

DEVELOPMENTS AND APPLICATIONS OF BULK METALLIC GLASSES

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Received: February 28, 2008

Abstract. This paper aims to review our recent data on the development researches and application examples of bulk glassy alloys. Very recently, the glass-forming ability of metallic alloys is significantly enhanced by further multiplication of alloy components based on the three component rules for bulk glass formation. The maximum diameter for glass formation exceeds 10 mm in a variety of alloy systems such as Zr, Pd, Pt, Mg, La, Fe, Co, Ni, and Cu bases. By use of high glass-forming ability, good castability, good printability and unique characteristics, application examples of bulk glassy alloys have been extended to much valuable fields in which conventional crystalline alloys cannot be used. These novel advantages for bulk glassy alloys allow us to conclude that bulk glassy alloys are used in much wider application fields in the near future.

1. INTRODUCTION

Metallic materials have been used for several thousands years. However, the metals used before the industrial revolution in the 18th century had been limited to eleven kinds, e.g., gold, silver, copper, iron, mercury, *etc.* For several hundreds of years after the industrial revolution, the number of metals which have been used by human beings increases significantly through the progresses of refinement and metallurgy techniques and reaches over 75 kinds at present. However, metallic materials in a bulk form with thicknesses of over several millimeters had been limited to a crystalline structure. This is due to the inevitable principles of phase transformation and solidification in metals and alloys. That is, in metals and alloys having metallic bonding nature, the atomic diffusion is extremely easy in a supercooled liquid region at high temperatures just below melting temperature, resulting in instantaneous nucleation and growth reactions of a crystalline phase. The easy transformation had lead to the limited state where bulk

metallic materials were composed of only a crystalline structure.

In such an enclosed state, it was found around 1990 that the phase transformation from supercooled liquid to crystalline phase was retarded by 8 to 9 orders for special multi-component metallic alloys [1-3]. The selection of their novel alloy components has enabled us to produce glassy (disordered) metallic alloys in a bulk form because the transformation of supercooled liquid to crystalline phase for their alloys can be suppressed even at a very low cooling rate of the order 0.01 K/s [4]. By the increase in the stability of supercooled liquid against crystallization by 8 to 9 orders, we can control and utilize the supercooled liquid state in their special metallic alloys, leading to the production of various non-equilibrium bulk alloys exhibiting highly functional characteristics as well as unique workability and castability. These non-equilibrium bulk alloys have attracted increasing interest over the world as an innovative metal in scientific and technological aspects.

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Table1. Typical bulk glassy alloy systems reported up to date together with the calendar years when the first paper or patent of each alloy system was published.

1. Nonferrous alloy system

Mg-Ln-M (Ln=Lanthanide Metal, M=Ni,Cu,Zn)*	1988
Ln-Al-TM(TM=Fe,Co,Ni,Cu)*	1989
Zr-Al-TM *	1990
Zr-Ln-Al-TM *	1992
Ti-Zr-TM *	1993
Zr-Ti-TM-Be	1993
Zr-(Ti,Nb,Pd)-Al-TM*	1995
Pd-Cu-Ni-P*	1996
Pd-Ni-Fe-P	1996
Ti-Ni-Cu-Sn *	1998
Ca-Cu-Ag-Mg*	2000
Cu-Zr*,Cu-Hf*	2001
Cu-(Zr,Hf)-Ti*	2001
Cu-(Zr,Hf)-Ti-(Y,Be) *	2001
Cu-(Zr,Hf)-Ti-(Fe,Co,Ni)*	2002
Cu-(Zr,Hf)-Al*	2003
Cu-(Zr,Hf)-Al-(Ag,Pd)*	2004
Pt-Cu-Ni-P	2004
Ti-Cu-(Zr,Hf)-(Co,Ni)*	2004
Au-Ag-Pd-Cu-Si	2005
Ce-Cu-Al-Si-Fe*	2005
Cu-(Zr,Hf)-Ag*	2006

2. Ferrous alloy system

Fe-(Al,Ga)-(P,C,B,Si,Ge)*	1995
Fe-(Nb,Mo)-(Al,Ga)-(P,B,Si)*	1995
Co-(Al,Ga)-(P,B,Si)*	1996
Fe-(Zr,Hf,Nb)-B*	1996
Co-(Zr,Hf,Nb)-B*	1996
Ni-(Zr,Hf,Nb)-B*	1996
Fe-Co-Ln-B*	1998
Fe-Ga-(Cr,Mo)-(P,C,B)	1999
Fe-(Nb,Cr,Mo)-(C,B)*	1999
Ni-(Nb,Cr,Mo)-(P,B)*	1999
Co-Ta-B*	1999
Fe-Ga-(P,B)*	2000
Ni-Zr-Ti-Sn-Si	2001
Ni-(Nb,Ta)-Zr-Ti*	2002
Fe-Si-B-Nb*	2002
Co-Fe-Si-B-Nb*	2002
Ni-Nb-Sn	2003
Co-Fe-Ta-B-Si*	2003
Ni-Pd-P *	2004
Fe-(Cr,Mo)-(C,B)-Ln (Ln=Y, Er, Tm)	2004
Co-(Cr,Mo)-(C,B)-Ln (Ln=Y, Er, Tm)*	2006

Alloy systems which are marked with * were found by Sendai Group.

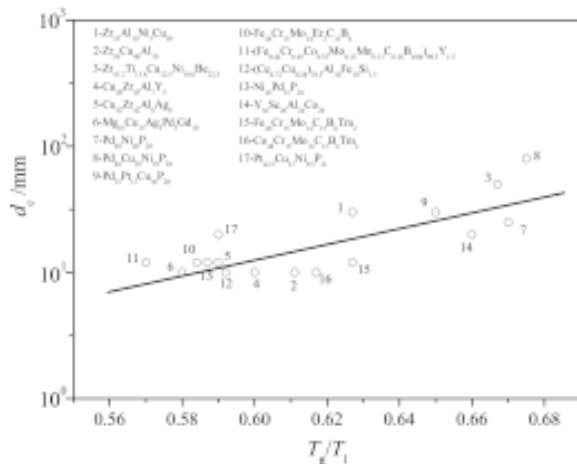


Fig. 1. Critical maximum diameters (d_{max}) as a function of T_g/T_l for glassy alloys with critical diameters of over 1 cm.

2. FORMATION CRITERION AND DEVELOPMENT HISTORY OF BULK METALLIC GLASSES

Table 1 summarizes alloy component systems of bulk glassy alloys found for the past 18 years since the first discovery in 1988 [5,6]. The alloy systems can be classified to non-ferrous and ferrous alloy groups. The former group is composed of Mg, lanthanide metal (Ln), Zr, Ti, Hf, Pd, Ca, Cu, Pt, and Au base systems and the latter group consists of Fe, Co, and Ni base systems. The table indicates clearly the following three features, i.e., (1) considering that the Ln element consists of more than 15 kinds, the total number of bulk glassy alloy systems reaches about 1000 kinds, (2) 5 to 6 years between 1988 and 1993 are an incubation period in the research field of bulk glassy alloys, and (3) more than 50% in the number of bulk glassy alloy systems were found for the last 10 years after 1995 and hence this research field has developed significantly even at present.

The extraordinary increase in the number of bulk glassy alloy systems for the past one decade is attributed to active searches for a new alloy system based on the following three component rules for stabilization of supercooled liquid [1,3], i.e., (1) multi-component consisting of more than three elements, (2) significant atomic size mismatches of over about 12% among the main three elements, and (3) negative heats of mixing among the main

three elements. The series of successes in finding a large number of bulk glassy alloy systems demonstrate the validity of the three component rule as a search guide of a new bulk glassy alloy system. In addition, the multi-component alloys with the three component rules always belong to a eutectic type system. Thus, we can recognize a tendency that the highest glass-forming ability is obtained at the composition near the multi-component eutectic point with the lowest melting temperature.

More recently, the establishment of the validity of multi-component for the glass-forming ability has led to the syntheses of a number of bulk glassy alloys consisting of more multi-components, resulting in a further improvement of glass-forming ability. As summarized in Table 2, bulk glassy alloys with diameters of over 10 mm have been produced in various alloy systems such as Zr, Pd, Pt, Mg, La, Fe, Co, Ni, and Cu bases. Judging from experimental data on the maximum diameter for glass formation, the glass-forming ability of these bulk glassy alloys is concluded to be the highest for Pd-based alloys and decreases in the order of $Zr > Pt > Mg=Ln > Fe=Cu > Ni=Co$. The glass-forming ability has been investigated on the basis of some thermal stability parameters, e.g., (1) reduced glass transition temperature (T_g/T_l) [1,2,7], (2) temperature interval of supercooled liquid region ($\Delta T_x (= T_x - T_g)$) [1,2,8], and (3) $\gamma (= T_x / (T_g + T_l))$ [9]. As shown in Fig. 1, the smallest scattering is recognized in the relation between the critical diameter (d_c) and T_g/T_l . Its relation can be formulized by $\text{Log}_{10} d_c = -2.57 + 6.16 T_g/T_l$. Based on the validity of the three component rules and the formulized relation, much larger bulk glassy alloys with diameters of over 20 mm are expected to be formed in a number of alloy systems through the further detailed studies on the multiplication of alloy components. Thus, the maximum diameters of bulk glassy alloys approach the dimension of conventional crystalline metallic materials.

3. FUNDAMENTAL CHARACTERISTICS OF BULK GLASSY ALLOYS

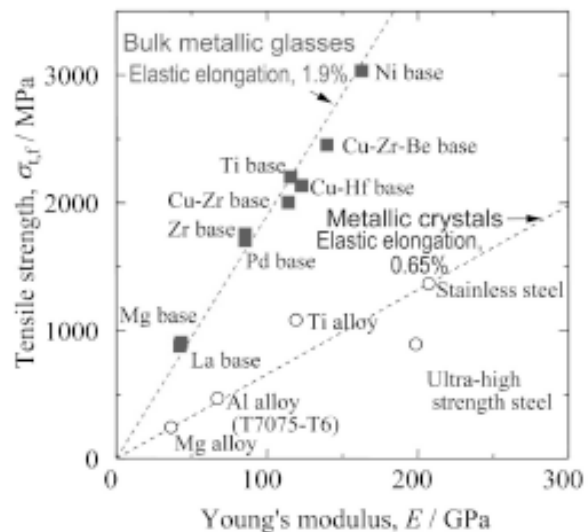
Fig. 2 shows the relation between tensile strength and Young's modulus for typical bulk glassy alloys, together with the data for conventional crystalline alloys. As unique mechanical properties of bulk glassy alloys in comparison with crystalline metallic alloys, one can recognize the following points [1-3], i.e., (1) the tensile strength of bulk glassy al-

Table 2. Critical diameter and preparation method for bulk glassy alloy systems with critical diameters of over 1 cm.

Alloy system	Critical diameter (mm)	Preparation method
Zr-Al-Cu	15	tilt-casting
Pd-Ni-P	25	water quenching
Ni-Pd-P	15	water quenching
Zr-Al-Ni-Cu	30	suction casting
Pd-Cu-Ni-P	>80	water quenching
Pd-Pt-Cu-P	>50	water quenching
Pt-Cu-Ni-P	20	water quenching
Cu-Zr-Al-Y	10	copper mold casting
Cu-Zr-Al-Ag	>25	copper mold casting
Y-Sc-Al-Co	20	copper mold casting
Mg-Cu-Ag-Gd	25	copper mold casting
Zr-Ti-Ni-Cu-Be	50	copper mold casting
Ce-Cu-Al-Si-Fe	20	copper mold casting
Fe-(Cr,Mo)-C-B-Y	12	copper mold casting
Fe-(Cr,Mo)-C-B-Tm	12	copper mold casting
Co-(Cr,Mo)-C-B-Y	10	copper mold casting
Co-(Cr,Mo)-C-B-Tm	10	copper mold casting
Fe-Co-(Cr,Mo)-C-B-Tm	16	copper mold casting

loys is about three times higher than that of crystalline alloys in case of the same Young's modulus, (2) the Young's modulus of bulk glassy alloys is about one-third as high as that of crystalline alloys in case of the same tensile strength, and (3) there is a good linear relation between tensile strength and Young's modulus, implying the satisfaction of Hook's law. The slope of this linear relation corresponds to an elastic elongation limit. This limit is measured to be about 2% which is about 3 times larger than that (about 0.65%) for crystalline alloys. In addition, the reversible maximum values of twist angle and shear stress under a torsional deformation mode are also 3 times larger than those for crystalline alloys.

In addition to static mechanical properties, the Charpy impact fracture energy is as high as over 100 kJ/m² for Zr-based bulk glassy alloys [10]. The fracture toughness measured for the ASTM-E399 standard size specimen is 40-50 MPa·m^{1/2} for Zr-based alloys, 68 MPa·m^{1/2} for Cu-based alloys and 22 MPa·m^{1/2} for Ti-based alloys [11,12], indicating that these bulk glassy alloys have simultaneously high strength and high dynamic ductility. Furthermore, the fatigue endurance limit after 10⁷ cycles

**Fig. 2.** Relationship between tensile strength and Young's modulus for typical transition metal base bulk glassy alloys. The data of conventional crystalline alloys are also shown for comparison.

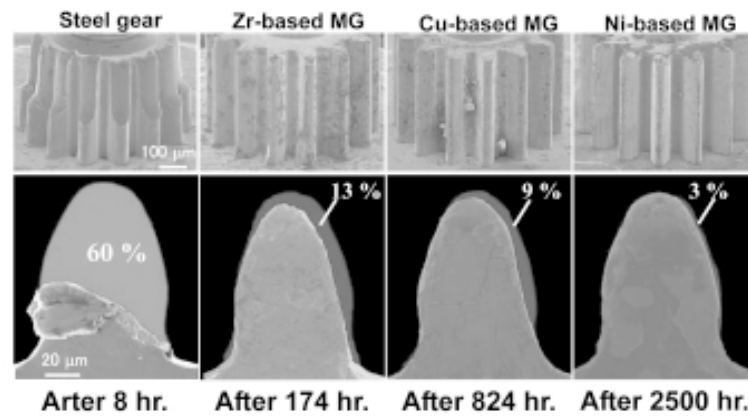


Fig. 3. The durability of Ni-based glassy alloy gears of 2.4 mm in diameter in comparison with SK4.

is in the range from 0.15 to 0.38 for Zr-, Ti- and Cu-based bulk glassy alloys [13,14]. Reflecting the high strength values of bulk glassy alloys, the minimum amplitude stress corresponding to the fatigue endurance limit after 10^7 cycles shows relatively high values of about 1000 MPa for Cu-based alloys and about 1500 MPa for Ti-based alloys which are comparable to or higher than those for conventional Cr-Mo steel and SKD tool steel.

The bulk glassy alloys containing Nb, Ta, Cr or Mo element can form highly dense passive surface film with high stability and homogeneity through the enrichment of these elements and exhibit excellent corrosion resistance [15-17]. For instance, the corrosion resistance of engineering important Fe-, Co- and Ni-based bulk glassy alloys in various chemical corrosive liquids is 100 to 10000 times higher than that for the highest class of stainless steel (SUS316L).

4. HIGH WEAR RESISTANCE OF BULK GLASSY ALLOYS AS MACHINERY PARTS

Recently, we have succeeded in producing high torque geared motors with outer diameters of 1.5 and 2.4 mm which are composed of sun-carrier, planet-type gears, gear axis and bearing made of Zr-, Cu-, Ni-, and Fe-based glassy alloys [18]. When the endurance time of these gears is measured in comparison with conventional tool steel (SK4), it is noticed that the time is much longer than that of

SK4 and the difference reaches 32 times for Zr-based alloys, 107 times for Cu-based alloys and over 1000 times for Ni-based alloys. As an example, Fig. 3 shows the durability of Ni-based glassy alloy gears used in the geared motor with an outer diameter of 2.4 mm in comparison with SK4. The 2.4 mm diameter geared motor is the minimum size which can be produced by mechanical machining processes. As seen in the figure, significant wear loss is seen for the SK4 gear. The rotation of the motor using SK4 gears stopped after 8 h ($6 \cdot 10^6$ revolutions), while the Ni-based glassy alloy gear kept the original shape even after 2500 h ($1875 \cdot 10^6$ revolutions) and could be used continuously. These wear data were obtained by using the same kind of metallic alloy gears. On the other hand, the wear resistance against Ni-based glassy alloy bearing is the highest for Fe-based glassy alloy, followed by Ni-based glassy alloy, Cu-based glassy alloy, Zr-based glassy alloy and then SK4. The wear loss of Fe-based glassy alloy is about one-third smaller than Ni-based glassy alloy, indicating that the wear resistance of their bulk glassy alloys is much better than that of SK4 steel. The reason for the excellent wear resistance is presumably due to the combination effect of smooth outer surface without grain boundary, highly homogenized structure without appreciable component segregation and high corrosion resistance, in addition to unique mechanical characteristics of high hardness, low Young's modulus, large elastic elongation, high toughness and high fatigue strength.

5. HIGH DUCTILITY OF BULK GLASSY ALLOYS BY COEXISTENCE WITH CRYSTALLINE PHASE

By adding special elements leading to the deviation from the three component rule to bulk glassy alloys, we can produce bulk glassy alloys containing nano-crystalline [19], nano-quasicrystalline [20,21] or micrometer-scale dendrite crystalline[22] phase in a number of alloy systems such as Zr, Cu, Ni, Pd, Ti, and Fe bases. These composite-type bulk glassy alloys exhibit various useful properties which cannot be obtained for single phase bulk glassy alloys. Among these properties, it is noticed that the elongation under a uniaxial compressive deformation increases significantly. This improvement is due to the easy generation of shear bands caused by stress concentration effect at the interface between glassy and crystalline phases having significantly different strength and Young's modulus characteristics. This can be regarded as the homogeneous dispersion effect of easy deformation sites. Besides, the nanocrystal-dispersed bulk glassy alloys can have improved values of both yield strength and ductility, though the bulk glassy alloys containing dendritic crystalline phase exhibit the reduced yield strength. Furthermore, Fe-based composite bulk glassy alloys exhibit improved soft magnetic properties in case of nanoscale bcc-Fe phase dispersion state [23] and hard magnetic properties in case of nanoscale bct-FePt phase dispersion state [24].

6. FORMATION OF POROUS BULK GLASSY ALLOYS

As shown in Fig. 4, it was also found that the ductility of a $\text{Pd}_{40}\text{Cu}_{30}\text{Ni}_{10}\text{P}_{20}$ bulk glassy alloy increased even by homogeneous dispersion of spherical pores into glassy matrix [25]. In addition to the increase in ductility, the porous bulk glassy alloys exhibited the decreases in specific weight, Young's modulus and strength as well as the increase in absorption energy required up to fracture [26]. The porous bulk glassy alloys containing spherical pores with sizes from 15 to 33 μm in a wide volume fraction range from 2 to 70% can be formed by use of significant difference in solubility limit of hydrogen between supercooled liquid and glassy solid. The 0.2% proof stress and Young's modulus as a function of porosity can be estimated by taking the stress concentration effect around pores into consideration. The simultaneous achievement of high

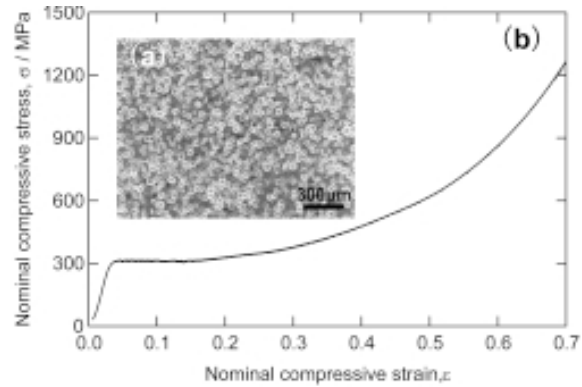


Fig. 4. Scanning-electron micrographs of a transverse cross section (a) and compressive stress-strain curves (b) of $\text{Pd}_{42.5}\text{Cu}_{30}\text{Ni}_{7.5}\text{P}_{20}$ glassy alloy rod with a porosity of about 60%.

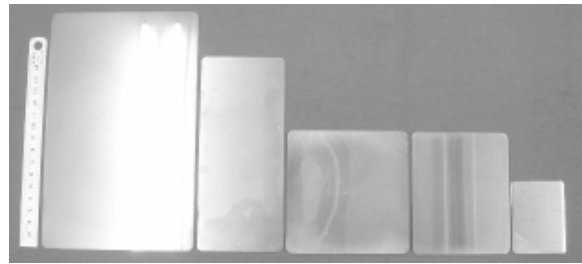


Fig. 5. Outer shape of the glassy alloy sheets with dimensions of 0.3-2x125x200 mm.

ductility and high fracture absorption energy as well as light weight and low stiffness is important for future extension of application fields for bulk glassy alloys. Porous bulk glassy alloys have been also produced by the precompaction and sintering method [27], by the sequent processes of homogeneous mixing of bulk glassy alloy powder and sodium chloride, followed by dissolution of sodium chloride with water [28], and by the formation of immiscible type glassy alloy, followed by dissolution of less-noble glassy phase field with some acids [29].

7. PRODUCTION OF GLASSY ALLOYS IN VARIOUS FORMS OF ROD, PLATE AND BALL

Recently, various casting techniques of molten alloy to produce bulk glassy alloys with desired outer shapes have been developed to full fill their engineering needs [18]. As a result, we can utilize glassy alloy rods with diameters of 3 to 5 mm and a length

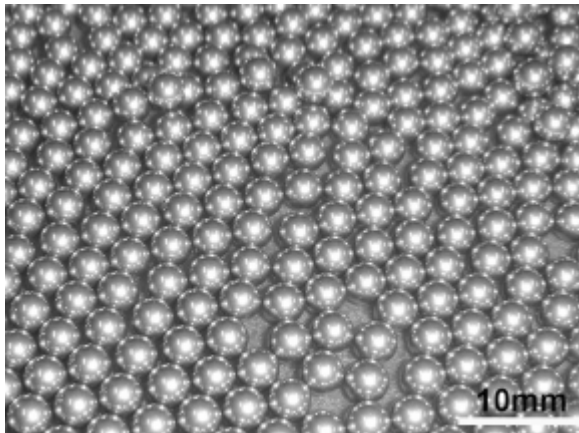


Fig. 6. Outer shape of the glassy alloy balls with a diameter of 3 mm.

of about 1 m, glassy alloy sheets with dimensions of 0.3-2x125x200 mm (Fig. 5) and spherical glassy alloy balls with diameters of 3-10 mm (Fig. 6). The combination effect of unique properties, these useful forms and simple net-shape production process is expected to open up much wider application fields.

8. UNIQUE WORKING PROCESSES

Glass-type alloys always exhibit the glass transition, followed by a supercooled liquid region and then crystallization reaction in case of continuous heating. The Newtonian flow can be recognized in the supercooled liquid region and the strain rate sensitivity exponent (m value) during the Newtonian flow deformation is 1.0, indicating the achievement of ideal super-plasticity [1,3]. As a result, we can achieve extremely large pull deformation with elongation reaching several million percentages. By use of the Newtonian flow, the surface pattern with a minimum size of about 22 nm can be produced by the pressing treatment in the supercooled liquid, indicating that the bulk glassy alloy possesses good nanoscale imprint ability [30] which cannot be achieved for conventional crystalline alloys. Owing to these advantages, the bulk glassy alloys have attracted much interest as a new material for nanotechnology processing.

In addition, when a bulk glassy alloy is formed by casting, the specific volume of supercooled liquid decreases continuously with decreasing tem-

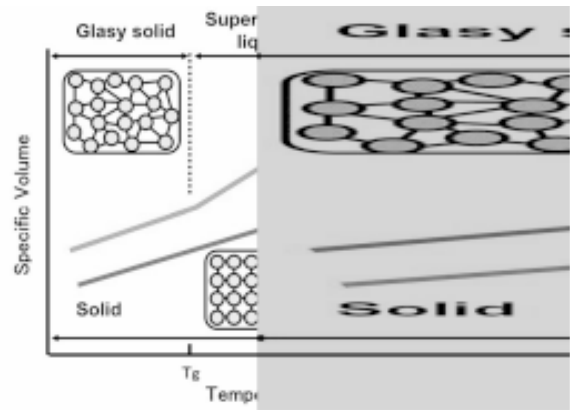


Fig. 7. Schematic illustration of the temperature dependence of specific volume of supercooled liquid for glassy alloys during continuous cooling, together with the data of crystalline alloys.

perature and the deviation of its temperature dependence occurs only at the glass transition temperature, as illustrated in Fig. 7. This is in good contrast to the significant discontinuous shrinkage in volume at melting temperature for all crystalline alloys. The continuous decrease in specific volume for glassy type alloys has enabled the production of nanoscale imprinted (mirror-image) pattern which corresponds very accurately to the inner surface pattern of cavity in copper mold by applying an appropriate level of stress to supercooled liquid [5,6], indicating good casting-induced imprintability on a nanometer scale. This implies that net-shape glassy alloy materials can be produced directly from melt. These unique casting and working characteristics have lead to the significant extension of application fields for bulk glassy alloys.

9. APPLICATIONS

As application fields of bulk glassy alloys, one can list the following fields, e.g., (1) striking face plate in golf clubs, (2) frame in tennis rackets, (3) various shapes of optical mirrors, (4) casing in cellular phones, (5) casing in electro-magnetic instruments, (6) connecting part for optical fibers, (7) shot penning balls, (8) electro-magnetic shielding plates, (9) soft magnetic choke coils, (10) soft magnetic high-frequency power coils, (11) high torque geared motor parts (Fig. 8), (12) high corrosion resistant

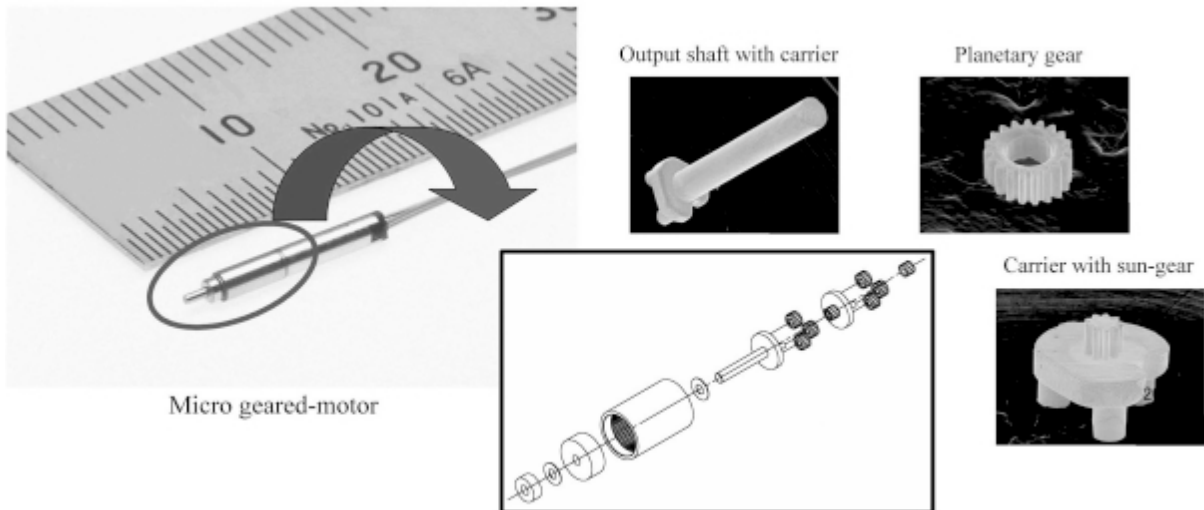


Fig. 8. Micro-geared motor with the world smallest size of 1.5 mm in diameter constructed by Ni-based glassy alloy geared parts and illustration of its construction diagram.

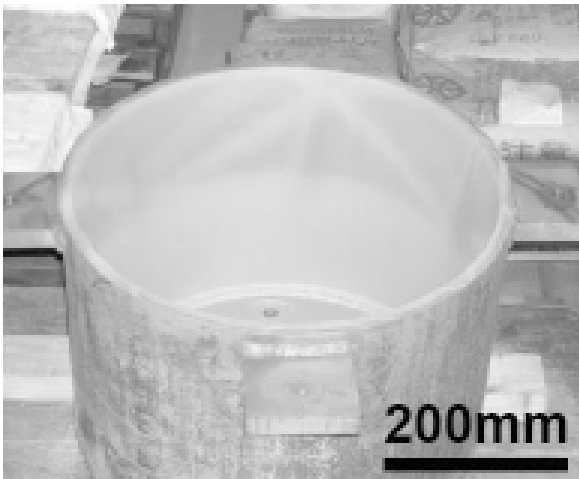


Fig. 9. Outer shape of a vessel for lead-free soldering.

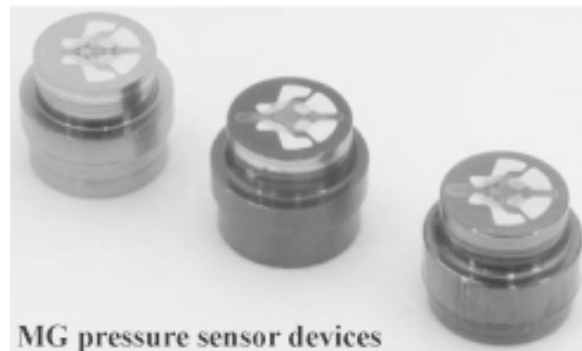


Fig. 10. Outer shape of the metallic glass pressure sensors.

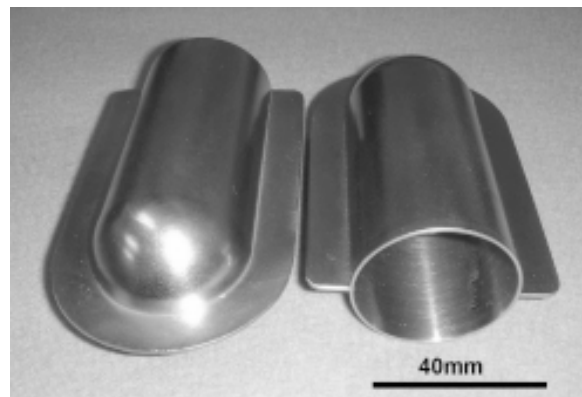


Fig. 11. Outer shape of a slat truck cover for air plane.

coating plates, (13) vessels for lead-free soldering (Fig. 9) etc. Furthermore, as article examples which lie at the final stage for applications, one can notice (1) higher sensitivity type, higher load type and smaller size type pressure sensors in various vehicles including automobile (Fig. 10), (2) Colliori type liquid flow meter, (3) spring, (4) slat truck cover for air plane (Fig. 11), (5) in-printing plate, (6) high density of information-storage material, (7) yoke material for linear actuator, (8) high-frequency type antenna material, (9) magnetic iron core for high

rotation speed motor, (10) biomedical instruments such as endoscope parts, and (11) tool etc.

10. FUTURE PROSPECTS

Although no bulk glassy alloy was found in any alloy systems of Fe, Co, Ni, and Cu bases before 1994, we can produce, at present, bulk glassy alloys with diameters of over 10 mm in all these alloy systems. Considering the significantly rapid progress of bulk glass-forming ability for the last one decade, it is expected that the subsequent study leads to the production of bulk glassy alloys with diameters of 30 to 50 mm. When we consider the future prospects of bulk glassy alloys, the key point is attributed to the possibility that much larger scale bulk glassy alloys with diameters of over 30 mm are formed in engineering important alloy systems such as Fe-, Co-, Ni-, and Cu-based alloys. Thus, we are approaching a new metallic age that bulk glassy alloys can be used widely as base materials in social life.

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