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METHOD TO IDENTIFY RHEOLOGICAL CONSTITUTIVE MODEL ADAPTED FOR POWDER INJECTION MOULDING PROCESS USING INVERSE METHOD

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Abstract

The aim of this paper is to show a method to identify rheological parameters of constitutive models for powder injection moulding process. The constitutive rheological model used is generalized model mixing several rheological laws such as Carreau-Yasuda, Maron-Pierce and Arrhenius. Thus, the constitutive model takes accounts shear rate, powder volume loading, temperature and particle size.

The material used for this study is Inconel 718, nickel-chromium-based superalloy is typically used in high-temperature and high-performance applications, particularly in the aeronautic industry. To elaborate the feedstock, powder was mix with a formulation composed of three different binder ingredients: polypropylene (PP), polyethylene glycol (PEG) and a stearic acid (SA).

Then, a rheological characterization on the powder and feedstock was carried out. The rheological properties of the resulting binder formulations and feedstocks were characterized using a capillary rheometer. First all the binder granules were filled into rheometer barrel heated to 170, 180 or 190°C.More than, the powder particle size distribution was measured by laser scattering particle analyser.

Then, the data collected from the first characterization were used to identify parameters of the model. Then, this identification of parameters could be used to carry out numerical injection simulations. Also, sensitivity of parameters analysis was carrying out to determine influence of each of rheological law.

Key words: rheological model, metal injection moulding, identification parameter

1. INTRODUCTION

The Metal Injection Moulding (MIM) process is an economically attractive method of producing large amounts of small and complex metallic parts. This process is expanding regarding the numerous scientist papers published these 20 last years (Shivashankar et al., 2013; Özgün et al., 2013; Quinard et al., 2011). The dimensions and mechanical properties of MIM components are influenced by the feedstock characteristics, the process parameters of the injection moulding, as well as the atmosphere and kinetics of debinding and the sintering.

Numerical simulations are a very important feature of the beginning of any product or technology development. It requires also accurate constitutive models describing material behaviour at large shear rates up to 10^5 s^{-1}. The choice of a rheological model and the determination of its parameters should be made from tests generating such conditions.

To identify rheological constitutive models of loaded polymer feedstocks, one generalized constitutive law is applied. The binder was composed of three polymers: polypropylene (PP), Polyethylene glycol (PEG) and stearic acid (SA) (Urterkin et al., 2011). It was prepared by twin screw mixing and mixed with powders. After mixing, the binder has

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been granule and use for rheometer analyses. The super alloy powdersused in the elaborated feedstock are Inconel 718 for airplane or automotive applications. The formulation is based on thermal and solvent debinding (Omar et al., 2003; Onbattuvelli et al., 2014).

The rheological properties of the resulting binder formulations and feedstocks were characterized by a capillary rheometer. In the rheometer, shear rates were varied from 100 to 105 s^{-1}.

Then, comparisons are carried out among the experimental and analytical results. Thus, several models were used such as power law, Williams-Landel-Ferry, Arrhenius, Maron-Pierce and Carreau-Yasuda. In the wake of this comparison, identifications of parameters used in models were performed. This parameter identification carried out with non-linear least squares method and a trust-region algorithm.

Finally, identified parameters were used to complete identification of a more complex law. This equation takes account of several parameters such as particle size, temperature, shear rate and powder volume loading and take back identifications of parameters from previous laws.

Then, was performed sensitivity analysis on parameters of the rheological model, in order to determine the main parameters that need to be precisely estimated.

2. MATERIAL CONSTITUTIVE BEHAVIOUR LAWS

In the present work, comparisons are made between experimental results and those obtained from analytical models. Thus, several models more or less complicated were used such as power law, Arrhenius, Chong and Carreau-Yasuda. In the wake of this comparison, identification of parameters used in models and sensitivity analysis were performed, see table 1. In this paper, Ratkovich et al. (2013) show several rheological models suitable for non-Newtonian material among the numerous models predicting the apparent viscosity with different degrees of complexity. Table 2 shows several models suitable for viscosity of load polymers. The symbols in the tables mean: τ_0 the yield stress (Pa), n the flow behaviour index (–), k the flow consistency index (Pa·s^n), γ the shear rate (/s), μ the apparent viscosity (Pa·s), μ_∞ the infinite rate apparent viscosity (Pa·s), μ_0 the zero shear apparent viscosity (Pa·s), λ the (Cross) time constant (s) and m the Cross rate constant (–) in table 1, and η the viscosity (Pa·s), η_0 the apparent viscosity (Pa·s), ϕ the powder load rate and ϕ^m the maximal powder load rate in table 2, respectively.

The Carreau-Yasuda model is a generalized model of Carreau for which n = 2 (Yasuda et al., 1981). As shown in figure 1, different power law models may be appropriate for different shear rate regimes. The Carreau-Yasuda model allows getting a best link between the power-law model and the Newtonian plateau at low shear rates. It is why this model has been chosen for the identification. However, the identification of the power-law model has been done before in the aim to determine the flow behavior index n.

Table 1. *Empirical models for viscosity of polymers.*

Model	Equation
Power-law (Ostwald de Waele)	$\tau = k\dot{\gamma}^n$
Bingham	$\tau = \tau_0 + k\dot{\gamma}$
Herschel and Bulkley	$\tau = \tau_0 + k\dot{\gamma}^n$
Casson	$\tau^{0.5} = \tau_0^{0.5} + \mu_\infty^{0.5}\dot{\gamma}^{0.5}$
Sisko	$\mu = \mu_\infty + K\dot{\gamma}^{n-1}$
Cross	$\frac{\mu - \mu_\infty}{\mu_0 - \mu_\infty} = \frac{1}{1 + (\lambda\dot{\gamma})^m}$
Carreau	$\frac{\mu - \mu_\infty}{\mu_0 - \mu_\infty} = (1 + (\lambda\dot{\gamma})^2)^{\frac{n-1}{2}}$

Table 2. *Empirical models for viscosity of load polymers.*

Model	Equation
Eilers (1941)	$\eta/\eta_0 = \left(1 + \frac{1.25\phi}{1 - \phi/\phi_m}\right)^2$
Mooney (1951)	$\eta/\eta_0 = exp\left(\frac{2.5\phi}{1 - \phi/\phi_m}\right)$
Krieger and Dougherty (1959)	$\eta/\eta_0 = \left(1 - \frac{\phi}{\phi_m}\right)^{-2.5\phi_m}$
Chong and al (1971)	$\eta/\eta_0 = \left(1 + 0.75\frac{\phi/\phi_m}{1 - \phi/\phi_m}\right)^2$
Quemada (1977)	$\eta/\eta_0 = \left(1 - \frac{\phi}{\phi_m}\right)^{-2}$
Mills (1985)	$\eta/\eta_0 = 1 - \phi/\left(1 - \frac{\phi}{\phi_m}\right)^2$
Koda and Furuse (2006)	$\eta/\eta_0 = \frac{1 + 0.5.k.\phi - \phi}{(1 - k.\phi)^2(1 - \phi)}$

In the same way of Senapati et al. (2010) and Hidalgo Garcia (2013), a generalized model (see equation (1)) is elaborated from Arrhenius, Carreau-Yasuda and Maron-Pierce laws:

$$\eta = \frac{C_u}{D_{50}} \cdot \exp\left(\frac{E_a}{R \cdot T}\right) \cdot \eta_0 \left(1 + (\lambda \dot{\gamma})^a\right)^{\frac{n-1}{a}} \cdot \left(\frac{\varphi_m}{\varphi_m - \varphi}\right)^m \tag{1}$$

where E_a is the Arrhenius activation energy ($kJ \cdot mol^{-1}$), R is perfect gas constant ($J \cdot K^{-1} \cdot mol^{-1}$), Cu the D60/D10 , D60 the particle size at 60%, D10 the particle size at 10% and D50 the particle size at 50%.

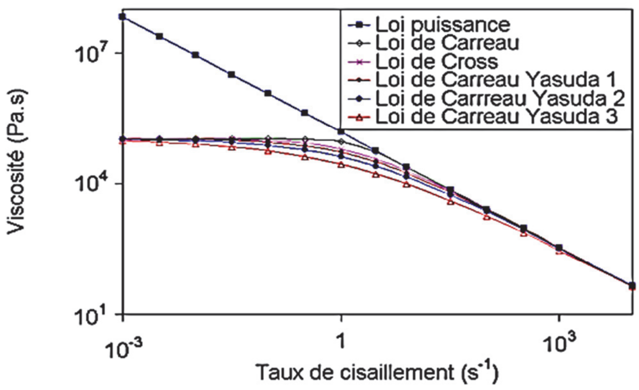

Fig. 1. Representation of different rheological models (El Otmani, 2009).

3. RESULTS AND DISCUSSIONS

3.1. Powder particle size

Figure 2 illustrates the particle size distribution for Inconel super alloy powders. The particle size distribution was shown in terms of the number of particles. The graph shows the d10, d50 and d90 diameters equivalent to 3,53 μm, 6,24 μm and 10,97 μm, respectively. The standard deviation in this case, was 0,86 μm. The measure of SSA indicates a specific area of 0,095 m^2/g.

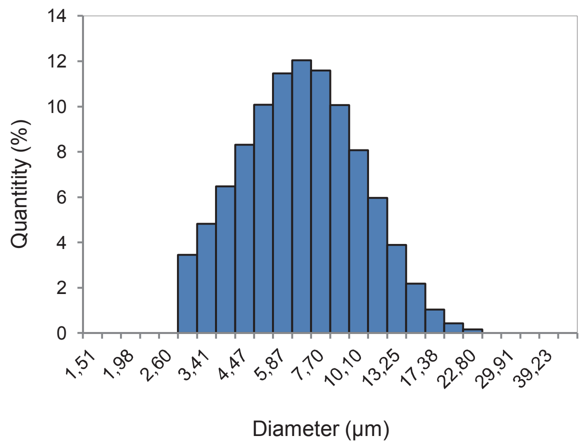

Fig. 2. Particle size distribution for Inconel super alloy powders.

3.2. Shear viscosity

The results of the shear rate viscosity measurements vs shear rate and temperature of the Inconel superalloy elaborated feedstocks are shown in figure 3. It exhibits pseudo-plastic flow behaviour and the viscosity decreased as the shear rate increased at all test temperatures. The values of shear viscosity are less 100 Pa·s in injection range, the developed and elaborated feedstock is very low and very easily injectable for injection and micro-injection process. This viscosity is plotted for 170, 180 and 190°C. Shear viscosity decreases with shear rate for all temperatures. This behaviour matches with pseudoplastic behaviour flow. So, the Carreau-Yasuda model is suitable to model this viscosity behaviour curves.

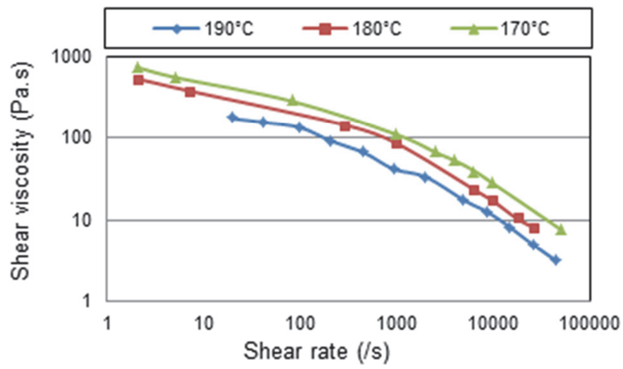

Fig. 3. Feedstock apparent viscosity obtained at different temperatures.

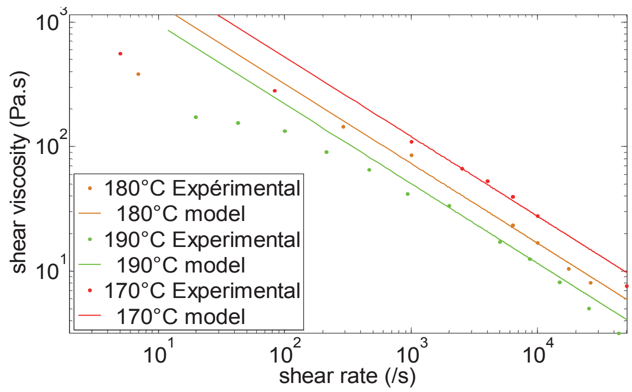

Fig. 4. Power-law modeling of shear viscosity vs. shear rate after parameter identification of shear viscosity for Inconel feedstock.

3.4. Identification of the rheological parameters

The first identification by inverse method (Szeliga et al., 2006) was carried out with the power-law model and is presented in figure 4. This identification allows to define the flow behaviour index equivalent to a value of 0,36. For this model, only the experimental measurements with shear rates biggest than 10^3 s^{-1} were taken into account for identification.

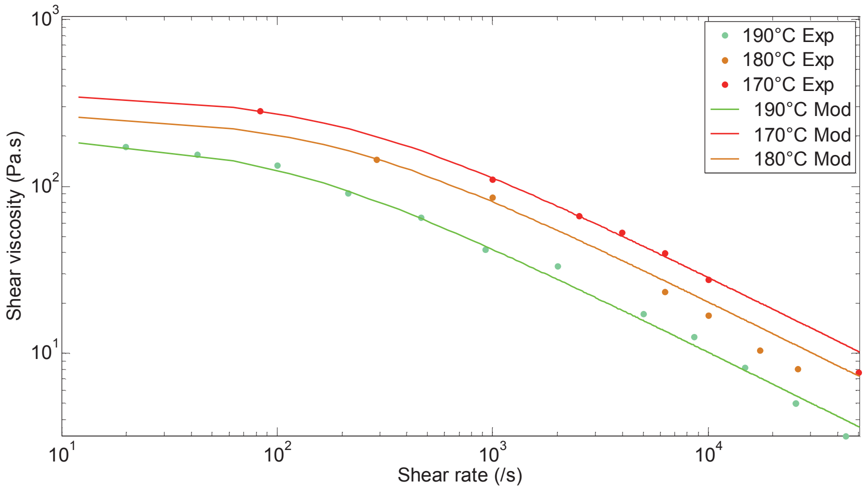

Fig. 5. Comparison of generalized law modeling after parameter identification of the evolution of shear viscosity vs shear rate and temperature for Inconel loaded polymer.

Table 3. Identified parameters using a generalized full model (see equation (1)).

T (°C)	Cu/d50	E_a (kJ·.mol^{-1})	η_0 (Pa·s)	λ	ϕ	ϕ_m	m	a
170	0.498	20.22	683.7	0.005031	0.7	0.72	0.009	1
180	0.498	20.26	521.4	0.005597	0.7	0.72	0.009	1
190	0.498	20.06	376.8	0.01005	0.7	0.72	0.009	1

The second identification was performed with the Carreau-Yasuda model and is given on the figure 5. With this model, the experimental measurements and analytical predictions are almost similar. The parameters allowing this good correlation between analytical and experimental curves are given in table 3.

3.5. Sensitivity analysis

To determinate the influence of each parameter of the Carreau-Yasuda model, a sensitivity analysis was carried out (figure 6 and figure 7). For each curve of this analysis, only one parameter is increased

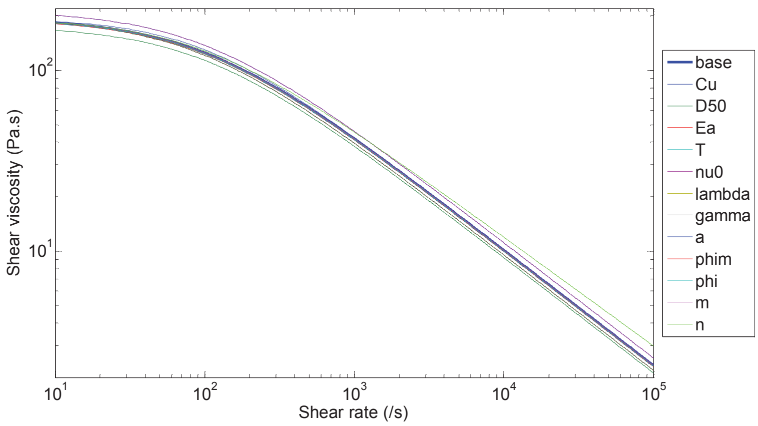

Fig. 6. Generalized law coefficients variation vs shear rate.

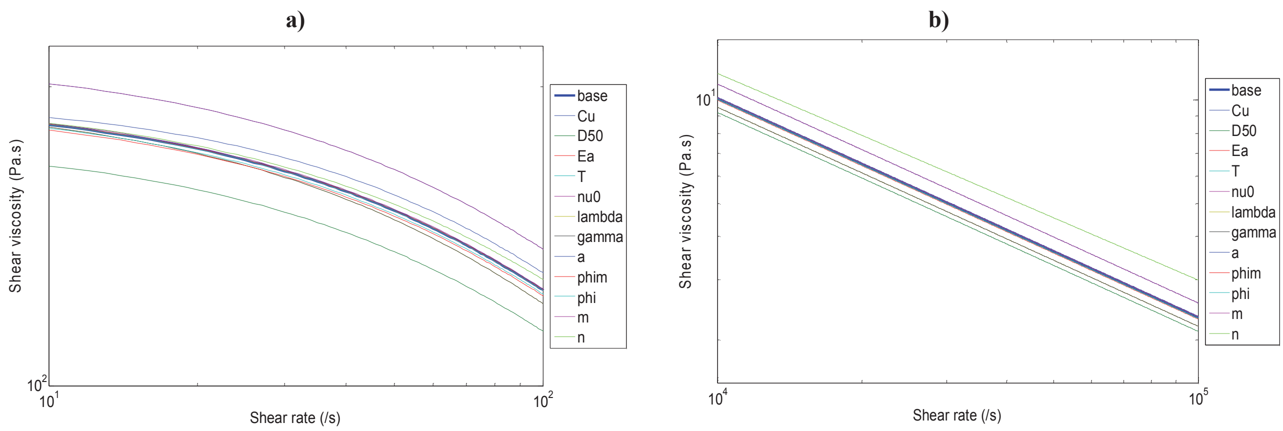

Fig. 7. Zoom in generalized law coefficients variation curve: on poor shear rate area (a), on high shear rate area (b).

of 10% with regards to the initial value. This application allows to see the influence of each parameter with regards to the baseline.

The *a* and *n* parameter influences on the viscosity curve are depending of the shear rate. These parameters are the most important when shear rate is superior to 1000/s. Inversely, they have no influence with low shear rates and η_0 becomes the most influent in this situation.

4. CONCLUSIONS

The determination of rheological parameters using data obtained through standard viscometric flows opens the possibility of building reliable analytical models, which can be used in the injection simulations. The present work aims at the identification of rheological parameters associated to a non-linear constitutive equation. Firstly, a robust analysis has been used, in order to determine the constitutive coefficient values, starting from experimental data obtained by rheological tests. Then, an extensive sensitivity analysis has been performed. The aim of this work is to examine a proper constitutive model to more accurately describe the influence of the shear rates and temperature on the feedstock behavior in large shear rates ranges.

To improve the rheological sensitivity on the injection simulations, in a future work we propose to analyze the influence of a more complex law. It is then necessary to take into account the solid loading, shapes and particle size of powder grains and then interaction between powders and binders.

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METODA IDENTYFIKACJI KONSTYTUTYWNEGO MODELU REOLOGICZNEGO ZASTOSOWANEGO W PROCESIE ODLEWANIA METODĄ WTRYSKIWANIA PROSZKU PRZY UŻYCIU METODY ODWROTNEJ

Streszczenie

Celem pracy jest przedstawienie metody identyfikacji parametrów reologicznych modelu konstytutywnego dla procesu wtryskiwania proszku. Reologiczny model konstytutywny jest uogólniony model będący połączeniem wielu praw reologicznych, takich jak Carreau-Yasuda, Maron-Pierce'a i Arrheniusa. Zatem model konstytutywny bierze pod uwagę prędkość ścinania, objętość proszku, temperaturę i wielkość cząstek.

Analizowanymi materiałami były Inconel 718 i nadstop na bazie niklu i chromu, stosowane w procesach wysokotemperaturowych i o wysokiej wydajności, szczególnie w przemyśle lotniczym. Materiał wsadowy wytworzono z proszku składającego się z trzech składników: polipropylenu (PP), glikolu polietylenowego (PEG) i kwasu stearynowego (SA).

Następnie opracowano charakterystykę reologiczną dla proszku i materiału wsadowego. Reologiczne własności spoiwa i materiału wsadowego zostały wyznaczone reometru kapilarnego. Na początku spoiwo umieszczono w reometrze, który podgrzano do temperatury 170, 180 lub 190°C. Następnie został zmierzony rozkład wielkości cząstek proszku z użyciem laserowego analizatora rozmiaru cząstek.

W kolejnym kroku badań zebrane dane zostały wykorzystane do identyfikacji parametru modelu. Oszacowane parametry modelu mogą zostać zastosowane w symulacjach numerycznych proce-

su wtryskiwania. Ponadto w pracy przeprowadzono analizę wrażliwości, określając wpływ parametrów poszczególnych modeli na wartości wyjściowe z modeli.

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