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**Comment on "Reversible phase transition between amorphous and crystalline in Zr<sub>41.2</sub>Ti<sub>13.8</sub>Cu<sub>12.5</sub>Ni<sub>10</sub>Be<sub>22.5</sub> under high pressure at room temperature" [Appl. Phys. Lett. 76, 2874 (2000)]**

**Jiang, Jianzhong; Gerward, Leif; Olsen, J. Staun**

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## COMMENTS

### Comment on "Reversible phase transition between amorphous and crystalline in $\text{Zr}_{41.2}\text{Ti}_{13.8}\text{Cu}_{12.5}\text{Ni}_{10}\text{Be}_{22.5}$ under high pressure at room temperature" [Appl. Phys. Lett. 76, 2874 (2000)]

J. Z. Jiang and L. Gerward<sup>a)</sup>

Department of Physics, Technical University of Denmark, Fysikvej 307, DK-2800 Kgs. Lyngby, Denmark

J. Staun Olsen

Niels Bohr Institute, Oersted Laboratory, University of Copenhagen, DK-2100 Copenhagen, Denmark

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In their recent letter, Sun *et al.*<sup>1</sup> reported a reversible pressure-induced phase transition between the amorphous and crystalline state of the bulk metallic glass (BMG)  $\text{Zr}_{41.2}\text{Ti}_{13.8}\text{Cu}_{12.5}\text{Ni}_{10}\text{Be}_{22.5}$  at room temperature. Using *in situ* high-pressure x-ray diffraction and the energy-dispersive method in conjunction with synchrotron radiation, Sun *et al.* found that the bulk metallic glass partially crystallizes at 24 GPa upon compression, and that it reverts to the amorphous state upon decompression. All experiments were performed at room temperature. However, in our opinion, the authors have based their finding of this unusual behavior on the presence of one or two very weak peaks in their observed diffraction patterns (Sun *et al.*, Fig. 1).<sup>2</sup> Moreover, their experimental conditions are far from being optimized, perhaps as a result of the limited photon energy range of their synchrotron radiation source. Therefore, we have repeated the x-ray diffraction experiment under optimum conditions and, as the results will show, we observe no crystallization of the bulk metallic glass.

The white-beam, energy-dispersive method is based on the use of a fixed scattering angle,  $2\theta$ , where  $\theta$  is the Bragg angle.<sup>3</sup> Sun *et al.*<sup>1</sup> have used a rather large scattering angle,  $2\theta=22.8^\circ$ , which means that possible diffraction features are occurring at photon energies less than 15 keV (the broad amorphous peak is centered at 13.3 keV). At these low energies, there are problems with absorption in the diamonds of the high-pressure cell and in the sample itself. Another problem is caused by the presence of x-ray fluorescence peaks (Zr  $K\alpha$  and Zr  $K\beta$ ) in the same energy range.

Our high-pressure x-ray diffraction experiments were performed at the Hamburg Synchrotron Radiation Laboratory (HASYLAB). This is a powerful source and we have used radiation in the 20–70 keV photon energy range. The diffractometer, working in the energy-dispersive mode, has been described elsewhere.<sup>4</sup> High pressures were obtained in a Syassen–Holzapfel type diamond-anvil cell. The sample and a small ruby chip were enclosed in a hole of diameter 0.2 mm in an inconel gasket. For the pressure-transmitting me-

dium, we have used a 16:3:1 methanol-ethanol-water mixture, i.e., the same medium as Sun *et al.*<sup>1</sup> The pressure was determined by the ruby fluorescence method, using the non-linear pressure scale of Mao *et al.*<sup>5</sup> The Bragg angle, associated with each experimental run, was determined from a zero-pressure spectrum of powdered NaCl in the diamond-anvil cell.

In our experiment, the scattering angle is  $2\theta=7.29^\circ$ . At ambient pressure, the broad amorphous peak of the bulk metallic glass is found to be centered at 41.05 keV (Fig. 1). The peak energy corresponds to a  $d$  spacing of 2.37 Å in good agreement with the value 2.36 Å reported by Sun *et al.*<sup>1</sup>

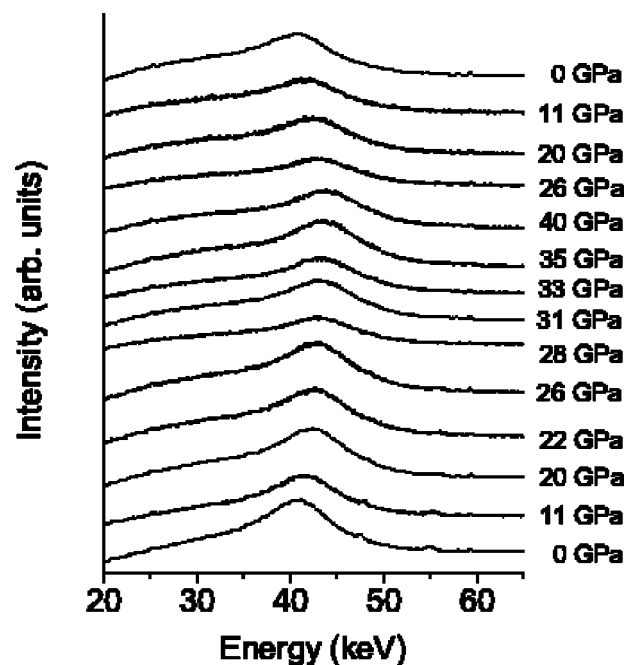


FIG. 1. *In situ* synchrotron radiation x-ray diffraction patterns of  $\text{Zr}_{41.2}\text{Ti}_{13.8}\text{Cu}_{12.5}\text{Ni}_{10}\text{Be}_{22.5}$  bulk metallic glass under pressure at room temperature. Note the shift of the broad amorphous peak with pressure. The small peaks at about 47.5 and 54.8 keV are inconel 111 and 200 lines due to the gasket. Also barely seen are the W  $K\alpha_1$  and W  $K\alpha_2$  peaks at 59.3 and 58.0 keV. They are caused by the slit system. The Zr  $K\alpha$  and Zr  $K\beta$  fluorescence peaks at 15.7 and 17.7 keV have been eliminated by the lower-level discriminator of the multichannel pulse-height analyzer.

<sup>a)</sup>Electronic mail: gerward@fysik.dtu.dk

Upon applying pressure, the amorphous peak is shifted to higher energies, corresponding to lower  $d$  values, as expected. We have applied pressures up to 40 GPa, and in Fig. 1 we give some examples of the observed diffraction patterns. In particular, several diffraction patterns were recorded at pressures around 24 GPa. In none of our runs have we observed any sign of crystallization of the bulk metallic glass. Upon decompression, the amorphous peak is found to revert to its original position.

In conclusion, we are unable to reproduce the experimental findings of Sun *et al.*<sup>1</sup> In particular, we have found no indications of any pressure-induced crystallization of the bulk metallic glass  $\text{Zr}_{41.2}\text{Ti}_{13.8}\text{Cu}_{12.5}\text{Ni}_{10}\text{Be}_{22.5}$  at room temperature.

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<sup>1</sup>L. L. Sun, W. K. Wang, D. W. He, W. H. Wang, Q. Wu, X. Y. Zhang, and Z. X. Bao, *Appl. Phys. Lett.* **76**, 2874 (2000).

<sup>2</sup>Sun *et al.* (see Ref. 1) have also performed resistance measurements showing some interesting features, but in the present note we will comment on the x-ray diffraction results only.

<sup>3</sup>B. Buras and L. Gerward, in *International Tables for Crystallography*, 2nd ed., edited by A. J. C. Wilson and E. Prince (Kluwer, Dordrecht, 1999), Vol. C, p. 84.

<sup>4</sup>J. S. Olsen, *Rev. Sci. Instrum.* **83**, 1058 (1992).

<sup>5</sup>K. H. Mao, P. M. Bell, J. W. Shaner, and D. J. Steinberg, *J. Appl. Phys.* **49**, 3276 (1978).