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The effect of IIIB and IVB elements on the superconducting properties of zirconium-based amorphous alloys

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Zr-based amorphous alloys exhibiting superconductivity have been found in ternary $Zr_{85}Si_{15-x}M_x$ and $Zr_{85}Ge_{15-x}M_x$ (M = B, Al, C, Si, Ge or Sn) systems by a melt-spinning technique. Specimens are in the form of continuous ribbons of 1 mm wide and 0.02 mm thick. The M content in the amorphous alloys is limited to less than 10 at % B, 7 at % Al, 3 at % C, 9 at % Sn for the Zr–Si–M system and less than 7 at % B, 7 at % Al, 3 at % C and 9 at % Sn for the Zr–Ge–M system. The hardness and crystallization temperature increase significantly with the substitution of B, C or Al for Si or Ge, but the effect of the other elements is less significant. The superconducting transition temperature, T_c, rises with the substitution of C, Al, Sn or B for Si, whereas the substitution by Ge causes a slight lowering of T_c. The effectiveness of element M on the rise of T_c decreases in the order C > Al > Sn > B > Si > Ge. The upper critical field gradient at T_c, $(dH_{c2}/dT)_{T_c}$, and the electrical resistivity at 4.2 K, ρ_n, decrease from 2.30×10^6 to 1.97×10^6 A m^{-1} K^{-1} and from 2.70 to 2.00 $\mu\Omega$m, respectively, with the amount of element M. The coefficient of low-temperature electronic specific heat, γ, and the dressed density of electronic states at the Fermi level, $N^*(E_f)$, were calculated from the experimentally measured values of ρ_n and $(dH_{c2}/dT)_{T_c}$ using the strong-coupling theories. From the comparison of T_c with their calculated parameters, it was found that the rise of T_c with the substitution of Si by B, C, Al or Sn is caused by the increase in γ and $N^*(E_f)$. The GL parameter, κ, estimated by using the GLAG theory, decreases from 100 to 78 by the substitution of elements M for Si and it is inferred that the decrease in the dirtiness is also attributable to the rise of T_c.

1. Introduction

Among the amorphous superconductors found to date, the alloy composition of the superconductors exhibiting relatively high superconducting transition temperatures (T_c) is limited to the metal–metalloid type [1, 2]. It has been demonstrated for La [3–5], Nb [6, 7], Mo [8–11] and Ti–Nb [12, 13] based amorphous alloys that the superconducting properties are sensitively affected by both the metal and metalloid components. However, the effect of metalloid elements on T_c is significantly different among their alloy system, e.g., the lowering of T_c by 1 at % addition becomes large in the order of Ge > Si > As > B > P for the Nb-, Mo- and Ti–Nb-base alloys, but that for the La-based alloys is in the order of Si > Ga > Ge. Thus, there is no systematic information about the change in T_c with metalloid composition.

Recently, it has been reported [14, 15] that melt-quenched Zr–(Si or Ge) binary alloys form an amorphous phase in wide composition ranges and exhibit a superconducting transition whose

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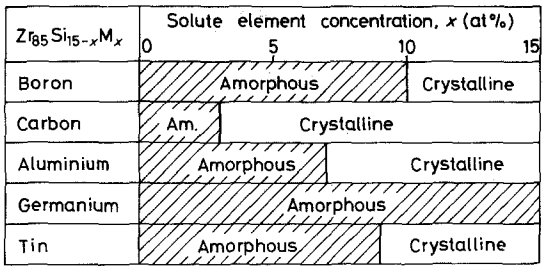

Figure 1 Composition range for the formation of an amorphous phase in the $Zr_{85}Si_{15-x}M_x$ (M = B, C, Al, Ge or Sn) alloys.

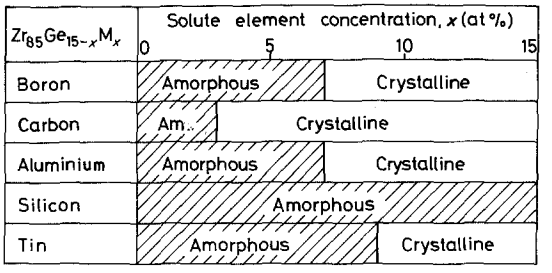

Figure 2 Composition range for the formation of amorphous phase in the $Zr_{85}Ge_{15-x}M_x$ (M = B, C, Al, Si or Sn) alloys.

temperature is in the range 2.05 to 3.20 K. These alloys appear to be favourable alloy systems for studying systematically the effect of metalloid elements on the superconducting properties and for comparing the present results with those [3–13] of the La-, Nb-, Mo- and Ti–Nb-base amorphous alloys. The purpose of this paper is to clarify the effect of group IIIB and IVB elements (M = C, Al, B, Si, Ge or Sn) on the superconducting properties and the related parameters of $Zr_{85}Si_{15-x}M_x$ and $Zr_{85}Ge_{15-x}M_x$ amorphous alloys prepared by the melt-quenching technique.

2. Experimental procedure

The alloy systems selected for the present work are $Zr_{85}Si_{15-x}M_x$ and $Zr_{85}Ge_{15-x}M_x$ (M = C, Al, B, Si, Ge or Sn). The crystalline alloys, from which the specimens used in this experiment were prepared, were made by melting each pure element of Zr, Si, Ge and M under an argon atmosphere in an arc furnace. To achieve homogeneity, the alloys were repeatedly turned over and remelted. The compositions of alloys reported are the nominal ones since the losses of Si and Ge during melting were negligible. Long ribbon specimens were produced by squirting a stream of molten melted with a levitation furnace under argon through a quartz nozzle on a single spinning roller which was rotating at a high speed of about 4000 rpm. The ribbons thus produced were about 1 mm wide by about 20 μm thick. The methods of characterizing the as-quenched phases by X-ray and electron metallographic techniques are described elsewhere [6]. Crystallization temperature (T_x) was evaluated in a differential scanning calorimeter (DSC) at a heating rate of 20 K min^{-1}. Hardness (H_v) was measured by a Vickers microhardness tester with a 100 g load.

All measurements of superconducting properties, T_c, $J_c(H)$ and $H_{c2}(T)$ were done resistively using a conventional four-probe technique. The temperature was measured with an accuracy of ± 0.01 K using a calibrated germanium thermometer. The magnetic field up to 7.16×10^6 A m^{-1} was applied perpendicularly to the specimen surface and feed current.

3. Results

3.1. Formation range of the amorphous phase

The formation of the amorphous phase by the melt-quenching technique was examined in the binary systems of Zr–B, Zr–Al, Zr–C, Zr–Si, Zr–Ge and Zr–Sn and it was demonstrated [14, 15] that the Zr–Si and Zr–Ge alloys showed an amorphous phase with good bend ductility, which is shown by the 180° bending, whereas no amorphous phase was found in the other alloy systems. The formation range of the binary amorphous alloys is in the range 12 to 24 at% Si and 12 to 20 at% Ge and these alloys appear to have the highest amorphous phase-forming tendency in the vicinity of 15 at% Si or Ge. Therefore, the metalloid content of the samples used in the present work was fixed at 15 at%.

The composition ranges in which the totally amorphous phase formed in the $Zr_{85}Si_{15-x}M_x$ (M = B, C, Al, Ge or Sn) and $Zr_{85}Ge_{15-x}M_x$ (M = B, C, Al, Si or Sn) ternary systems are shown in Figs. 1 and 2. It can be seen that the range is limited to less than 10 at% B, 7 at% Al, 3 at% C or 9 at% Sn for $Zr_{85}Si_{15-x}M_x$ alloys and less than 7 at% B, 7 at% Al, 3 at% C or 9 at% Sn for $Zr_{85}Ge_{15-x}M_x$ alloys, whereas in the $Zr_{85}(Si–Ge)_{15}$ alloys an amorphous phase is formed over the whole composition region. Thus, the effect of elements M on the amorphous phase-forming tendency of the Zr-based alloys is greatest for Si followed for Ge, Sn, B, Al and then C, suggesting the tendency that the amorphous phase-forming

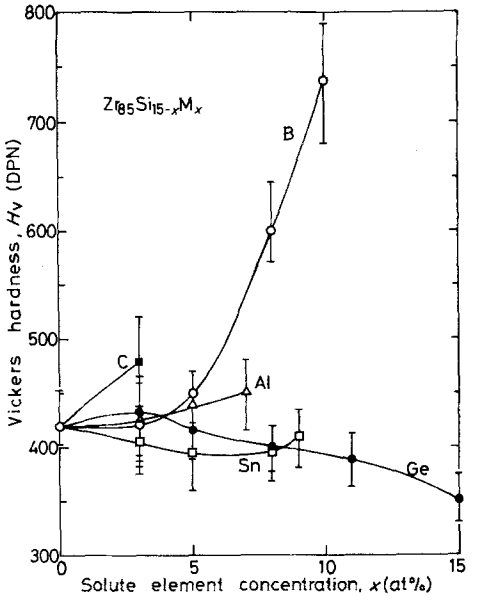

Figure 3 Change in Vickers hardness (H_V) for $Zr_{85}Si_{15-x}M_x$ (M = B, C, Al, Ge or Sn) amorphous alloys with concentration of element M.

ability enhances by the addition of elements M having a large average outer electron concentration and a moderately large atomic size. This is consistent with the previous results that the formation of an amorphous phase is significantly influenced by atomic configuration [16] and chemical bonding nature [17] of constituent elements.

3.2. Hardness and crystallization temperature

Figs. 3 and 4 show the change in Vickers hardness (H_V) value of amorphous $Zr_{85}Si_{15-x}M_x$ and $Zr_{85}Ge_{15-x}M_x$ alloys as a function of M concentration. The change appears to be divided into the following three groups: (1) C, B and Al which enhance the H_V value, (2) Ge which causes a decrease in H_V, and (3) Sn which results in no significant change. On the other hand, the value of H_V of $Zr_{85}Ge_{15-x}M_x$ alloys increases with the substitution of C, B, Al, Sn or Si for Ge. From the compositional dependence of H_V shown in Figs. 3 and 4, it is concluded that the effect of additional elements M on the increase in hardness decreases in the order C > B > Al > Sn > Si > Ge.

Additionally, the effect of the elements M on the crystallization temperature (T_x) of amorphous $Zr_{85}Si_{15-x}M_x$ and $Zr_{85}Ge_{15-x}M_x$ alloys was examined and the T_x values are plotted as a function of M concentration in Figs. 5 and 6. The effect of elements M is very similar in both the

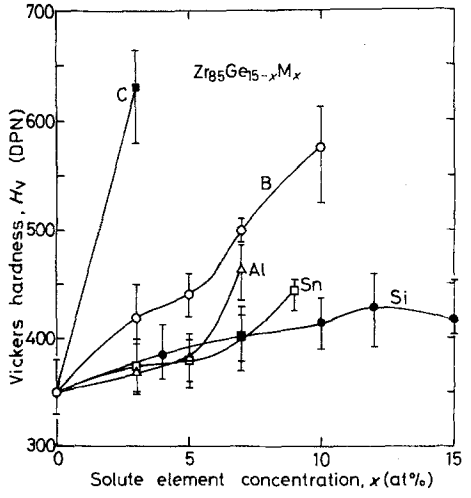

Figure 4 Change in Vickers hardness (H_V) for $Zr_{85}Ge_{15-x}M_x$ (M = B, C, Al, Si or Sn) amorphous alloys with concentration of element M.

alloy systems and decreases in the order C > B > Al > Sn > Si > Ge, in good agreement with the tendency for hardness.

3.3. Superconducting properties

Fig. 7 shows the reduced electrical resistance curves in the vicinity of T_c in the case of no applied magnetic field for the amorphous $Zr_{85}Si_{15-x}M_x$ (M = C, Al, Ge, or Sn) alloys. The transition occurs sharply with a temperature width (ΔT_c) of less than 0.2 K from the relatively high electrical resistivity values ranging from 2.00 to 2.70 $\mu\Omega$m and there is no system change in ΔT_c with the substitution of elements M for Si even

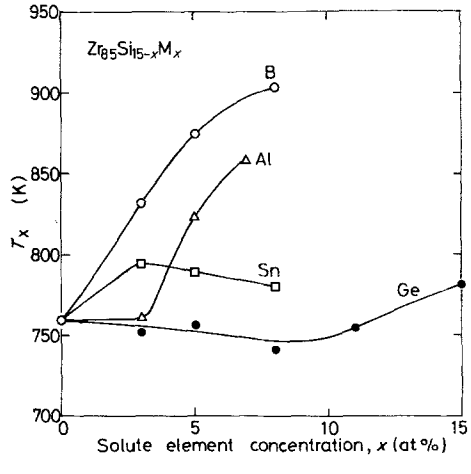

Figure 5 Change in the crystallization temperature (T_x) for $Zr_{85}Si_{15-x}M_x$ (M = B, Al, Ge or Sn) amorphous alloys with concentration of element M.

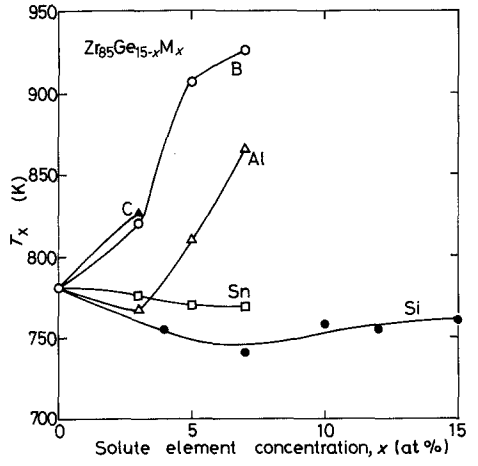

Figure 6 Change in crystallization temperature (T_x) for $Zr_{85}Ge_{15-x}M_x$ (M = B, C, Al, Si or Sn) amorphous alloys with concentration of element M.

though the T_c value rises significantly. Figs. 8 and 9 show the plots of T_c and ΔT_c of the amorphous $Zr_{85}Si_{15-x}M_x$ and $Zr_{85}Ge_{15-x}M_x$ alloys as a function of M content. The T_c value is 2.65 K for $Zr_{85}Si_{15}$, rises with the replacement of Si by C, Al, Sn or B and reaches the highest value of 3.91 K for $Zr_{85}Si_8Al_7$. Conversely, the substitution of Ge for Si results in a slight lowering of T_c. The similar compositional dependence is recognized in $Zr_{85}Ge_{15-x}M_x$ alloys and the T_c value rises with the replacement of Ge by C, Al, Sn or Si and the highest value attained is 3.80 K for $Zr_{85}Ge_8Al_7$. The above results indicate that the effect of elements M on the rise of T_c is similar between the $Zr_{85}Si_{15-x}M_x$ and $Zr_{85}Ge_{15-x}M_x$ alloys and the effectiveness decreases in the order Al > Sn > B > C > Si > Ge.

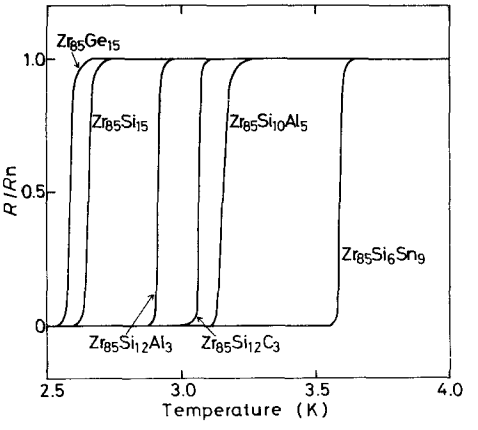

Figure 7 Change in the reduced electrical resistance as a function of temperature for $Zr_{85}Si_{15}$ and $Zr_{85}Si_{15-x}M_x$ (M = C, Al, Ge or Sn) amorphous alloys.

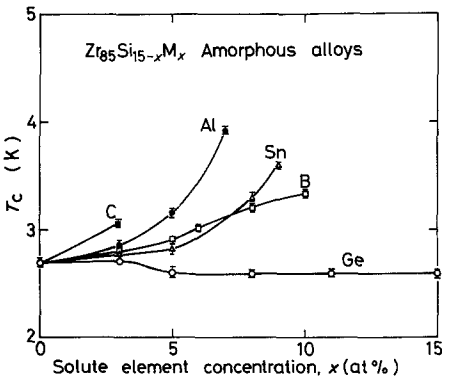

Figure 8 Change in the superconducting transition temperature (T_c) and the transition width (ΔT_c) for $Zr_{85}Si_{15-x}M_x$ (M = B, C, Al, Ge or Sn) amorphous alloys with concentration of element M.

The upper critical magnetic field, H_{c2}, was measured at various temperatures ranging from 1.5 K to T_c. Fig. 10 shows a plot of the H_{c2} value of amorphous $Zr_{85}Si_{15-x}M_x$ (M = B, Al, C, Ge or Sn) alloys as a function of temperature. Here, H_{c2} was defined as being the applied magnetic field at which the resistance of the samples begins to deviate from its normal value. The solid lines in the figure represent a linear extrapolation at T_c. H_{c2} increases linearly with decreasing temperature over almost the whole temperature range and the gradient at T_c, $(dH_{c2}/dT)_{T_c}$, is 2.06×10^6 A m^{-1} K^{-1} for $Zr_{85}Si_5B_{10}$, 2.11×10^6 A m^{-1} K^{-1} for $Zr_{85}Si_{10}Al_5$, 1.97×10^6 A m^{-1} K^{-1} for $Zr_{85}Si_{12}C_3$, 2.29×10^6 A m^{-1} K^{-1} for $Zr_{85}Ge_{15}$ and 1.97×10^6 A m^{-1} K^{-1} for $Zr_{85}Si_6Sn_9$, indicating the tendency that the substitution of elements M for Si results in a decrease in the gradient.

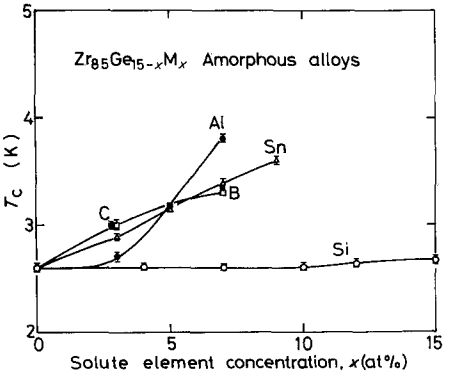

Figure 9 Change in the superconducting transition temperature (T_c) and the transition width (ΔT_c) for $Zr_{85}Ge_{15-x}M_x$ (M = B, C, Al, Si or Sn) amorphous alloys with concentration of element M.

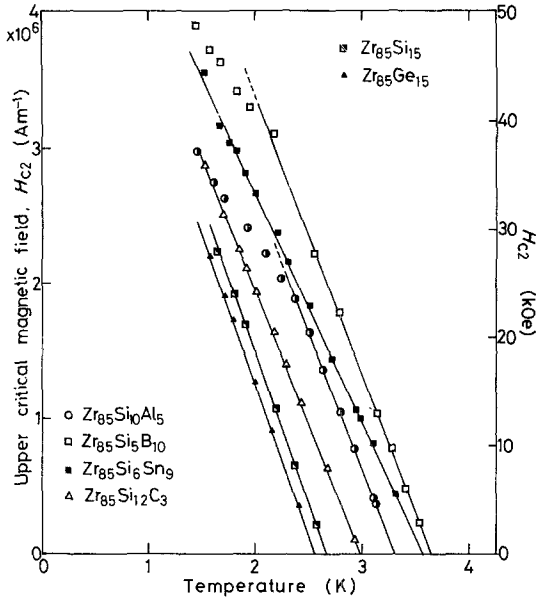

Figure 10 Change in the upper critical magnetic field H_{c2} as a function of temperature for $Zr_{85}Si_{15}$ and $Zr_{85}Si_{15-x}M_x$ (M = B, C, Al, Ge or Sn) amorphous alloys.

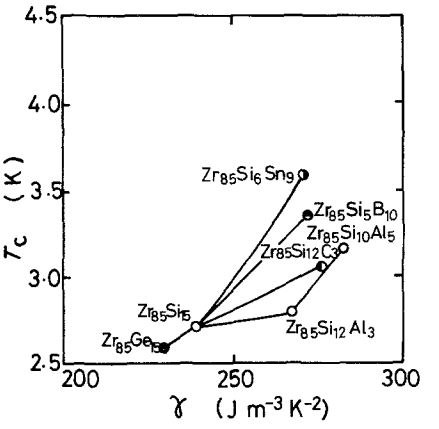

Figure 11 Relationship between T_c and γ for $Zr_{85}Si_{15}$ and $Zr_{85}Si_{15-x}M_x$ (M = B, C, Al, Ge or Sn) amorphous alloys.

4. Discussion

As shown in Figs. 8 and 9, the T_c of amorphous $Zr_{85}Si_{15}$ alloy rises significantly with the substitution of B, Al, C or Sn for Si, whereas the substitution of Ge for Si results in a slight lowering of T_c. In this section, we shall investigate the origin of such a compositional dependence of T_c by evaluating some fundamental superconducting parameters. From the measured values of the upper critical field gradient at T_c, $(dH_{c2}/dT)_{T_c}$, and residual electrical resistivity, ρ_n, at 4.2 K, we can estimate some fundamental parameters dominanting superconductivity such as the electronic dressed density of states $N^*(E_f)$, at the Fermi level, the coefficient of low-temperature electronic specific heat, γ, the Ginzburg–Landau (GL) coherence length $\xi_{GL}(0)$, the extrinsic GL parameter κ, the penetration depth, λ_0, and the electronic diffusivity, D, by using the Ginzburg–Landau–Abrikosov–Gorkov (GLAG) theory for "dirty" superconductors (e.g. [18]). The details of the expressions derived from the GLAG theory have been already presented elsewhere [19]. Values of γ and $N^*(E_f)$ thus obtained are summarized in Table I, together with the values of T_c, ΔT_c, ρ_n and dH_{c2}/dT at T_c. The γ and $N^*(E_f)$ values for $Zr_{85}Si_{15}$ amorphous alloys are 239 J m^{-3} K^{-2} and 1.91×10^{47} states m^{-3} J^{-1} spin^{-1}, respectively, and tend to increase with the substitution of B, Al, C, or Sn for Si. On the other hand, the substitution of Ge for Si results in the decreases of γ and $N^*(E_f)$. It is well known [20] that the T_c value for a strong coupling superconductor is dominated by the Debye temperature, θ_D, the electron–phonon coupling constant, λ, and/or the bare density of states at the Fermi level, $N(E_f)$, and there is a close relation whereby the larger the values of θ_D, λ and $N(E_f)$ the higher is T_c. Considering the fact [18] that there exist the relations of $N^*(E_f) = N(E_f)(1 + \lambda)$ and $\gamma = \frac{2}{3}\pi^2 k_B^2 N(E_f)(1 + \lambda)$, it is expected that T_c is closely related to γ and $N^*(E_f)$. Here k_B is Boltzmann's constant. Fig. 11 shows the relation between T_c and γ for the $Zr_{85}Si_{15-x}M_x$ (M = B, Al ,C, Ge or Sn) alloys. Although a completely linear relation is not well satisfied in both the parameters, it can be seen that the values of γ reflect on T_c: that is, the larger the values of γ and/or $N^*(E_f)$, the higher is T_c. It is therefore concluded that the rise of T_c with the substitution of B, Al, C or Sn for Si is due to the increase in γ and/or $N^*(E_f)$. On the other hand, the lowest T_c value of the $Zr_{85}Ge_{15}$ alloy is attributed to the low values of γ and $N^*(E_f)$. Although the reason for the increase in γ and $N^*(E_f)$ with the substitution of B, Al, C or Sn for Si is not clear at present, it may be due to the result that the interaction between metal and metalloid atoms is weaker for Zr–(B, Al, C or Sn) than for Zr–(Si or Ge) evidence for which is obtained from the facts that an amorphous phase is obtained only in Zr–Si [14] and Zr–Ge [15] alloys and the value of $(T_p - T_e)/X_e$ corresponding to the slope of the liquidus line is larger for the latter alloys [21]. Here T_p is the melting point of

TABLE I Superconducting and the related properties of $Zr_{85}Si_{15}$ and $Zr_{85}Si_{15-x}M_x$ (M = B, C, Al, Ge or Sn) amorphous alloys

Alloy (at%)	T_c (K)	ΔT_c (K)	ρ_n, 4.2 K ($\mu\Omega$m)	$-(dH_{c2}/dT)_{T_c}$ (A m^{-1} K^{-1})	γ (J m^{-3} K^{-2})	$N^*(E_f)$ $\times 10^{47}$ (states m^{-3} J^{-1} spin^{-1})	κ	$\xi_{GL}(0)$ (nm)	λ_0 (nm)	D (mm^2 sec^{-1})
$Zr_{85}Si_{15}$	2.71	0.05	2.70	2.30×10^6	239	1.91	100	7.6	1050	38.0
$Zr_{85}Si_5B_{10}$	3.33	0.07	2.10	2.04×10^6	272	2.17	82	7.3	834	42.9
$Zr_{85}Si_{12}Al_3$	2.81	0.10	2.30	2.19×10^6	267	2.13	89	7.6	950	39.9
$Zr_{85}Si_{10}Al_5$	3.16	0.08	2.10	2.11×10^6	282	2.25	84	7.3	857	41.5
$Zr_{85}Si_{12}C_3$	3.06	0.10	2.00	1.97×10^6	276	2.20	78	7.7	850	44.4
$Zr_{85}Ge_{15}$	2.59	0.05	2.80	2.29×10^6	229	1.82	101	7.8	1090	38.2
$Zr_{85}Si_6Sn_9$	3.59	0.08	2.05	1.97×10^6	271	2.16	80	7.1	793	44.2

Zr, T_e is the eutectic temperature and X_e is the eutectic composition represented in at%. The weak interaction is considered to result in an increase in the number of atoms contributing to the superconductivity through the decrease in the number of atoms which contribute to the covalent-like bonding between metal and metalloid atoms. It is well known that the larger the interaction among the constituent elements in alloys and the steeper the slope of the liquidus line the easier is the amorphous-phase formation of the alloys.

Furthermore, one can notice the tendency that T_c rises with decreasing residual electrical resistivity. Such a relationship between T_c and ρ_n has also been recognized in the amorphous alloys of Zr–Si [14], Zr–Ge [15] and Zr–Nb–Si [19], etc. These results indicate that as the degree of disorder in atomic configuration increases, T_c is decreased. Therefore, it is considered that the substitution of B, Al, C or Sn for Si causes a decrease in the degree of disorder of the amorphous structure, and hence results in a rise of T_c.

As shown in Table I, by the substitution of B, Al, C or Sn for Si, the κ and λ_0 decrease from 100 to 78 and 1050 to 735 nm, respectively, and D increases from 38.0 to 44.4 $m^2\,sec^{-1}$. No systematic change of the $\xi_{GL}(0)$ by changing the compositions is seen and the value is about 7.4 nm. The decreases in κ and λ_0 and the increase in D appear to originate from the increase of the electron mean-free path, l, due to the decrease in the degree of disordered state near atomic scale, evidence for which is given by the significant decrease in the electrical resistivity from 2.70 to 2.00 $\mu\Omega$m. The reason for the decrease in electrical resistivity with increasing Al or Sn content for $Zr_{85}Si_{15-x}Al_x$ and $Zr_{85}Si_{15-x}Sn_x$ alloys appears to be caused by the decrease of the metalloid (Si) content because Al and Sn belong to metal elements. It has been demonstrated for Zr–Si [14] and Zr–Ge [15] amorphous alloys that the decrease in Si or Ge content results in a significant decrease in electrical resistivity. However, the reason for the decrease in ρ_n with the substitution of B or C for Si is considered to be different from that in the case of the substitution of Al or Sn for Si. The dissolution of B or C appears to result in a lowering of the degree of disorder by occupying the holes in the amorphous polyhedron which was constructed with Zr and Si having atomic sizes much larger than B or C. A more detailed investigation of the structure of $Zr_{85}Si_{15-x}M_x$ amorphous alloys will shed some light upon such a compositional dependence of T_c.

Although the values of κ, $\xi_{GL}(0)$ and λ_0 decrease and D increases with the substitution of B, Al, C or Sn for Si, the absolute values of κ, $\xi_{GL}(0)$ and λ_0 are extremely large and D is extremely small. These results indicate that the present $Zr_{85}Si_{15-x}M_x$ amorphous superconductors are typical type-II materials characterized as extremely high degree of dirtiness. As described above, the decrease in the degree of dirtiness is probably because atomic configurations become less random on the scale much smaller than $\xi_{GL}(0)$ by the substitution of B, C, Al or Sn for Si.

5. Conclusions

The compositional dependences of the formation range, H_v, T_x and superconducting properties of $Zr_{85}Si_{15-x}M_x$ and $Zr_{85}Ge_{15-x}M_x$ (M = B, Al, C, Si, Ge or Sn) amorphous alloys prepared by the melt-quenching technique were examined as a function of M content. The results obtained are summarized below.

(1) The effect of elements M on the formation tendency of the amorphous phase is very similar between $Zr_{85}Si_{15-x}M_x$ and $Zr_{85}Ge_{15-x}M_x$ alloys and becomes smaller in the order Si $\simeq$ Ge > B $\simeq$ Al $\simeq$ Sn > C.

(2) The substitution of B, C or Al for Si or Ge results in significant increases in H_v and T_x, whereas the effect of the other elements on the increases in H_v and T_x is less significant.

(3) T_c rises with the substitution of B, Al, C or Sn for Si, whereas the substitution of Ge causes a slight lowering of T_c. Thus, the effectiveness of elements M on the rise of T_c decreases in the order of C > Al > Sn > B > Si > Ge.

(4) The dH_{c2}/dT at T_c and ρ_n decrease from 2.30×10^6 to 1.97×10^6 $A\,m^{-1}\,K^{-1}$ and 2.70 to 2.00 $\mu\Omega$m, respectively, with the substitution of B, Al, C or Sn for Si. The values of γ, $N^*(E_f)$, κ, $\xi_{GL}(0)$ and λ_0 were estimated from the data of $(dH_{c2}/dT)_{T_c}$ and ρ_n by using the extend GLAG theory, and it is concluded that the rise of T_c with the substitution of B, C, Al or Sn is attributable to the increases in γ and $N^*(E_f)$ and is also clearly related to the decrease in the degree of dirtiness caused by the decrease in electrical resistivity.

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