

Invited Paper

Icosahedral Clusters and Composition Rules for Zr-based Bulk Metallic Glasses

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Abstract

In this paper, we will describe the formation rules of Zr-based bulk metallic glasses, originally developed for quasicrystals, by combining cluster structures with phase diagram features. We will introduce the e/a -constant and e/a -variant criteria for ternary systems, and e/a -constant and atomic size constant criteria for quaternary systems. We will show how the glass forming composition optimization is realized by applying these rules in the Zr-Al-Ni and Zr-Al-Ni-Cu systems. In both systems the optimized glass-forming composition is related to a common binary icosahedral cluster Zr_9Ni_4 derived from the fcc Zr_2Ni phase.

Introduction

Quasicrystals (QCs) and amorphous alloys have close structural connections, particularly in some Zr-based systems with extremely high glass-forming abilities where the primary devitrification phases are often quasicrystalline [1-5]. Such an observation reflects the fact that icosahedral short-range orders are widely present in amorphous alloys [6-8]. However, the abundant discussions on the relationship between QCs and amorphous alloys have not been directed towards their probably similar composition rules. A clue towards this objective might be to use the icosahedral clusters.

We have developed two composition criteria for QCs. The first was the e/a -constant criterion, i.e. QCs and their approximants share similar electron concentrations [9-10], and the second is the e/a -variant criterion, i.e. the ternary QC is located along the line linking a binary QC and the third element [11]. Later we recognized that the binary QC composition coincided with the basic icosahedral cluster of that binary QC [12] so that this line actually reflects the cluster growth pathway. The very same idea was then extended to a few Zr-based bulk metallic glass (BMG) forming systems [13-15]. The revelation of these simple rules, common to both QCs and BMGs in many respects, have led to the composition optimization of a few known BMG systems as well as the discovery of a series of new BMG-forming systems and compositions [14, 16-20]. The present paper will summarize these composition rules.

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Composition rules of ternary QCs

Stable quasicrystals have been found in many ternary alloy systems. For example, in Al-Cu-Fe, there exists a stable ternary icosahedral quasicrystal with composition close to $Al_{62.3}Cu_{24.9}Fe_{12.8}$ [21]. Figure 1 is the phase diagram at room temperature as reported in reference [21]. In this phase diagram there are two important lines serving the composition criteria for ternary QC, namely the e/a -constant line and the e/a -variant line.

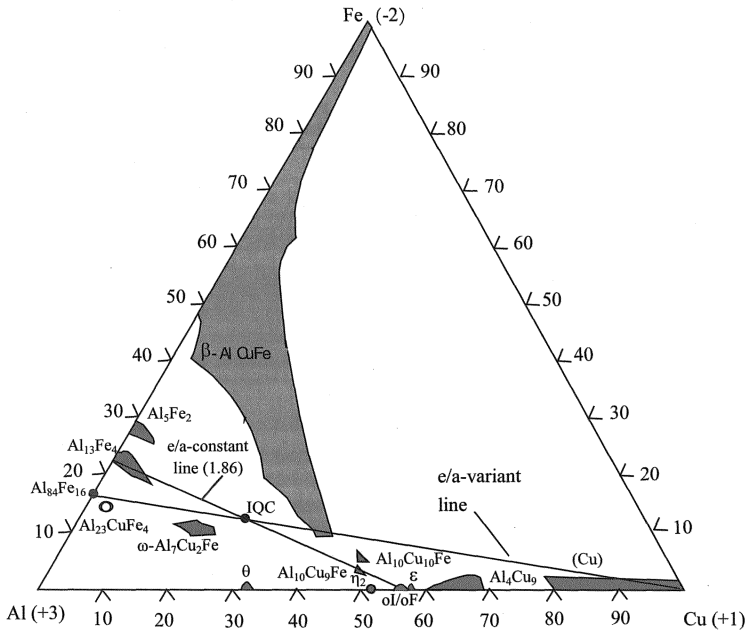


Figure 1. Al-Cu-Fe phase diagram showing the e/a -constant and e/a -variant lines.

Taking the valence contribution from Al, Cu, and Fe respectively as $N_{Al} = 3$, $N_{Cu} = 1$, and $N_{Fe} = -2$, the $Al_{62.5}Cu_{25}Fe_{12.5}$ quasicrystalline phase has $e/a = 1.86$ and the constant $e/a = 1.86$ line follows equation $C_{Fe} = 0.23 - 0.4C_{Cu}$. Notice that the $Al_{62.5}Cu_{25}Fe_{12.5}$ quasicrystals and its approximants λ - $Al_{13}Fe_4$, ϕ - $Al_{10}Cu_{10}Fe$ are all located near this e/a -constant line. [9-10]

The other characteristic line is defined by linking a binary QC DQC- $Al_{84}Fe_{16}$ and the third element Cu, the ternary QC being located on this line [11]. We have further verified that the compositions of binary QCs are determined by their basic icosahedral clusters, and the structural information of these clusters can be derived from nearby crystalline compounds. For example, the approximant $Al_{13}Fe_4$ contains an icosahedron with composition $Al_{10.7}Fe_2$, or $Al_{84.3}Fe_{15.7}$ in atomic percentage, and the binary QC is presumably constructed mainly with this cluster so that the overall composition of this binary QC is close to that of its basic cluster. Therefore this line reflects the growth

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pathway from a binary icosahedral cluster to the structure of a ternary QC by adding a third element. [12] Since the e/a -ratios along this line change in general, in order to distinguish it from the e/a -constant criterion, it is termed the e/a -variant criterion for ternary QCs. Both rules are universal in ternary quasicrystalline phase diagrams. We have applied these rules to determine the exact composition zone of the Al-Ni-Fe [11] and Zr-Ti-Ni quasicrystals [22].

It should be pointed out however that these rules have nothing to do with quasi-periodicity. They are only correlated with icosahedral clusters. Therefore, similar rules may be present in materials whose structures are dominated by icosahedral clusters, examples being Zr-base amorphous alloys.

Composition rules of BMGs using icosahedral clusters

The e/a -constant criterion for BMG forming systems

In the Zr-Al-Ni-Cu alloys system, the $Zr_{65}Al_{7.5}Ni_{10}Cu_{17.5}$ BMG has a high glass forming ability with a large ΔT_x of 127K [23]. In an investigation on the microstructure of an as-cast $Zr_{65}Al_{7.5}Ni_{10}Cu_{17.5}$ ingot, we identified five crystalline phases in coexistence with the glassy phase [24]. What is remarkable about these phases, as we have noted, is that they are all Hume-Rothery phases with nearly constant e/a ratios. This is strikingly similar to the e/a -constant phenomenon in quasicrystalline systems shown previously. Thereof we propose the first criterion for the BMG formation: a BMG and its crystalline counterparts share constant e/a ratios specific in a given alloy system. In a ternary system, the e/a -constant compositions correspond to a straight composition line, or the e/a -constant line. In a quaternary system, it is an e/a -constant plane. We notice that the known amorphous phases in the Zr-Al-Ni-Cu systems, and the crystalline phases in Zr-Al-Ni-Cu system all have e/a ratios close to 1.3-1.5, as shown in table 1.

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Table 1 Electron concentration (e/a) and average atomic size R_{av} of the amorphous, quasicrystalline and crystalline phases in the as-cast $Zr_{65}Al_{7.5}Ni_{10}Cu_{17.5}$ alloy.

Phases	Structures and origins	e/a	R_{av}/nm
$Zr_{65}Al_{7.5}Ni_{10}Cu_{17.5}$	BMG	1.38	0.1496
hP-Zr _{65.4} Al _{11.7} Ni _{11.6} Cu _{11.3}	Hexagonal Al ₃ NiZr ₆ type, observed in as-cast $Zr_{65}Al_{7.5}Ni_{10}Cu_{17.5}$	1.45	0.1502
oP-Zr _{65.4} Al _{11.7} Ni _{11.6} Cu _{11.3}	Orthorhombic, superstructure of Al ₃ NiZr ₆ , observed in as-cast $Zr_{65}Al_{7.5}Ni_{10}Cu_{17.5}$	1.45	0.1502
tI-Zr _{66.7} Al _{11.7} Ni _{8.4} Cu _{22.9}	Tetragonal CuZr ₂ type, observed in as-cast $Zr_{65}Al_{7.5}Ni_{10}Cu_{17.5}$	1.28	0.1487
fcc-Zr _{67.9} Al _{5.1} Ni _{15.2} Cu _{11.8}	fcc Zr ₂ Ni phase (NiTi ₃ type)	1.29	0.1499
QC-Zr _{69.5} Al _{7.5} Ni ₁₁ Cu ₁₂	Icosahedral quasicrystal	1.39	0.1509
$Zr_{60}Al_{20}Ni_{20}$	BMG	1.50	0.1496
$Zr_{55}Al_{20}Ni_{25}$	BMG	1.43	0.1479
$Zr_{60}Al_{15}Ni_{25}$	BMG	1.35	0.1487
$Zr_{65}Al_{7.5}Cu_{27.5}$	BMG	1.48	0.1499
$Zr_{60}Al_{10}Cu_{30}$	BMG	1.50	0.1487
$Zr_{73}Al_{13}Cu_{14}$	BMG	1.63	0.1533

The e/a -constant line exists in BMG-forming ternary systems. Take for instance the Zr-Al-Ni system, which is a typical BMG ternary system [25]. In the isothermal section (1073K) of the Zr-Al-Ni phase diagram, three known phases, Al₅₀Ni₅₀ (CsCl type), ZrAlNi (Fe₂P type), and pure Zr fall exactly along a straight line (figure 2). Among these phases, Al₅₀Ni₅₀ is the ideal phase composition for the β phase with $e/a = 1.5$. The electronic effect for its phase stability has been discussed, for example in reference [26]. For the ZrAlNi phase, the X-ray diffraction gives the strongest {210} reflections. The wave number is $k_{210} = 27.8 \text{ nm}^{-1}$. By using the effective e/a values of the constituent elements [15], the Fermi radius is calculated to be $k_f = 14.0 \text{ nm}^{-1}$. Thus, $2k_f \approx k_{210}$, i.e. the Hume-Rothery matching condition of this phase is fulfilled. It is noticeable that the reported composition $Zr_{60}Al_{20}Ni_{20}$ [25] together with these electron phases fall precisely on the composition line with $e/a=1.5$. The Zr-Al-Co system manifests similar phase diagram characteristics (figure 2).

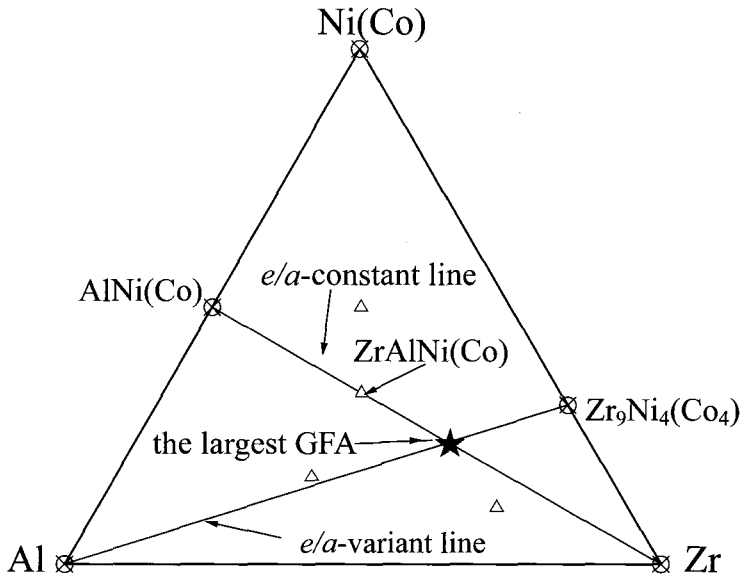


Figure 2. The e/a -constant line ($\text{Al}_{50}\text{Ni}(\text{Co})_{50}$ -Zr) and the e/a -variant line ($\text{Zr}_9\text{Ni}(\text{Co})_4$ -Al) in the Zr-Al-Ni (Co) composition charts. Open triangles stand for ternary phase positions.

The e/a -variant line criterion for ternary BMGs

By analogy to the rules for QCs, the e/a -variant criterion for BMGs is defined by linking the 3rd element to a specific binary cluster composition near a deep eutectic point of a binary subsystem. For instance, Zr-Al-Ni and Zr-Al-Co each contains a typical eutectic subsystem, Zr-Ni and Zr-Co, in which metallic glasses are formed over a broad composition range. For these two subsystems, the Zr_2Ni and Zr_2Co metallic glasses crystallize primarily to the fcc Ti_2Ni -type $\text{Zr}_2\text{Ni}(\text{Co})$ phase [27, 28]. The fcc Ti_2Ni -type phase contains a Ni(Co)-centered 1st-shell icosahedral cluster, $\text{Zr}_9\text{Ni}(\text{Co})_4$, which is close to the phase composition and to the deep eutectic point. Therefore, this specific composition is taken as the starting point to construct the e/a -variant line in the Zr-Al-Ni (Co) phase diagram.

Linking the composition $\text{Zr}_9\text{Ni}(\text{Co})_4$ to the third constituent Al, a series of alloys are constructed with an e/a span from 1.1 to 3. Here the e/a values of 1.5, 3 and 0 are assigned to Zr, Al, and Ni(Co), respectively [13, 15]. BMGs obtained on the two composition lines are listed in Table 2. The e/a span of our BMG samples is from 1.30 to 1.50, and outside this span the alloys are partially crystallized.

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Table 2 Compositions, conduction electron concentration (e/a), glass transition temperature (T_g), onset temperature of crystallization (T_x), undercooled liquid region ΔT_x ($\Delta T_x = T_x - T_g$), onset temperature of melting T_m , offset temperature of melting T_l^* , GFA indicators T_g/T_m , T_g/T_l , $\gamma = T_x/(T_g + T_l)$, $\Delta T_l = T_l - T_x$ and $\Delta T_g = T_l - T_g$ of the BMGs obtained along the e/a -constant and the e/a -variant lines in the Zr-Al-Ni(Co) systems. The temperature unit is K.

compositions	e/a	T_g	T_x	ΔT_x	T_m	T_l	T_g/T_m	T_g/T_l	γ	ΔT_l	ΔT_g
Zr _{58.6} Al _{15.4} Ni ₂₆	1.34	707	789	82	1188	1274	0.595	0.555	0.398	485	567
Zr _{57.5} Al _{16.9} Ni _{25.6}	1.37	721	801	80	1186	1280	0.608	0.563	0.400	479	559
Zr _{55.8} Al _{19.4} Ni _{24.8}	1.42	735	805	70	1184	1277	0.621	0.576	0.400	472	542
Zr ₅₄ Al ₂₂ Ni ₂₄	1.47	751	813	62	1183	1284	0.635	0.585	0.400	471	533
Zr ₅₃ Al _{23.5} Ni _{23.5}	1.50	771	822	51	1180	1288	0.653	0.599	0.401	466	517
Zr _{58.4} Al _{20.8} Ni _{20.8}	1.50	720	776	56	1184	1289	0.608	0.559	0.386	513	569
Zr ₆₀ Al ₂₀ Ni ₂₀	1.50	713	766	53	1183	1294	0.603	0.551	0.382	528	581
Zr ₆₃ Al _{18.5} Ni _{18.5}	1.50	702	747	45	1193	1315	0.588	0.534	0.370	568	613
Zr ₆₀ Al _{13.5} Co _{26.7}	1.30	718	757	39	1234	1254	0.582	0.573	0.384	497	536
Zr _{58.8} Al _{15.4} Co ₂₆	1.34	724	773	49	1232	1282	0.586	0.565	0.385	509	558
Zr _{57.5} Al _{16.9} Co _{25.6}	1.37	744	786	42	1236	1300	0.604	0.572	0.385	514	556
Zr _{55.8} Al _{19.4} Co _{24.8}	1.42	751	820	69	1232	1302	0.612	0.577	0.399	482	551
Zr ₅₄ Al ₂₂ Co ₂₄	1.47	768	843	75	1230	1316	0.624	0.584	0.404	473	548
Zr ₅₃ Al _{23.5} Co _{23.5}	1.5	783	849	66	1230	1320	0.637	0.590	0.404	471	537
Zr _{55.4} Al _{22.5} Co _{22.5}	1.5	767	815	48	1233	1326	0.622	0.578	0.389	511	559
Zr _{57.6} Al _{21.2} Co _{21.2}	1.5	761	794	33	1234	1324	0.617	0.574	0.381	530	563
Zr ₆₀ Al ₃₀ Co ₂₀	1.5	731	780	49	1231		0.594				
Zr _{61.2} Al _{19.4} Co _{19.4}	1.5	720	771	51	1232		0.584				

Note: the offset temperatures (T_l) of several BMGs are almost undetectable in the present DTA measurement due to the sensitivity limit of the instrument.

From the table we see that for the BMGs obtained along the Zr₉Ni₄-Al line, both T_g and T_x increase with increasing e/a , signifying that the thermal stability is enhanced at higher e/a . The largest thermal stability is found in the vicinity of the composition Zr₅₃Al_{23.5}Ni_{23.5} with the maximum e/a value of 1.5. This composition is located exactly at the intersection of the two lines.

Many criteria for GFA have been proposed for practical and reliable reasons based on the characteristic temperatures obtainable easily by thermal analysis. In this work, the GFA indicators, T_g/T_m , $T_x = T_x - T_g$, T_g/T_l , $\gamma = T_x/(T_g + T_l)$, $T_l = T_l - T_x$ and $T_g = T_l - T_g$ are all taken into account. The data of the present BMGs are listed in Table 2. The indicators, except T_x , manifest consistent variation tendencies in quantifying the GFAs of these BMGs. The BMGs on the Zr₉Ni₄-Al line shows a monotonous increase in GFA against e/a , and the largest GFA is in the vicinity of Zr₅₃Al_{23.5}Ni_{23.5} with an upper e/a limit of 1.5. For the e/a -variant BMGs, the T_m values are nearly constant, indicating near eutectic meltings, and T_g is very composition sensitive showing a temperature span about 60 K. As a sequence, the indicator, T_g/T_m , reveals much more distinctly the GFA differences among these compositions. Furthermore, the BMG with the highest T_g/T_m behaves a large melting span ($T_l - T_m$) about 108K, indicating that the exact ternary eutectic composition would not correspond to the largest GFA. The Al addition to the Zr₉Ni₄ cluster composition increases the e/a ratio and the largest thermal stability and GFA were obtained at an upper e/a limit of 1.5. It should be mentioned that the atomic packing effect, in particular, large atomic size ratios among constituents are a necessity for a

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BMG formation. The constituent elements Zr, Al and Ni manifest a large-intermediate-small atomic size sequence fulfilling the atomic size requirement. It is worth mentioning that the Ti_2Ni -type phases and quasicrystals are primary crystallization products of many Zr-based multi-component BMGs. These complex phases share nearly the same electron concentration per unit volume as that of their corresponding glassy phases [15]. The e/a -variant line indicates that Al favors the stabilization of the initial binary $Zr_9Ni(Co)_4$ cluster, which may in turn stabilize the present BMGs after a certain e/a ratio is reached.

The average atomic size rule for quaternary BMGs

We further notice that the Zr-Al-Ni-Cu amorphous and crystalline phases are distributed close to a specific straight line on the e/a -constant plane in the phase diagram. This special composition distribution reflects the influence from factors other than the electron concentration. It is known that atomic size is an important factor in forming amorphous structures. By analogy to the calculation of the average electron number per atom, e/a , we introduce the concept of average atomic size R_{av} as the second criterion to judge the glass forming compositions. R_{av} is defined as the summary of multiplications of atomic fraction C_i and Goldschmidt atomic radius R_i of each element, $R_{av} = \sum C_i * R_i$. We then calculate the R_{av} values for the phases mentioned above. As shown in table 3, the constant R_{av} phenomenon is obvious; all the compositions have R_a close to 0.15 nm. The specific composition line, near which all the phases are located, is the intersection of two planes, one being e/a -constant and the other R_a -constant.

Table 3 Amorphous phase formation in Zr-Al-Ni-Cu system by suction casting at different electron concentration e/a and average atomic size R_{av} .

e/a	R_{av}	Sample state
1.25	1.492	crystalline
1.375	1.486	amorphous
	1.492	amorphous
	1.496	amorphous (inc. the Inoue alloy)
1.4	1.492	amorphous
1.5	1.486	amorphous
	1.492	amorphous
	1.496	amorphous
1.65	1.492	crystalline

By using these two criteria we have carried out the composition optimization for the Zr-Al-Ni-Cu BMGs [14]. Compositions with different combinations of e/a and R_{av} have been prepared by suction casting into 3mm diameter bars. BMGs are obtained with an e/a range of 1.3-1.5, similar to the Zr-Al-Ni subsystem. The R_{av} is in the range of 0.1486-0.1496 nm. The results are summarized in table 3. The optimum BMG forming zone corresponds to $e/a = 1.5$ and $R_{av} = 0.1486-0.1492$ nm (tables 3 and 4). Notice that the e/a value is exactly the same as that of the best BMG $Zr_{53}Al_{123.5}Ni_{23.5}$. This coincidence seems to indicate that the quaternary Zr-Al-Ni-Cu BMG is originated from the ternary Zr-Al-Ni by adding the third element Cu, while maintaining the constant e/a ratio of 1.5. The average atomic radius R_{av} range 0.1486-0.1492 nm falls close to that of the icosahedral

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cluster Zr_9Ni_4 ($R_{av} = 0.1492$ nm), which is the binary starting composition of the e/a -variant line in the Zr-Al-Ni system. These results again confirm our basic idea that this BMG is constructed with an icosahedral short-range order based on the Zr_9Ni_4 binary cluster. Therefore we see that the quaternary BMG is the alloying result of its ternary subsystem BMG Zr-Al-Ni, and the multi-component BMGs are actually all related to binary clusters. The composition rules of BMG, as well as QCs, reflects the composition evolution from simple subsystem up to a multi-component system, while maintaining some basic electronic and atomic size features of the basic clusters.

Table 4 Optimum glass-forming composition zones of Zr-Al-Ni-Cu BMGs. The highest GFA is found at $Zr_{58}Al_{16}Ni_{11}Cu_{15}$ ($e/a=1.5$, $R_{av}=0.1486$ nm) for the largest T_g/T_l . The temperature unit is in K.

e/a	R_{av}/nm	Compositions	T_g	T_m	T_l	T_x	T_g/T_m	T_g/T_l	γ
1.5	0.1486	Zr58.3Al14.6Ni8.3Cu18.8	695	1060	1165	78	0.656	0.597	0.416
		Zr58Al16Ni11Cu15	705	1078	1134	79	0.654	0.622	0.426
		Zr57.5Al17.5Ni13.8Cu11.3	717	1136	1157	63	0.631	0.620	0.416
		Zr57Al19Ni16.5Cu7.5	727	1132	1237	52	0.642	0.588	0.397
		Zr56.6Al20.5Ni19.2Cu3.8	741	1151	1277	44	0.644	0.580	0.389
1.375	0.1496	Zr65Al17.5Ni10Cu17.5 (the Inoue alloy)				127	0.58		
		Inoue data							
		Zr65Al17.5Ni10Cu17.5 (the Inoue alloy) our data				100	0.59	0.56	0.416

Conclusions

This paper presents the composition rules of QCs and metallic glasses, which consist of two composition criteria, the e -constant criterion and the e/a -variant criterion. The former one reflects the electron structure stabilization mechanism for intermetallics compounds, and the latter one is correlated with a cluster growth process from a binary basic icosahedral cluster a multi-component phase. The optimum compositions are located in ternary phase diagrams at the intersections of these two lines. For quaternary system, the composition rules of metallic glasses involve another criterion, namely, the atomic size constant criterion. These criteria have been validated in some Zr-base systems. Our results demonstrate that icosahedral short-range orders, or icosahedral clusters, can guide the composition design of QCs and BMGs after incorporating appropriate criteria in relation to atomic size and electron concentration factors.

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