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Characterisation of phase segregation during back extrusion of ZA27 semisolid alloy

J. Mola*, H. Aashuri and B. Shalchi Amirkhiz

Effort was made to characterise segregation tendency of mechanically stirred ZA27 alloy with back extrusion as a thixoforming process. At sufficiently high ram speeds, at which liquid phase flows forcefully, pooling of the heavy liquid phase owing to gravitational segregation contributes to homogeneity of back extruded products in terms of solid phase distribution. The amount of this contribution depends on the contact time of this liquid with solid skeleton during liquid exertion to the clearance. Hence, at high ram speeds, small rams which demand taller initial slugs are more efficient in minimising the segregation. However, in the case of low speed back extrusion tests, liquid impotency gives rise to higher performance of the ram which sooner induces combined flow of both phases and thus, thick rams are advantageous over thin ones.

Keywords: Semisolid processing, Mechanical stirring, Phase segregation, Back extrusion, ZA27 alloy

Introduction

Semisolid processing of alloys is fraught with difficulties such as segregation of phases which, as a result, has heterogeneity of products in terms of solid phase distribution. This heterogeneity signifies composition departure from the average and giving up the advantages owing to which a specific alloy designation has been developed. Although several researches have been performed to shed light on the phase segregation observed during thixoforming processes,1–5 a deeper insight into this matter demands further experimental work.

Chen et al.6 proposed a model to describe deformation behaviour of semisolid alloys under compression. Their phenomenological model was consisted of four mechanisms with varying degrees of dominance depending on solid fraction, strain and deformation rate, namely, an increase in solid fraction and strain increases the dominance of plastic deformation mechanism while increasing deformation rate, promotes combined flow of solid and liquid phases once a given strain is exceeded. Using the presented model, it was therefore possible to explain squeezing out of liquid phase towards radial edge of the compressed samples, a phenomenon modelled and assessed experimentally by many authors.1,3,6–8

Loué et al.5 deduced data on viscosity of semisolid slurries subjected to back extrusion tests, by simply registering force versus displacement of ram. Besides its rheological contribution, back extrusion test can also serve as a means of assessing segregation tendency of slurries at higher shear rates than those made possible with simple compression test. For instance, in the case of A356 alloy prepared by magnetohydrodynamic (MHD) route, the critical extrusion speed with no discernible segregation was of the order of 530 mm s⁻¹. The extrusion speed was defined to be product of the ram speed and extrusion ratio, where the latter was a function of geometrical conditions of the setup, increasing with increasing ram diameter at a fixed cavity size. Therefore, it is possible to induce homogeneous flow by simply reducing the clearance between the ram and the cavity.

In sharp contrast to above observations, Basner et al.4 reported unsuitability of back extrusion technique as a viscometry test in the case of thin walled products; since at a similar ram speed, these products had a higher average liquid fraction in their walls than thick walled ones. The alloy they examined was a Sn–15.6 wt-%Pb alloy which, during the soaking time, had undergone liquid pooling owing to gravitational segregation.

Recently, in a study on the segregation of back extruded samples, Vieira et al.2 have accounted for the higher segregation tendency of A356 alloy at low ram speeds. Explanation was based on the well known theory of Darcy which, at a given temperature, predicts a percolation velocity dependent on the permeability of solid phase as well as the pressure gradient within the liquid phase. Segregation was found to become obvious when percolation velocity exceeded the ram speed.

This work is aimed at providing further information on the phase segregation characteristics of alloys consisted of phases with noticeable density difference, notably ZA27 alloy, during back extrusion. The presented model also accounts for the scatter in reported data of other experimental works.

Experimental procedure

Material

Material used for this study was the mechanically stirred zinc based ZA27 alloy (Zn–27.8±0.01 wt-%Al–2.5±
Preparation of the precursor materials for back extrusion tests was accomplished by mechanically stirring the alloy during its continuous cooling from the fully liquid state to a temperature within its mushy zone, followed by quenching in a water cooled steel mould. Stirring was performed in a bottom poured ceramic crucible with teeth on its internal wall while the stirrer was made of stainless steel. Processing parameters applied during stirring are summarised in Table 1.

**Back extrusion**

Back extrusion tests on slugs cut from mechanically stirred billets of ZA27 alloy were performed using a hydraulic jack system. Experiments were carried out at three constant ram speeds of 175, 100 and 60 mm s\(^{-1}\). Before each test, samples were resistively heated up to 452 ± 1°C, corresponding to a solid volume fraction of ∼0-45 and held for 55 min. Temperature was measured by means of a thermocouple inserted into a lead filled pocket located on the outer surface of the die nearest to the samples (Fig. 1a). Temperature homogeneity was once examined by directly measuring the sample temperature and comparing it with the one read from the lead filled pocket.

Three different ram diameters of 18, 20.4 and 22 mm were chosen while cavity had always a diameter of 25 mm. The semisolid slug height was adjusted so as to result in samples with an equal height of ∼5 cm at a final bottom thickness of 3 mm. After each test, samples were water quenched in order to minimise formation of voids and enhance the contrast between phases. Figure 1 displays some of the equipments used in the back extrusion step (Fig. 1a) as well as the stirrer and the crucible used in the mechanical stirring step (Fig. 1b and c).

**Characterisation**

After sectioning the cup shaped samples, they were examined in terms of average solid fraction \( f_s \) at the bottom as well as at different heights upwards the wall, using standard metallography and image analysis equipments. The sketch in Fig. 2 depicts the examined positions of a typical sample. Energy dispersive spectroscopy (EDS) quantitative analysis was also performed to measure aluminium contents at the bottom of some representative samples.

**Results and discussion**

Table 2 lists the data on normalised solid content, i.e. solid content at a given point divided by the average solid content of the slugs, at points A, B, C and D as shown in Fig. 2. The last column is a measure of severity of segregation, calculable as the absolute gradient of linear fit to the data of normalised solid content versus height (in metres). Comparing measures of segregation for the tests carried out with rams of identical diameters (Table 2), phase segregation becomes less severe as the ram speed increases. The results obtained herein regarding effect of deformation rate on homogeneity of back extruded samples are in accordance with those disclosed in the case of back extrusion and compression tests performed by other workers.\(^{1,2,5,7,9}\)

For the purpose of better comprehensibility, the results are comparatively summarised in Fig. 3.

### Table 1 Some processing parameters applied in mechanical stirring step

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rotational speed</td>
<td>600±20 rev min(^{-1})</td>
</tr>
<tr>
<td>Stirring start temperature</td>
<td>500±1°C</td>
</tr>
<tr>
<td>Withdrawal temperature</td>
<td>465±1°C</td>
</tr>
<tr>
<td>Cooling rate</td>
<td>1 K min(^{-1})</td>
</tr>
</tbody>
</table>

1 Some of equipments employed in this investigation of back extruded samples are in accordance with those disclosed in the case of back extrusion and compression tests performed by other workers.\(^{1,2,5,7,9}\)
Surprisingly, altering ram diameter has opposite effects on the degree of segregation in the cases of low and high ram speeds; at low ram speeds, decreasing ram diameter accentuates the segregation whereas at high ram speeds, the decrease leads to reduced phase segregation.

Before justification of results, it is necessary to address a difference among various alloy types examined so far with back extrusion, which is of importance to the present discussion. A356 alloy is basically consisted of Al and Si elements which have no important density difference. Therefore, even prolonged holding times do not produce any discernible gravitational segregation. Conversely, some alloy types such as Sn–Pb and Zn–Al, easily undergo gravitational segregation. As per the Zn–Al binary phase diagram of Fig. 4, upon solidification of ZA27 alloy, Al rich fcc \(a\) is the first phase to nucleate from the liquid. Thus, zinc content of the liquid gradually increases as solidification progresses. Partitioning of light aluminium in the solid and dense zinc in the liquid phase leads to liquid pooling on prolonged holding of the alloy in the temperature range between the peritectic reaction temperature (443°C) and that of the liquidus (490°C).\(^{10}\) As shown in Fig. 5, after holding ZA27 alloy for 55 min at 452°C, the heavy liquid phase has accumulated at the bottom of cavity owing to possession of a higher zinc content compared with the \(a\)-Al primary phase.

### Initial microstructure

The microstructure shown in Fig. 6 is the typical initial microstructure of slugs used in this work. It is basically consisted of primary phase in two different forms, i.e. large rosette like particles and small equiaxed dendrites. The latest theory to justify the rosette like morphology of primary phase is the one proposed by Fan and his co-workers\(^{11–13}\) and other modellers.\(^{14,15}\) The theory associates such morphologies with the change in the thickness of diffusion boundary layer and thus, in the solute transport pattern owing to stirring action. In fact, an increase in the vigorousness of agitation results in further facilitation of solute transport from between dendrite arms to the bulk liquid. Therefore, morphology gradually changes from dendritic to rosette like and

### Table 2  Back extrusion data under different conditions

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Ram diameter, mm</th>
<th>Ram speed, (\text{mm s}^{-1})</th>
<th>Extrusion ratio</th>
<th>Normalised solid content at different heights</th>
<th>Measure of segregation</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>(h=0) mm</td>
<td>(h=15) mm</td>
</tr>
<tr>
<td>BE28</td>
<td>18</td>
<td>175</td>
<td>2.08</td>
<td>1.24</td>
<td>0.91</td>
</tr>
<tr>
<td>BE27</td>
<td>20.4</td>
<td>175</td>
<td>2.99</td>
<td>1.11</td>
<td>1.00</td>
</tr>
<tr>
<td>BE29</td>
<td>22</td>
<td>175</td>
<td>4.43</td>
<td>1.11</td>
<td>0.84</td>
</tr>
<tr>
<td>BE12</td>
<td>18</td>
<td>100</td>
<td>2.08</td>
<td>1.99</td>
<td>1.20</td>
</tr>
<tr>
<td>BE14</td>
<td>20.4</td>
<td>100</td>
<td>2.99</td>
<td>1.33</td>
<td>0.98</td>
</tr>
<tr>
<td>BE8</td>
<td>22</td>
<td>100</td>
<td>4.43</td>
<td>1.67</td>
<td>1.11</td>
</tr>
<tr>
<td>BE23</td>
<td>18</td>
<td>60</td>
<td>2.08</td>
<td>2.00</td>
<td>1.44</td>
</tr>
<tr>
<td>BE25</td>
<td>20.4</td>
<td>60</td>
<td>2.99</td>
<td>1.93</td>
<td>0.42</td>
</tr>
<tr>
<td>BE13</td>
<td>22</td>
<td>60</td>
<td>4.43</td>
<td>1.90</td>
<td>0.89</td>
</tr>
</tbody>
</table>
eventually to globular. However, the rotational speed of the stirrer in this study has not been high enough to promote globular morphology and has only created large rosette like particles. Stirring action has additionally sped up the growth rate of primary phase and thus affected its size.

Darcy’s law
The main origin of segregation during thixoforming processes is connectivity of the solid globules in spite of their isolated and separate appearance in 2D metallographic sections. Therefore, liquid phase drains from the networked solid, provided its flow is not forceful enough to disagglomerate and carry the solid phase with itself. The most accepted theory describing percolation of a liquid from a porous medium is the one proposed by Darcy. Darcy’s law in the case of horizontal unidirectional flow may be written in form of

\[ V_X = \frac{K}{\eta f_L} \frac{dP}{dX} \]  

(1)

where \( K \) is permeability of the solid phase, \( \eta \) and \( f_L \) are viscosity and volume fraction of liquid phase respectively, and \( dP/dX \) is pressure gradient within the liquid phase. Since segregation degree is to a large extent controlled by solid-liquid interactions at the early moments of deformation application, and owing to similarity of liquid viscosity, solid fraction and permeability of samples just before the tests, it is mostly effects of extrusion ratio and ram speed on the pressure gradient term which lead to differences in segregation characteristics of samples.

Low ram speeds
The results of three tests, which were carried out at a ram speed of 60 mm s\(^{-1}\), are analogue of the original observations of Loué et al., since segregation is most severe in the case of the smallest ram (Fig. 3). These results can be justified in view of the fact that pressure gradient in the case of high extrusion ratios attains the critical value which is the onset of combined flow of solid and liquid phases sooner than when applying lower extrusion ratios. In other words, immediately after initial contact of ram and slug, interglobular and pooled liquid flow upwards the wall by passing through the networked solid. Later on, pressure gradient in the remained liquid becomes important enough to disagglomerate and carry the solid phase. However, this mechanism becomes active sooner for the thin walled sample than for the thick walled one. Therefore, combined flow of liquid and solid phases becomes active when the overall solid fraction of the unextruded part of the slug is less for the highest extrusion ratio than the lower ones. The sooner activation of the combined flow of phases which is associated with less segregation, along with another effect pointed out in the next paragraph, leads to more homogeneous products at higher extrusion ratios. Further deformation is accommodated through sliding and rolling of the solid phase followed by plastic deformation of solid under the ram.

There is also another effect controlling the degree of segregation which is usually overlooked in spite of its vital importance. This is the effect of initial height of the slugs which is inevitably different for different ram diameters, provided equal bottom thicknesses and wall heights are to be attained. Consequently, bottom of thick walled products experiences more strain than that of thin walled ones and at the final stages of experiments, they are more likely to undergo plastic deformation and densification of solid phase, the mechanism dominant at large strains.

High ram speeds
The less significant segregations encountered for the back extrusion tests performed at high ram speeds are a
result of steeper pressure gradients induced within the liquid phase. Hence, a higher percolation velocity is expected according to the Darcy’s law which, on the other hand, increases the chance of disagglomeration of the solid phase. As a result, the accompanying flow of both phases becomes dominant sooner for the tests performed at high ram speeds and their products are of more homogeneity.

Comparing measures of segregation relevant to the tests performed with different ram diameters at the ram speed of 175 mm s$^{-1}$, one realises that unlike low speed tests, enlargement of ram diameter has not contributed to homogeneity of products. Although a similar effect has been previously reported for another gravitationally segregated alloy (Sn–15.6 wt-%Pb), the effect of higher $dP/dX$ for the thick walled sample has not counteracted these two effects.

Considering proximity of solid fraction at the bottom of thick walled sample to the overall average solid fraction (Table 2), no deformation accommodation through plastic deformation of the solid phase has taken place. Figure 7b shows microstructure at the bottom of the sample extruded at 175 mm s$^{-1}$ which gives credence to the absence or negligibility of plastic deformation, whereas the sample extruded with the same ram but at the ram speed of 60 mm s$^{-1}$, displays significant solid deformation at the bottom (Fig. 7a). Aluminium contents relevant to regions shown in Fig. 7a and b were 55.63±0.5 and 35.41±0.5 wt-% respectively, both higher than the average aluminium content of the alloy, as per quantitative element analysis through SEMEDS. However, accumulation of Al rich primary phase at the bottom is far more pronounced for the slowly deformed sample.

**Intermediate ram speeds**

Back extrusion experiments performed with a ram speed of 100 mm s$^{-1}$ show features in common with two other ram speeds. In other words, results of this series of tests can be interpreted with regard to both phenomenological models already explained for high and low ram speeds. According to Fig. 3, neither the thick (22 mm diameter) nor the thin (18 mm diameter) rams have shown capability of the ram with an intermediate diameter of 20.4 mm, in minimising segregation. The best explanation for this observation is to consider the fact that in ram speeds near a particular level, the ram diameter becomes crucial in that its small variation may promote dominance of either of two models already stated for deformation at low and high ram speeds. In view of the results, rams with diameters of 20.4 and 18 mm diameter becomes combined flow of both phases as the ram speed increases. In view of the results, a ram speed of 175 mm has been high enough to turn the ineffective flow at 60 mm s$^{-1}$ ram insertion speed into an effective one which is the basis for the following discussion.

Once the contact between the ram and the slug is established, liquid pressure gradient becomes significant enough to cause disagglomeration and conveyance of the primary grains with the flow. At such high ram speeds, pooling of the liquid phase plays an important role and leads to an anomalous segregation characteristic. With insertion of ram into the slug, liquid phase tends to flow forcefully into the clearance. Since a large proportion of liquid is in form of gravitationally segregated at the bottom, the amount of solid carried by this effective liquid is a function of duration, it remains in contact with the solid skeleton. Hence, the initial height of the slugs becomes a paramount factor, since it determines how long it takes for the liquid to pass the solid network. In addition to longer passage times, the amount of pooled liquid also increases for taller initial slugs. Combination of a higher extent of pooled liquid and longer passage time of liquid through the solid skeleton gives rise to lesser extents of segregation in the case of thick walled samples. It must be noted that even the effect of higher $dP/dX$ for the thin walled sample, has not counteracted these two effects.
ram diameters result in low liquid pressure gradients, there is enough justification for ineffectiveness of the liquid in the case of 18 mm diameter ram.

Schematic representation of the models outlined for the high and low ram speed tests are respectively shown in Figs. 8 and 9 which illustrate solid distribution at different stages during back extrusion using both small and large rams.

Conclusions

1. Mechanical stirring of ZA27 alloy at 600 rev min⁻¹ during cooling from fully liquid state to its mushy zone followed by quenching results in a microstructure consisting of primary phase in two different forms: large rosette like particles and fine dendritic features.
2. Initial height of the slugs should not be overlooked when characterising segregation of back extruded products, since tall slugs undergo more strain than short ones at the same final bottom thickness.
3. Under some conditions, pooled liquid in the case of gravitationally segregated alloys such as ZA27 can contribute to homogeneity of microstructure of back extruded products.
4. At sufficiently high deformation rates, liquid phase becomes effective in that it can disagglomerate and carry the solid phase. Under these circumstances, tall slugs corresponding to thick walled products lead to more homogeneous products since contact time of the pooled liquid with the solid skeleton is longer.
5. At low deformation rates, liquid phase is ineffective and merely exudes from the solid skeleton. As a result, enlargement of ram reduces the phase segregation by inducing a higher pressure gradient within the liquid phase.

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