Formation and Properties of New La-Based Bulk Glassy Alloys with Diameters up to Centimeter Order

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New La-based bulk glassy alloys with superior glass-forming ability (GFA) were synthesized in La-Al-Ag-(Cu,Co,Ni) system. The addition of 5 at%Ag to La-Al-Cu ternary base system greatly increases the stability of supercooled liquid as well as the GFA. The largest supercooled liquid region and largest diameter in La-Al-Cu-Ag quaternary system were of 83 K and 6.0 mm, respectively, for $\text{La}_{62.5}\text{Cu}_{20}\text{Al}_{12.5}\text{Ag}_5$ alloy. The addition of Ni or Co to La-Al-Cu-Ag alloy is effective for the further increase in GFA. The maximum sample diameter of over 12 mm was fabricated for $\text{La}_{62.5}\text{Al}_{12.5}\text{Cu}_{10}\text{Ni}_5\text{Co}_5\text{Ag}_5$ alloy. The GFA has better correlation with reduced glass transition temperature. The new La-based bulk glassy alloys exhibit high fracture strength of 640–858 MPa with distinct plastic strains of 0.2–0.4%. [doi:10.2320/matertrans.48.68]

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1. Introduction

Since the syntheses of bulk glassy alloys in multi-component Ln^{-1} (Ln: lanthanide metal) and Mg^{-2} based alloys by the copper mold casting method in 1989, much effort has been devoted to develop a new bulk glassy alloy exhibiting high glass-forming ability (GFA) and useful engineering properties. As a result, bulk glassy alloy systems have been widely extended to $\text{Zr}^{-3,4}$, Fe^{-5} , Pd^{-6} , Ti^{-7} , Co^{-8} , Ni^{-9-11} , Cu^{-12-14} , Pt^{-15} and Au^{-16} based alloys. These bulk glassy alloys exhibit unique properties such as high strength, large elastic strain limit, high hardness, good soft magnetic properties, and excellent corrosion resistance. The compositions of the bulk glassy alloys with high GFA are typically close to a deep eutectic composition.¹⁷⁾ Consequently, their melting temperatures are much lower than those estimated from interpolation of the alloy constituents' melting temperatures. The resulting low liquidus temperature (T_l) is an attractive parameter for finding a new bulk glassy alloys, because the reduced glass transition temperature (T_g/T_l , T_g : glass transition temperature) is an important factor parameter for GFA.¹⁸⁾ The stabilization of bulk glassy alloys against crystallization also results in a large supercooled liquid region, ΔT_x , ($\Delta T_x = T_x - T_g$, T_x : crystallization temperature), in which the glassy phase first relaxes into a highly viscous liquid before eventually crystallizing. In this temperature region, BMG's are amenable to superplastic processing using netshape processing methods similar to those employed for thermoplastics.¹⁹⁾ In addition, the ΔT_x is also an important parameter that interprets GFA of bulk glassy alloys.²⁰⁾

Recently, the Ln-based bulk glassy alloys have gained considerable interest again because the glassy alloys have lower T_g ,²¹⁾ large ΔT_x ,²¹⁾ high GFA,²²⁻²⁴⁾ low melting temperature,²⁵⁾ good mechanical properties,^{24,26)} and hard magnetic properties.²²⁾ It is important for scientific and engineering aspects to develop a new Ln-based glassy alloy with higher GFA and larger ΔT_x . Very recently, Ln-based glassy alloy rods with diameters of over one centimeter were

fabricated. However, the new bulk glassy alloys exhibited narrow ΔT_x which were below 45 K.^{23,24)}

In this paper, we report on the formation, stabilization of supercooled liquid and mechanical properties of a new series of Ln-based glassy alloys in the La-Al-Cu-Ag and La-Al-Cu-Ag-(Co,Ni) systems, which show the critical diameters range from 6 to 12 mm, low T_g of 398–425 K, large ΔT_x of 65–96 K and high fracture strength of 640–858 MPa with plastic strain of ~0.4%.

2. Experimental Procedure

The alloy ingots were prepared by arc melting the mixtures of pure La, Cu, Al, Ag, Co, and Ni metals in an argon atmosphere. The purity of metals was over 99.5 mass%. Alloy ingots were re-melted five times to ensure chemical homogeneity. The mass losses were measured for each ingot after melting and were less than 0.1 mass%. The glassy alloy was produced by copper mold casting for bulk cylindrical rods with diameters of 2–15 mm and by melt spinning for ribbons with a cross section of $0.02 \times 1.2 \text{ mm}^2$. The glassy phase was identified by X-ray diffractometer using $\text{Cu-K}\alpha$ radiation and thermal stability was examined by differential scanning calorimetry (DSC) at a heating rate of 0.67 K/s. The melting and liquidus temperatures were measured with a differential thermal analyzer (DTA) at a heating rate of 0.17 K/s. Mechanical properties were measured with an Instron testing machine. The gauge dimension of specimens was 2 mm in diameter and 4 mm in height for compressive test and the strain rate was $5 \times 10^{-4} \text{ s}^{-1}$. The fracture surface was examined by scanning electron microscopy (SEM).

3. Results and Discussion

3.1 Ternary La-based glassy alloys

Table 1 lists some examples of the newly discovered La-based bulk glassy alloys in an order that reflects the sequential optimization of successive alloy additions which resulted in the improvement of the critical sample diameters.

It has been reported that ternary $\text{La}_{55}\text{Al}_{25}\text{Cu}_{20}$ glassy alloys exhibit a large supercooled liquid region (ΔT_x) of

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Table 1 Summary of the properties and the critical sample diameters of the new La-based bulk glassy alloys developed in this work.

Alloy composition (at%)	T_g (K)	T_x (K)	T_l (K)	ΔT_x (K)	T_g/T_l	γ	d_c (mm)
La ₅₅ Al ₂₅ Ag ₂₀	498	527	1059	29	0.47	0.339	2<
La ₅₅ Ag ₂₅ Cu ₂₀	384	412	752	27	0.511	0.362	2<
La ₅₅ Al ₂₅ Cu ₂₀	443	498	889	56	0.498	0.374	2.0
La ₆₀ Al ₂₀ Cu ₂₀	415	486	811	71	0.512	0.396	3
La _{62.5} Al _{17.5} Cu ₂₀	401	471	772	70	0.519	0.402	3
La ₅₅ Al ₂₀ Cu ₂₀ Ag ₅	429	503	823	74	0.521	0.399	4
La ₅₅ Al _{17.5} Cu ₂₀ Ag _{7.5}	425	498	852	73	0.499	0.390	3
La ₅₅ Al ₁₅ Cu ₂₀ Ag ₁₀	416	483	893	67	0.467	0.361	2
La ₆₀ Al ₁₅ Cu ₂₀ Ag ₅	410	481	759	80	0.530	0.412	5
La _{62.5} Al _{12.5} Cu ₂₀ Ag ₅	389	472	721	83	0.539	0.417	6
La _{62.5} Al _{12.5} Cu ₁₅ Ni ₅ Ag ₅	391	487	717	96	0.545	0.440	8
La _{62.5} Al _{12.5} Cu ₁₅ Co ₅ Ag ₅	397	474	700	77	0.567	0.432	9
La _{62.5} Al _{12.5} Cu _{12.5} Ni _{2.5} Co ₅ Ag ₅	402	477	699	75	0.575	0.433	10
La _{62.5} Al _{12.5} Cu ₁₀ Ni ₅ Co ₅ Ag ₅	404	469	691	65	0.585	0.428	12

Table 2 The mechanical properties of the new La-based bulk glassy alloys.

Alloy composition (at%)	$\sigma_{c,f}$ (MPa)	E (GPa)	$\varepsilon_{c,e}$ (%)	$\varepsilon_{c,p}$ (%)	H_v
La ₅₅ Al ₂₅ Cu ₂₀	858	36	1.8	0.2	208
La ₅₅ Al ₂₀ Cu ₂₀ Ag ₅	758	42	1.8	0.2	226
La _{62.5} Al _{12.5} Cu ₂₀ Ag ₅	640	36	1.9	0.3	201
La _{62.5} Al _{12.5} Cu ₁₅ Ni ₅ Ag ₅	665	38	1.7	0.3	198
La _{62.5} Al _{12.5} Cu ₁₅ Co ₅ Ag ₅	670	38	1.7	0.4	204
La _{62.5} Al _{12.5} Cu ₁₀ Ni ₅ Co ₅ Ag ₅	678	39	1.7	0.2	201

over 50 K, leading to the formation of glassy rod specimens.²⁶⁾ We discovered new La-Cu-Ag and La-Al-Ag ternary glassy alloys, which also show a distinct glass transition, followed by a supercooled liquid region and then exothermic reactions due to crystallization. Figure 1 show DSC curves of the melt-spun La₅₅Ag₂₅Cu₂₀, La₅₅Al₂₅Ag₂₀ and La_{80-x}Al_xCu₂₀ ($x = 17.5, 20$ and 25) glassy alloys. The La-Cu-Al glassy alloys exhibit large ΔT_x of over 55 K, and the largest ΔT_x was 71 K for $x = 20$ alloy. The La-Cu-Ag and La-Al-Ag alloys show small ΔT_x , *e.g.*, 29 K for La₅₅Al₂₅Ag₂₀ and 27 K for La₅₅Ag₂₅Cu₂₀ alloy. We examined the GFA of the ternary alloys by copper mold casting. Although the critical rod diameters for formation of the glassy phase are below 3.0 mm for the ternary alloys as summarized in Table 1, higher GFA and larger ΔT_x are expected to be obtained for La-Al-Cu-Ag quaternary alloys.^{17,19)}

3.2 Quaternary La-Al-Cu-Ag glassy alloys

We first examined the effect of Ag addition on the thermal stability of supercooled liquid and GFA of the ternary La-Cu-Al alloys. Figures 2 and 3 show DSC and DTA curves of the quaternary La-Cu-Al-Ag glassy alloys. From these DSC scans and Table 1, it is seen that all the ΔT_x remarkably increase by the addition of a small amount of Ag. With addition of Ag to La₅₅Al₂₅Cu₂₀, the ΔT_x increases from 56 K at 0 at%Ag to 74 K at 5 at%Ag, and then decreases with a further increase in Ag content. By the 5 at% Ag addition to La₆₀Al₂₀Cu₂₀ and La_{62.5}Al_{17.5}Cu₂₀ alloys, the ΔT_x also increases from 71 to 80 K and 70 to 83 K, respectively. It is

Fig. 1 DSC curves of melt-spun La-Al-Cu, La-Al-Ag and La-Ag-Cu glassy alloys.

therefore concluded that the addition of a small amount Ag to La-Cu-Al ternary glassy alloys is very effective for an increase in ΔT_x , *i.e.*, stabilization of supercooled liquid against crystallization.

From Fig. 3 and Table 1, it is seen that the T_l values of the ternary La-Cu-Al alloys significantly decrease by the addition of a small amount of Ag. By 5% Ag addition to La₅₅Al₂₅Cu₂₀, La₆₀Al₁₅Cu₂₀ and La_{62.5}Al_{12.5}Cu₂₀ alloys, the T_l decreases from 889 to 832 K, 811 to 759 K and 772 to 721 K, respectively, resulting in an increase of reduced glass transition temperature (T_g/T_l) from 0.497 to 0.521, 0.512 to 0.530 and 0.519 to 0.539, respectively. In addition, the La_{62.5}Al_{12.5}Cu₂₀Ag₅ alloy shows an even lower T_l of 721 K and only one distinct endothermic event, indicating that this alloy is rather close to the quaternary eutectic point among the present alloys.

Fig. 2 DSC curves of melt-spun La-Al-Cu-Ag glassy alloys.

Fig. 3 DTA curves of the La-Al-Cu-Ag alloys.

Figure 4 shows X-ray diffraction patterns of the as-cast La-Cu-Al-Ag alloy rods with diameters ranging from 3.0 to 7.0 mm. Without Ag, the La-Cu-Al ternary alloys are distinctly crystallized for the rods of 3.0 and 4.0 mm in diameter and their critical diameters are below 3 mm as shown in Table 1. With 5 at% of Ag addition, the glassy alloy rods with larger diameters have been successfully prepared, and the best GFA is achieved for $\text{La}_{62.5}\text{Al}_{12.5}\text{Cu}_{20}\text{Ag}_5$ which has a critical casting diameters above 6 mm. This indicates that the appropriate amount of Ag addition prevents the

Fig. 4 X-ray diffraction patterns of cast La-Al-Cu and La-Al-Cu-Ag alloy rods with diameters of 3–7 mm.

formation of intermetallic compounds, yielding fully glassy samples. It has been noticed that the highest GFA of the present quaternary alloy series, especially the $\text{La}_{62.5}\text{Al}_{12.5}\text{Cu}_{20}\text{Ag}_5$ alloy, comes in most part from the alloying effect of Ag, which lowers the liquidus temperature of the matrix alloy and brings the composition to a deeper eutectic valley (Fig. 3). From a kinetic aspect, there are multiple ordered phases competing with each other for eutectic alloys in multicomponent systems, and the crystallization of the liquid requires simultaneous rearrangements of different species of atoms, which significantly suppresses the kinetics of the crystallization process and thus promotes glass formation.²⁷⁾ The empirical component rules drawn from experimental results²⁰⁾ have demonstrated that certain atomic-size mismatch and efficient atomic packing enhance GFA of multicomponent systems. In the La-Cu-Al-Ag system, the atomic radii of the component elements are La, 1.88 Å; Ag, 1.45 Å; Al, 1.43 Å and Cu, 1.27 Å, respectively,²⁸⁾ and hence the atomic size ratios are 1.48 for La/Cu, 1.30 for La/Ag, and 1.32 for Ag/Cu. Therefore, the present combination of atomic sizes can produce an efficiently packed local structure, which is often associated with low internal energy and high viscosity of liquid. In addition, in the present system, this criterion is also satisfied because the heats of mixing for La-Al, La-Cu, and La-Ag pairs are -38 , -22 , and -21 kJ = mol, respectively.²⁹⁾ These large negative values of the heat of mixing enhance the interactions among the components and promote chemical short range ordering in liquids, which can improve the local packing efficiency and restrain long range diffusion of atoms.

3.3 Multi-component La-Al-(Cu,Co,Ni)-Ag glassy alloys

A small amount of Co or Ni addition was found to further

Fig. 5 Shape and outer surface appearance of cast La-Al-(Cu,Co,Ni)-Ag alloy rods with diameters of 8–12 mm. (a) $\text{La}_{62.5}\text{Al}_{12.5}\text{Cu}_{15}\text{Ni}_5\text{Ag}_5$, (b) $\text{La}_{62.5}\text{Al}_{12.5}\text{Cu}_{12.5}\text{Ni}_{2.5}\text{Co}_5\text{Ag}_5$, (c) $\text{La}_{62.5}\text{Al}_{12.5}\text{Cu}_{10}\text{Ni}_5\text{Co}_5\text{Ag}_5$.

Fig. 6 X-ray diffraction patterns of cast La-Al-(Cu,Co,Ni)-Ag alloy rods with diameter of 8–12 mm.

improve the GFA. Figures 5 and 6 show the outer shape and surface appearance and X-ray diffraction patterns of the as-cast La-Al-Ag-(Cu, Co, Ni) alloy rods with diameters ranging from 8 to 12 mm. The surfaces of the rods are smooth and neither concaveness nor cavity can be seen. All XRD patterns consist only of broad peaks, and no diffraction peak corresponding to a crystalline phase is seen, indicating that the glassy alloy rods are formed in the diameter range up to 12 mm. Figures 7 and 8 show DSC and DTA curves of the La-Al-Ag-(Cu, Co, Ni) glassy alloys. As seen from these DSC scans as well as from Table 1, the ΔT_x remarkably increases by a small amount of Ni addition and the largest

Fig. 7 DSC curves of melt-spun La-Al-(Cu, Co, Ni)-Ag glassy alloys.

Fig. 8 DTA curves of the La-Al-(Cu, Co, Ni)-Ag alloys.

ΔT_x of 96 K was obtained for $\text{La}_{62.5}\text{Al}_{12.5}\text{Cu}_{15}\text{Ni}_5\text{Ag}_5$ alloy. However, the ΔT_x value decrease by a small amount of Co addition. In addition, it is noticed that all the T_g slightly increase by the Ni or Co addition. From Fig. 8 and Table 1, it is seen that the T_i values of the La-Cu-Al-Ag alloys decrease by a small amount of Ni or Co addition. The $\text{La}_{62.5}\text{Al}_{12.5}\text{Cu}_{10}\text{Ni}_5\text{Co}_5\text{Ag}_5$ glassy alloy shows the lowest T_i of 691 K, resulting in the highest T_g/T_i of 0.585, leading to the formation of the glassy sample with largest critical diameter above 12 mm. Based on Table 1, the critical sample

diameters (d_c) of the present glassy alloys are plotted as a function of T_g/T_l , as shown in Fig. 9. A good correlation between the d_c and T_g/T_l is recognized, suggesting that the T_g/T_l is closely related to the GFA of the La-based glassy alloys. On the other hand, with the aim of obtaining at a better description of the GFA of various glassy alloys, the parameters, including $\Delta T_x^{(20)}$ and $\gamma(T_x/(T_g + T_l))^{(30)}$ have been utilized by different researchers. However, as is evident from Table 1, we cannot see that the present glassy alloys have good correlation between GFA and ΔT_x or γ .

3.4 Mechanical properties of La-based bulk glassy alloys

Figure 10 shows compressive stress-strain curves of the La-based glassy alloy rods with a diameter of 2.0 mm. The glassy alloys exhibit high fracture strengths of over 640 MPa and distinct plastic strains. Table 1 summarizes mechanical properties of bulk glassy La-based glassy alloys. The bulk glassy alloys possess good mechanical properties, *i.e.*, compressive fracture strength ($\sigma_{c,f}$) of 640–858 MPa, Young's modulus (E) of 36–46 GPa, compressive elastic

strain ($\varepsilon_{c,e}$) of 1.7–1.9%, plastic strain ($\varepsilon_{c,p}$) of 0.2–0.4% and Vickers hardness (H_v) of 201–228. The fracture behavior and fracture surface appearance are exemplified for the $\text{La}_{55}\text{Al}_{20}\text{Cu}_{20}\text{Ag}_5$ glassy alloy rod in Fig. 11. The fracture occurs along the maximum shear stress plane, which is declined by about 45 degrees to the direction of applied load (Fig. 11(a)), and the fracture surface consists mainly of a well-developed vein pattern (Fig. 11(b)) typical to other bulk glassy alloys with good ductility.^{12,20} Many shear deformation bands are also observed on the rod surface (Fig. 11(c)). The distinct plastic strain can thus be attributed to the generation of many shear deformation bands.

4. Conclusions

We examined the thermal stability, GFA and mechanical properties of La-based La-Al-Cu-Ag and La-Al-(Cu, Ni, Co)-Ag glassy alloys, based on the fundamental knowledge

Fig. 9 The critical diameters as a function of T_g/T_l for the La-based bulk glassy alloys.

Fig. 10 Compressive stress-strain curves of cast La-based glassy alloy rods with a diameter of 2.0 mm. (a) $\text{La}_{55}\text{Al}_{25}\text{Cu}_{20}$, (b) $\text{La}_{55}\text{Al}_{20}\text{Cu}_{20}\text{Ag}_5$, (c) $\text{La}_{62.5}\text{Al}_{12.5}\text{Cu}_{15}\text{Ni}_5\text{Ag}_5$, (d) $\text{La}_{62.5}\text{Al}_{12.5}\text{Cu}_{15}\text{Co}_5\text{Ag}_5$, (e) $\text{La}_{62.5}\text{Al}_{12.5}\text{Cu}_{10}\text{Ni}_5\text{Co}_5\text{Ag}_5$.

Fig. 11 Fracture surface appearance of the cast $\text{La}_{55}\text{Al}_{20}\text{Cu}_{20}\text{Ag}_5$ glassy alloy rod.

La-Al-Cu ternary glassy alloys, with the aim of searching for a new bulk glassy system with high GFA. The results obtained are summarized as follows.

- (1) The addition of 5 at% Ag to La-Al-Cu alloys greatly increases the stability of supercooled liquid and T_g/T_l . The largest ΔT_x of 83 K and T_g/T_l of 0.539 were obtained for the $\text{La}_{62.5}\text{Cu}_{20}\text{Al}_{12.5}\text{Ag}_5$ alloy, leading to the formation of the glassy rods with diameters up to 6 mm by copper mold casting.
- (2) The addition of a small amount of Ni or Co to La-Al-Ag-Cu alloys increases T_g and lowers T_l , resulting in the higher T_g/T_l values of over 0.540. The best GFA was achieved for $\text{La}_{62.5}\text{Al}_{12.5}\text{Cu}_{10}\text{Ni}_5\text{Co}_5\text{Ag}_5$ alloy and its critical sample diameter was over 12 mm.
- (3) The GFA of the La-based alloys is more closely related with T_g/T_l values rather than the ΔT_x or γ .
- (4) The bulk glassy alloys exhibited good mechanical properties, *i.e.*, compressive fracture strength of 640–858 MPa, Young's modulus (E) of 36–46 GPa, plastic strain of 0.2–0.4% and Vickers hardness (H_v) of 201–228. The new La-based glassy alloys with high GFA, low T_g , wide ΔT_x and good mechanical properties may have useful engineering applications in the future.

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