

Response of acoustic and elastic properties to pressure and crystallization of Ce-based bulk metallic glass

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(Received 12 May 2005; revised manuscript received 13 July 2005; published 28 September 2005)

The density and acoustic velocities of a $\text{Ce}_{70}\text{Al}_{10}\text{Ni}_{10}\text{Cu}_{10}$ bulk metallic glass (BMG) under hydrostatic-pressure (up to 0.5 GPa) and in crystallized state in ambient conditions were measured *in situ* by a pulse echo overlap method. The pressure derivatives of velocities and Grüneisen parameters as well as the equation of state (EOS) of the BMG were determined and compared to those of various other BMGs and nonmetallic glasses. Surprisingly, the BMG, unlike other BMGs with normal mode stiffness, exhibits an anomalous soft longitudinal acoustic mode under pressure similar to that of typical oxide glasses. An unusually large softening of longitudinal acoustic phonons in the BMG, relative to its crystalline state, is also observed, analogous with that in oxide glasses. The possible origin for the anomaly is the intrinsic glassy structure containing short-range covalent bonds.

DOI: [10.1103/PhysRevB.72.104205](https://doi.org/10.1103/PhysRevB.72.104205)

PACS number(s): 61.43.Dq, 81.05.Kf

I. INTRODUCTION

The universal structural feature of glasses is long-range disorder. However, different glasses based on van der Waal, covalent or metallic bonds have different microstructure or short-range orders.¹ For example, polymers have a chainlike structure;² oxide glasses are in continuous random networks³ and bulk metallic glasses (BMGs) have a random close packed atomic configuration.^{4–6} Different microstructures lead to the variety of physical properties of different glasses. Interest in the elastic properties of glasses is longstanding because the study can provide important and unique information on structural and vibrational characteristics of glasses.^{6–8} Ultrasonic is useful for probing subtle structural and elastic properties changes in glasses. The elastic properties under pressure have been studied in the nonmetallic glasses such as oxide glasses,^{7,9–11} and amorphous carbon.¹² They usually exhibit soft-mode behaviors under compression, i.e., the negative pressure derivatives of acoustic velocities or elastic constants. Metallic glasses' elastic features under pressure are poorly understood compared with those of non-metallic glasses due to the difficulties in preparing bulk samples of the metallic glasses. Our previous studies on various BMGs (including Zr-, Pd-, and Cu- based systems) have shown that BMGs have a normal positive pressure derivative of acoustic velocities and positive Grüneisen parameters similar to those of metallic crystalline materials.^{13–15} It is a common view that the metallic glasses should become stiffer under pressure. On the other hand, the differences between the glassy and crystalline states in elastic behaviors are also important in understanding the unique microstructural characteristics and physical properties of BMGs. It has been shown that the crystallization significantly affects the longitudinal mode for oxide glasses and the shear mode for metallic glasses.¹⁵

Recently, we developed a family of cerium-based BMGs which show excellent glass-forming ability, extremely low glass transition temperature ($\sim 60^\circ\text{C}$), low elastic moduli (close to those of nonmetallic glasses such as some poly-

mers), large supercooled liquid temperature region, high stability and a very large electron effective mass.¹⁶ We find that, in near-boiling water, these materials can be repeatedly shaped, and can thus be regarded as metallic thermoplastics. Their resistance to crystallization permits extended forming times above T_g and ensures adequate lifetime at room temperature. Such materials, combining polymer-like thermoplastic behavior with the distinctive properties of metallic glasses, have potential in structural and functional applications and can facilitate studies of the supercooled liquid state. It would be intriguing to study the structural features of such metallic glass.

In this work, we report an unusual response of acoustic and elastic properties to hydrostatic pressure (up to 0.5 GPa) and crystallization in a recently developed $\text{Ce}_{70}\text{Al}_{10}\text{Ni}_{10}\text{Cu}_{10}$ BMG. The pressure derivatives of acoustic velocities and Grüneisen parameters of this BMG are anomalously negative and comparable with those of silicate glasses such as fused quartz. The volume change of the BMG under pressure [i.e., the equation of state (EOS)] is also very similar to that of typical oxide glasses but quite different from that of other known BMGs. Furthermore, we found that there is a markedly softening of both long-wavelength longitudinal and transverse acoustic phonons in this BMG relative to its corresponding crystalline state. The large softening of longitudinal mode in the Ce-based BMG is much larger than that in other reported metallic glasses and comparable with that of oxide glasses. The experimental results provide evidences for the existence of special covalent bonded short-range ordering structures in the BMG, which should be primarily responsible for such unusual elastic responses to pressure. The results shed new light for the better understanding of the unique properties in this BMG. The results are also significant for understanding of the glass-forming ability in BMG-forming alloy and the relationship between properties and microstructure in metallic glasses.

II. EXPERIMENTAL

The $\text{Ce}_{70}\text{Al}_{10}\text{Ni}_{10}\text{Cu}_{10}$ BMG was prepared by copper mold cast method, and the details of the preparation can be

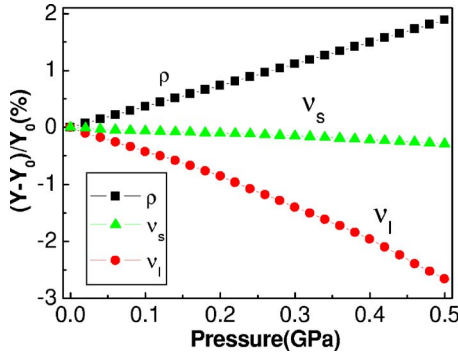


FIG. 1. (Color online). Relative variations $\Delta Y(p)/Y(p_0) = [Y(p) - Y(p_0)]/Y(p_0)$ of ρ , v_l , and v_s with pressure up to 0.5 GPa for the BMG at room temperature. p_0 is the ambient pressure.

seen in Ref. 16. The amorphous nature of the BMGs was ascertained by x-ray diffraction, differential scanning calorimeter, and transmission electron microscopy.¹⁶ The crystalline sample of the BMG was obtained by annealing the cast sample in vacuum chamber (better than 10^{-3} Pa) at a temperature above crystallization temperature 100 K for 1 h. The amorphous and crystalline rods were cut to a length of about 7 mm, and their ends were carefully polished flat and normal to the longitudinal axis. The acoustic longitudinal velocity (v_l) and shear velocity (v_s) of the BMG under high pressure and in crystallized state were measured at room temperature using a pulse echo overlap method.^{17,18} Quartz transducers with harmonic frequency 10 MHz provided the excitation and detection of the ultrasonic pulses. The travel time of ultrasonic waves propagating through the sample was measured using a MATEC 6600 ultrasonic system with a measuring sensitive of 0.5 ns. The carrying frequency of the ultrasonic is 10 MHz. The transducers were bonded to the specimen using honey. The high pressure experiment was performed in a piston-cylinder pressure apparatus. The sample with a bonded transducer was immersed in electric insulating oil (as the pressure transmitting media, for which

hydrostaticity has already been determined.) The high-pressure measurement was performed in situ for several pressure load-unload cycles to examine the reproducibility and to minimize error. Pressure-induced changes in the sample dimensions were accounted for by using Cook's method.¹⁹ The density ρ was measured by the Archimedeian principle with an accuracy of about 0.005 g/cm^3 . The elastic constants (including Young's modulus E , the shear modulus G , the bulk modulus K and Poisson's ratio σ are derived from the acoustic data and density.^{17,18}

III. RESULTS AND DISCUSSIONS

Figure 1 shows the pressure variations $\Delta Y(p)/Y(p_0) = [Y(p) - Y(p_0)]/Y(p_0)$ ($Y = \rho, v_l, \text{ or } v_s$) for the Ce-based BMG at room temperature, where p_0 is the ambient pressure. The changes are reproducible under pressure cycling and show no measurable hysteresis effect up to 0.5 GPa, indicating that acoustic measurement is within the elastic limit of the BMG; and no irreversible pressure-induced changes are observed. The ρ follows a linear relation to pressure with an unusually high relative increase (up to 1.89% at 0.5 GPa). The value is much higher than that of other BMGs and similar to or even larger than that of the nonmetallic glasses such as oxides glasses as shown in Table I. Surprisingly, the relative change of acoustic velocities as a function of pressure is negative, which is markedly different from other BMGs but rather similar to nonmetallic glasses. From Fig. 1, one can see that both v_l and v_s decrease roughly linearly with increasing pressure, i.e., $dv_l/dp < 0$, $dv_s/dp < 0$. Table I shows the relative variations of ρ , v_l , v_s , G , E , K , and σ for various BMGs, oxide glasses, and amorphous carbon (CG) at 0.5 GPa.^{13,15,20} Figures 2(a) and 2(b) compare the pressure variations of v_l and v_s , respectively, for various typical BMGs, nonmetallic glasses. Their pressure derivatives (dv_l/dp and dv_s/dp) are listed in Table II. Except for the Ce-based BMG having negative dv_l/dp and dv_s/dp , all the BMGs have a positive pressure dependence of acoustic ve-

TABLE I. Relative pressure-induced variations of ρ , v_l , v_s , G , E , K , and σ for various BMGs, oxide glasses, and amorphous carbon. All the variations for these glasses are measured at 0.5 GPa except for fused quartz at 0.43 GPa, water white glass, and window glass at 0.42 GPa. The data except for $\text{Ce}_{70}\text{Al}_{10}\text{Ni}_{10}\text{Cu}_{10}$ BMG are taken from Refs. 13–15.

Glasses	$\Delta\rho/\rho_0$ (%)	$\Delta v_l/v_{l0}$ (%)	$\Delta v_s/v_{s0}$ (%)	$\Delta K/K_0$ (%)	$\Delta G/G_0$ (%)	$\Delta E/E_0$ (%)	$\Delta\sigma/\sigma_0$ (%)
Fused quartz (0.43 GPa)	1.24	-2.90	-2.49	-5.47	-3.75	-4.14	-2.87
Amorphous carbon	4.09	2.18	-3.09	20.30	-2.24	1.77	26.36
Water white glass (0.42 GPa)	0.92	-0.08	-0.64	1.71	-0.37	-0.01	1.87
Window glass (0.42 GPa)	1.08	-0.06	-1.19	3.14	-1.31	-0.48	4.81
$\text{SiO}_2 + \text{TiO}_2$ glass	1.58	4.57	-3.69	-9.41	-5.77	-6.59	-5.92
$\text{Zr}_{41}\text{Ti}_{14}\text{Cu}_{12.5}\text{Ni}_{10.2}\text{Be}_{22.5}$	0.46	0.57	0.30	1.84	1.07	1.14	0.28
$\text{Zr}_{48}\text{Nb}_8\text{Cu}_{12}\text{Fe}_8\text{Be}_{24}$	0.44	0.55	0.23	1.59	0.91	0.99	0.31
$\text{Zr}_{50.6}\text{Ti}_{5.2}\text{Cu}_{18.8}\text{Ni}_{14.1}\text{Al}_{14.3}$	0.44	0.64	0.36	1.97	1.16	1.23	0.27
$\text{Pd}_{39}\text{Ni}_{10}\text{Cu}_{30}\text{P}_{21}$	0.32	0.75	0.51	1.98	1.34	1.38	0.15
$\text{Cu}_{60}\text{Zr}_{20}\text{Hf}_{10}\text{Ti}_{10}$	0.39	0.68	0.39	1.97	1.17	1.24	0.27
$\text{Ce}_{70}\text{Al}_{10}\text{Ni}_{10}\text{Cu}_{10}$	1.89	-2.66	-0.29	-6.08	1.30	0.34	-3.91

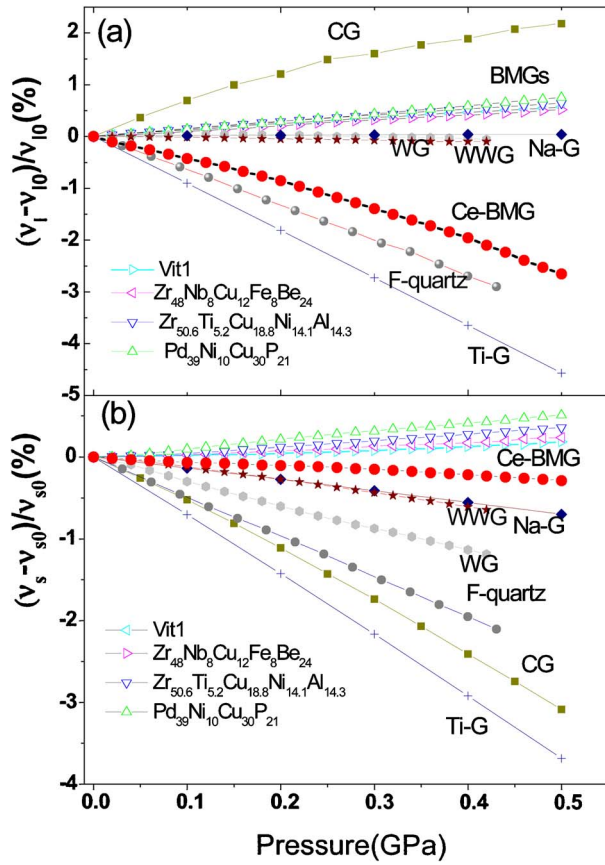


FIG. 2. (Color online). Pressure variations of v_l (a) and v_s (b) for various BMGs, oxide glasses, and amorphous carbon (CG). The oxide glasses are window glass (WG), water white glass (WWG), fused quartz (F quartz), float glass (Na-G), and $\text{SiO}_2 + \text{TiO}_2$ glass (Ti-G).

locities. As shown in Fig. 2, the pressure variation of v_l of the Ce-based BMG is very close to that of fused quartz (F quartz) and the pressure variation of v_s close to that of float glass (Na-G) and water white glass (WWG). These results suggest that the Ce-based BMG should have certain structure similar to that of silicate glasses. It should be noted that the pressure-induced decrease of v_l is about 20 times larger than that of v_s : $|dv_l/dp| \approx 20|dv_s/dp|$, while for other BMGs, $|dv_l/dp| \approx 5|dv_s/dp|$, and such a large difference between $|dv_l/dp|$ and $|dv_s/dp|$ has not been observed in oxide glasses either. This indicates that the longitudinal phonon mode is much softer than the shear mode in the Ce-based BMG under pressure.

Figure 3 presents the pressure dependence of the elastic constants G , K , and σ for the Ce-based BMG. The shear modulus G has a comparably small increase with pressure while K and σ exhibit the largest decreases up to -6.1% and -3.9% , respectively, at 0.5 GPa. According to the relation $G = \rho v_s^2$, the increased G comes in large part from the large increase of ρ under pressure because $dv_s/dp < 0$. The Poisson ratio σ , as a density independent parameter, directly reflects the relative change of v_l/v_s , so it is very sensitive to microstructure.¹⁷ The large variation of σ under pressure reveals a pressure-induced structural change in the BMG. This

phenomenon is not observed in Zr-, Cu-, and Pd-based BMGs,^{13–15} whose Poisson ratios remain constant or show small variations under pressure as listed in Table I. Figure 3 and Table I contrasts the variations $\Delta Y(0.5 \text{ GPa})/Y(p0)$ of ($Y = \rho, v_l, v_s, G, K$, and σ) for the Ce-based BMG and the typical $\text{Zr}_{41}\text{Ti}_{14}\text{Cu}_{12.5}\text{Ni}_{10}\text{Be}_{22.5}$ BMG (Vit1).¹⁵ Apparently, the Ce-based BMG exhibits much larger and different variations of ρ, v_l, K , and σ compared to those of Vit1, strongly indicating that the Ce-based BMG has a unique structure that is quite different from those of other BMGs.

Further physical insight into the acoustic vibrational behaviors of this BMG can be gained by investigating the anharmonicity of the long-wavelength acoustic modes. The Grüneisen parameters of this BMG are determined using the following equations:^{17,21}

$$\gamma_l = -\frac{K}{6C_{11}} \left(3 - \frac{3C_{12}}{K} - \frac{3dK}{dp} - 4\frac{dG}{dp} \right), \quad (1)$$

$$\gamma_s = -\frac{1}{6G} \left(2G - 3K\frac{dG}{dp} - \frac{3}{2}K + \frac{3}{2}C_{12} \right), \quad (2)$$

and

$$\gamma_{av} = \frac{1}{3}(\gamma_l + 2\gamma_s), \quad (3)$$

where γ_l , γ_s , and γ_{av} are the longitudinal, shear and average Grüneisen parameters, respectively, and $C_{11} = \rho V_l^2$, $C_{12} = C_{11} - 2C_{44}$, $G = C_{44} = \rho V_s^2$. From the linear fit of the elastic constants versus pressure, the γ_l , γ_s , and γ_{av} obtained using Eqs. (1)–(3) for the Ce-based BMG are -1.10 , 0.20 , and -0.23 , respectively. The BMG has large negative γ_l value but comparably small positive γ_s and thus negative γ_{av} . The available Grüneisen parameters of various BMGs, oxide glasses, and amorphous carbon are collected in Table II. The shear Grüneisen parameter γ_s of the Ce-based BMG is positive but much smaller than that of the Zr-, Pd-, and Cu-based BMGs. The large negative γ_l of the Ce-BMG is comparable to that of silicate glasses. In other words, the softening vibrational behaviors in the long-wavelength limit of Ce-based BMG under high-pressure are mainly controlled by the longitudinal acoustic mode. The opposite signs of γ_l and γ_s in the Ce-BMG are similar to that of carbon glass, which has a special mixed structure: the C—C linkage is a mixture of covalent and metallic bonds, while the linkage between layers is van der Waals bonding.¹² Previous structural analyses have demonstrated the covalently bonded short-range ordering structures in Al—Ni—Ce melt-spin glasses.²² The covalently bonded short-range ordering is the “rigid” structural units such as tetrahedra in oxide glasses and icosahedral cluster in some BMGs. The glass with a structure composed of the weakly linked “rigid” structural units.¹⁴ The soft modes are low energy deformation of these “rigid” structural units. Thus, the normal local structure similar to that of other BMGs and covalent bonding structures similar to those of oxide glasses may coexist in the Ce-based BMG, and the intrinsic glassy structure containing short-range covalent bonds causes the anomalous acoustic behaviors under pressure.

TABLE II. Pressure derivatives of longitudinal velocity v_l and transverse velocity v_s and Grüneisen parameters γ_l , γ_s , and γ_{av} for different glasses including BMGs, silicate glasses and CG. All the data except for $\text{Ce}_{70}\text{Al}_{10}\text{Ni}_{10}\text{Cu}_{10}$ BMG are taken from Refs. 13–15 and 20.

Glasses	dV_l/dP (km/s GPa)	dV_s/dP (km/s GPa)	γ_l	γ_s	γ_{av}
$\text{Zr}_{41}\text{Ti}_{14}\text{Cu}_{12.5}\text{Ni}_{10}\text{Be}_{22.5}$ (vit1)	0.055	0.014	1.65	0.86	1.12
$\text{Zr}_{48}\text{Nb}_8\text{Cu}_{12}\text{Fe}_8\text{Be}_{24}$	0.055	0.011	1.54	0.88	1.10
$\text{Zr}_{50.6}\text{Ti}_{5.2}\text{Cu}_{18.8}\text{Ni}_{14.1}\text{Al}_{14.3}$	0.062	0.017	1.76	0.67	1.03
$\text{Pd}_{39.1}\text{Ni}_{10.1}\text{Cu}_{29.9}\text{P}_{20.9}$	0.072	0.021	2.75	2.02	2.26
$\text{Cu}_{60}\text{Zr}_{20}\text{Hf}_{10}\text{Ti}_{10}$	0.063	0.016	2.10	1.34	1.59
$\text{Ce}_{70}\text{Al}_{10}\text{Ni}_{10}\text{Cu}_{10}$	-0.133	-0.007	-1.11	0.20	-0.23
Amorphous carbon	0.150	-0.153	0.76	-0.45	-0.04
Window glass	-0.009	-0.097	0.27	-0.78	-0.43
Water white glass	-0.015	-0.053	0.22	-0.38	-0.18
Float glass	0.008	-0.046	0.27	-0.42	-0.19
$\text{SiO}_2 + \text{TiO}_2$ glass	-0.462	-0.273	-2.25	-2.10	-2.15
Fused quartz	-0.403	-0.218	-2.10	-1.75	-1.86

Based on the bulk modulus B and its pressure dependence, an isothermal EOS is established in terms of the Murnaghan formula:²³

$$\ln\left(\frac{V_0}{V(p)}\right) = \frac{1}{K'_0} \ln\left(\frac{K'_0}{K_0}p + 1\right), \quad (4)$$

where K_0 and K'_0 are the bulk modulus and its pressure derivative at ambient pressure, respectively. Assuming a linear relationship between sound velocities and pressure, the fitted values of K'_0 and K_0 for the Ce-based BMG are -3.328 and 27.79 GPa, respectively. The EOS is presented in Fig. 4. As seen in Fig. 4, the compressibility of the Ce-based BMG is also similar to that of silicate glasses but much higher than that of the other BMGs. This result further confirms that the Ce-based BMG contains short-range covalent bonds similar to those of silicate glasses.

The intrinsic glassy structure of the Ce-based BMG has also demonstrated by its unique acoustic and elastic responses to crystallization. The relative changes $\Delta Y = (Y_c - Y_g)/Y_g$ ($Y = \rho, v_l, v_s, E, G$, and K) between the fully crys-

tallized state (Y_c) and glassy state (Y_g) for the Ce-based BMG, typical Vit1 BMG²⁴ and oxide glasses¹⁵ are shown in Fig. 5. The large changes in v_l (14.4%), v_s (20.9%), E (43.6%), G (47.8%), and K (22.9%) between the glassy and crystallized states for the Ce-based BMG can be seen in Fig. 5. The remarkably large changes of acoustic velocities and elastic moduli in the BMG relative to its crystalline state mean that marked softening of long-wavelength acoustic phonons, both transverse and longitudinal, in the Ce-based BMG. However, for other BMGs such as vit1, large changes in v_s (13.5%), E (27.2%), and G (30.3%) but much smaller changes in v_l (5.2%) and K (3.9%) between the two states indicating that only the marked softening of the transverse elastic modulus can be observed in the BMG relative to the crystallized state.^{12,24} For the Ce-based BMG, the relative change of v_l is about 2 times larger than that of the vit1, comparable with that of oxide glasses as shown in Fig. 5. Due to the remarkably large softening of longitudinal phonons a huge increase of K (about 5 times larger than that

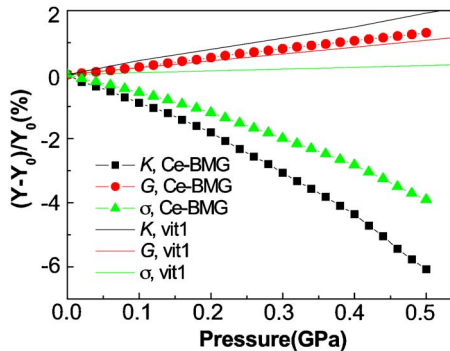


FIG. 3. (Color online). Comparison of the relative variations of G , K , and σ with pressure up to 0.5 GPa for the Ce BMG and vit 1 at room temperature.

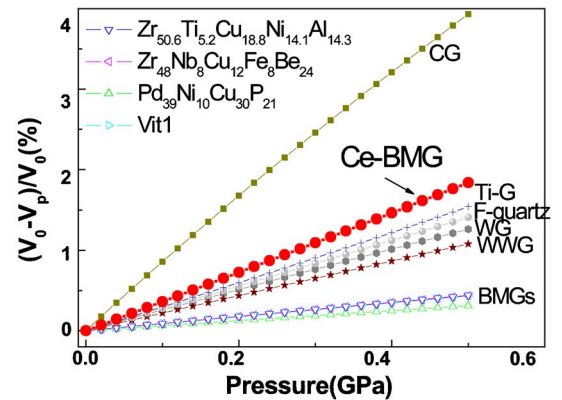


FIG. 4. (Color online). EOS of Ce-based BMGs. The EOS of other BMGs, silicate glasses, and amorphous carbon are also presented for comparison.

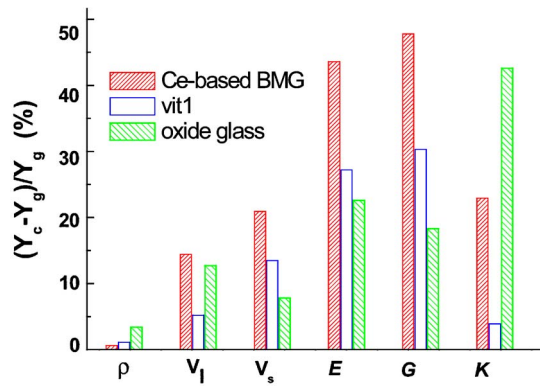


FIG. 5. (Color online). Relative changes $\Delta Y = (Y_c - Y_g)/Y_g$ ($Y = \rho, v_l, v_s, E, G$, and K) between the fully crystallized state (Y_c) and glassy state (Y_g) for the Ce-based BMG, Vit1, and oxide glasses.

of vit1 alloy) in the BMG is observed upon crystallization. Therefore, the softening especially in longitudinal acoustic mode, in the Ce-based BMG relative to its crystalline counterpart is quite different from that of other BMGs but similar to that of oxide glasses.^{15,24} Cerium has a special variable electronic structure and dual valency states, and it is believed that complex bonding structures including metallic and co-

valent bonds coexist in the Ce-based BMG. This unusual softening concerning the long-wavelength acoustic phonons can be attributed mainly to its covalently bonded local structure.

IV. CONCLUSIONS

The Ce-based BMG exhibits unusual soft-mode behaviors under pressure and relative to its crystalline state that are similar to that of some typical oxide glasses but markedly different from those of other BMGs. The unusual acoustic and elastic behaviors confirm that the intrinsic glassy structure contains covalent bonds in this BMG, which will be fairly important for understanding the nature of its microstructure, elastic properties and their relationship to bulk metallic glasses in general. The structural features may be helpful for understanding the unique properties in this metallic glass.

ACKNOWLEDGMENTS

Financial support from the National Natural Science Foundation of China (No. 50321101) is gratefully acknowledged. The useful discussion and experimental assistance of Dr. M. X. Pan, D.Q. Zhao, and F.Y. Li are appreciated.

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