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COMMENTS

Comment on “Unusual transition phenomenon in Zr-based bulk metallic glass upon heating at high pressure”
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In a recent letter, Sun *et al.*¹ report on the phase transformations of a $\text{Zr}_{41.2}\text{Ti}_{13.8}\text{Cu}_{12.5}\text{Ni}_{10}\text{Be}_{22.5}$ bulk glass during heating at 10 GPa studied by *in situ* energy-dispersive x-ray powder diffraction. In their experiment, sharp diffraction peaks are observed in the sample annealed at 528 K for 300 s in Fig. 1. The strongest peak at 47.66 keV first increases in intensity with temperature up to 653 K for 200 s and then decreases at 689 K for 200 s. In the sample at 689 K for 400 s, the peak essentially disappears. On further temperature increase up to 724 K, the amorphous structure remains unchanged. Sun *et al.* interpret the observations in terms of the formation of a fcc phase within the temperature range of 528–689 K and a fcc-to-amorphous phase transition within the temperature range of 689–724 K. The transition is caused by the enhancement of internal energy for the fcc phase due to lattice distortion. While we have no reservations about the experimental observations, we point out an alternative point of view about their experimental observations.

(1) We first address lattice distortion of the fcc phase. In Fig. 3, Sun *et al.*, claim that the increase of the (111) d spacing in a temperature range of 580–700 K is due to lattice distortion as a result of atomic migration.¹ However, due to pure thermal expansion, the lattice constant of the fcc phase could also increase. The thermal expansion coefficient of the phase in the temperature range studied, could be estimated from Fig. 3 to be in the order of 10^{-5} K^{-1} , which is in good agreement with pure copper and other Zr-based glasses.^{2,3} Although atomic migration into the fcc phase could cause lattice distortion, more experiments are essential to reveal the nature of the expansion of the (111) d spacing for the fcc phase.

(2) For a fcc structure with a lattice constant of 4.24 Å mentioned in the letter,¹ one expects d -spacing values are 2.448 Å for (111), 2.120 Å for (200), 1.499 Å for (220), 1.278 Å for (311), and 1.224 Å for (222). The (111), (311), and (222) peaks are indeed consistent with the experimental results of 2.456 ± 10 , 1.281 ± 10 , and 1.237 ± 10 Å in the sample at 618 K for 300 s while the diffraction peak of d -spacing value of 1.386 ± 10 Å mentioned in the letter¹ cannot be indexed to the fcc phase. It should be mentioned that

the (200) and (220) peaks of the fcc phase, which are not overlapped with NaCl peaks, were not detected in Fig. 1. This fact might indicate a strong anisotropy crystallographic orientation of the fcc crystal. By comparing the (111) peak width with the peaks for NaCl, one can conclude that the crystal size of the fcc-crystal formed in the sample is similar to NaCl crystals, usually about a few tens of micrometers. These results further lead us to suggest that the reduction of the diffraction peak (111) within the temperature range of 689–724 K in Fig. 1 could be due to a geometric effect of the experimental setup used in the letter.¹ The reasons are given as follows.

(3) For an energy-dispersive x-ray powder diffraction measurement, the detector angle is fixed, 3° used in the letter.¹ Between sample and detector (usually having a distance of about 200 mm), solid slits (about 0.5 mm) are usually employed to reduce the background. Note that the x-ray powder diffraction patterns recorded in Fig. 1 have low background. When one high-orientated micrometer-sized crystal slightly rotates during experiments, e.g., about 0.3° , the diffraction peak could disappear in the energy-dispersive x-ray powder diffraction pattern. Such a phenomenon is often observed by energy-dispersive x-ray powder diffraction measurements in the systems where a high-orientated crystal slightly rotates in a low-viscosity medium, e.g., during melting process of multicomponent alloys. Based on the fact that the temperature range of 689–724 K used in the letter¹ is in the supercooled liquid region (low-viscosity region) of the $\text{Zr}_{41.2}\text{Ti}_{13.8}\text{Cu}_{12.5}\text{Ni}_{10}\text{Be}_{22.5}$ bulk glass,⁴ we believe that due to off-Bragg reflection of the (111) plane in the high-orientated fcc crystal, no diffraction peak might be detected by the energy-dispersive x-ray powder diffraction used in the letter.¹ *But, the fcc crystal could still exist in the sample.* Therefore, to confirm the existence of a fcc-to-amorphous phase transition reported in the letter,¹ more experiments, e.g., by changing detector angle or rotating sample, are required.

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