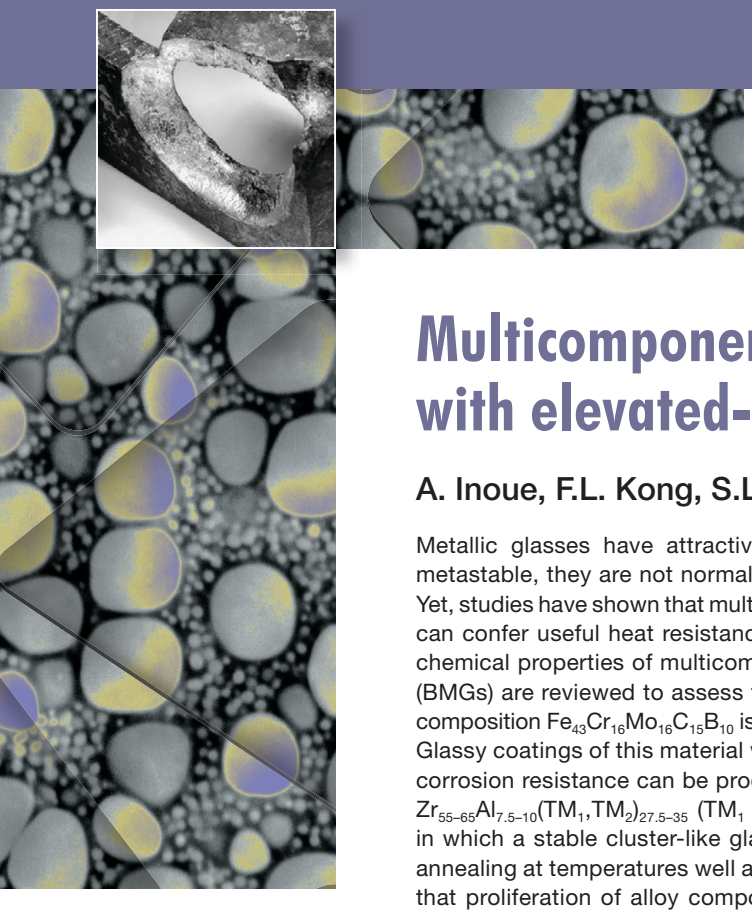


Multicomponent bulk metallic glasses with elevated-temperature resistance

A. Inoue, F.L. Kong, S.L. Zhu, and A.L. Greer

Metallic glasses have attractive properties, but since the glassy state is inherently metastable, they are not normally considered for applications at elevated temperatures. Yet, studies have shown that multicomponent and pseudo high-entropy (PHE) compositions can confer useful heat resistance. The formation, thermal stability, and mechanical and chemical properties of multicomponent Fe-(Cr, Mo)- and Zr-based bulk metallic glasses (BMGs) are reviewed to assess their potential as heat-resistant structural materials. The composition $Fe_{43}Cr_{16}Mo_{16}C_{15}B_{10}$ is castable and fully glassy with rod diameters up to 2.7 mm. Glassy coatings of this material with low porosity, good mechanical properties, and good corrosion resistance can be produced by high-velocity spray coating. The compositions $Zr_{55–65}Al_{7.5–10}(TM_1,TM_2)_{27.5–35}$ (TM_1 = Fe, Co, Ni, TM_2 = Cu, Pd, Ag, Au) yield PHE BMGs, in which a stable cluster-like glassy phase without crystalline precipitates is formed by annealing at temperatures well above the first calorimetric transformation. It is suggested that proliferation of alloy components is an effective method to synthesize metastable metallic materials that retain high strength at elevated temperatures.

Introduction

Most of the common metals have a simple crystal structure, body-centered-cubic (bcc), face-centered-cubic (fcc), or hexagonal closed-packed (hcp), though their alloys may include compounds with more complex structures. It is of interest to synthesize novel alloys with different structures that may be expected to give unique and useful properties. One effective method to change structures is by alloying with more metallic elements. This can change the structure from crystalline to glassy even in a cast bulk form.[1–3]

The formation of bulk metallic glass (BMG) is associated with alloys in which the selection of components follows three rules, namely: (1) at least three elemental components, (2) significant atomic size mismatches above 12% among the main three elements, and (3) negative heats of mixing among the three elements.[1,4] In addition, such multicomponent alloys have deep eutectic valleys and permit access to a highly supercooled liquid state, resulting in the formation of BMGs, which have an atomic configuration that is disordered at long range and ordered at a medium range of 1.7–2.0 nm.[5] These BMGs exhibit unique combinations of properties that cannot be obtained for crystalline alloys (e.g., ultrahigh strength, large elastic strain, high corrosion resistance, high wear resistance, surface smoothness, viscous flowability, net castability, and precise formability).[1,4] In addition, Fe- and Co-based BMGs exhibit good soft magnetic properties with low coercivity, high permeability, and low core loss since their atomic configuration is uniform on the scale of the magnetic domains.[1,4] To exploit these advantages, BMGs have been used in many fields and applications such as structural, soft magnetic, coatings, ornamental, casing, springs, sensors, mirrors, gears, hinges, bolts, and cutlery.[1,4]

In general, BMGs have compositions selected to optimize their glass-forming ability ([GFA], which is used to describe the tendency for materials to form glassy phase). An equiatomic multicomponent BMG was synthesized for $Ti_{20}Zr_{20}Hf_{20}Ni_{20}Cu_{20}$ in 2002.[6] Two years later, equiatomic multicomponent crystalline alloys with fcc and bcc structures were also formed for $Fe_{20}Co_{20}Ni_{20}Cr_{20}Mn_{20}$[7] and $Al_xFeCoNiCrCu$ (x = 0–3),[8] respectively. The latter was named as a high-entropy (HE) alloy in the paper. Since these syntheses, much attention has also been paid to equiatomic and near-equiatomic multicomponent alloys since

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doi:10.1557/mrs.2019.253

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these show metastable supersaturated solid-solution phases with bcc and fcc structures in the absence of compounds, or show a glassy structure.[9,10] Subsequently, pseudo-high-entropy (PHE) glassy alloys, where the definition is (1) five elements or more; (2) solvent element of 40–65 at.%; (3) solute elements of 5–15 at.%; and (4) positive heat of mixing and large atomic size mismatching among the solute elements, which is different from HE alloys, have been synthesized.[11–13] The PHE type $Zr_{55–65}Al_{7.5–10}(TM_1,TM_2)_{27.5–35}$ (TM_1 = Fe, Co, Ni, TM_2 = Cu, Ag, Pd, Au) glassy alloys exhibit a novel phase decomposition behavior leading to the formation of a cluster-like glassy structure.

This article reviews the formation, thermal stability, mechanical and chemical properties, and applications of multicomponent Fe- and PHE Zr-based BMGs with high resistance to phase decomposition and investigates the usefulness of these BMGs as heat-resistant materials.

Development of amorphous alloy steels

Alloy steels containing Cr and Mo are well known as low-cost heat-resistant materials. Since the first synthesis of an amorphous alloy (in the Au-Si system) by rapid solidification in 1960,[14] amorphous alloys have attracted significant attention as potential novel advanced materials. The metastable nature of amorphous or glassy alloys, however, does not make them obvious candidates as heat-resistant materials.

When considering possible heat-resistant amorphous alloys for structural applications, Fe-based systems with alloy components similar to conventional heat-resistant materials are attractive as precursor materials. In the development of structural Fe-based amorphous alloys, Fe-(Cr,Mo,W)-C ternary and quaternary amorphous alloys were synthesized by rapid solidification in 1978.[15–17] Among these, $Fe_{82–x–y}Cr_xMo_yC_{18}$ alloys had a wide amorphous composition range of 0–50 at.% Cr and 0–26 at.% Mo, and were named amorphous alloy steels. These amorphous steels exhibit a high crystallization temperature (T_x) of 765–935 K, good bending plasticity, high tensile fracture strength of 3450–3790 MPa, high Vickers hardness (Hv) of 910–1130, and high corrosion resistance in various solutions. There is a clear tendency for T_x and Hv to increase with increasing Cr and Mo contents. In scanning calorimetry, these alloys do not show a glass transition, and have been termed amorphous-type alloys. The absence of a detectable glass transition reflects the instability of the supercooled liquid (SL) state and shows that these systems do not have high GFA.

Much effort was made to develop a highly heat-resistant material by using these amorphous alloy steels as a precursor.[18,19] These heat-resistant alloys were produced in two simple stages: (1) production of amorphous alloy powders by high-pressure gas atomization or pulverization of melt-spun amorphous alloy ribbons subjected to annealing-induced embrittlement treatment, followed by (2) uniaxial hot pressing, hot isostatic pressing, or spark plasma sintering of the amorphous powders. The resulting press-sintered bulk materials have high packing densities above 99% and a mixed structure of fine carbides dispersed homogeneously in bcc or tempered martensite matrix phase. The Hv is dependent on the pressing temperature (T_p), showing a maximum near the T_p of 1273 K and decreasing rapidly with further increase in T_p, owing to the grain growth of the constituent phases. Press-sintered bulk alloys of the Fe-Cr-Mo-C and Fe-Cr-Mo-V-C systems were tested as heat-resistant high-speed cutting or bolt materials and were confirmed to exhibit favorable characteristics, such as high hardness even at elevated temperatures.[20] However, no significant applications have been realized so far because of the relatively high cost of these materials.

Fe-based BMGs and their applications

In a 1995 breakthrough, the first Fe-based BMGs were synthesized. Multicomponent Fe-(Al,Ga)-(P,B,Si,C) alloys, with compositions following the three rules previously noted[21] were made by copper mold casting. These pioneering Fe-based BMGs were, however, not usable as structural materials owing to their low corrosion resistance. Much effort has since been devoted to develop a structural Fe-based BMG using previous data[15–17] on Fe-(Cr,Mo)-C amorphous alloys. In 2002, new BMGs based on the Fe-Cr-Mo-C-B system were synthesized by copper mold casting into rods with diameters up to 2.7 mm.[22] The glassy phase was formed over a wide composition range of 0–30 at.% Cr, 7.5–22.5 at.% Mo, and a wide C/B concentration ratio range, as shown in **Figure 1**. These BMGs exhibit high glass-transition temperature (T_g) (860–940 K), wide SL region (40–90 K), high T_x (900–1010 K), and high corrosion resistance.[23] The high corrosion resistance originates from the spontaneous formation of a thin, highly passive $Cr_x(OH)_y$ surface layer.

The bulk GFA has been significantly improved by adding small amounts of Y or lanthanide elements (Er and Tm), as exemplified by the maximum rod diameters of Fe-Cr-Mo-C-B-Er,[24] Fe-Cr-Mo-Mn-C-B-Y,[25] and (Fe-Co)-Cr-Mo-C-B-Tm[26] alloys shown in **Figure 2**. The maximum rod diameter for glass formation reaches above 10 mm for these alloy systems and 16 mm for the (Fe-Co)-Cr-Mo-C-B-M (M = Tm or Y) system.[26,27] The high GFA for these Fe-based alloys is reportedly related to the development of medium-range-ordered atomic configurations. For example, Fe-Cr-Mo-C-B and Fe-Cr-Mo-C-B-Tm BMGs reflect, respectively, the complex structures of cubic $M_{23}(C,B)_6$ and $M_6(C,B)$ (M = Fe, Cr, Mo).[24,28] These compounds have a large unit cell that includes icosahedral-like local atomic configurations. The addition of Ln has been interpreted to cause the formation of denser atomic configurations through greater adherence to the three component rules for the formation of BMGs and stabilization of the SL.[28,29]

High-velocity oxy-fuel (HVOF) spraying has been widely used for thermal-coating processes. A mixture of fuel and oxygen is ignited to provide heat and high pressure. The coating powder is heated and accelerated in the spray gun and then

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sprayed at a high velocity onto the surface of the substrate. Exploiting the high GFA that enables the production of BMGs with diameters above several millimeters, glassy deposits with thickness of 0.2–0.5 mm have been applied over the surface by HVOF spraying onto diverse substrates such as stainless steels, plain carbon steels, Al alloys, and Mg alloys.[30,31] **Figure 3**a shows the cross-sectional structure of a Fe-Cr-Mo-C-B glassy coating that is fully dense and has a high compressive strength.[31] The coating has good corrosion resistance (Figure 3b). There is no appreciable exfoliation of the coating from the substrate for samples subjected to repeated bending deformation (Japanese Industrial Standards: JIS H 8504:1999, Methods of adhesion test for metallic coatings, Figure 3c). Figure 3d shows coatings applied to pipes.[4] The wear resistance test was conducted by the ball-on-disk wear test with a water soluble lubricant for 1 h with a load of 60 kgf. The metallic glass coating shows excellent wear resistance exceeding that of commercial CrN coatings and hard chromium plating (**Table I**).[32]

The Fe-Cr-Mo-C-B glassy coating has found use in applications, because its combination of favorable properties cannot be obtained for any crystalline coating. **Figure 4** shows the stainless-steel SUS304 test piece with and without a glassy coating after the erosion test and an example of the glassy coating on a vessel used for the melting of lead-free solder.[33] The elimination of lead increases the temperature required for liquid processing and greatly increases the corrosive attack on the steel vessel. The Fe-Cr-Mo-C-B glassy coating demonstrates retention of corrosion and erosion resistances, and of strength, at an elevated temperature.[33] It can be used for at least six months, much longer than the usable life of the best-performing stainless steel.[33] Severe erosion can be observed for the uncoated vessel used for three years, but no sign of erosion can be observed for the coated vessel used for four years under the same condition. The transition to the use of

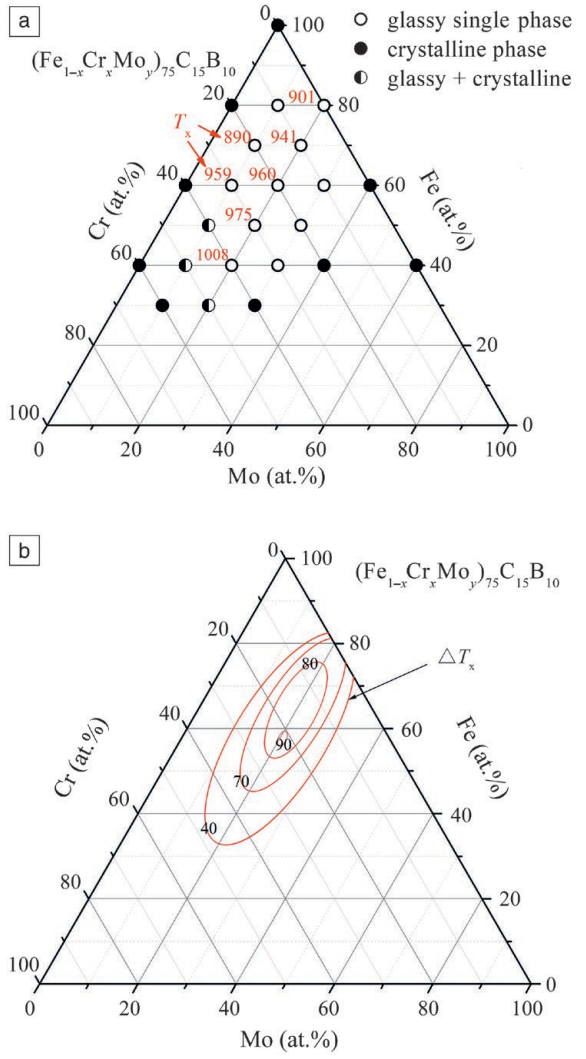


Figure 1. (a) Glass-formation range and crystallization temperature T_x for as-spun Fe-Cr-Mo-C-B alloys, and (b) compositional dependence of the supercooled liquid region, $\triangle T_x (= T_x - T_g$, where T_g is the glass-transition temperature), of these glassy alloys.[23] The alloys can form a single glassy phase in a wide composition range and exhibit high T_x as well as T_g.

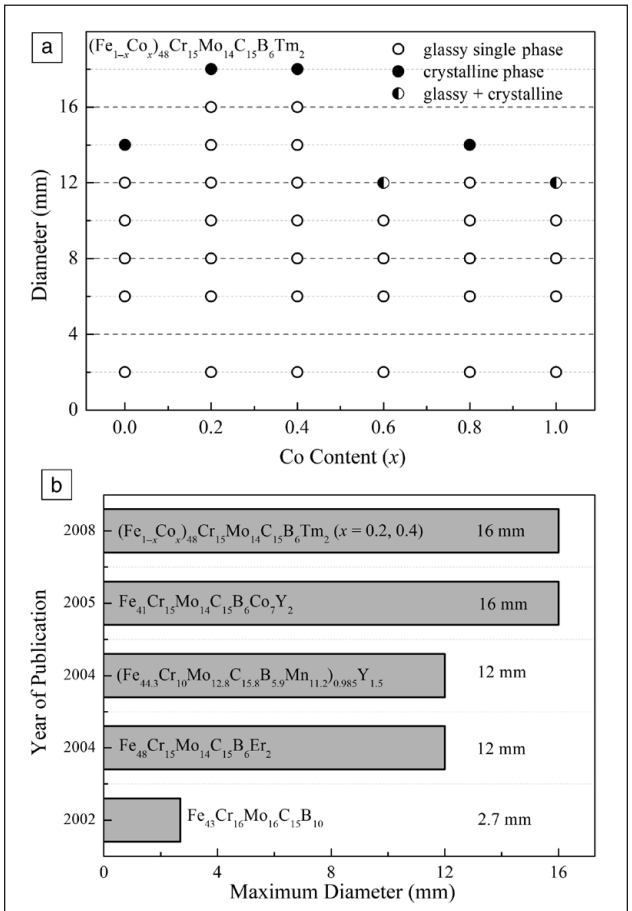


Figure 2. (a) Maximum diameters for (Fe,Co)-Cr-Mo-C-B-Tm bulk metallic glass (BMG) rods prepared by copper mold casting. With a 2% addition of Tm element, the maximum rod diameter reaches 16 mm, and (b) maximum diameters of (Fe,Co)-Cr-Mo-C-B(-Ln) BMGs.[24–26] The glass-forming ability can be significantly improved by adding small amounts of Y, Tm, or Er.

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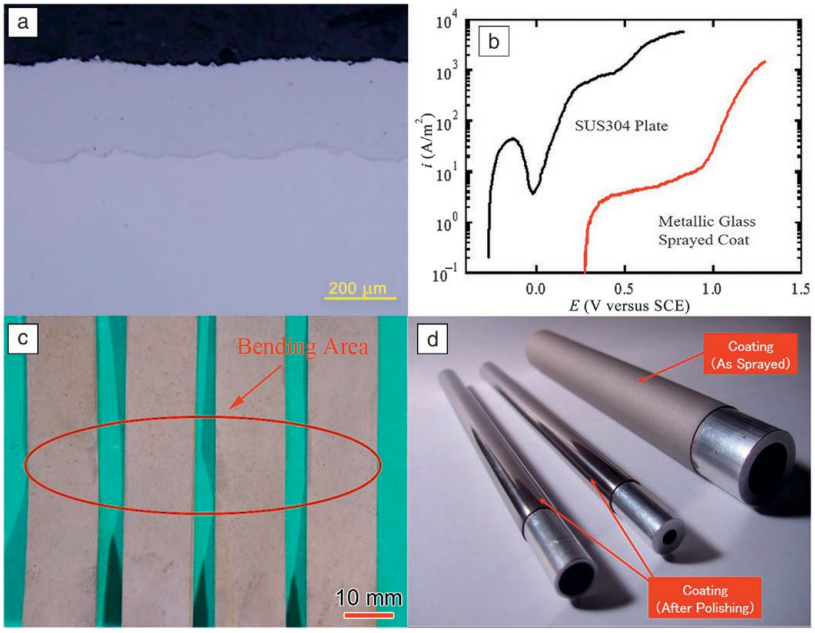


Figure 3. (a) Optical micrograph of the cross section of Fe-Cr-Mo-C-B metallic glass (MG) coating. A dense coating with thickness of 200–250 μm was obtained. (b) Corrosive potential (*E*) and anodic current density (*i*) curve for stainless-steel SUS304 (with 18 wt% Cr and 8 wt% Ni) plate and Fe-Cr-Mo-C-B MG coating. The MG coating shows a wide stable potential region Note: SCE, saturated calomel electrode. (c) Photograph of an Fe-Cr-Mo-C-B glassy coating prepared by high-velocity oxy-fuel spray coating after repeated bending deformation. Neither abrasion nor swelling can be observed after the bending test. (d) The appearance of MG coatings sprayed onto aluminum pipes.[4,31]

lead-free solder has clear environmental benefits, and the use of the glassy coating facilitates cleaner processing, relevant for sustainability.

Zr-based clustered glassy alloys

The primary precipitation phase for $Zr_{65}Al_{7.5}Ni_{10}Cu_{17.5}$ BMGs changes from a crystalline compound to an icosahedral (I) phase by the partial replacement of Cu with noble metals (NM = Pd, Pt, Ag or Au).[34] Several studies on I-phase precipitation from the amorphous phase upon annealing have focused on similar Zr-based BMGs with 65–70 at.% Zr and 3–10 at.% NM.[35,36] I-phase precipitation has been interpreted to reflect the generation of Ni–NM atomic pairs with positive heats of mixing, leading to the partial destruction of medium-range-ordered atomic configurations. However, there had been no data on the phase decomposition behavior for Zr-rich BMGs containing much larger amounts of NM.

Recently, it was reported that the addition of larger amounts of NMs causes a distinct change in the primary precipitation phase from I-phase to extremely fine cluster-like precipitate; this can be regarded as the formation of a cluster-like glassy phase.[12] The cluster-like glassy alloys have the following compositional features: (1) at least four elements (Zr, Al, TM_1, and TM_2); (2) large atomic-size mismatches among the main three elements, Zr, Al, and TM; (3) negative heats of mixing among the three elements; (4) large atomic-size mismatches between TM_1 and TM_2; and (5) positive heats of mixing between TM_1 and TM_2. Factors (1–3) are the same as those for the formation of ordinary BMGs. The additional (4) and (5) factors seem to be essential for the formation of the cluster-like glassy phase. Multicomponent BMGs with these compositional and structural features are termed as PHE BMGs.[37]

The PHE glassy alloys, $Zr_{65}Al_{7.5}Ni_{10}Cu_{2.5}Ag_{15}$, $Zr_{65}Al_{7.5}Ni_{15}Ag_{12.5}$, and $Zr_{65}Al_{7.5}Co_{12.5}Ag_{15}$,[12,13] exhibit two exothermic peaks in the differential scanning calorimetry (DSC) curves. **Figure 5**a shows x-ray diffraction patterns of PHE $Zr_{65}Al_{7.5}Co_{27.5-x}Ag_x$ glassy alloys annealed for 7.2 ks at 713 K, which is above the onset temperature of the first-stage exotherm peak (T_{x1}). Apart from the $x = 15$ alloy that shows precipitation of the I-phase, the patterns retain the broad haloes that are typical for a glassy phase, and upon annealing, no distinct change is recognized. In addition, as exemplified for the annealed $Zr_{65}Al_{7.5}Co_{17.5}Ag_{10}$ alloy in Figure 5b–e,[13] transmission electron microscopy data show the material to be fully amorphous. No appreciable crystalline precipitates were observed even after annealing for a long period of time at temperatures well above T_{x1}, indicating that the PHE amorphous alloys have extremely high stability against phase decomposition. The DSC curves of these annealed cluster-like glassy alloys show only the second exothermic peak.[13] The disappearance of the first exothermic peak indicates that calorimetrically, the effect of annealing is equivalent to completion of the first-stage crystallization reaction. There appear to be no earlier reports of this unusual phenomenon. The change to the cluster-glass phase also increases *Hv* by approximately 1.5× as compared with as-prepared glassy alloys.

The absence of an appreciable crystalline phase in the cluster-like glassy phase may be due to extremely sluggish crystal grain growth, implying that the PHE alloys

Table I. Results of wear tests for metallic glass coating, hard chromium plating, and CrN coating.[32]

Layer Material	Weight of Specimen		Wear Loss (b–a)/g
	(a) Before/g	(b) After/g	
Metallic glass coating	74.04	74.03	0.01
Hard chromium plating	73.48	73.07	0.41
CrN coating	71.02	70.91	0.11

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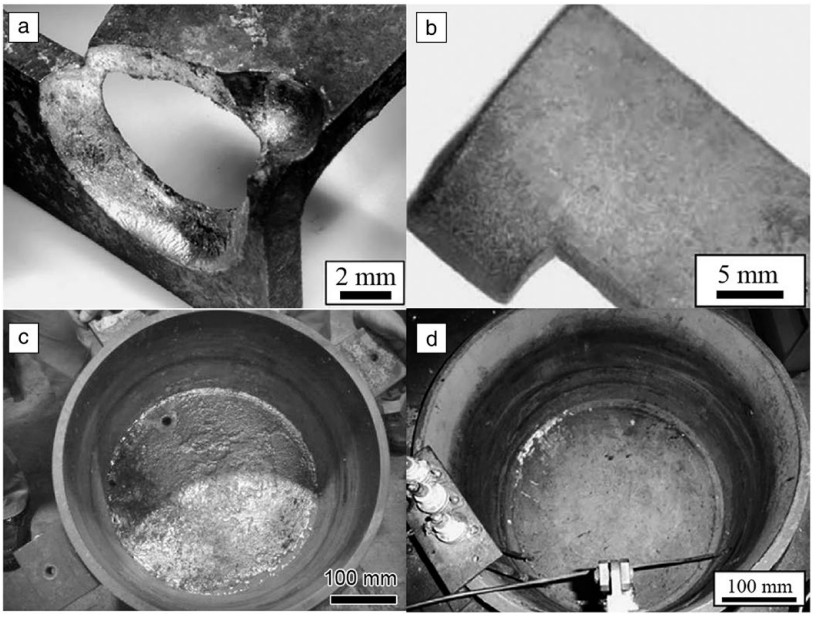


Figure 4. Stainless-steel SUS304 test piece subject to an erosion test (a) without Fe-Cr-Mo-C-B metallic glass (MG) coating after 500 h and (b) with MG coating after 2000 h. The photograph of a commercial stainless-steel vessel used for lead-free soldering: (c) three years without MG coating, and (d) four years with MG coating. Severe erosion was observed for the uncoated sample, but no obvious erosion was seen for the coated sample.[4,32]

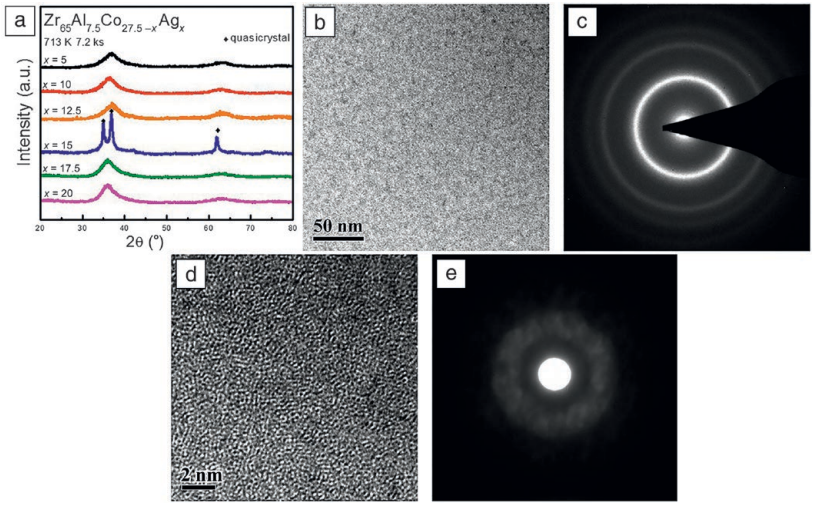


Figure 5. (a) X-ray diffraction patterns of $Zr_{65}Al_{7.5}Co_{27.5-x}Ag_x$ (x = 5–20 at.%) melt-spun ribbons, where θ is the scattering angle; only the alloy with x = 15 showed an icosahedral phase; (b) transmission electron microscopy (TEM) bright-field image, (c) selected-area electron diffraction pattern, (d) High-resolution TEM image, and (e) nanobeam diffraction pattern of $Zr_{65}Al_{7.5}Co_{17.5}Ag_{10}$ alloy annealed for 7.2 ks at 713 K, a temperature above the first exothermic peak in differential scanning calorimetry traces.[13]

also retain bulk GFA, as is evident from the formation of BMG rods with diameters of up to several millimeters.[11–13] These BMGs do not exhibit the first-stage exothermic peak, but no appreciable contrast corresponding to a crystalline phase is observed over the transverse cross section, indicating the formation of Zr-based BMGs with higher resistance to crystallization as compared with ordinary BMGs.[13]

Summary

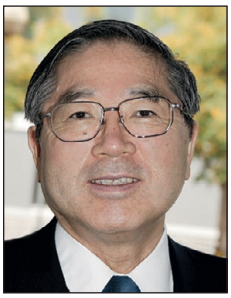
Studies on the potential heat-resistant properties of multicomponent Fe- and Zr-based glassy alloys have been reviewed. Fe-Cr-Mo-C-B glassy alloys have high GFA, which enables the formation of BMG rods with diameters up to 2.7 mm, exhibiting high mechanical strength and good corrosion resistance. The high GFA also enabled glassy coatings with a thickness of at least 0.5 mm to be applied to a diverse range of substrates by a high-velocity spray-coating technique. Such a glassy coating has been applied to vessels that are used for melting of lead-free solder liquid, an application that requires elevated-temperature resistance to corrosion and erosion. Novel cluster-like BMGs with significantly enhanced strength were formed after annealing for long holding times at temperatures well above T_{x1} for PHE $Zr_{55-65}Al_{7.5-10}(TM_1,TM_2)_{27.5-35}$ (TM_1 = Fe, Co, Ni, TM_2 = Cu, Pd, Ag, Au) alloys. Multicomponent Fe-based and PHE BMGs are promising for future developments of heat-resistant structural materials that retain strength at elevated temperatures.

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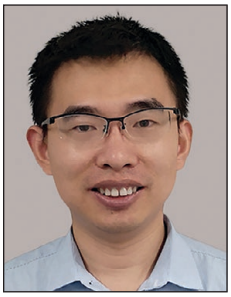
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https://doi.org/10.1557/mrs.2019.253 Published online by Cambridge University Press