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Processing of MIM feedstocks based on Inconel 718 powder and partially water-soluble binder varying in PEG molecular weight

Eva Hnatkova [a,b], Berenika Hausnerova [a,b,*], Andrew Hales [c], Lukas Jiranek [d], Fatos Derguti [c], Iain Todd [c]

[a] Department of Production Engineering, Faculty of Technology, Tomas Bata University in Zlín, nám. T. G. Masaryka 5555, 760 01 Zlín, Czech Republic
[b] Centre of Polymer Systems, University Institute, Tomas Bata University in Zlín, Tř. Tomáše Bati 5678, 760 01 Zlín, Czech Republic
[c] The University of Sheffield, Department of Materials Science and Engineering, Sir Robert Hadfield Building, Mappin Street, Sheffield S1 3JD, United Kingdom
[d] Beckett MIM, Tinsley Industrial Park Shepcote Way Sheffield, S9 1TH, United Kingdom

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ABSTRACT

This paper deals with a complex characterization of metal injection molding (MIM) feedstocks based on Inconel 718 powder and partially water-soluble binder systems. Polyethylene glycol (PEG) is a well-known component in design of feedstock formulations due to its low viscosity, solubility in the water, eco-friendly nature and commercial availability. The main objective of this paper is to investigate the influence of PEG molecular weight on overall MIM process chain including its eventual impact on final mechanical properties. For this purpose, 7 feedstocks with a fixed amount of powder loading (59 vol%) and the binder composition differing in molecular weight of PEG (in a range from 1500 to 20,000 g/mol) were investigated. The PEG molecular weight was found to have the considerable impact on the feedstock moldability and debinding kinetics.

1. Introduction

Polymeric binder plays an important role in MIM process. First, binder has to ensure uniform distribution of metal particles, sufficient cohesion, and stable flow properties during injection molding. At this stage, it has to guarantee dimensional and shape stability of injected parts. Second, binder removal should be carried out step-by-step without shape distortions or cracking [1,2]. In order to fulfil these requirements, polymeric binders are designed as multi-component systems in which each component performs a specific task. Typical binder system is composed of (i) major component/s providing low viscosity, (ii) backbone ensuring the strength, and (iii) small amount of additive/s improving the wettability of feedstocks.

Adoption of water-soluble solvents in debinding process appears as a valuable alternative to organic solvents, which are often environmentally unacceptable. This also illustrates the fact that since 1976 over 130 patents concerning water-soluble binders have been submitted [3]. In this respect, polyethylene glycol (PEG) is one of the most relevant components in the design of partially soluble binders for MIM due to its environmentally benign nature and commercial availability. PEG is a semicrystalline polyether available in various molecular weights. At the molecular weights smaller than 1000, PEGs are viscous, colorless liquids, while higher molecular weight PEGs are waxy, white solids. Molecular weights typically employed in MIM range from hundreds to 20,000, and they are proportional to melting temperatures [4].

Binder system based on PEG was firstly reported by Cao et al. [5]. Then, Hens and German [6] examined the role of PEG in feedstocks aimed at simplifying processing, and shape retention during debinding. Hence, the water-soluble binders based on PEG have been widely investigated for both metal and ceramic feedstocks. The dimensional variation of alumina injection molded compacts containing PEG binders during water debinding was examined by Yang and Hon [7]. Sidambe et al. [8] analyzed the possibility to produce titanium components for biomedical applications using a binder system consisting of PEG, poly (methyl methacrylate) (PMMA) and stearic acid (SA), while Hidalgo et al. [9] tested water-soluble binder based on PEG for low pressure injection molding of water-soluble zirconia feedstocks. Recently, Royer et al. [10] investigated degradation effect of PEG during MIM process and analyzed the miscibility of binder components. Although, PEG based feedstocks are well documented in literature, there is only a limited scientific research relating to the effect of PEG molecular weight on a complex MIM process.

In order to select optimum feedstock composition, Liu et al. [11] examined the effect of PEG 600 and PEG 4000 in feedstock composition for fabrication of large-section ceramic parts. They found out some drawbacks of each PEG during debinding, therefore proposed to use a combination of both (66.7% PEG 600 + 33.3% PEG 4000). Rheological behavior and temperature dependence of feedstocks containing various PEGs (1000; 1500; 4000 and 20,000) were studied also by Yang et al. [18].

* Corresponding author.
E-mail address: hausnerova@ft.utb.cz (B. Hausnerova).

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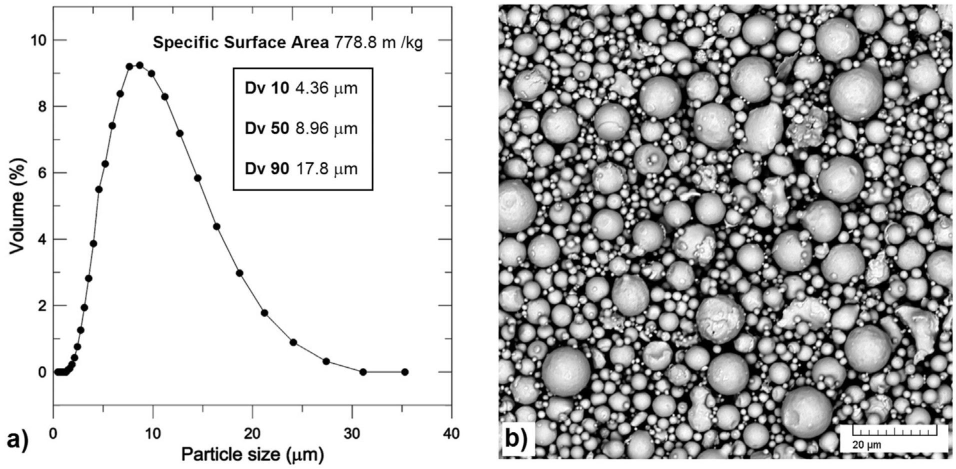

Fig. 1. Particle size distribution (a), and SEM image of Inconel 718 powder (b).

Based on the results, they proposed PEG 20,000 as the most suitable for the feedstock formulation due to a stronger adhesion to powder bringing a better flow stability, despite of the feedstock lower fluidity. Recently, Hayat et al. [13] reported on the impact of PEGs (1500; 4000; 10,000 and 20,000) in terms of feedstock rheology and debinding behavior, where the most suitable PEG for their feedstock formulation was PEG 10,000 with not only excellent properties, but appropriate debinding performance as well.

The objective of this study is to carry out a comprehensive investigation of PEG molecular weight impact on feedstock rheology, debinding, densities and mechanical properties of sintered and heat-treated Inconel 718 samples. A deeper understanding helps to provide an appropriate selection of PEG for further binder design of emerging MIM feedstocks based on nickel superalloys such as Inconel 718 [14–15]. This interest is driven by automotive and aerospace industries in the field of production of complex components (as wheels, buckets, spacers and bolts) exposed to high temperatures.

2. Experimental procedure

2.1. Powder and binder characteristics

Gas atomized nickel based superalloy Inconel 718 powder provided by Sandvik Osprey Ltd. (UK) was used in this study. The particle size distribution was determined using a particle size laser analyzer (Malvern Mastersizer 3000; with a hydro EV dispersion unit). Refractive indexes 1.98 for nickel and 1.33 for water (a solvent) were used as the input parameters for the calculation of the size distribution by volume. Data is presented as average numbers of three discrete measurements of each powder (Fig. 1a). Shape of particles (Fig. 1b) was observed from scanning electron microscopy (SEM) using Vega II LMU Tescan. As it can be seen from Fig. 1b, powder particles have a high spherical uniformity as well as surface purity. Chemical composition of the Inconel 718 powder was analyzed by energy dispersive X-ray spectroscopy (EDX) and obtained values are summarized in Table 1.

Wellestablished three-component binder system based on PEG/PMMA/SA was used in this study. The main component PEG provides low viscosity during injection molding and easy removal by water leaching, while the backbone PMMA retains part shape and strength during and after water debinding. To improve compatibility between the metallic particles and other binder components, SA was added as a surfactant. Detailed composition of binder is depicted in Table 2.

2.2. Feedstock preparation

In this work, seven feedstocks were formulated with the constant amount of powder loading (59 vol%) and binder composition differing only in PEG number-average molecular weight, Table 3. Differential scanning calorimetry (DSC) measurement was carried out in order to obtain the melting range of each PEG (Perkin Elmer Pyris 6 DSC under argon atmosphere 20 ml/min at a heating rate 10 °C/min), Fig. 2.

The mixing procedure of Inconel 718 powder and three-component binder system was performed at a room temperature in a centrifugal SpeedMixer™, Dual Axial Centrifuge DAC 5000. Inconel 718 and binder densities of 8.19 g/cm^3 and 1.1 g/cm^3, respectively, were considered for the calculation of the powder/binder ratio. The components underwent five stages of mixing (Table 4), excluding PMMA emulsion which was added after the third stage of mixing. The final mixture was in a paste form. This is due to the dissipation energy generated by the shear action within mixed feedstock, causing the binder components to melt. Among the particular compounding stages the mixing centrifuge was stopped, and the tin was removed from the machine to scrape off the material stacked on the inner wall. With increasing molecular weight of PEG in feedstocks, the rotation speed and duration in mixing profile had to be prolonged.

After mixing, the feedstocks were removed from the container, and dried in a laboratory oven at 30 °C for 24 h. To ensure an appropriate homogenization, dry feedstocks were broken down into small pieces. Then, extruded and pelletized twice using an injection unit of Arburg 320C Allrounder at 120 °C, and finally pelletized. As it is shown in Fig. 3, SEM of prepared feedstocks confirmed no agglomerates within the feedstocks.

Table 1
Chemical composition of super-alloy Inconel 718 powder.

Element	Ti	Cr	Fe	Ni	Nb	Mo
Weight (%)	1.77	19.17	20.84	50.57	4.89	2.76
Stand. dev. (%)	0.20	1.70	1.70	1.31	0.41	1.67

Table 2
Composition of binder system.

Role in binder	Component	Supplier	wt%
Main component	PEG	Fluka, Sigma-Aldrich	83
Backbone	PMMA[a]	Scott Bader Co Ltd	15
Surfactant	SA	Prolabo Chemicals	2

[a] Emulsion with finely dispersed 0.1–0.2 μm PMMA particles (M_w ~ 10^6 g/mol).