Cavitation characteristics in AZ31 Mg alloys during LTSP or HSRSP

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Abstract

The cavitation behaviors in heavily extruded fine-grained AZ31 Mg alloys loaded at 200–400 °C and 6×10^{-4}–1×10^{-2} s^{-1} have been analyzed, covering the low temperature or high strain rate superplasticity (LTSP or HSRSP) regimes. Cavities smaller than 2 μm are basically nearly spherical in shape and randomly dispersed in the matrix, suggesting the involvement of diffusion or superplasticity diffusion controlled cavity growth mechanism. In contrast, the larger cavities tend to be elongated with the long axis aligned toward the loading axis, implying the plasticity controlled cavity growth mechanism. However, it is also recognized that coalescence may play an important role in the elongation of cavities. The cavitation in AZ31 Mg appears to be much less severe than those observed in Al based alloys or composites as a result of higher grain boundary diffusion rate, well-defined grain structure with a high volume fraction of high angle grain boundaries, smooth operation of grain boundary sliding, and the absence of second phases in this solution-hardening alloy.

Keywords: Magnesium alloy; Cavitation; Superplasticity; Grain boundary sliding

1. Introduction

Magnesium alloys have been used in a wide variety of structural and nonstructural applications due to their unique properties such as low density and high specific strength and elastic modulus [1–3]. Among many Mg based alloys, the AZ (Mg–Al–Zn), AM (Mg–Al–Mn) and ZK (Mg–Zn–Zr) based alloys seem to be the most popular; with the AZ91 and AZ31 alloys being cheaper and occupying the highest market. There have been numerous efforts in processing metallic alloys into fine-grained materials, so as to exhibit high strain rate superplasticity (HSRSP) and/or low temperature superplasticity (LTSP). Due to the hexagonal nature of Mg alloys, rolling at room or elevated temperatures tends to cause side cracking. In contrast, extrusion at elevated temperatures will result in satisfactory products in the form of bars, plates, sheets or tubes [4].

The lattice and grain boundary diffusion rates of Mg base alloys are around 3× and 18× faster, respectively, than those of Al base alloys at the typical processing and heat treatment temperature of ~300 °C [5]. Owing to the relatively higher diffusion rate, recrystallization in Mg alloys can occur rapidly during extrusion at elevated temperatures. The nearly fully recrystallized grains, measuring around 1–20 μm, in the extruded products has been shown to exhibit supreme LTSP and HSRSP behavior [6–8] at 200–450 °C and 10^{-4}–10^{-1} s^{-1}. Such superplastic capability of the commercial Mg alloys can be applied in the press forming [9] or press forging process for making Mg enclosures for electromagnetic shielding purpose in the 3C industry. With the nearly fully recrystallized grains possessing a large fraction of high angle grain boundaries (HAB) in many thermomechanically processed Mg alloys [10], grain boundary sliding (GBS) can be more readily operated during superplastic deformation. Thus, cavitation might be suppressed. Cavitation is an essential issue, since not all fine-grained materials with high strain rate sensitivity values would exhibit high superplastic elongations.
Theoretical modeling on cavitation can be classified into crack nucleation [11–13], growth [12,14–18], and coalescence [19–23]. Among these three, the growth stage appears to be most critical, since most materials deform plastically during processing or usage under this stage. Upon entering the coalescence stage, a drastic failure would usually occur immediately. The crack growth mechanisms during superplastic straining were mostly derived from those developed for creep deformation, which are basically classified into three categories: (i) diffusion (DIF) controlled growth [12,14–16], (ii) plasticity (PLA) controlled growth [17], and (iii) superplasticity diffusion (SPD) controlled growth [18].

For DIF cavity growth, the crack radius growth rate \( \frac{dr}{dt} \) and the crack volume growth rate \( \frac{dV}{dt} \) can be derived as [12,15]

\[
\frac{dr}{dt} = \frac{\Omega \delta D_{gb} \sigma}{5kT r^2} \left( \frac{\sigma - 2\gamma/r}{\dot{\varepsilon}} \right) \tag{1}
\]

and

\[
\frac{dV}{dt} = 2\pi \delta D_{gb} kT \left[ \frac{(\sigma + p - 2\gamma/r)(1 - r^2/f^2)}{\ln \left( \frac{r_0}{r} \right) - \frac{1}{3} + \frac{\pi}{6} \left( 1 - \frac{2}{r_0} \right) } \right], \tag{2}
\]

where \( \Omega \) is the atomic volume, \( \delta \) is the grain boundary thickness, \( D_{gb} \) is the grain boundary diffusion coefficient, \( kT \) is the usual meaning, \( \sigma \) is the tensile stress, \( \gamma \) is the crack surface energy, \( \dot{\varepsilon} \) is the strain rate, \( p \) is the cavity internal pressure, and \( 2l \) is the interspacing between two cracks. In parallel, the equations for PLA crack growth are given as [17]

\[
\frac{dr}{dt} = r - \frac{3\gamma}{2\sigma} \tag{3}
\]

and

\[
\frac{dV}{dt} = A \varepsilon \left( 1 - \frac{3\gamma}{2\sigma} \right). \tag{4}
\]

By integrating Eq. (4), the cavity volume fraction is given by the well known form

\[
C_v = C_0 \exp(\eta \varepsilon), \tag{5}
\]

where \( \eta \) is the cavity growth exponent. For fine-grained materials with a grain size \( d \) deformed at lower strain rates, the crack growth rate under the SPD regime is given by [18]

\[
\frac{dr}{dt} = \frac{45 \Omega \delta D_{gb} \sigma}{d^2 kT \dot{\varepsilon}}. \tag{6}
\]

Previous studies on cavitation during superplastic deformation were predominantly centered on the fine-grained Al alloys strained at high temperatures (450–550 °C) and lower strain rate \( (10^{-3} \text{–} 10^{-4} \text{ s}^{-1}) \) [24–29]. Studies on the cavitation characteristics and suppression methods in Al based composites under the HSRSP regime have been relatively less [30–32]. There has been rarely any report on the cavitation in Mg based alloys subject to LTSP (200–300 °C) and HSRSP \( (10^{-2} \text{–} 10^{-1} \text{ s}^{-1}) \). In this paper, the results obtained from the AZ31 alloys are presented.

2. Experimental methods

The AZ31B alloys, purchased from the CDN Company, Deltabc, Canada, were produced by the semi-continuous casting method, with a chemical composition of Mg–3.02 wt% Al–1.01 wt% Zn–0.3 wt% Mn. The average grain size under the as-received condition was 75 μm. The ingot was then warm extruded at \( \sim 300 \text{ °C} \) and \( \sim 1 \times 10^{-3} \text{ s}^{-1} \), with a fixed extrusion ratio of 100:1. The extrusion strain rate \( \dot{\varepsilon}_{\text{ext}} \) was calculated by [33]

\[
\dot{\varepsilon}_{\text{ext}} = \frac{6v_{\text{ext}}}{D_{\text{ext}}} \ln R, \tag{7}
\]

where \( v_{\text{ext}} \) is the extrusion speed, \( D_{\text{ext}} \) the input material diameter, and \( R \) the extrusion ratio.

Tensile specimens with 5.5 mm gauge length were machined from the as-extruded plates, with the loading axis parallel to the extrusion direction. Tensile tests at room (25 °C) and elevated temperatures (200, 250, 300, 350, 400, and 450 °C) and strain rates from \( 6 \times 10^{-4} \text{ to } 1 \times 10^{-1} \text{ s}^{-1} \) were conducted using an Instron 5582 universal testing machine, with the temperature controlled within \pm 2 °C. Among these conditions, the analyses of cavity growth mechanism were performed for the six representative cases, namely, at 200 °C (lower temperature, LT), 300 °C (medium temperature, MT), and 400 °C (higher temperature, HT) with a strain rate of \( 6 \times 10^{-4} \text{ s}^{-1} \) (lower strain rate, LR) or \( 1 \times 10^{-2} \text{ s}^{-1} \) (higher strain rate, HR). The jump strain rate tests [34] were performed at various temperatures and strain rates for measuring the apparent strain rate sensitivity, \( m \), as a function of strain level.

The tensile tests were terminated at selected strain levels, and the specimens were sampled for optical microscopy (OM) and scanning electron microscopy (SEM) characterization. The samples were first carefully polished step by step, with the final polishing using 0.05 μm Al2O3 powders, to reveal the cavities, and then cleaned ultrasonically in acetone. Then the specimens were photographed using a high resolution digital camera and the images were analyzed by the Optimas image analysis software [35]. In order to quantitatively measure the cavities, a threshold gray level of the digital image needs to be defined out of the 0 (completely white) to 255 (254, completely black) level. After numerous confirmations, the background appears to be at a gray level below 50 (mostly 20–35), precipitates around 60–90, and cavities above 140. In this study, the pixels with a gray level greater than 140 are classified as cavities. The cavity volume fraction \( C_v \) is assigned to be equal to the area fraction, assuming that the cavities are present in a 3D uniform manner. During the study, it was found
that the $C_v$ data measured from the specimen surface are slightly lower than those obtained from the specimen center layer. The data presented in this paper are all measured from the center region, i.e., with the plate thickness being ground away for around 40–50%.

The grain structure and the identification for the relation between cavities and grain boundaries (or grain triple points) were revealed by further etching of the above polished samples in a solution of 100 ml ethanol + 5 g picric acid + 5 ml acetic acid + 10 ml distilled water. The misorientation angle distribution of the as-extruded alloys was constructed using electron back-scattered diffraction (EBSD) attached with the FEG-SEM. The specimens need to be carefully electrochemically polished, and examined soon after polishing. Due to the low efficiency of backscattered electrons, the intensity of Kikuchi patterns from the low atomic number element Mg will be low, and longer accumulation time is needed. The pixel size was typically around 1 μm, and around 4000 pixel points were measured for each mapping and the construction of grain misorientation distribution. On average, approximately 800 grains were included for each case.

3. Results

3.1. Grain structures

Fig. 1 shows the nearly equiaxed grain structure in the AZ31 plate extruded at 300 °C and 1×10⁻³ s⁻¹, measuring a grain size ($d$) of 2.9 μm. From the well-defined clear grain boundaries exposed by the chemical etching, they appear to be HAB. It was found that a lower extrusion temperature (~250 °C) and a higher extrusion strain rate (10⁻²–10⁹ s⁻¹) would result in a finer grain size [4]. The finest grain size achieved through high ratio extrusion of the AZ31 alloy was ~0.7 μm, but the grain boundaries sometimes were low angle boundaries. In all cases, the grain size can be effectively refined from the initial 75 μm through one-pass warm extrusion. All cavitation studies described below were performed on the plate extruded at 300 °C and 1×10⁻³ s⁻¹.

Upon static annealing and superplastic loading at 200–400 °C, the grain size would gradually increase, as shown in Fig. 2, with more rapid grain growth occurring under the dynamic tensile loading condition. It is noted that the grains have grown to a size of about 20 μm at the later stage of superplastic straining at a higher temperature of 400 °C and a lower strain rate of 6×10⁻⁴ s⁻¹. The larger grain size in this very specimen might result in different cavitation mechanisms. In contrast, the grain size remains nearly unchanged (or even refined further) at lower holding temperatures of 200 and 250 °C, irrespective of static annealing or dynamic straining at low or high strain rates.

The grain mutual misorientation distribution was measured by EBSD, and one typical result for such recrystallized grains under the as-extruded condition is displayed in Fig. 3. The histogram of the occurrence frequency as a function grain boundary relative misorientation angle obtained by SEM-EBSD in the fine-grained Mg alloys. The HABs with misorientation angles greater than 15° occupy 70–90% of the total population, significantly higher than the Al counterparts.
depicted in Fig. 3. The HAB fraction with misorientation angles greater than 15° was scattered within 70–90%, which is appreciably higher than the ~55% measured from the 5083 Al alloy which was subjected to severe rolling type TMT processes [36]. This might be one of the main reasons that cause a drastic distinction between the cavitation behaviors in Mg and Al based alloys.

3.2. Superplastic results

The room temperature yield stress, ultimate tensile stress, and elongation of the as-extruded AZ31 plate specimens are 268 MPa, 329 MPa, and 43%, respectively, which are appreciably higher than the 100 MPa, 160 MPa, and 9% for the as-received ingot. The tensile results at elevated temperatures are presented in Fig. 4. The highest LTSP elongations at 200 and 250 °C at a low strain rate of $6 \times 10^{-4}$ s$^{-1}$ are ~300% and ~450%, while the highest HSRSP elongations at $1 \times 10^{-2}$ and $1 \times 10^{-1}$ s$^{-1}$ are 510% and 260%, respectively. The SP elongation showed a maximum peak of ~740% at 300 °C and $1 \times 10^{-3}$ s$^{-1}$.

Selected specimens were strained and then stopped at true strain levels of ~0.3, 0.7, 1.1, and 1.5 (2.0 if applicable) for cavitation examinations. For most cases, the apparent initial hardening and subsequent softening are seen except for those loaded at higher temperatures and lower strain rates. The average apparent strain rate sensitivity $m$ data determined by the jump strain rate tests in the range of 0.20–0.55 are summarized in Table 1. After considering the threshold stress [37], the true strain rate sensitivity was mostly around 0.45–0.6 (except for the cases at 200 °C and higher strain rates), suggesting that GBS was the dominant deformation mechanism over most of the current test regimes.

<table>
<thead>
<tr>
<th>$T$ (°C)</th>
<th>Low strain rate</th>
<th>High strain rate</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$5 \times 10^{-4}-8 \times 10^{-4}$ s$^{-1}$</td>
<td>$9 \times 10^{-3}-2 \times 10^{-2}$ s$^{-1}$</td>
</tr>
<tr>
<td>200</td>
<td>0.31</td>
<td>0.20</td>
</tr>
<tr>
<td>300</td>
<td>0.46</td>
<td>0.37</td>
</tr>
<tr>
<td>400</td>
<td>0.55</td>
<td>0.40</td>
</tr>
</tbody>
</table>

3.3. Cavity examinations (OM and SEM)

The typical surface topography SEM micrographs taken from the specimens loaded at 200, 300 and 400 °C to a strain around 0.7 are shown in Fig. 5. Some microcracks are evident at the grain boundaries or triple points. The grains still remained nearly equiaxed throughout the straining until fracture. Fig. 6 shows the variation of cavity number density (per unit area)
as a function of strain level. In all cases, the cavity number density increased rapidly over $\varepsilon = 0.5$, and gradually reached the nearly saturated level at $\varepsilon \sim 0.5–1.0$, suggesting that the crack nucleation predominantly occurred at the very early stage and crack growth dominated most of the straining process. The saturated number density, $\#$, was mostly scattered within 1000–2000 mm$^{-2}$. The $\#$ values measured from OM and SEM for the 200 °C specimens are subjected to greater uncertainty due to the many small cavities less than 0.5 μm; the $\#$ values are greater than 1000 mm$^{-2}$ (not shown in Fig. 6). Basically, the $\#$ values increase with increasing strain rate and decreasing loading temperature.

The representative variations of cavity size distribution for different strain levels, loading temperatures, and strain rates are shown in Fig. 7. In general, cavities smaller than 2 μm are consistently occupied around 60–65%, except for strain levels greater than 1.8 where the fraction dropped to 40–50%. With increasing strain level and loading temperature, the cavity size tends to shift to a higher level. The effect from strain rate was relatively minor. The increasing trend of cavity size with increasing temperature might lead to confusion since it is commonly believed that a higher temperature would enhance accommodation and suppress cavitation. This phenomenon was actually a result of pronounced grain growth occurred at 350–450 °C, as depicted in Fig. 2. The larger grain size would lead to less smooth operation of GBS and a longer distance for diffusion or dislocation accommodation.

The cavity long to short axis aspect ratio $\rho$ and the angle $\psi$ for the cavity long axis with respect to the loading axis have also been determined. In order to demonstrate the difference in various cases more clearly, the cavity characteristics were assessed for the small group (<2 μm) and the largest 20 cavities. For the small group, $\rho$ was consistently scattered around 1.3–1.5, with a random distribution for the $\psi$ angle (or an average $\psi$ angle of around 45°) with respect to the loading axis, as shown in Fig. 8(a) and listed in Table 2. In other words, the small cavities are basically nearly spherical in shape and randomly dispersed in the matrix, suggesting the involvement of DIF [12,14–16] or SPD [18] cavity growth mechanism. In contrast, the largest 20 cavities tend to show an increasing trend for the aspect ratio $\rho$ (toward 2.5), with a greater tendency to align along the loading axis (or a decreasing average $\psi$ angle toward 10–20°) upon increasing the strain level (Fig. 8(b) or Table 2). Such behavior would imply the PLA cavity growth mechanism [17] during which the cavities are influenced by the plastic metal flow.

With the cavity number density and size distribution, the cavity volume fraction $C_v$ is ready to be determined. Fig. 9 compares the variation of $C_v$ as a function of strain level. For most cases, $C_v$ data were around 2% or less. But the specimens loaded at a higher temperature of 400 °C and a lower strain rate of $6 \times 10^{-4}$ s$^{-1}$ showed an apparent increasing trend at $\varepsilon > 1.5$, due to the significant growth of grain size (10–20 μm) during the long time exposure at higher temperatures. This situation would not occur for loading at 400 °C and $1 \times 10^{-2}$ s$^{-1}$, since the total loading time until failure was only a few minutes.
The cavity appearance near the fracture tip for the 300 (MT) and 400 °C (HT) specimens is presented in Fig. 10. Coalescence of the closely nucleated cavities can be seen. It is recognized that coalescence should play an important role in the elongation of cavities. Apparent local necking was observed in specimens loaded at 200 °C (LT, not shown in Fig. 10), suggesting the dislocation slip deformation as evident from the lower apparent m-values of 0.2–0.35. On the other hand, more diffuse necking and minimum necking were seen in the 300 and 400 °C specimens, respectively. The cavities in specimens loaded at $1 \times 10^{-2}$ s$^{-1}$ (HR) were higher in number density and smaller in size, as compared with those loaded at $6 \times 10^{-4}$ s$^{-1}$ (LR). Finally, the cavity location is of concern. Fig. 11 shows an example of the polished specimens followed by further etching. The cavities are seen to be present predominantly at the grain boundaries or triple points, with few occurrences in the grain interior. Some of the cavities located at the grain triple points appeared to be spherical in shape, consistent with the superplastic diffusion controlled cavity growth mechanism [18].

**Table 2**

A typical example measured for the AZ31 plate loaded at 300 °C and $6 \times 10^{-4}$ s$^{-1}$ for the cavity aspect ratio $\rho$, angle $\psi$ with respect to the loading axis, and the average cavity size for the small (<2 µm) and the large (the largest 20) cavity groups.

<table>
<thead>
<tr>
<th>Strain</th>
<th>Cavities &lt;2 µm</th>
<th>Largest 20 cavities</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Aspect ratio $\rho$</td>
<td>$\psi$ (°) Average diameter (µm)</td>
</tr>
<tr>
<td>0.24</td>
<td>1.4 45</td>
<td>1.2</td>
</tr>
<tr>
<td>0.71</td>
<td>1.3 43</td>
<td>1.1</td>
</tr>
<tr>
<td>1.28</td>
<td>1.4 49</td>
<td>1.0</td>
</tr>
<tr>
<td>1.80</td>
<td>1.4 46</td>
<td>1.0</td>
</tr>
<tr>
<td>2.03</td>
<td>1.4 46</td>
<td>1.1</td>
</tr>
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</table>
nucleation which is experimentally observed after $\varepsilon = 0.3$) and multiplying the cavity number density $N$, the cavity volume fraction $C_v$ as a function of strain $\varepsilon$ can be written as

$$C_v = C_0 + \frac{N4\pi\delta D_{gb} \sigma}{5kT \dot{\varepsilon}^2}$$

where $C_0$ is the cavity volume fraction at $\varepsilon = 0$, and is estimated to be $\sim 0.4\%$ induced by severe warm extrusion.

Secondly, $d\dot{\varepsilon}/d\varepsilon$ and $C_v$ for the PLA cavity growth have been given by Eqs. (3) and (5). Finally, $d\dot{\varepsilon}/d\varepsilon$ for the SPD mechanism has been derived as Eq. (6). Using the integration procedure, Eq. (6) can be written as

$$r = \frac{45\Delta D_{gb} (\frac{\sigma}{\dot{\varepsilon}})}{d^2 kT} e + r_0$$

Again, assuming that the cavity is a sphere and the initial cavity radius is small enough to neglect the $r_0$ term, the cavity volume fraction $C_v$ as a function of strain can be expressed as

$$C_v = C_0 + \frac{N4\pi}{3} \left( \frac{45\Delta D_{gb} \sigma}{d^2 kT \dot{\varepsilon}^2} \right)^{3/2}$$

Note that there is a grain size dependence $d^{-1/2}$ on the $C_v$ term; the larger grain size would lead to lower superplastic diffusion and lower $C_v$. In summary, the experimental results of the cavity growth rate $d\dot{\varepsilon}/d\varepsilon$ and the cavity volume fraction $C_v$ can be assessed in terms of Eqs. (1) and (8) for the DIF model, Eqs. (3) and (5) for the PLA model, and Eqs. (6) and (11) for the SPD model.

4.2. Data analyses

Previous cavitation analyses on the superplastic materials were all centered on the high temperature regime, e.g., 450–550 °C for the Al based alloys or composites.

<table>
<thead>
<tr>
<th>$\dot{\varepsilon}$</th>
<th>$\varepsilon \sim 0.7$</th>
<th>$\varepsilon \sim 1.5$</th>
<th>$\dot{\varepsilon}$</th>
<th>$\varepsilon \sim 0.7$</th>
<th>$\varepsilon \sim 1.5$</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>200 °C</strong></td>
<td></td>
<td></td>
<td><strong>300 °C</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Small cavities $d_{ave} \sim 1.1$</td>
<td>$\mu m; \rho \sim 1.6; \psi \sim 33^\circ$</td>
<td>–</td>
<td>Small cavities $d_{ave} \sim 1.1$</td>
<td>$\mu m; \rho \sim 1.8; \psi \sim 27^\circ$</td>
<td>–</td>
</tr>
<tr>
<td>Largest 20 ones $d_{ave} \sim 8.4$</td>
<td>$\mu m; \rho \sim 1.8; \psi \sim 25^\circ$</td>
<td>(1)</td>
<td>Largest 20 ones $d_{ave} \sim 4.7$</td>
<td>$\mu m; \rho \sim 2.3; \psi \sim 19^\circ$</td>
<td>(2)</td>
</tr>
<tr>
<td><strong>300 °C</strong></td>
<td></td>
<td></td>
<td><strong>400 °C</strong></td>
<td></td>
<td></td>
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<tr>
<td>Small cavities $d_{ave} \sim 1.3$</td>
<td>$\mu m; \rho \sim 1.3; \psi \sim 43^\circ$</td>
<td>(1)</td>
<td>Small cavities $d_{ave} \sim 1.3$</td>
<td>$\mu m; \rho \sim 1.4; \psi \sim 48^\circ$</td>
<td>(1)</td>
</tr>
<tr>
<td>Largest 20 ones $d_{ave} \sim 8.7$</td>
<td>$\mu m; \rho \sim 1.6; \psi \sim 29^\circ$</td>
<td>(2)</td>
<td>Largest 20 ones $d_{ave} \sim 12.0 \mu m; \rho \sim 1.7; \psi \sim 21^\circ$</td>
<td>(2)</td>
<td>Largest 20 ones $d_{ave} \sim 9 \mu m; \rho \sim 1.6; \psi \sim 26^\circ$</td>
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<td><strong>400 °C</strong></td>
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<td><strong>400 °C</strong></td>
<td></td>
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<tr>
<td>Small cavities $d_{ave} \sim 1.3$</td>
<td>$\mu m; \rho \sim 1.3; \psi \sim 49^\circ$</td>
<td>(1)</td>
<td>Small cavities $d_{ave} \sim 1.3$</td>
<td>$\mu m; \rho \sim 1.5; \psi \sim 46^\circ$</td>
<td>(1)</td>
</tr>
<tr>
<td>Largest 20 ones $d_{ave} \sim 10.7 \mu m; \rho \sim 1.6; \psi \sim 35^\circ$</td>
<td>(2)</td>
<td>Largest 20 ones $d_{ave} \sim 51.6 \mu m; \rho \sim 1.9; \psi \sim 26^\circ$</td>
<td>(2)</td>
<td>Largest 20 ones $d_{ave} \sim 11.8 \mu m; \rho \sim 1.8; \psi \sim 19^\circ$</td>
<td>(2)</td>
</tr>
<tr>
<td>Small cavities $d_{ave} \sim 1.2$</td>
<td>$\mu m; \rho \sim 1.4; \psi \sim 35^\circ$</td>
<td>(1)</td>
<td>Small cavities $d_{ave} \sim 1.2$</td>
<td>$\mu m; \rho \sim 1.4; \psi \sim 36^\circ$</td>
<td>(1)</td>
</tr>
<tr>
<td>Largest 20 ones $d_{ave} \sim 19.5 \mu m; \rho \sim 2.2; \psi \sim 15^\circ$</td>
<td>(2)</td>
<td>Largest 20 ones $d_{ave} \sim 19.5 \mu m; \rho \sim 2.2; \psi \sim 15^\circ$</td>
<td>(2)</td>
<td>Largest 20 ones $d_{ave} \sim 19.5 \mu m; \rho \sim 2.2; \psi \sim 15^\circ$</td>
<td>(2)</td>
</tr>
</tbody>
</table>
In what follows, the analyses of the six representative loading conditions, namely, three temperatures of 200 °C (LT), 300 °C (MT), and 400 °C (HT) and two strain rates of $6 \times 10^{-4}$ s$^{-1}$ (LR) and $1 \times 10^{-2}$ s$^{-1}$ (HR), are described. For simplicity, two strain levels are selected for demonstrating the cavity evolution characteristics, i.e., $\varepsilon \sim 0.7$ (lower strain) and $\varepsilon \sim 1.5$ (higher strain).

4.2.1. Condition: 200 °C and $6 \times 10^{-4}$ s$^{-1}$ (LT and LR)

The SP elongation for this case is 300%, with an average grain size $D$ of 2.5 μm and an average apparent $m$ of 0.31. Using the relationship proposed by Stowell et al. [19]

$$\eta = \frac{3}{2} \left( \frac{m + 1}{m} \right) \sinh \left( \frac{2(2 - m)}{3(2 + m)} \right),$$  \hspace{1cm} (12)

the theoretically predicted $\eta$ is 3.22. At $\varepsilon \sim 0.7$, $C_v$ is $\sim 0.65\%$ and the saturated cavity density $#$ is greater than 1000 mm$^{-2}$ (the exact datum is difficult to define owing to the presence of numerous cavities smaller than 0.5 μm).

Due to the low loading temperature (200 °C, or 0.5 $T_m$), the DIF mechanism would not be dominant except for very small cavities ($< 1$ μm). Using the appropriate parameters listed in Table 3, the calculated $C_v$ from diffusion model (even with $C_0 = 0.4\%$) still accounts only for $\sim 0.4\%$, i.e., nearly no contribution and much lower than the experimental data, as compared in Fig. 12(a). This is also true for the calculated $dr/d\varepsilon$ based on the DIF model, as shown in Table 4. The DIF will provide contribution only to very small cavities. The same situation also applies to all other five loading conditions.

Fig. 12. Comparison of the experimentally measured cavity volume fraction with the theoretical predictions based on the diffusion model (DIF), plasticity model (PLA), and superplasticity diffusion model (SPD) with various grain sizes for the AZ31 specimens loaded at six different conditions. The initial cavity volume fraction before tensile loading, $C_0$, is $\sim 0.4\%$. 
Judging from the medium values for cavity aspect ratio \( \rho \) and cavity long axis aligning angle \( \psi \) for small cavities \( \leq 2 \mu m \) (1.6° and 33°) and the largest 20 cavities (1.8° and 25°) in Table 3, both SPD and PLA mechanisms are operative. This is consistent with the trend for \( C_v \) in Fig. 12(a) and for \( dr/d\varepsilon \) in Table 4, where the experimental data fall in between those predicted by SPD and PLA, but closer to SPD, implying that the cavity growth was mainly controlled by SPD, with minor contribution from the PLA mechanism, as summarized in Table 5 for the roughly estimated relative contributions.

4.2.2. Condition: 200 °C and \( 1 \times 10^{-2} \) s^{-1} (LT and HR)

Under this condition, the SP elongation drops to 180%, with an average \( D \) of ~2.5 \( \mu m \), an average \( m \) of ~0.20, and a theoretically predicted \( \eta \) (by Eq. (12)) of 5.11. At \( \varepsilon \sim 0.7 \), the saturated cavity density is greater than 1000 mm^{-2} and \( C_v \) is ~0.25%.

At such a higher loading rate, the DIF contribution becomes even less, as evident from the theoretically predicted \( dr/d\varepsilon \) values of 0.003–0.006 \( \mu m \) in Table 4. From the \( \rho \) and \( \psi \) values for larger cavities (2.3 and 19°) in Table 3, PLA should have been dominant. The experimental \( dr/d\varepsilon \) value for larger cavities also favors the PLA mechanism. However, the \( C_v \) experimental data in Fig. 12(b) seem to fit better to the SPD mechanism. It should be noted that the \( C_v \) term includes all cavities, small and large, thus the trend in Fig. 12(b) cannot delineate fully the growth mechanism for large cavities. The controversy might also be caused by the overestimation of \( \eta \) and the underestimation of the \( C_v \) and \# values for those micro-cavities smaller than 0.5 \( \mu m \). From the direct observation of the cavity shape and alignment, it is postulated that SPD and PLA dominate the growth of small and large cavities, respectively, as shown in Table 5.

4.2.3. Condition: 300 °C and \( 6 \times 10^{-4} \) s^{-1} (MT and LR)

Under this condition, the SP elongation is 660%, with an average \( D \) of ~5 and 9 \( \mu m \) at \( \varepsilon \sim 0.7 \) and 1.5, an average \( m \) of ~0.46, and a theoretically predicted \( \eta \) of 2.05. The saturated cavity density is ~1850 mm^{-2} and \( C_v \) is ~0.8% and 1.0% at \( \varepsilon \sim 0.7 \) and 1.5.

At \( \varepsilon \sim 0.7 \) and 1.5, both DIF and SPD should have dominated for small cavities owing to the data of \( \rho = 1.3 \) and 1.4 and \( \psi = 43° \) and 48°; and SPD plus minor PLA would control the growth of those largest 20 cavities with \( \rho = 1.6 \) and 1.7 and \( \psi = 29° \) and 21°. The trends of \( C_v \) in Fig. 12(c) and \( dr/d\varepsilon \) in Table 4 reveal a similar information; SPD overwhelms throughout the straining, with minor contribution from PLA at higher strain levels (Table 5).

4.2.4. Condition: 300 °C and \( 1 \times 10^{-2} \) s^{-1} (MT and HR)

Under this condition, the SP elongation is 500%, with an average \( D \) of ~3 and 4 \( \mu m \) at \( \varepsilon \sim 0.7 \) and 1.5, an average \( m \) of ~0.37, and a theoretically predicted \( \eta \) of 2.64. The saturated cavity density is ~2000 mm^{-2} and \( C_v \) is ~1.1% and 1.9% at \( \varepsilon \sim 0.7 \) and 1.5.

For small cavities, SPD overwhelms, and DIF becomes much less important due to the high strain rate. For large cavities, PLA strengthens its role as seen from higher \( \rho \) (2.1) and lower \( \psi \) (16°) for the HR specimens strained to \( \varepsilon \sim 1.5 \) (Table 3). Fig. 12(d) and Table 4 also show this situation, the experimental \( C_v \) and \( dr/d\varepsilon \) data

<table>
<thead>
<tr>
<th>Table 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Summary of the operating cavity growth rate ( dr/d\varepsilon ) (in unit of ( \mu m )) for the small (&lt;2 ( \mu m )) and the largest 20 cavities in specimens strained to 0.5 and 1.5 under various loading conditions</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>( \dot{\varepsilon} = 6 \times 10^{-4} ) s^{-1} (LR)</th>
<th>( \dot{\varepsilon} = 1 \times 10^{-2} ) s^{-1} (HR)</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \varepsilon \sim 0.7 )</td>
<td>( \varepsilon \sim 0.7 )</td>
</tr>
<tr>
<td>( \varepsilon \sim 1.5 )</td>
<td>( \varepsilon \sim 1.5 )</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>200 °C</th>
<th>300 °C</th>
<th>400 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>(1) Small cavities DIF: 0.08 PLA: 0.55 SPD: 0.89</td>
<td>(1) Small cavities DIF: 0.006 PLA: 0.55 SPD: 0.89</td>
<td>(1) Small cavities DIF: 0.14 PLA: 0.55 SPD: 0.78</td>
</tr>
<tr>
<td>Exp: 0.2</td>
<td>Exp: 0.7</td>
<td>Exp: 0.1</td>
</tr>
<tr>
<td>(2) Largest 20 ones DIF: 0.01 PLA: 4.20 SPD: 0.89</td>
<td>(2) Largest 20 ones DIF: 0.003 PLA: 2.39 SPD: 0.81</td>
<td>(2) Largest 20 ones DIF: 0.001 PLA: 4.90 SPD: 0.81</td>
</tr>
<tr>
<td>Exp: 1.91</td>
<td>Exp: 0.7</td>
<td>Exp: 0.7</td>
</tr>
<tr>
<td>(1) Small cavities DIF: 0.54 PLA: 0.65 SPD: 1.06</td>
<td>(2) Largest 20 ones DIF: 0.01 PLA: 5.74 SPD: 0.80</td>
<td>(2) Largest 20 ones DIF: 0.001 PLA: 5.37 SPD: 0.78</td>
</tr>
<tr>
<td>Exp: 0.24</td>
<td>Exp: 0.1</td>
<td>Exp: 0.55</td>
</tr>
<tr>
<td>(2) Largest 20 ones DIF: 0.01 PLA: 4.40 SPD: 1.06</td>
<td>Exp: 2.65</td>
<td>Exp: 3.31</td>
</tr>
<tr>
<td>Exp: 0.75</td>
<td>Exp: 0.78</td>
<td>Exp: 0.55</td>
</tr>
<tr>
<td>(1) Small cavities DIF: 4.96 PLA: 0.65 SPD: 3.26</td>
<td>(1) Small cavities DIF: 5.09 PLA: 0.65 SPD: 2.50</td>
<td>(1) Small cavities DIF: 1.18 PLA: 0.60 SPD: 1.37</td>
</tr>
<tr>
<td>Exp: 1.39</td>
<td>Exp: 2.54</td>
<td>Exp: 0.31</td>
</tr>
<tr>
<td>(2) Largest 20 ones DIF: 0.09 PLA: 5.31 SPD: 3.26</td>
<td>(2) Largest 20 ones DIF: 0.006 PLA: 20.74 SPD: 4.96</td>
<td>(2) Largest 20 ones DIF: 0.004 PLA: 9.24 SPD: 4.60</td>
</tr>
<tr>
<td>Exp: 7.95</td>
<td>Exp: 25.00</td>
<td>Exp: 1.41</td>
</tr>
</tbody>
</table>

Values from the three theoretical models are referred as DIF, PLA, and SPD, and the experimental data are referred as Exp.
deviate more toward the PLA prediction. The relative contributions are shown in Table 5.

### 4.2.5. Condition: 400 °C and $6 \times 10^{-4}$ s$^{-1}$ (HT and LR)

Under this condition, the SP elongation is 600%, with an average $D$ of $\sim$14 and 18 μm at $\varepsilon \sim 0.7$ and 1.5, an average $m$ of $\sim$0.55, and a theoretically predicted $\eta$ of 1.64. The saturated cavity density is $\sim$1140 mm$^{-2}$ and $C_v$ is $\sim$0.75% and 4.5% at $\varepsilon \sim 0.7$ and 1.5.

Due to the prolonged exposure at the higher temperature of 400 °C, the grain size increases to over 10 μm. This would significantly affect the diffusion process during cavity growth. On the basis of $\rho$ and $\psi$ values, and the trends shown in Fig. 12(e) and Table 4, it is concluded that DIF and SPD would still control the growth for small cavities, but PLA takes over for the large cavities, especially for those at higher strain levels where experimental $C_v$ and $d\gamma/d\varepsilon$ (with $\eta$ of 1.72 and $d\gamma/d\varepsilon$ of 35.90 μm) are both close to the PLA predic-

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**Table 5**

Summary of the estimated relative contributions by the three cavity growth mechanisms for the small (< 2 μm) and the largest 20 cavities in specimens strained to 0.5 and 1.5 under various loading conditions

<table>
<thead>
<tr>
<th>Condition</th>
<th>$\dot{\varepsilon}$</th>
<th>Loading Conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$=6 \times 10^{-4}$ s$^{-1}$ (LR)</td>
<td>$\varepsilon \sim 0.7$</td>
</tr>
<tr>
<td></td>
<td>$=1 \times 10^{-4}$ s$^{-1}$ (HR)</td>
<td>$\varepsilon \sim 0.7$</td>
</tr>
<tr>
<td>200°C</td>
<td>(1) Small cavities</td>
<td>![Bar Chart]</td>
</tr>
<tr>
<td></td>
<td>(2) Largest 20 ones</td>
<td>![Bar Chart]</td>
</tr>
<tr>
<td>300°C</td>
<td>(1) Small cavities</td>
<td>![Bar Chart]</td>
</tr>
<tr>
<td></td>
<td>(2) Largest 20 ones</td>
<td>![Bar Chart]</td>
</tr>
<tr>
<td>400°C</td>
<td>(1) Small cavities</td>
<td>![Bar Chart]</td>
</tr>
<tr>
<td></td>
<td>(2) Largest 20 ones</td>
<td>![Bar Chart]</td>
</tr>
</tbody>
</table>

The vertical axis is referred to the contribution percentage.
tions (with $\eta$ of 1.64 and $dr/d\varepsilon$ of 20.74 $\mu$m). The contributions from the three mechanisms are also presented in Table 5. At an even higher strain near failure, $\varepsilon \sim 2.0$, coalescence is involved.

4.2.6. Condition: 400 °C and 1 $\times$ 10^{-2} s^{-1} (HT and HR)

Under this condition, the SP elongation decreases to 330%, with an average $D$ of ~5 and 7 $\mu$m at $\varepsilon \sim 0.7$ and 1.5, an average $m$ of ~0.40, and a theoretically predicted $\eta$ of 2.41. The saturated cavity density is ~1980 mm^{-2} and $C_v$ is ~1.4% and 2.1% at $\varepsilon \sim 0.7$ and 1.5.

The grain growth effect at HR was not as severe as the LR case at 400 °C. The $p$ and $\psi$ values, coupled with $C_v$ and $dr/d\varepsilon$ da data presented in Fig. 12(f) and Table 4, suggest that DIF and SPD together control the growth for small cavities, and PLA governs for the large cavities especially at later straining stage, as compared in Table 5. No apparent evidence for the coalescence effect for the HR specimens is observed.

4.3. Factors influencing cavitation behaviors

The major factors imposing apparent influence on the cavitation behavior of the extruded fine-grained AZ31 alloy during LTSP or HSRSP are the loading temperature, strain rate, strain level, and grain size. Due to the pronounced grain growth only at 400 °C and low strain rates, the analyses on the cavity growth mechanisms become complicated. A comparison cannot be made under the constant structure (grain size) condition.

If the grain size is fixed, the cavity number density would generally decrease with increasing the loading temperature; the cavity size and volume fraction would also increase. Also, under the constant grain size condition, with increasing strain rate (or increasing flow stress level), all of the cavity number density, cavity size and volume fraction would increase. With increasing strain level, the cavity number density increases initially and becomes saturated after $\varepsilon > 1.0$, the cavity size and volume fraction also increases. With increasing cavity size, the atom-diffusion influence decreases, the dislocation plasticity effect increases, and the cavity becomes elongated along the loading axis. The above trend might be reversed when the grain size is appreciably increased; the cavity growth behavior would be distinctly different in materials with a grain size of 2 and 20 $\mu$m. Fig. 13 shows the summarized cavitation map for 5 and 20 $\mu$m grains. With increasing grain size, the PLA zone shifts to a lower strain rate, and the DIF zone moves to a lower temperature.

4.4. Cavitation of AZ31 at 200% tensile elongation

The superplasticity capability has long been utilized in superplastic gas blowing forming at temperatures around 0.7–0.8 $T_m$ in aerospace or aircraft industry. Recent applications have extended to press forming [9], press forging, or hydroforming at much lower temperatures and higher strain rates. The deformation strain during these forming methods is usually less than 200% in terms of uniaxial tensile elongation, or equivalent to ~1.1 true strain. The cavity characteristics at this straining stage at a lower temperature around 0.5–0.7 $T_m$ will be of great concern. Table 6

Table 6
The cavity development in the extruded AZ31 plate deformed to $\varepsilon \sim 1.1$

<table>
<thead>
<tr>
<th>$T$ (°C)</th>
<th>$\dot{\varepsilon}$ (s^{-1})</th>
<th>Average cavity size for all cavities (μm)</th>
<th>Average cavity size for largest 20 cavities (μm)</th>
<th>Cavity density (mm^{-2})</th>
<th>Average growth rate $dr/d\varepsilon$ (μm)</th>
<th>Volume fraction, $C_v$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>$6 \times 10^{-4}$</td>
<td>1.7</td>
<td>9</td>
<td>$&gt;1000$</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td></td>
<td>$1 \times 10^{-2}$</td>
<td>1.4</td>
<td>5</td>
<td>$&gt;1000$</td>
<td>0.5</td>
<td>0.3</td>
</tr>
<tr>
<td>300</td>
<td>$6 \times 10^{-4}$</td>
<td>2.5</td>
<td>10</td>
<td>~1800</td>
<td>1.5</td>
<td>1.0</td>
</tr>
<tr>
<td></td>
<td>$1 \times 10^{-2}$</td>
<td>2.4</td>
<td>9</td>
<td>~2000</td>
<td>1.5</td>
<td>1.5</td>
</tr>
<tr>
<td>400</td>
<td>$6 \times 10^{-4}$</td>
<td>3.8</td>
<td>20</td>
<td>~1100</td>
<td>5.0</td>
<td>2.0</td>
</tr>
<tr>
<td></td>
<td>$1 \times 10^{-2}$</td>
<td>3.3</td>
<td>15</td>
<td>~1900</td>
<td>2.5</td>
<td>1.7</td>
</tr>
</tbody>
</table>

Fig. 13. Proposed map for the dominating cavity growth mechanisms in the current fine-grained AZ31 alloy. The solid lines are for a grain size of ~5 μm, and the lighter double lines are for a grain size of ~20 μm.
lists the cavity measurement data in this study for the extruded fine-grained AZ31 Mg alloy under the LTSP or HSRSP condition. The average size for all cavities is around 1.5–3.5 μm (with few large ones measuring 5–20 μm), the cavity number density around 1000–2000 mm⁻², the growth rate around 0.5–5.0 μm, and the volume fraction around 1–2%. The optimum superplasticity for this alloy has been shown to occur at \( \sim 300 ^\circ C \) and \( 1 \times 10^{-3} \text{ s}^{-1} \), and the resulting cavitation situation can be estimated from the data in Table 6. It is assured that the cavity behavior of the current AZ31 Mg alloy is superior to those observed in Al base alloys subjected to high or low temperature superplastic forming [24–29,38,39].

5. Conclusion

1. The as-extruded AZ31 plate possesses equiaxed grains measuring 2.9 μm in size, with a high fraction of HAB, favorable for GBS and resulting in satisfactory LTSP or HSRSP.
2. The cavity number density increased rapidly over \( \varepsilon = 0–0.5 \), and gradually reached the saturated level at \( \varepsilon \sim 0.5–1.0 \), suggesting that the cavity nucleation only occurred at the very early stage and cavity growth dominated most of the straining process.
3. For cavities smaller than 2 μm, they are basically nearly spherical in shape and randomly dispersed in the matrix, suggesting the involvement of diffusion or superplasticity diffusion controlled cavity growth mechanism. In contrast, the larger cavities tend to be elongated with the long axis aligned toward the loading axis, implying the plasticity controlled cavity growth mechanism. However, it is also recognized that coalescence may play an important role in the elongation of cavities.
4. For small cavities less than 2 μm, SPD with a minor contribution from DIF control the growth. For large cavities, PLA takes over for most cases, but SPD still controls in the case of lower strain rate and small grain size. The relative contributions from the three mechanisms and the cavity map for this alloy are established.
5. The major factors affecting the cavitation behavior are the loading temperature, strain rate, strain level, and grain size. The influencing trends are established.
6. The low degree of cavity development in AZ31 during LTSP or HSRSP to a strain of 1.1 (or 200% elongation) provides a positive factor for this alloy to be applied during superplastic gas forming, press forming, or hydroforming.
7. The cavitation in AZ31 appears to be much less severe than those observed in Al based alloys during HTSP or LTSP, due to the higher grain boundary diffusion rate, the well-defined grain structure with a high HAB fraction, the smooth operation of GBS, and the absence of second phases in this solution-hardening alloy.

Acknowledgements

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References