Analysis of the rheological behavior of copper metal injection molding (MIM) feedstock


Abstract

Metal Injection Molding (MIM) technique allows for the production of highly porous metallic foams with porosity levels up to 90%. It makes use of the pressure built up by the decomposition of a foaming agent which is incorporated in a foamable precursor copper material obtained by powder compaction. Rheological is one of the key factors to ensure the successful of MIM technique and to predict failure, whether due to the binder component and compositions, powder loading or unsuitable process parameters. The balanced ratio feedstock contains of 63 vol.% of copper powder, different percentage of potassium carbonate; Batch 1 (0.4 vol.%), Batch 2 (0.5 vol.%) and Batch 3 (0.6 vol.%), and the remaining volume percentage of binder system has been mixed to form copper feedstock. The rheological behaviors were investigated using a capillary rheometer (CFT-500D, Shimadzu) at various temperature and loads. From the experiments, it was concluded that the MIM feedstock exhibit a shear thinning or pseudo-plastic behavior based on the trend of graph which is suitable for MIM process. This result is within the ideal range of viscosity theoretical for MIM feedstock which is in the range of between 10 Pa.s to 1000 Pa.s at all temperature tested. The viscosity of a pseudo-plastic substance decreases as the shear rate increases (shear thinning). This could be due to particle orientation and ordering with flow as well as breakage of particle agglomerates released together with the binder.

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Keywords: Copper; rheology; feedstock
1. Introduction

Metallic foams or cellular materials are unique materials with controlled pore structure and properties that can be applied to various applications; such as low density, high stiffness in conjunction with very low specific weight and high gas permeability combined with high thermal conductivity. Many metals have been researched and developed to produce metallic foam such as aluminium, copper, titanium and so on. Copper foam has been used in various industrial applications, such as thermal conductors, catalysts, and batteries\textsuperscript{1,2}.

Many different methods have been used to manufacture copper metal foams, among which, the methods that use metal powders are the most widely used. These methods have a good control over the cell shape, cell size and porosity distribution. Some investigators have used the Sintering and Dissolution Process (SDP), and recently, the Lost Carbonate Sintering (LCS) process\textsuperscript{3,4}. This research aims to produce the copper foam by using MIM as this process has the ability to produce complex shaped powder parts with near net shape at high production capacity.

In MIM process, the molding stage is a critical step for the fabrication of sound parts without cracks and distortions. Non-homogenous flow and powder-binder separation can produce defects during molding, resulting in cracking and warpage during debinding and sintering, and ultimately poor physical and mechanical properties of the final MIM component\textsuperscript{5}. So this step requires specific rheological behavior; viscosity, density, thermal properties and pseudoplastic behavior of a feedstock that determine its performance for successful manufacturing process.

With reference to recent results on the development of copper foam for MIM\textsuperscript{6,7}, the present study is specifically focus on investigation of the rheological behavior and stability of Copper feedstock. The data can be used not only to predict the flow behavior during injection molding and to determine viscosity, but also to reveal the stability and homogeneity of feedstock and the extent of powder-binder separation.

### Nomenclature

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
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<tbody>
<tr>
<td>μm</td>
<td>micrometer</td>
</tr>
<tr>
<td>vol.%</td>
<td>volume percentage</td>
</tr>
<tr>
<td>ºC</td>
<td>degree Celcius</td>
</tr>
<tr>
<td>s⁻¹</td>
<td>per second</td>
</tr>
<tr>
<td>Pa.s</td>
<td>Pascal second</td>
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2. Experimental Procedure

A commercially available Copper spherical shape powder as shown in Fig. 1 is provided by Guangzhou Jiechuang Trading Co. Ltd., China and its characteristics is tabulated in Table 1. The 22 μm particle size distribution was determined using a Cilas particle size analyzer and it can be seen that the powder had a relative wide particle size distribution which is desirable for efficient particle packing.

Fig. 1. A scanning electron micrograph of Copper powder.
Table 1. Characteristics of Copper powder

<table>
<thead>
<tr>
<th>Powder Type</th>
<th>Particle shape</th>
<th>D20 (µm)</th>
<th>D50 (µm)</th>
<th>D80 (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper</td>
<td>Sphere</td>
<td>6.06</td>
<td>16.61</td>
<td>24.47</td>
</tr>
</tbody>
</table>

The combination of Copper powder and the binder system were referred by their powder constitution and optimum solid loading of 63 vol.% This 63 vol.% of Copper will be mixed with various percentage of Potassium Carbonate; A1 (0.4 vol.%), A2 (0.5 vol.%) and A3 (0.6 vol.%) and the remaining content are the binder system consists of Polyethylene (PE), Paraffin Wax and Stearic Acid (SA), this mixture were blend together using a Winkworth MZ-3 Z-blade mixer for duration of 2 hours at the temperature of 160 °C in order to produce a mixture which defines as feedstock. The feedstock material will be cut into small pieces as to be used on the Capillary Rheometer Shimadzu CFT-500D for the rheological study which based on the compatibility and homogeneity of the mixture.

The rheological study of the fine feedstock was analyzed based on Flow Rate, Shear Rate, Viscosity and Melting Flow Rate. A die with 1 mm diameter (D) and 20 mm length (L), giving a ratio (L/D) of 20 was used. Each feedstock will be placed in the rheometer barrel and allowed to preheat at varying temperatures for 160 °C, 170 °C, 180 °C and 190 °C under shear stress of various test loads for initiating testing.

3. Results and discussion

3.1. Thermal analysis

The melting and evaporation temperature of PE, PW and SA are tabulated in Table 2. The heating cycle for the removal of the binder system can be indicated based on these results. The heating cycle is required to ensure that the binder system that contains in the Copper samples are totally extracted before it undergoes sintering process, which is the final stage of MIM process.

Table 2. Melting and evaporation temperature of PE, PW and SA.

<table>
<thead>
<tr>
<th>Binder Material</th>
<th>Melting Temp. (°C)</th>
<th>Evaporation Temp. (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PE</td>
<td>125.9</td>
<td>488.0</td>
</tr>
<tr>
<td>PW</td>
<td>63.6</td>
<td>405.0</td>
</tr>
<tr>
<td>SA</td>
<td>79.1</td>
<td>345.5</td>
</tr>
</tbody>
</table>

3.2. Mixture of Copper powder, Potassium Carbonate and PE/PW/SA binder system

Based on optimum solid loading of Copper powder, the balanced ratio feedstock that contains of 63 vol.% combination of Copper powder and different percentage of Potassium Carbonate; A1 (0.4 vol.%), A2 (0.5 vol.%) and A3 (0.6 vol.%) and the remaining volume percentage of binder system has been mixed to form Copper feedstock. The scanning electron micrograph of Copper feedstocks is shown on Fig. 2 where it can be observed that the Copper powder particle were surrounded with PE/PW/SA binder system and Potassium Carbonate, proof that the feedstock were mixed homogenously at 160 °C.

The grey and and white colours of the feedstock structure represent the PE/PW/SA binder system that were fill homogenously within the Copper and Potassium carbonate powder particle. The positions of Potassium Carbonate powder particle and its quantity needed to be well balanced in the feedstock as it plays a major role to ensure that the pore structure are well formed. The pore has to be relatively homogenous in all part of metallic foam as according to the required percentage of porosity.
3.3. Rheological study: Effect of shear rate

The feedstocks are considered to be shear thinning or pseudoplastic fluids. The main characteristic of a pseudoplastic fluid is that viscosity decreases with the increase of shear rate. Empirical studies have shown that the shear rate during moulding usually ranges between 100 and 10,000 s⁻¹ and maximum viscosity for moulding is 1000 Pa.s at the moulding temperature. The effect of temperature and shear rate on the viscosity of feedstock A1, A2 and A3 is shown in Fig. 3. All feedstock apparently exhibited pseudoplastic flow behaviour and the shear viscosity decreased with increasing shear rate for all working temperatures.

![Fig. 3. Effect of temperature and shear rate on the viscosity of feedstock (a) A1, (b) A2 and (c) A3.](image)

The decrease in viscosity with increasing shear rate indicates particle (or binder molecule) orientation and ordering with flow, and may reflect an improved homogeneity. From the figure, it clearly shows that all feedstock exhibit good rheological behaviour, however these feedstocks exhibited higher viscosity, which is higher than 1000 Pa.s particularly at lower shear rate. Additionally, the variation of viscosity versus shear rate as recorded in Fig. 3.
(a) and Fig. 3 (b), shows an indicator of feedstock stability.

The viscosity data shown all feedstock in Fig. 3 indicates the flow ability of the MIM feedstock. The lower the value of viscosity, the easier it is for a MIM feedstock to flow. Nevertheless, too low the viscosity tends to lead to slumping phenomenon, whereby the feedstock is too soft to handle. The viscosity of the feedstock A2 can be considered as the most appropriate for injection based on comparison of graph in Fig. 4, since all data obtained indicates small changes of viscosity at all temperature. Based on the theoretical range of viscosity of the feedstock, it should be within the range of 10 Pa.s and 1000 Pa.s at all temperature tested.

![Fig. 4. Effect of temperature and shear rate on the viscosity of feedstocks at temperature of 160 °C](image)

4. Conclusion

From the experiments, it was concluded that the A2 Copper feedstock exhibit a shear thinning or pseudoplastic behavior based on the trend of graph in Fig. 4 which is the most suitable for MIM process. The viscosity of a pseudoplastic substance decreases as the shear rate increases (shear thinning). This could be due to particle orientation and ordering with flow as well as breakage of particle agglomerates re-leased together with the binder. This result is within the ideal range of viscosity theoretical for MIM feedstock which in the range of between 10 Pa.s to 1000 Pa.s at temperature tested between 160 °C and 170 °C.

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References