Evaluation of the pressures measured in the double stage diamond anvil cell technique Evaluation of the pressures measured in the double stage diamond anvil cell technique

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The double stage diamond anvil cell (dsDAC) is claimed as an advance technique to generate a TPa static pressure (Dubrovinskaia et al. 2016). Although the nanocrystalline diamond semi-balls were used for the TPa generation, it is need to prepare an desired anvil shape; i.e., the culet and bevel size and so on, in order to control the pressure distribution at the tip of the 2nd anvils. We have been trying to develop the dsDAC as a well-controlled experiment device using the focused ion beam (FIB) system (Sakai et al. 2015).

Here we report the results of the dsDAC experiments using the newly synthesized nanopolycrystalline diamond (NPD) with a single-nano grain size as a micro-anvils material. The 2^{nd} stage micro-anvils are shaped with 3 um culet and 10 um bevel using FIB system (Scios, FEI). The tiny rhenium disc (3 um diameter and 1 um thickness) was used as a sample. The other experimental procedure was generally same as in Sakai et al. (2015). The X-ray diffraction (XRD) experiments were performed at SPring-8 BL10XU in order to determine the generated pressure from the lattice parameter of rhenium. The diffraction patterns from the rhenium sample showed very broad peaks due to the large pressure gradient at the tip of the micro-anvils as contrasted with the sharp peaks observed in previous works (Dubrovinsky et al. 2012, 2015; Dubrovinskaia et al. 2016). The deconvolution of the peak results that the rhenium was compressed to be V/V₀ = 0.633. According to the equation of state of rhenium (Re-EoS) reported by Anzellini et al. (2014), it corresponds to about 430 GPa. On the other hand, it is 630 GPa if we adapt the Re-EoS by Dubrovinsky et al. (2012). In the term of V/V₀ value, we reproduced the result of Dubrovinsky et al. (2012) although we observed the X-ray diffraction peaks with large FWHM as an unavoidable result from the large pressure gradient at the tip of the 2nd stage micro-anvils.

The compression behavior of NPD micro anvils shows a monotonous volume decrease along the equation of state of the diamond (Dewaele et al. 2008); however it becomes incompressible when the compression by the 2nd stage micro-anvils started. It is a well-known phenomenon induced by the uniaxial stress, i.e., the overestimation of the volume. This large effect of uniaxial stress was released when the 2nd stage micro-anvils failed, which means the sudden volume decrease occurred although the pressure was dropped. This "incompressible" feature is the opposite of the "compressible" behavior which reported in Dubrovinsky et al. (2012).

The dsDAC certainly has a potential ability to generate an ultra-high pressure. Since the X-ray beam is comparable in size to the culet of the 2nd stage micro-anvils, it needs further evaluation of the

relationships between the X-ray beam intensity profile and the pressure distribution and the sample distribution to interpret the XRD patterns measured in the dsDAC experiments correctly.

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