Microstructures and mechanical behaviors of Mg$_{58}$Cu$_{31}$Gd$_{11}$ and Mg$_{65}$Cu$_{25}$Gd$_{10}$ amorphous alloys synthesized by injection casting and melt spinning

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**Abstract**

Mg$_{58}$Cu$_{31}$Gd$_{11}$ and Mg$_{65}$Cu$_{25}$Gd$_{10}$ alloys were synthesized via two processing routes, injection casting and melt spinning. The diameter of the injection-cast bars was 4 mm in diameter. The XRD results obtained for the Mg$_{58}$Cu$_{31}$Gd$_{11}$ are nearly identical to those for the Mg$_{58}$Cu$_{31}$Gd$_{10}$, showing amorphous-like broad characteristic peaks. All the four characteristic temperatures, $T_g$, $T_x$, $T_m$ and $T_f$ of the Mg$_{58}$Cu$_{31}$Gd$_{10}$ are essentially lower than those of Mg$_{58}$Cu$_{31}$Gd$_{11}$, for both injection-cast rods and melt-spun ribbons. The glass forming abilities of the Mg$_{58}$Cu$_{31}$Gd$_{10}$ are similar to those of Mg$_{58}$Cu$_{31}$Gd$_{11}$, for both injection-cast rods and melt-spun ribbons, indicating by $T_g = 0.60$ and $\gamma = 0.42$. The average microhardness of the Mg$_{58}$Cu$_{31}$Gd$_{10}$ is 2.41 GPa and 2.27 GPa for injection-cast bars and melt-spun ribbons, respectively, which are significantly lower than 2.84 GPa and 2.49 GPa of the Mg$_{58}$Cu$_{31}$Gd$_{11}$. The nanohardness at the maximum load from the multiple loading is 3.5 GPa for Mg$_{58}$Cu$_{31}$Gd$_{10}$, which is lower than 3.9 GPa for Mg$_{58}$Cu$_{31}$Gd$_{11}$. The curves of load vs. the depth obtained from the nanoindentation tests all show stepwise behavior due to the pop-in events, and the step width increases as the indentation rate decreases. The modulus at the maximum load from the multiple loading obtained from the nanoindentation tests is 64.9 GPa for Mg$_{58}$Cu$_{31}$Gd$_{10}$, which is lower than 70.7 GPa for Mg$_{58}$Cu$_{31}$Gd$_{11}$. The fracture stress and strain of the Mg$_{58}$Cu$_{31}$Gd$_{10}$ BMG rod at room temperature are 490 MPa and 3%, respectively, smaller than those of the Mg$_{58}$Cu$_{31}$Gd$_{11}$ BMG rod, 548 MPa and 3.2%, respectively. The Mg$_{58}$Cu$_{31}$Gd$_{11}$ BMG rod is stronger at room temperature, and also shows higher yield stress and less deformable at elevated temperature, than the Mg$_{58}$Cu$_{31}$Gd$_{10}$ BMG rod.

1. Introduction

A minute amorphous Au–Si alloy was first synthesized through extremely rapid solidification technique (over 10$^6$ K/s) in 1960 [1]. Over the years, bulk amorphous alloys (BMGs) with larger dimensions have been intensively studied and become promising candidates for industrial applications. The advances of BMG have been mainly on the path of finding new glass-forming compositions with a critical cooling rate less than 100 K/s. Among these BMGs, Mg-based BMGs possess maximum specific strength and are suitable for fabricating light component parts [2–6].

The most intensively studied Mg BMGs are Mg–TM–RE (TM: e.g. Ni, Cu, Zr; RE: e.g. Gd), which have large undercooled liquid region and high glass forming ability (GFA), since they obey the three principles of forming bulk metallic glasses [7], which are (1) a alloy system comprising at least 3 elements; (2) the difference between the sizes of the major elements are larger than 12% and (3) the heat of mixing is a negative value. Mg$_{65}$Cu$_{25}$Y$_{10}$ BMG was developed by Inoue group with 4 mm in diameter via copper mold casting [2]. Recently 2 Mg systems, Mg$_{65}$Cu$_{25}$Gd$_{10}$ and Mg$_{58}$Cu$_{31}$Gd$_{11}$ were found to have better glass forming ability than Mg$_{65}$Cu$_{25}$Y$_{10}$. However, there is no published research work done on the microstructural properties at elevated temperatures for these 2 materials. In this work, the mechanical properties of these 2 systems in the forms of injection-cast BMG bars and melt-spin ribbons at room and elevated temperatures were studied and compared.

2. Experimental methods

Raw materials used in this study were all 99.9% in purity. Cu–Gd ingots were prepared by arc melting under Ti-getting argon atmosphere. The ingots and Mg chips were remelted by induction melting four times to attain the homogeneity under argon atmosphere to make Mg$_{65}$Cu$_{25}$Gd$_{10}$ and Mg$_{58}$Cu$_{31}$Gd$_{11}$ ingots. The Mg–Cu–Gd
ingots were subsequently crushed into small pieces, which were then put into a quartz tube under argon atmosphere for melting and injection-casting into rods with 4 mm in diameter. Ribbons with the same chemical compositions were also prepared by a melt-spinning equipment with a Cu wheel running at a surface speed of 20 m/s. X-ray (Cu Kα) diffraction patterns were obtained from angles of 20–70° at a scanning speed of 4°/min. The thermal analysis experiments were performed on a differential scanning calorimetry (DSC) at a heating rate of 0.67 K/s. The micro-Vickers hardness tests were conducted at a load of 300 g and a dwell time of 10 s. The nanoindentation tests were performed under two conditions at room temperature. One is single loading condition, in which the indentor was pressed down under a constant strain rate and stopped at the depth of 1 μm. Two strain rates, 0.05 s⁻¹ and 0.001 s⁻¹, were used. The other one is multiple loading condition, in which indentor was pressed down continuously with various load rates: 0.2 mN/s → 0.4 mN/s → 0.8 mN/s → 1.67 mN/s → 3.33 mN/s, until the load reached 50 mN. For each load, the load was allowed to first increase for 15 s, held at a constant for 30 s, and then relieved for 15 s. The compressive tests were done using specimens of 4 mm in diameter and 8 mm in height with a Shimadzu Universal Testing Machine. Room temperature tests were done at a strain rate of 5 × 10⁻⁴ s⁻¹, and high temperature tests were done at three strain rates, 5 × 10⁻² s⁻¹, 5 × 10⁻¹ s⁻¹ and 5 × 10⁻⁰ s⁻¹, at temperatures of (T_g + T_x)/2.

3. Results and discussion

The X-ray diffraction patterns of injection-cast rod and melt-spun ribbon of Mg₆₅Cu₂₅Gd₁₀ and Mg₅₈Cu₃₁Gd₁₁ are shown in Fig. 1a and b, respectively. The amorphous-like broad characteristic peaks are shown at angles of 20–45° for all injection-cast and melt-spun Mg₆₅Cu₂₅Gd₁₀. The XRD results obtained for the Mg₅₈Cu₃₁Gd₁₁ are nearly identical to those for the Mg₆₅Cu₂₅Gd₁₀. Fig. 2 presents the DSC traces for the injection-cast bar and melt-spun ribbon at heating rate of 0.67 K/s, in which glass transition temperature (T_g) and onset crystallization temperature (T_x) are also marked, along with melting temperature (T_m) and liquidus temperature (T_l). Fig. 2 summarizes the results of DSC, which also lists ΔT_x = T_x – T_g, and three GFA factors, T_rg = (T_g/T_l) [8], γ_x = (T_x/T_g + T_l) [9] and γ_m = (2T_x − T_g)/T_l [10]. It can be seen that all the four characteristic temperatures of the injection-cast rods are higher than those of the melt-spun ribbons for both Mg₆₅Cu₂₅Gd₁₀
Table 1
Thermal properties and micro-Vickers hardness of Mg$_{65}$Cu$_{25}$Gd$_{10}$ and Mg$_{58}$Cu$_{31}$Gd$_{11}$.

<table>
<thead>
<tr>
<th>Temp. (K)</th>
<th>$T_g$</th>
<th>$T_x$</th>
<th>$T_m$</th>
<th>$T_l$</th>
<th>$\Delta T_x$</th>
<th>$\gamma$</th>
<th>$\gamma_m$</th>
<th>VHN (kgf/mm$^2$)</th>
<th>Micro-Vickers (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mg$<em>{65}$Cu$</em>{25}$Gd$_{10}$</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\Phi = 4$ mm</td>
<td>434</td>
<td>497</td>
<td>716</td>
<td>727</td>
<td>63</td>
<td>0.59</td>
<td>0.42</td>
<td>0.770</td>
<td>241</td>
</tr>
<tr>
<td>Ribbon</td>
<td>423</td>
<td>470</td>
<td>700</td>
<td>704</td>
<td>47</td>
<td>0.60</td>
<td>0.41</td>
<td>0.734</td>
<td>227</td>
</tr>
<tr>
<td>Mg$<em>{58}$Cu$</em>{31}$Gd$_{11}$</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\Phi = 4$ mm</td>
<td>440</td>
<td>495</td>
<td>721</td>
<td>729</td>
<td>55</td>
<td>0.60</td>
<td>0.42</td>
<td>0.754</td>
<td>284</td>
</tr>
<tr>
<td>Ribbon</td>
<td>438</td>
<td>495</td>
<td>712</td>
<td>728</td>
<td>57</td>
<td>0.60</td>
<td>0.42</td>
<td>0.758</td>
<td>249</td>
</tr>
</tbody>
</table>

and Mg$_{58}$Cu$_{31}$Gd$_{11}$. All the four characteristic temperatures of the Mg$_{65}$Cu$_{25}$Gd$_{10}$ are essentially lower than those of Mg$_{58}$Cu$_{31}$Gd$_{11}$, for both injection-cast rods and melt-spun ribbons. The glass forming abilities shown by $T_g$ and $\gamma$ of the injection-cast rods are similar to those of the melt-spun ribbons for both Mg$_{65}$Cu$_{25}$Gd$_{10}$ and Mg$_{58}$Cu$_{31}$Gd$_{11}$. The $T_g$ and $\gamma$ of the Mg$_{65}$Cu$_{25}$Gd$_{10}$ are also similar to those of Mg$_{58}$Cu$_{31}$Gd$_{11}$, for both injection-cast rods and melt-spun ribbons. However, there is significant difference in the $\gamma_m$ factor between the bar and the ribbon for the Mg$_{65}$Cu$_{25}$Gd$_{10}$, but no difference for the Mg$_{58}$Cu$_{31}$Gd$_{11}$.

The microhardnesses of injection-cast rods and melt-spun ribbons of Mg$_{65}$Cu$_{25}$Gd$_{10}$ and Mg$_{58}$Cu$_{31}$Gd$_{11}$ are also shown in Table 1. The average hardness of the injection-cast bars is larger than those of the melt-spun ribbon, indicating a stronger material for the latter. The microhardnesses of injection-cast rods and melt-spun ribbons of Mg$_{65}$Cu$_{25}$Gd$_{10}$ and Mg$_{58}$Cu$_{31}$Gd$_{11}$ are also shown in Table 1.

Fig. 3 shows the curves of load vs. the depth obtained from the nanoindentation tests at a strain rate of 0.05 s$^{-1}$ for Mg$_{65}$Cu$_{25}$Gd$_{10}$ and Mg$_{58}$Cu$_{31}$Gd$_{11}$ BMG rods. All the curves show stepwise behavior. These serrations were caused by the pop-in events, in which the load nearly did not increase as the depth of the indentation increased, and became more significant as the load increased. This phenomenon is associated with the nucleation and propagation of the shear bands [11–13]. When shear bands form, they propagate and stop by the obstacles, where new shear bands nucleate and start to form, and subsequently propagate. The widths of the steps represent the propagating distance of the shear bands before they stop at the obstacles. According to Nieh et al., the step width increases as the indentation rate decreases [14]. This behavior was also found in this study for both materials. In particular, for the Mg$_{65}$Cu$_{25}$Gd$_{10}$, at the strain rate of 0.05 s$^{-1}$, the step width between the depths of 580–610 nm is about 14 nm, while at the lower strain rate of 0.001 s$^{-1}$, the step width between the depths of 550–675 nm increases significantly to about 100 nm. This also happened for the Mg$_{58}$Cu$_{31}$Gd$_{11}$, at the strain rate of 0.05 s$^{-1}$, the step width between the depths of 575–590 nm is about 10 nm, while at the lower strain rate of 0.001 s$^{-1}$, the step width between the depths of 550–610 nm also increases significantly to about 100 nm.

Table 2 lists the modulus and hardness of the Mg$_{65}$Cu$_{25}$Gd$_{10}$ and Mg$_{58}$Cu$_{31}$Gd$_{11}$ calculated from the nanoindentation data for both multiple and single loadings, using Oliver and Pharr’s method [15]. The modulus at the maximum load from the multiple loading and the average modulus from the single loading are lower for Mg$_{65}$Cu$_{25}$Gd$_{10}$, and the average modulus from the single loading are lower for Mg$_{58}$Cu$_{31}$Gd$_{11}$, respectively. The average modulus from the single loading are lower for the strain rate of 0.001 s$^{-1}$ than those for the higher strain rate of 0.05 s$^{-1}$. The hardness at the maximum load from the multiple loading and the average hardness from the single loading are lower for Mg$_{65}$Cu$_{25}$Gd$_{10}$, and the average hardness from the single loading are lower for Mg$_{58}$Cu$_{31}$Gd$_{11}$, respectively. The average hardness from the single loading are lower for the strain rate of 0.001 s$^{-1}$ than those for the higher strain rate of 0.05 s$^{-1}$, respectively. These results obtained from the nanoindentation testing are consistent with those from the microhardness testing.

Table 3 shows that the true compressive fracture stress and strain of the Mg$_{65}$Cu$_{25}$Gd$_{10}$ BMG rod tested at a strain rate of $5 \times 10^{-4}$ s$^{-1}$ at room temperature are 490 MPa and 3%, both of which are smaller than those of the Mg$_{58}$Cu$_{31}$Gd$_{11}$ BMG.
rod, which are 548 MPa and 3.2%, respectively. This indicates that the Mg₅₈Cu₃₁Gd₁₁ BMG rod is stronger and a bit more ductile than the Mg₆₅Cu₂₅Gd₁₀ BMG rod at room temperature.

The Mg₆₅Cu₂₅Gd₁₀ BMG rods tested at lower strain rates of 5 × 10⁻⁴ s⁻¹ and 5 × 10⁻³ s⁻¹ behaved superplastically at the supercooling temperature of (Tₚ + Tₐ)/2 = 193 °C, and were compressed into flakes. The true strain as large as about 160% without fracturing was achieved. However, at the highest strain rate of 5 × 10⁻² s⁻¹, the specimen fractured at a strain about 40%, and the stress started to inject downward. Table 3 lists the yield stresses of the Mg₆₅Cu₂₅Gd₁₀ BMG rods for various strain rates. The yield stress increases as the strain rate increases, and the yield stresses are 17 MPa, 49 MPa and 106 MPa for strain rates of 5 × 10⁻⁴ s⁻¹, 5 × 10⁻³ s⁻¹ and 5 × 10⁻² s⁻¹, respectively.

The Mg₅₈Cu₃₁Gd₁₁ BMG rods tested at the lowest strain rate of 5 × 10⁻⁴ s⁻¹ behaved superplastically at the supercooling temperature of (Tₚ + Tₐ)/2 = 195 °C, and were compressed into flakes. The true strain as large as about 160% without fracturing was also achieved. However, at the higher strain rate of 5 × 10⁻³ s⁻¹, the specimen fractured prematurely, and the stress started to injection downward after the yielding. The yield stress increases as the strain rate increases, and the yield stresses are 60 MPa and 89 MPa for strain rates of 5 × 10⁻⁴ s⁻¹ and 5 × 10⁻³ s⁻¹, respectively, as shown in Table 3. At the highest strain rates of 5 × 10⁻² s⁻¹, the rod failed right at the initial yielding, which rendered the specimen nearly undeformed at all. The brittle behavior at higher strain rate is due to simultaneous nucleation and propagation of multiple shear bands at such a high strain rate, which restricts the propagation of the shear bands [13].

It is shown that the yield stresses of the Mg₅₈Cu₃₁Gd₁₁ at elevated temperature are higher than those of the Mg₆₅Cu₂₅Gd₁₀, indicating that the Mg₅₈Cu₃₁Gd₁₁ BMG rod is stronger at room temperature, but also shows higher yield stress and less deformable at elevated temperature, than the Mg₆₅Cu₂₅Gd₁₀ BMG rod.

**Table 2**

Nanohardness and modulus of Mg₆₅Cu₂₅Gd₁₀ and Mg₅₈Cu₃₁Gd₁₁ from nanoindentation.

<table>
<thead>
<tr>
<th>Material</th>
<th>E at max load (GPa)</th>
<th>H at max load (GPa)</th>
<th>E average over defined range (GPa)</th>
<th>H average over defined range (GPa)</th>
<th>E from unload (GPa)</th>
<th>H from unload (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mg₆₅Cu₂₅Gd₁₀</td>
<td>64.9</td>
<td>3.5</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td>Multiple indentation mode</td>
<td>ε = 0.05 s⁻¹</td>
<td>N/A</td>
<td>65.6</td>
<td>3.7</td>
<td>64.3</td>
<td>3.5</td>
</tr>
<tr>
<td></td>
<td>ε = 0.001 s⁻¹</td>
<td>N/A</td>
<td>58.1</td>
<td>2.6</td>
<td>64.1</td>
<td>2.7</td>
</tr>
<tr>
<td>Mg₅₈Cu₃₁Gd₁₁</td>
<td>70.7</td>
<td>3.9</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td>Multiple indentation mode</td>
<td>ε = 0.05 s⁻¹</td>
<td>N/A</td>
<td>71.5</td>
<td>4.3</td>
<td>69.4</td>
<td>3.7</td>
</tr>
<tr>
<td></td>
<td>ε = 0.001 s⁻¹</td>
<td>N/A</td>
<td>58.6</td>
<td>2.7</td>
<td>65.8</td>
<td>2.8</td>
</tr>
</tbody>
</table>

E: modulus and H: hardness.

**Table 3**

Results from compression tests at RT, 193 °C and 195 °C.

<table>
<thead>
<tr>
<th>Temp. (°C)</th>
<th>Strain rate (s⁻¹)</th>
<th>True yield stress (MPa)</th>
<th>True fracture strain</th>
<th>True fracture stress (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mg₆₅Cu₂₅Gd₁₀</td>
<td>5 × 10⁻⁴</td>
<td>17</td>
<td>60</td>
<td>490</td>
</tr>
<tr>
<td>RT</td>
<td>193</td>
<td>49</td>
<td>89</td>
<td>548</td>
</tr>
<tr>
<td>5 × 10⁻³</td>
<td>106</td>
<td>89</td>
<td>57</td>
<td>490</td>
</tr>
<tr>
<td>5 × 10⁻²</td>
<td>106</td>
<td>89</td>
<td>57</td>
<td>490</td>
</tr>
<tr>
<td>Mg₅₈Cu₃₁Gd₁₁</td>
<td>5 × 10⁻⁴</td>
<td>0.030</td>
<td>4.3</td>
<td>69.4</td>
</tr>
<tr>
<td>RT</td>
<td>193</td>
<td>0.032</td>
<td>4.3</td>
<td>69.4</td>
</tr>
<tr>
<td>5 × 10⁻³</td>
<td>89</td>
<td>4.3</td>
<td>69.4</td>
<td>548</td>
</tr>
<tr>
<td>5 × 10⁻²</td>
<td>57</td>
<td>4.3</td>
<td>69.4</td>
<td>548</td>
</tr>
</tbody>
</table>

4. Conclusions

The XRD results obtained for the Mg₅₈Cu₃₁Gd₁₁ are nearly identical to those for the Mg₆₅Cu₂₅Gd₁₀, showing amorphous-like broad characteristic peaks.

All the four characteristic temperatures, Tₚ, Tₚ, Tₘ and Tᵣ, of the Mg₆₅Cu₂₅Gd₁₀ are essentially lower than those of Mg₅₈Cu₃₁Gd₁₁, for both injection-cast rods and melt-spun ribbons.

The glass forming abilities of the Mg₆₅Cu₂₅Gd₁₀ are similar to those of Mg₅₈Cu₃₁Gd₁₁, for both injection-cast rods and melt-spun ribbons, indicated by Tₚ = 0.60 and γ = 0.42.

The average microhardness of the Mg₆₅Cu₂₅Gd₁₀ is 2.41 GPa and 2.27 GPa for injection-cast bars and melt-spin ribbons, respectively, which are significantly lower than 2.84 GPa and 2.49 GPa of the Mg₅₈Cu₃₁Gd₁₁, indicating a stronger material for the latter.

The curves of load vs. the depth obtained from the nanoindentation tests all show stepwise behavior due to the pop-in events, and the step width increases as the indentation rate decreases.

The modulus at the maximum load from the multiple loading obtained from the nanoindentation tests is 64.9 GPa for Mg₆₅Cu₂₅Gd₁₀, which is lower than 70.7 GPa for Mg₅₈Cu₃₁Gd₁₁. The average modulus from the single loading obtained from the nanoindentation tests are lower for the strain rate of 0.001 s⁻¹ that those for the higher strain rate of 0.05 s⁻¹.

The nanohardness at the maximum load from the multiple loading is 3.5 GPa for Mg₆₅Cu₂₅Gd₁₀, which is lower than 3.9 GPa for Mg₅₈Cu₃₁Gd₁₁. The average nanohardness from the single loading are lower for the strain rate of 0.001 s⁻¹ that those for the higher strain rate of 0.05 s⁻¹.

The fracture stress and strain of the Mg₆₅Cu₂₅Gd₁₀ BMG rod at room temperature are 490 MPa and 3%, respectively, smaller than those of the Mg₅₈Cu₃₁Gd₁₁ BMG rod, 548 MPa and 3.2%, respect-
tively, indicating that the Mg$_{58}$Cu$_{31}$Gd$_{11}$ BMG rod is stronger and a bit more ductile than Mg$_{65}$Cu$_{25}$Gd$_{10}$ BMG rod at room temperature.

The Mg$_{65}$Cu$_{25}$Gd$_{10}$ BMG rods tested at lower strain rates of $5 \times 10^{-4}$ s$^{-1}$ and $5 \times 10^{-3}$ s$^{-1}$ behaved superplastically at 193°C, and the true strain as large as about 160% without fracturing was achieved. However, at the highest strain rate of $5 \times 10^{-2}$ s$^{-1}$, the specimen fractured at a strain about 40%. The yield stress increases as the strain rate increases, which are 17 MPa, 49 MPa and 106 MPa for strain rates of $5 \times 10^{-4}$ s$^{-1}$, $5 \times 10^{-3}$ s$^{-1}$ and $5 \times 10^{-2}$ s$^{-1}$, respectively.

The Mg$_{58}$Cu$_{31}$Gd$_{11}$ BMG rods tested at the lowest strain rate of $5 \times 10^{-4}$ s$^{-1}$ behaved superplastically at 195°C, and the true strain as large as about 160% without fracturing was also achieved. However, at the higher strain rate, the specimen fractured prematurely, and the stress started to injection downwards after yielding.

The Mg$_{58}$Cu$_{31}$Gd$_{11}$ BMG rod is stronger at room temperature, and also shows higher yield stress and less deformable at elevated temperature, than the Mg$_{65}$Cu$_{25}$Gd$_{10}$ BMG rod.

**Acknowledgement**

This research is funded by National Science Council, Taiwan, ROC (NSC95-2221-E-006-061-MY3).

**References**