Extrusion of Cu Feed Materials

Rattana WICHIANRAK¹, Monnapas MORAKOTJINDA², Thanyaporn YODKAEW², Nattaya TORSANGTUM², Rungrit KRATAITHONG², Anan DARAPHAN², Ornmamee COOVATTANACHAI², Bhanu VETAYANUGUL², Nandh THAVARUNGKUL¹, Nuchthana POOLTHONG¹ and Ruangdj TONGSRI²

¹ Division of Materials Technology, Faculty of Energy, Environment and Materials, King Mongkut’s University of Technology Thonburi, Bangkok 10140, Thailand
² Powder Metallurgy R&D Unit (PM_RDU) National Metal and Materials Technology Center, 114 Paholyothin Rd., Klong 1, Klong Luang, Pathum Thani 12120, Thailand.

Abstract

Experimental works have been carried out to determine (i) extrusion limits (ii) feed materials property (flowability of the extrudable feed materials) and (iii) mechanical properties of sintered Cu rods shaped by extrusion. The works were divided into 4 processing steps such as (i) preparation of particulate-binder mixtures (feed materials), (ii) shaping of the feed materials by extrusion, (iii) removal of the binders and (iv) sintering of the debinded parts. In the step of feed material preparation, Cu powders with different shapes and sizes were mixed with a binder, comprising of low density polyethylene (LDPE): paraffin wax (PW): stearic acid (SA). Volume ratios between the Cu powder and the binder were varied, e.g., 60:40, 65:35 and 70:30. Shaping of the feed materials was performed by downwardly direct extrusion, with an extrusion ratio of 10:1, into a rod-shape part with a diameter of 5.0 mm. Extrusion limits were determined. For spherical Cu powders, feed materials prepared from different Cu particle sizes (<45 μm, 45-75 μm and 75-125 μm) could be extrudable when the metal powder charges were ≤ 70 vol. %. For irregular Cu powders, feed materials prepared from the powder with particle sizes < 45 μm could be extrudable when the metal powder charges were up to 65 vol. %. When the irregular Cu powders with particles sizes of 45-75 μm were employed, the powder to binder ratio was limited at 60:40. Rheological property test results showed that all extrudable feed materials exhibited pseudo-plastic behavior, i.e., their viscosity values decreased with increasing shear rates. The extrudates were further debinded and sintered. Mechanical properties of the sintered Cu extrudates depended on original Cu particle size and shape and sintering time.

Key words : Feed materials, Extrusion, Plastic rheological behavior, Debinding, Sintering

Introduction

Extrusion is one of various metal forming processes involving squeezing of a metal billet to flow through a die and to form a desired part. In a bulk metal extrusion, a metal can be deformed and flowed only when a high pressure is exerted to a metal billet, made from either a metal ingot or a powdered metal compact, under considerably high temperature.¹, ² For example, pressures required for extrusion of aluminium alloys are up to 1000 MPa and extrusion temperatures for the same materials are around 400-500 °C.² Because of high pressure and high temperature, a heavy extrusion press machine and high heating instrument are needed. Energy consumption for a conventional metal extrusion is therefore high. A new approach for powder extrusion (PE), based on shaping of metal powder-binder mixes, not only reduces energy consumption but also provides opportunity for producing porous parts with a high aspect ratio.

The PE process involves processing steps of (i) mixing of the metal powder with a binder granulate to form a “feed material”, (ii) extrusion of the feed material, (iii) removing of the binder (debinding) and (iv) sintering of the debinded or brown parts. The rheological property required for the feed material is that no plastic deformation of green body (extrudate) occurring due to its own weight or due to the necessary handling.³ Different shapes of powder affect rheology of the feed material and inter-particle friction of the green part (extrudate).⁴ Spherical metal powders from gas atomization are suitable component of the feed material because of their high packing density,
high flow rate and fluid-like characteristics. However, the cost of gas atomized metal powders is relatively high. Their low inter-particle friction also affects component shape retention. In contrast, the cost of irregular shape powders is low. The irregular shape results in a high inter-particle friction with improved shape retention. The packing density of irregular shape powders is low and shaping is difficult due to thigh viscosity of the feed material.\(^{(3)}\)

## Materials and Experimental Procedures

Starting materials used in this work were Cu powders with spherical and irregular shapes. The powder sizes were divided into fractions of <45, 45-75 and 75-125 \(\mu\)m. A binder consisted of 45\% low density polyethylene (LDPE), 50\% paraffin wax (PW) and 5\% stearic acid (SA). Volume ratios between the Cu powder and the binder were varied as 60:40, 65:35, 70:30 and 75:25. Mixing of the Cu powder with the binder was carried out in an internal mixer with counter rotating twin screw at 130\(^\circ\)C and speed of 25 rpm for 60 min. Rheological properties of the feed material were measured by a capillary rheometer (RH 2200 Rosand) with capillary diameter of 2 mm and long of 16 mm at 95\(^\circ\)C. The feed material was put into the extrusion chamber and then extruded with an extrusion pressure of 30 bar at 95\(^\circ\)C. Debinding was performed in two stages, (i) solvent debinding by immersing the extrudate in hexane solvent at 50 \(^\circ\)C for different times to identify the optimum solvent debinding conditions and (ii) thermal debinding with the following heating cycle: heating from room temperature to 100\(^\circ\)C with heating rate of 5\(^\circ\)/min and from 100 to 250\(^\circ\)C with lower heating rate of 1\(^\circ\)/min., holding at 250\(^\circ\)C for 90 min., heating from 250 to 450\(^\circ\)C with heating rate of 2\(^\circ\)/min., holding at 450\(^\circ\)C for 60 min., and finally cooling in the furnace. Sintering was performed at 1030\(^\circ\)C under H\(_2\) atmosphere. Holding times for sintering were varied from 60 to 180 min. A universal testing machine (Instron model 8801) was employed to measure mechanical properties of the sintered parts.

## Results and Discussion

### Extrusion Limits

Extrusion limit in this work means extrudability of the feedstocks. Limit diagram for extrusion of a metal billet is constructed from extrudability limited by extrusion ratio \((R)\) and by extrusion temperature.\(^{(2)}\) However, the feedstocks prepared in this works showed narrow working temperatures. Thus a limit diagram could not be constructed following the definition given in (Sheppard, 1999) for conventional metal extrusion. Experimental results (Table 1) showed that extrusion of Cu powders was limited by three parameters, namely powder shape, size and metal powder to binder ratio. For spherical powder, metal powder to binder ratio was a limiting factor. Shaping of the spherical Cu feed material was limited when binder content \(\leq 30\) vol. \%. For irregular powder, powder size was an important factor. Only the powders with particle sizes of < 75 could be extruded. Compared to MIM feed materials (Germon, 1998) the extrusion feed materials do not required finer powder particles. In the extrusion feed materials of spherical Cu powders, the binder content can be reduced to 30\% by volume. Moreover, the extrusion feed materials can also be prepared from irregular Cu powders.

### Rheological Property of the Extrudable Feed Materials

In previous report (Shi-Bo and Jian-Xin, 2007) different shapes of powder affected rheology of the feed material and inter-particle friction of the green part (extrudate). Spherical metal powders from gas atomization were suitable component of the feed material because of their high packing density, high flow rate and fluid-like characteristics. In our work, the effects of powder particle size and binder content were determined. It was clear that viscosity of the feed materials decreased with increasing shear rate (Figure 1). The feed materials showed pseudo-plastic rheological behavior during extrusion. The shear rates were in the range between 10 and \(10^3\) s\(^{-1}\). Within this shear rate range, the feed materials prepared from coarse spherical powders (> 45 \(\mu\)m) showed close viscosity values no matter was the binder content. For the feed materials prepared

### Table 1. Extrusion Limits

<table>
<thead>
<tr>
<th>Powder size range</th>
<th>Spherical powder</th>
<th>Irregular powder</th>
</tr>
</thead>
<tbody>
<tr>
<td>60% Cu 65% Cu 70% Cu 75% Cu</td>
<td>✓ ✓ ✓ ✓</td>
<td>❌ ❌ ❌ ❌</td>
</tr>
<tr>
<td>75-125 (\mu)m</td>
<td>✓ ✓ ✗ ✗</td>
<td>❌ ❌ ✗ ✗</td>
</tr>
<tr>
<td>45-75 (\mu)m</td>
<td>✓ ✓ ✓ ✓</td>
<td>✓ ✓ ✓ ✓</td>
</tr>
<tr>
<td>&lt;45\mu</td>
<td>✓ ✓ ✗ ✓</td>
<td>✓ ✓ ✓</td>
</tr>
</tbody>
</table>

### Table 1. Extrusion Limits

<table>
<thead>
<tr>
<th>Powder size range</th>
<th>Spherical powder</th>
<th>Irregular powder</th>
</tr>
</thead>
<tbody>
<tr>
<td>60% Cu 65% Cu 70% Cu 75% Cu</td>
<td>✓ ✓ ✓ ✓</td>
<td>❌ ❌ ❌ ❌</td>
</tr>
<tr>
<td>75-125 (\mu)m</td>
<td>✓ ✓ ✗ ✗</td>
<td>❌ ❌ ✗ ✗</td>
</tr>
<tr>
<td>45-75 (\mu)m</td>
<td>✓ ✓ ✓ ✓</td>
<td>✓ ✓ ✓ ✓</td>
</tr>
<tr>
<td>&lt;45\mu</td>
<td>✓ ✓ ✗ ✓</td>
<td>✓ ✓ ✓</td>
</tr>
</tbody>
</table>

### Rheological Property of the Extrudable Feed Materials

In previous report (Shi-Bo and Jian-Xin, 2007) different shapes of powder affected rheology of the feed material and inter-particle friction of the green part (extrudate). Spherical metal powders from gas atomization were suitable component of the feed material because of their high packing density, high flow rate and fluid-like characteristics. In our work, the effects of powder particle size and binder content were determined. It was clear that viscosity of the feed materials decreased with increasing shear rate (Figure 1). The feed materials showed pseudo-plastic rheological behavior during extrusion. The shear rates were in the range between 10 and \(10^3\) s\(^{-1}\). Within this shear rate range, the feed materials prepared from coarse spherical powders (> 45 \(\mu\)m) showed close viscosity values no matter was the binder content. For the feed materials prepared
from small powders (< 45 µm), lower binder content caused higher viscosity. Interrelationship between powder size, shape, binder content and viscosity is not understood yet. Further investigation on this issue needs to be conducted in the future.

**Mechanical Property of the Sintered Cu Material**

Mechanical properties of the sintered Cu extrudates were controlled by powder size and shape (Figure 2). The sintered materials produced from small Cu powders showed better mechanical properties than those of sintered coarse Cu powders. During powder shaping by the binder-assisted extrusion there is little powder particle deformation. Points/areas of contact, needed for sintering promotion, are resulted from powder particle packing. Powder particle surface areas and curvatures are important parameters for sintering promotion. Small or finer powder particles assist sintering process due to their high surface areas/curvatures or high surface energy (driving force for sintering).\(^7, 8\)

**Figure 1.** Viscosity VS shear rate of the Cu feed materials.

**Figure 2.** Mechanical properties of the sintered Cu extrudates.
Amongst small Cu powders, irregularity of the powder provided superior mechanical properties. Powder particle irregularity is important for forming metal powders by a ‘press and sinter’ process.\(^6\) Interparticle locking between irregular powders is important for compacted or green part strength. Points/areas of contacts are important for sintering process. However, in the case of binder-assisted extrusion, interparticle locking and points/areas of contact may be less important than surface areas/curvatures. It may be assumed here that small and irregular powders may have higher surface areas/curvatures than those of small and spherical powders.

Conclusions

Extrusion limits were observed. For spherical Cu powders, feed materials prepared from different Cu particle sizes (<45 \(\mu\)m, 45-75 \(\mu\)m and 75-125 \(\mu\)m) could be extrudable when the metal powder charges were \(\leq\) 70 vol. %. For irregular Cu powders, feed materials prepared from the powder with particle sizes < 45 \(\mu\)m could be extrudable when the metal powder charges were up to 65 vol. %. When the irregular Cu powders with particles sizes of 45-75 \(\mu\)m were employed, the powder to binder ratio was limited at 60:40. Mechanical properties of the sintered Cu extrudates were controlled by powder size and shape. The sintered materials produced from small Cu powders showed better mechanical properties than those of sintered coarse Cu powders. Amongst small Cu powders, irregularity of the powder provided superior mechanical properties.

Acknowledgements

The authors would like to express their sincere gratitude to Materials Technology Division, School of Energy Environment and Materials, King Mongkut University of Technology Thonburi, Bangkok, Thailand and National Metal and Materials Technology Center (MTEC), Pathum Thani, Thailand, for financial support.

References


