyethylene MIM Metal injection moulding

OA Oleic acid PE Polyethylene

PEG Poly(ethylene glycol)

PIM Powder injection moulding PMMA Poly(methyl methacrylate)

POM Polyoxymethylene

PP Polypropylene PW Paraffin wax SA Stearic acid (SA)

SEM Scanning electron microscopy ZrO₂ Zirconium dioxide (zirconia)

Symbols

A Pre-exponential factor

a, b, c, d Parameters

CSL Critical solid loading

D Diameter

E Young's modulus

 E_a Flow activation energy

 $E_{a,\dot{\gamma}}$ Flow activation energy at constant shear rate $E_{a,\tau}$ Flow activation energy at constant shear stress

F Force

 F_m Maximum force

L Capillary length

n Non-Newtonian index

 $R_{\rm m}$ Tensile strength $R_{\rm p0.2}$ Proof strength

SD Standard deviation

SP Switch point Temperature

 $T_{\rm m}$ Melting temperature

Greek letters

 Δp Total pressure drop

 $\Delta p_{\rm end}$ End effects

 ΔT Change in peak melting temperature

 η Viscosity of filled system

 $\eta_{\rm a}$ Apparent viscosity $\eta_{\rm c}$ Corrected viscosity $\dot{\gamma}_{a}$ Apparent shear rate $\dot{\gamma}_{c}$ Corrected shear rate

 ε Strain

 τ Shear stress

 τ_a Apparent shear stress τ_c Corrected shear stress

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