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Experimental study on moldability and segregation of Inconel 718 feedstocks used in low-pressure powder injection molding

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ABSTRACT

Moldability and segregation of feedstock are linked to the rheological behavior of the powder-binder mixture. In this study, the impact of binders on viscosity and segregation of feedstocks was investigated. The experiments were conducted on several feedstocks obtained by mixing Inconel 718 powder with paraffin wax-based binder systems. The viscosity of feedstocks was measured by a rotational rheometer while the segregation within green parts was evaluated using a thermogravimetric analyzer. It was demonstrated that the variation in solid loading within a molded part can be measured with a sensitivity of at least ±0.25 vol% of powder. The results indicated that the predominant powder-binder separation appears clearly at the top and the bottom of the molded part. It was also shown that the viscosity profiles of feedstocks and the intensity of segregation depends significantly on the binder constituents used in feedstock formulation. The mixture containing only paraffin wax produced the best trade-off between high moldability and low segregation for an injection process requiring an extended time range between injection and solidification of the part (e.g. up to 10 min). For a short processing time (e.g. <1 min spent in molten state), the feedstocks containing paraffin wax with stearic acid or small amount of ethylene vinyl acetate can be also considered as good candidates for LPIM process because their viscosity and segregation potential are relatively low.

1. Introduction

The cost-effective and small series fabrication of complex shape metallic parts with the proper dimensional tolerances is one of the main goals in advanced manufacturing industry. Low-pressure powder injection molding (LPIM) is a manufacturing technology covering all these requirements simultaneously [1,2]. Over the past 15 years, the conventional high-pressure powder injection molding (HPIM) requiring injection pressure of up to 200 MPa has transitioned to medium-pressure powder injection molding (MPIM) or LPIM by using low-viscosity feedstocks, requiring an injection pressure lower than 1 MPa [3,4]. This manufacturing process consists in four steps: mixing metallic or ceramic powder with molten wax-based binder to obtain a feedstock, injection molding of the feedstock into a mold cavity, debinding to remove completely the binder, and sintering to obtain a near-net shape dense metallic component [5,6].

The main difference between HPIM and LPIM manufacturing technologies lies in the binder viscosity used for the powder transportation during injection stage. Initially used in ceramics forming [7,8], the low-viscosity feedstocks formulated with low molecular weight polymers have been used in LPIM to increase moldability and shape complexity in the manufacture of metallic parts in the automotive, aerospace, and medical industries [9–13]. The binder constituents generally used in LPIM are waxes, surfactant and thickening agents. Paraffin wax, microcrystalline wax, beeswax, and carnauba wax are widely used as the main constituents to form commercial [14,15], and development feedstocks [16–18]. Surfactant agent is generally added in binder formulations to improve the powder surface characteristics in order to increase the powder wetting, to increase the powder-binder interaction, and to decrease the viscosity of the feedstocks. Stearic acid is one of the most commonly used surfactant to enhance the homogeneity and the mixing properties of the feedstock used in metal injection molding [1,19,20]. In counterpart, a thickening agent is

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generally used in binder formulations to increase the viscosity of the mixture in order to prevent powder-binder separation. Since ethylene vinyl acetate has a certain degree of solubility with paraffin wax, this binder constituent can thus be used to adapt the rheological properties of the mixture without inducing a significant increase in its melting temperature [21,22]. As it is well summarized by German and Bose [5], a feedstock is generally designed to minimize segregation, maximize its moldability, maximize the strength of the molded component, maximize the solid loading potential, and maximize the debindability potential. In this project, the emphasis is placed on the study of the moldability and segregation criteria.

Moldability is the ability of the feedstock to adequately fill up the mold cavity. Maximization of this parameter is of practical interest in producing complex shape or miniaturized components. The viscosity of PIM feedstock is an important property for evaluating the impact of the binder formulation on its flow behavior [23,24]. The rheological properties of several feedstocks used in conventional PIM are well known, as well as for some feedstocks used for LPIM [25–28]. However, the evaluation of the viscosity of LPIM Inconel feedstocks has received very little attention in the literature [29]. It is well accepted that an increase in feedstock temperature, in binder content, in powder sphericity and in shear rate applied on feedstock leads to a decrease in viscosity [30–33].

Segregation refers to the inhomogeneous distribution of powder particles in feedstocks. This undesirable effect must be minimized in order to prevent distortions, cracks, voids, warping and the heterogeneous shrinkage of sintered parts [5,34]. In general, the effect of segregation can be evaluated by several experimental methods including measurement of density, heat capacity, thermal conductivity, electrical conductivity, SEM/EDX analysis, tomographic analysis, or thermogravimetric analysis [35–38]. Separation of the binder from the powder is generated by high-pressure gradient during HPIM and LPIM process or by gravity for unmixed feedstock, the latter is mostly observed for LPIM mixtures.

During mold filling the feedstock is subjected to complex shear rate gradients which may lead to relative movement between the powder and binder, resulting in local change in powder solid loading within the injected parts. The study of segregation occurring during HPIM or micro-PIM injections is mainly focused on the development of numerical models to investigate the role of injection parameters on segregation. Gelin et al. [39] developed a biphasic simulation approach to assess the variation of powder volume fraction at different filling stages of the process and predicted the segregation effect through an injected part using a refined biphasic model [40]. Similar models were used by Wei et al. [41] and Greiner et al. [42] to investigate the influence of filling patterns on the powder-binder separation after molding. He et al. [43] used a granular model to predict the evolution of the density through a triangular mold due to the segregation effect. The role of injection parameters, the location and the intensity of segregation during injection were also studied using commercial and in development software [44–46]. Although some numerical simulation approaches were developed to predict the flow behavior of LPIM feedstocks [47,48], the segregation effects are generally not taken into account for these kind of low-viscosity mixtures. Recent studies on the segregation effects of LPIM or MPIM molded parts mainly focused on the development of experimental techniques to investigate the role of injection parameters or feedstock attributes on segregation. Hausnerova et al. [37,49] developed an experimental technique based on SEM/EDX to detect powder-binder separation. Demers et al. [38] used TGA technique to measure a nonuniform distribution of powder particles through rectangular and wedge shaped molded parts processed by LPIM. Shivashankar et al. [50] used capillary rheometer to investigate the segregation of low- and moderate-viscosity mixtures, while Mukund et al. [51] combined capillary rheology and density measurements to quantify powder-binder separation in MPIM feedstocks.

During injection dead times (e.g. between two injections), segregation may occur for low-viscosity feedstocks that remain idle within the injection press, producing a feedstock that could be inappropriate for further injections. Segregation generated by gravity has only been superficially examined in a conventional HPIM due to the inherently high viscosity of feedstocks preventing the occurrence of this phenomenon. For LPIM feedstocks, an experimental model was recently developed to highlight that the segregation of unmixed feedstocks depends of the binder constituents and the idle time [52].

Moldability and segregation are linked to the rheological behavior of the feedstock for which there is a compromise between these two parameters. Using too low feedstock viscosity should easily fill the mold cavity, but this would also lead to heterogeneity of solid loading within the green part. Conversely, moldability and segregation are both lowered if a too high feedstock viscosity is used. Therefore, an ideal feedstock viscosity minimizes the segregation parameter without compromising the moldability parameter, and vice versa. The aim of this study is to investigate the antagonistic impact of binders on the moldability and segregation of Inconel 718 superalloy feedstocks used in low-pressure powder injection molding.

2. Experimental procedures

2.1. Materials

The powder was gas-atomized spherical Inconel 718 superalloy provided by Osprey Sandvik (Neath, UK) with a typical average particle size of 12 µm (Fig. 1). Inconel 718 is a high-strength and corrosion-resistant nickel-based superalloy. This age-hardenable alloy generates a combination of good tensile strength, fatigue strength and creep resistance. It was demonstrated that PIM Inconel 718 powder can be consolidated up to a relative density value of 97.3%, providing high mechanical properties required for structural aerospace applications [53,54]. The physical properties of each constituent are listed in Table 1. Paraffin wax (PW), stearic acid (SA) and ethylene vinyl acetate (EVA) were selected as the major constituent, surfactant and thickening agents, respectively. These polymer constituents were mixed with the metallic powder to formulate nineteen (19) different feedstocks according to the powder-binder formulations given in Table 2. The feedstock identification is referenced by their volume fractions of polymer, which were determined at room temperature.

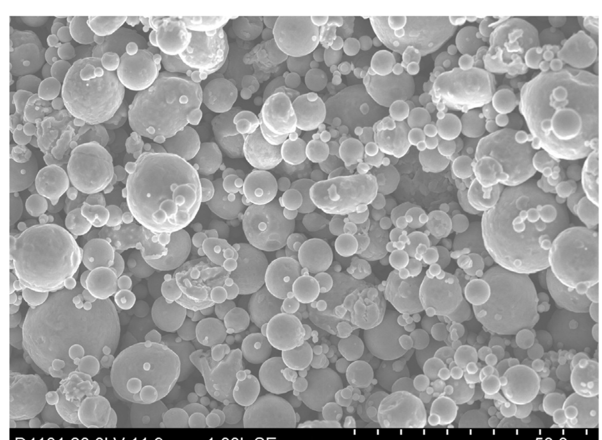

Fig. 1. SEM micrograph of Inconel 718 powder.

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