# LIQUID-QUENCHED METAL-METAL GLASSES

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## ABSTRACT

A wide variety of amorphous alloys (metallic glasses) containing only metals has been produced during the last few years. These metastable materials exhibit interesting thermal, electrical and magnetic properties and can be prepared in a very wide composition range. The composition ranges for the formation, thermal properties and crystallization behavior of the metal-metal glasses are presented in this review.

## 1. INTRODUCTION

The advent of splat quenching [1] in 1960 by Professor Pol Duwez of California Institute of Technology, Pasadena, USA, ushered in a new area of investigation in physical metallurgy. This technique, capable of producing cooling rates in excess of a million degrees per second in alloys cooled from the liquid state, has been found to produce several metastable effects. These include extension of solid solubility limits, formation of metastable crystalline intermediate phases and even amorphous phases at high enough cooling rates. The last category has attracted increasing interest in recent times since it was noticed that they have superior chemical, electrical, magnetic and mechanical properties in comparison to their crystalline counterparts. Several reviews are now available on this exotic category

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of metallic materials (see for example refs. 2-6). Although amorphous alloys can be produced by other techniques such as vapor deposition, sputtering, electrodeposition, etc., liquid quenching has by far been the most popular one and so the present paper will concentrate on results obtained only by this technique.

Broadly speaking, three categories of amorphous solids produced by liquid quenching are recognized:

(i) Pure metals      e.g., Ge, Ni, Fe

(ii) Metal -Metalloid type    e.g., $Pd_{80}Si_{20}$, $Fe_{80}P_{13}C_7$, $Fe_{81}B_{13}C_2Si_4$, $W_{35}Mo_{20}Cr_{15}$- $Fe_5Ni_5P_6B_6C_5Si_3$

(iii) Metal-Metal type     e.g., Ni-Nb, Cu-Zr, Mg-Zn, Ca-Mg

Amongst these, pure metals have very limited technological potential, while the metal-metalloid type (containing approximately 20 at.% of the metalloid, irrespective of the number of components involved have very useful properties to warrant commercial exploitation. The properties of these amorphous alloys can also be suitably modified by alloy additions and a large proportion of research in the field of amorphous alloys is concerned with this category. The third category of metal-metal type is relatively less explored and the present review aims at presenting some of the salient features of this category of amorphous alloys.

Although discovered [7] simultaneously with Fe-base metal-metalloid type (FePC) glasses in 1967 [8], some doubt existed about their real 'amorphousness'. In fact, initially, these were designated as 'microcrystalline'. Later x-ray diffraction studies indicated that these are also amorphous in the same sense as the metal-metalloid type glasses. Consequently, intensive research on different aspects of metal-metal glasses began only recently.

## 2. FORMATION

Table I lists the various metal-metal amorphous alloys produced till now. It may be noticed that the number of alloy systems is quite large and that the composition range in which the amorphous phase forms does not show any preferred compositions similar to the conventional metal-metalloid glasses. In addition to the above, amorphous phases were also detected in localised areas in some Al-base alloys (Al-Cr[36], Al-Cu[37], Al-Ni[38], and Al-Pd[39]), Au-Pb[40], Au-Sn[41] and Pb-Au[40] systems. Generally speaking, the following criteria have been recognized to favor easy glass formation: (a) presence of deep eutectics, (b) widely differing sizes of component atoms and (c) a high value of the reduced glass transition temperature, $T_{gr}(=T_g/T_m$, where $T_g$ is the glass transition temperature and $T_m$ is the melting point of the alloy).

As mentioned previously, the metal-metalloid type amorphous alloys form when the metalloid content is about 20 at.%. Based

Table I  Composition range for formation of amorphous phases in metal-metal systems

| System | Solute conc. at.% | Ref. | System | Solute conc. at.% | Ref. |
|---|---|---|---|---|---|
| Ca-Ag | 12.5-42.5 | 9 | Mg-Ga | 19 | 24 |
| Ca-Al | 12.5-47.5 | 9,10 | Mg-Zn | 25-32 | 25 |
| Ca-Cu | 12.5-62.5 | 9 | | | |
| Ca-Ga | 16 | 10 | Nb-Ni | 40-66 | 7,26 |
| Ca-Mg | 22.5-42.5 | 9,10 | Nb-Rh | 42-45 | 27 |
| Ca-Zn | 17.5-62.5 | 9,10 | | | |
| | | | $Nb_{48}Ni_{39}Al_{13}$ | — | 7 |
| Co-Ti | 21-23 | 11 | | | |
| Co-Zr | 9-16 | 12 | Ni-Zr | 10-11 | 12 |
| Cu-Cd | 33-45 | 13 | Sr-Al | 18,30 | 10 |
| Cu-Hf | 40 | 14 | Sr-Ga | 30 | 10 |
| Cu-Mg | ? | 13 | Sr-Mg | 30 | 10 |
| Cu-Ti | 30-35 | 15 | Sr-Zn | 25 | 10 |
| Cu-Zr | 25-60 | 15 | | | |
| | | | Ta-Ni | 40-70 | 7,26 |
| $Cu_{40}Hf_{30}Nb_{30}$ | — | 14 | | | |
| $Cu_{40}Ti_{30}Nb_{30}$ | — | 14 | Ti-Be | 37-41 | 28 |
| $Cu_{40}Zr_{30}Nb_{30}$ | — | 14 | Ti-Ni | 30-40 | 29 |
| Er-Fe | 32 | 16 | $Ti_{50}Be_{40}Zr_{10}$ | — | 30 |
| Fe-Th | 28-80 | 17 | U-Co | 20-40 | 31 |
| Fe-Zr | 9-11 | 12 | U-Cr | 27 | 31 |
| | | | U-Fe | 20-40 | 31 |
| Gd-Co | 40-50 | 18 | U-Mn | 20-35 | 31 |
| Gd-Fe | 32-50 | 16 | U-Ni | 20-40 | 31 |
| | | | U-V | 27 | 31 |
| Hf-Co | 22-91 | 19 | | | |
| Hf-Ni | 20-89 | 19 | Zr-Be | 30-50 | 28 |
| | | | Zr-Co | 22-90 | 32 |
| La-Ag | 25 | 20 | Zr-Fe | 33 | 33 |
| La-Al | 18-34 | 21 | Zr-Ni | 22-90 | 32 |
| La-Au | 18-26 | 22 | Zr-Pd | 20-35 | 15 |
| La-Cu | 30 | 22 | Zr-Rh | 18-26 | 34 |
| La-Ga | 16-28 | 23 | | | |
| La-Ge | 17-22 | 21 | $ZrBe_xNby$ | x=20-35 y=2.5-20 | 35 |
| La-Ni | 22 | 22 | | | |

on this observation, it was suggested that the Bernal model of dense random packing of hard spheres (DRPHS) is applicable to amorphous alloys as modified by Polk [42]. It was proposed that the larger size metal atoms form tetrahedra and the smaller size metalloid atoms occupy the interstices. Since all the vacant

space can be filled up by about 19 at.% of the metalloid atoms, it was considered that this model suitably describes the amorphous structure. However, in the case of metal-metal glasses, no such strict compositional requirements are observed. The amorphous phase can be produced in some binary systems from 10 to 90 at.% of solute content. Thus, it is doubtful whether the DRPHS model is applicable to metal-metal glasses.

Waseda et al. [43] performed a detailed analysis of x-ray scattering intensities on amorphous Cu-Zr alloys and came to the conclusion that the DRPHS model is applicable in this system. It must be stressed, however, that only very few detailed x-ray investigations have been carried out on metal-metal glasses; the scantily available data indicates that these amorphous alloys are similar to the metal-metalloid type.

The glass-forming compositions in the metal-metal category encompass eutectic compositions and in some cases intermediate phases with large unit cells and complex crystal structures. Based on these observations and (a) the presence of a sharp first peak in the radial distribution function (indicative of a high degree of short range order), (b) high thermal stability, (c) high hardness and (d) the close correspondence between the densities of amorphous alloys and the corresponding crystalline intermediate compounds, Wang [44] proposed another model involving random stacking of interpenetrating Kasper polyhedra. This model successfully explained the observations made on sputtered W-Fe, Ni-Nb, etc. amorphous alloys produced at equiatomic compositions. Finney [45] raised doubts about its applicability in three dimensions.

Zielinski et al. [33] on the other hand suggested that the glass-forming tendency is related to low values of $\Delta T$ (the difference between ideal liquidus and actual liquidus temperatures) and also to low values of $\Delta H^M/R$, where $\Delta H^M$ is heat of mixing and R is the gas constant. This was shown to explain the formation of amorphous phases in Zr-base alloys quite satisfactorily.

Thus, even though different models are proposed almost all of them seem to suffer from some disadvantage or other and a universally applicable model is yet to emerge.

## 3.  THERMAL PROPERTIES

Table II summarises the thermal properties of some metal-metal glasses. The glass transition temperature is detected only in some alloys. Another observation that can be made is that the crystallization temperature ($T_X$) is high. Giessen et al. [47] found that $T_g$ varies linearly with the solidus temperature and also with the melting point of the CaM$_2$ compounds in Ca-base metal-metal amorphous alloys. Chen and Krause [48] observed that $T_g$ increases with increasing Cu content in Zr-Cu glasses and that Young's modulus also follows a similar trend.

Table II   Thermal Properties of Metal-Metal Amorphous Alloys

| Alloy | $T_g$(K) | $T_{x1}$(K) | $T_{x2}$(K) | $T_{x3}$(K) | Heating Rate K/min | Ref. |
|---|---|---|---|---|---|---|
| $Co_{78}Ti_{22}$ | | 777 | 823 | | 5 | 11 |
| $Cu_{60}Zr_{40}$ | 729 | 753 | 771 | | 10 | 46 |
| $Cu_{50}Zr_{50}$ | 677 | 724 | 731 | | 10 | 46 |
| $Cu_{46}Zr_{54}$ | 663 | 704 | 747 | | 10 | 46 |
| $Hf_{78}Co_{22}$ | | 758 | | | 50 | 19 |
| $Hf_{60}Co_{40}$ | | 823 | | | 50 | 19 |
| $Hf_9Co_{91}$ | | 828 | | | 50 | 19 |
| $Hf_{80}Ni_{20}$ | | 738 | | | 50 | 19 |
| $Hf_{62}Ni_{38}$ | | 808 | | | 50 | 19 |
| $Hf_{36}Ni_{64}$ | | 923 | | | 50 | 19 |
| $Mg_{70}Zn_{30}$ | | 380 | 401 | 501 | 30 | 25 |
| $Ti_{63}Be_{37}$ | | 673 | 773 | | 20 | 28 |
| $U_{60}Fe_{40}$ | | 560 | 584 | | 40 | 31 |
| $U_{73}Co_{27}$ | 527 | 554 | 563 | 578 | 40 | 31 |
| $U_{73}Cr_{27}$ | | 684 | | | 40 | 31 |
| $U_{67}Ni_{33}$ | | 599 | 641 | 677 | 40 | 31 |
| $U_{70}Mn_{30}$ | | | 605 | 677 | 40 | 31 |
| $Zr_{65}Be_{35}$ | 623 | 648 | 663 | 683 | 20 | 28 |
| $Zr_{78}Co_{22}$ | | 643 | | | 50 | 32 |
| $Zr_{64}Co_{36}$ | | 740 | | | 50 | 32 |
| $Zr_{10}Co_{90}$ | | 833 | | | 50 | 32 |
| $Zr_{78}Ni_{22}$ | | 611 | | | 50 | 32 |
| $Zr_{64}Ni_{36}$ | | 730 | | | 50 | 32 |
| $Zr_{32}Ni_{63}$ | | 839 | | | 50 | 32 |

## 4.   CRYSTALLIZATION BEHAVIOR

The transformation behavior from the amorphous to the crystal-line equilibrium phases was investigated only for a few systems in the metal-metal category.  Even in those cases, the extent and details of investigations do not match those of the more con-ventional metal-metalloid type like Pd-Si, Fe-P-C, Fe-B-Si.  This is rather surprising in view of the fact that metal-metal glasses may be obtained in a much wider composition range than in metal-metalloid type.  One reason for this may be that in a large number of cases, under equilibrium conditions, the alloy systems consist of several complex phases, the crystal structures of some of which are not even determined.  This seems to be the case particularly for Mg-Zn and Cu-Zr, two systems quite extensively investigated.

The available data on the crystallization of metal-metal glasses is summarised in the following paragraphs.

Fig. 1 shows a bright-field micrograph and the corresponding diffraction pattern of liquid-quenched Co-22 at.% Ti alloy. The lack of contrast in the micrograph and the presence of diffuse haloes in the diffraction pattern clearly indicate that the as-quenched product is amorphous. It may so happen that sometimes even though the x-ray patterns indicate an amorphous phase, high-magnification electron microscopy may reveal the presence of fine particles/microcrystals. Hence, it may be advisable always to supplement the x-ray work with electron microscopy.
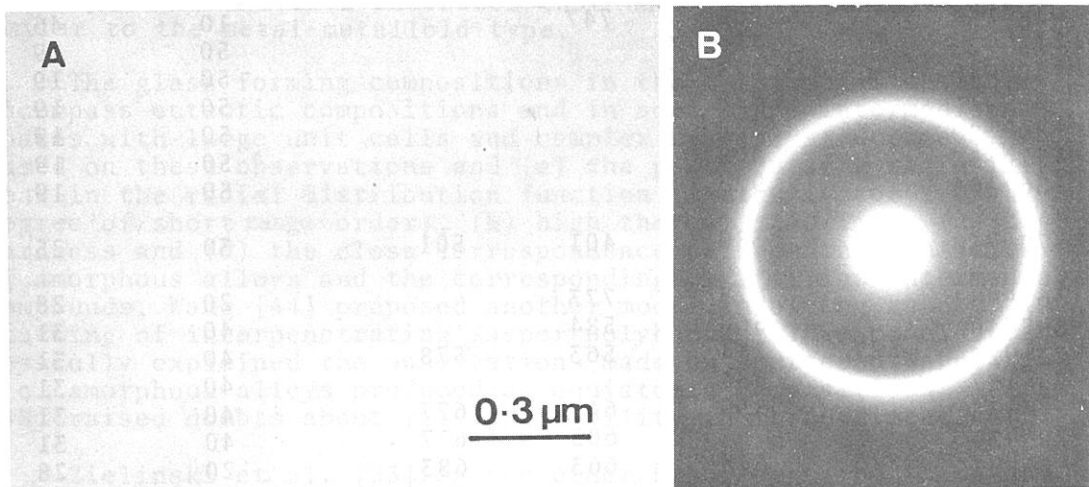


Fig. 1  (a) The as-quenched amorphous structure of a Co-22at.% Ti alloy (b) Electron diffraction pattern.

Structural relaxation at temperatures lower than the crystallization temperature, $T_x$, has been observed in some metal-metal glasses, e.g., Cu-Zr. However, contrary to the metal-metalloid glasses, Cu-Zr amorphous alloys remain completely ductile till the onset of crystallization [49]. Since several theories were proposed to explain temper embrittlement in metal-metalloid amorphous alloys - fast diffusion of smaller size metalloid atoms [50] or fine scale phase separation in the amorphous alloy [51] - it will be interesting to evaluate their applicability to metal-metal systems as well. Since this category of amorphous alloys does not contain any metalloid atoms, the latter theory may perhaps apply. However, the amount of data avilable is rather limited at present and so this problem may have to wait for some more time to be solved.

Vitek et al. [52] showed that $Cu_{10}Zr_7$ and a 'transformed non-crystalline structure' form when Cu-Zr amorphous alloys are annealed below $T_g$. Such a transformation was related to the compositional and structural reorientation. They had also observed

that particles of oblong shape and about 0.1 μm in diameter are formed initially. High resolution electron microscopy investigations suggested that the crystallites had a substructure. Lattice imaging results showed the subgrains, free of dislocation arrays, to have a size of about 30-50 nm. Tilt boundaries were also detected in support of the substructure hypothesis.

All the metal-metal glasses do not show a glass transition temperature. Calorimetric investigations indicate the presence of clear exothermic peaks. The number and height of the peaks varies from system to system. In metal-metalloid systems, the first peak is generally associated with the formation of a solvent-rich phase (MSI), while the second peak is associated with the formation of metal-metalloid complexes (MSII) [53]. Such a generalization appears difficult in the case of metal-metal glasses as the following observations show.

During the decomposition of Zr-Ni and Hf-Ni amorphous alloys, it was noticed that heat treatments corresponding to the first peak resulted in the formation of β-Zr and α-Hf, respectively [32, 19]. In both Zr-Be and Ti-Be systems only α-Zr and α-Ti precipitated [28]. This is similar (although one would have expected the high temperature β modification to form in all cases) to the metal-metalloid glasses where β-Ti precipitated out at $T_{x1}$ in Ti-Si alloys [54]. In no other case, the solvent-rich phase, with the crystal structure of the solvent is noticed in metal-metal glasses. In the Mg-Zn system, compositionally disordered and defective $Mg_7Zn_3$ having a relatively small unresolved grain structure formed [25, 55] while in the Co-Ti alloys the $Co_2Ti$ phase was found to form [11]. It is worth mentioning here that in all the cases reported above, the compositions are near the eutectic composition and in some cases they are slightly hypereutectic.

Heat treatments at temperatures corresponding to the second peak in the calorimetric curves indicated that mostly the equilibrium phases are formed, although in some cases metastable phases are detected. Generally speaking, it is noticed that the alloy compositions of interest correspond to a mixture of several intermetallic phases with complex crystal structures. In some cases, the crystal structures are unknown. Thus, a complete identification of the phases formed during decomposition is tedious and often tentative.

Fig. 2 shows a bright-field micrograph of a Co-22 at.% Ti amorphous alloy crystallized at 823 K for 600 s. One can notice two types of precipitates. The finely dispersed particles belong to the $Co_2Ti$ phase, while the larger, almost spherical, particles are of the $Co_3Ti$ phase. Most often it is noticed that the first crystalline phase to precipitate from the amorphous matrix is in the form of very fine particles and is uniformly distributed, while the second phase particles have a much larger size.

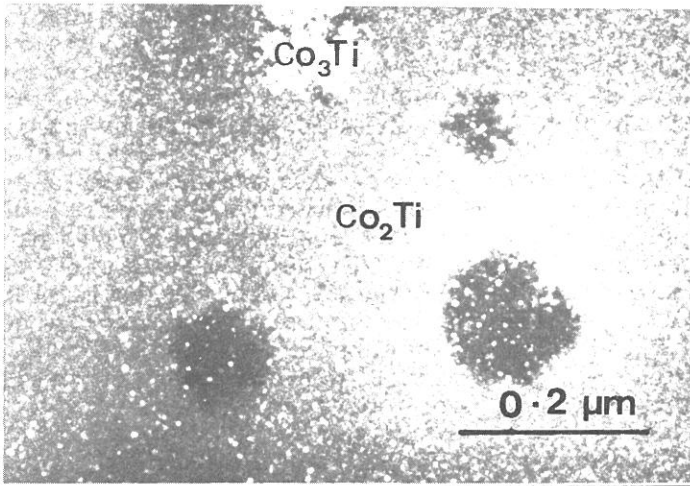Fig. 3 shows a typical electron diffraction pattern from a

Fig. 2   Co-22 at.% Ti alloy annealed for 600 s at 823 K showing the presence of $Co_2Ti$ and $Co_3Ti$ phases.
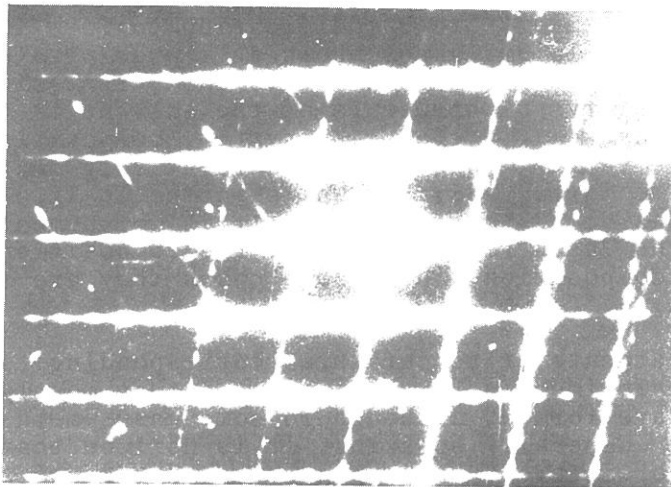


Fig. 3   Electron Diffraction pattern of a $Nb_{40}Ni_{60}$ amorphous alloy annealed for 3600 s at 873 K.

$Nb_{40}Ni_{60}$ amorphous alloy annealed for 3600 s at 873 K.  Even though the diffraction pattern looks complicated, in conjunction with careful x-ray analysis, it has been shown that one of the phases present has a tetragonal structure with the lattice constants a=1.106 and c=2.664 nm [56].  This appears to be a metastable phase preceding the formation of equilibrium $\mu$-NiNb and $Ni_3Nb$ phases.

$Cu_{40}X_{30}Nb_{30}$ (X=Ti, Zr or Hf) were quenched into the amorphous state by melt spinning.  In spite of the fact that alloys in the as-quenched state are not superconducting, it was noticed that annealing at high temperatures induces a superconducting transition in Ti- and Hf-containing alloys but not in the Zr-contain-

ing alloy. Careful electron microscopy was carried out on samples crystallized for different times at various temperatures to investigate the origin of this discrepancy. $Cu_{40}Zr_{30}Nb_{30}$ alloys, after crystallization gave rise to very complex diffraction patterns (consisting of $CuZr_2$ and at least another phase), whereas $Cu_{40}Ti_{30}Nb_{30}$ and $Cu_{40}Hf_{30}Nb_{30}$ alloys yielded apparently simpler patterns. The phase to form in both these alloys appears to be ordered on the basis of a b.c.c. unit cell with a lattice parameter of about 0.32 nm [57] [Fig. 4]. Although, it is tempting to assume that this b.c.c. phase may be the β-Ti or Hf containing niobium in solid solution, the size of the unit cell and the presence of ordering precludes this possibility. The mechanism of ordering and the crystal structure of the ordered phase are being determined at present. The detection of superconductivity in these two alloy systems appears to be related to the presence of this ordered phase.

Relatively little work has been carried out on the kinetics of crystallization in metal-metal glasses (see for example ref. 52).
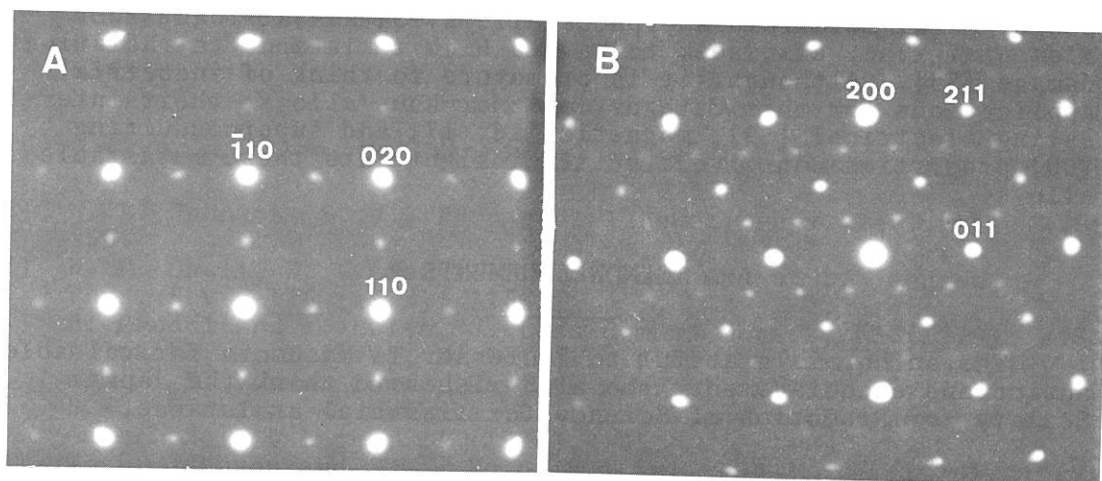


Fig. 4  Electron diffraction patterns showing the presence of ordered structures in (a) $Cu_{40}Ti_{30}Nb_{30}$ and (b) $Cu_{40}Hf_{30}Nb_{30}$ alloys.

## 5.  SUPERCONDUCTIVITY

Although first discovered only in 1975, many metal-metal glasses have been found to exhibit superconductivity either in the as-quenched state or after crystallization. (Since a comprehensive report appears on the superconductivity of liquid-quenched amorphous alloys separately in this proceedings, only the general outlines will be given here).

Amongst the metal-metal glasses, superconductivity is detected

in alloy systems based on La, Zr and Nb and the highest $T_C$ reported so far is only 6.85 K for a $Zr_{65}Nb_{15}Be_{20}$ alloy [35] (and 4.7 K for $Nb_{58}Rh_{42}$). The highest achieved $T_C$ for liquid-quenched amorphous alloys is 9.0 K for $Mo_{80}P_{10}B_{10}$. The presence of metalloid elements thus seems to raise the $T_C$ value. The critical magnetic field, $H_{c2}$, values are reasonably high, but the critical current density is very low. Such a combination of properties seems to preclude immediate applications for these alloys even though these can be readily produced in the form of long thin ribbons of uniform cross section and possessing good bend ductility.

## 6. CONCLUDING REMARKS

Metal-metal glasses do not differ essentially from the more extensively investigated metal-metalloid type glasses. However, it appears that in general they can be produced in the amorphous state in a much wider composition range and seem to have higher crystallization temperatures. Further, characterization of the phases formed during crystallization may be easier since most of these involve only two or in some cases three components, while useful metal-metalloid amorphous alloys contain at least four elements. They also seem to have a high resistance to radiation damage [58]. Although, it is premature to think of industrial applications for this category of amorphous alloys, their interesting electrical [59], magnetic [16, 17] and superconducting [60] properties can give a better insight into these metastable alloys.

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154

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