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High-pressure science with a multi-anvil apparatus at SPring-8

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Abstract

Since first opening its doors to public research in 1997, SPring-8 has seen the accomplishment of many important studies in a wide variety of fields through its stable operation and cutting edge technology. High-pressure experiments have been carried out on a number of beamlines using a diamond anvil cell or a multi-anvil press. Here, we review the multi-anvil presses installed on the SPring-8 beamlines and a few research projects currently utilizing this technology. The significant difference in post-spinel boundary between multi-anvil experiments and diamond anvil studies will also be discussed.

1. Facilities

SPring-8 is one of the third-generation synchrotron radiation sources at the Harima Science Garden City in Hyogo, Japan. Three multi-anvil presses have been installed on different beamlines at SPring-8 [1]. Their characteristics are summarized in table 1. Pressure–temperature conditions and experimental techniques available with these presses vary. Users can choose the apparatus best suited for their experimental purposes.

The largest press is installed on beamline BL04B1 (bending magnet source). This is a public beamline fully available for use by outside users with white x-rays (20–200 keV) available in the experimental hutch. The multi-anvil high-pressure–temperature device is named SPEED-1500 (SPring eight energy dispersive device with a 1500 ton press), and consists of a 1500 ton press, a guide-block, a horizontal goniometer, positioning stages, an SSD x-ray detector system, a set of fluorescence screens, a CCD camera, and computer control facilities (figure 1). Pressure generation is achieved using a two-stage compression method (the so-called 6–8 or Kawai system). This system is capable of generating pressures up to 27 GPa and temperatures of 2200 °C using tungsten carbide anvils. Recently, a significant effort has been made to extend the pressure region by replacing carbide anvils with sintered diamond ones, enabling pressures up to 50 GPa to be reached. The x-ray beam collimated by incident slits

Figure 1. A front view of the SPEED-1500 system installed on beamline BL04B1.

Table 1. Comparison of the multi-anvil presses installed at the SPring-8 beamlines.

	SPEED-1500	SMAP1	SMAP2
Compression method	6–8 two stage	Cubic anvil	Cubic anvil
Pressure (WC)	>27 GPa	>13 GPa	>13 GPa
Pressure (SD)	>50 GPa	>18 GPa	>18 GPa
Temperature	>2200 °C	>1800 °C	>1800 °C
Beamline	BL04B1	BL11XU	BL14B1
Light source	BM	Undulator	BM
X-ray	White	Monochromatic	White/monochromatic
Detector	Ge-SSD CCD camera	Imaging plate CCD camera	Ge-SSD
Measurement	EDX radiography	ADX	EDX XAFS

passes through small anvil gaps and irradiates the specimen. Diffracted x-rays pass through the horizontal gaps of anvils and are collected by a detector mounted on a horizontal goniometer. Since this high-pressure system is fixed in this beamline, and as only white x-rays are available, x-ray diffraction experiments are carried out by the energy dispersive method (EDX). A high-resolution CCD camera has made it possible to obtain a radiographic image of samples under high pressures. This technology has been applied to viscosity measurements via the falling sphere method.

The other two large-volume presses are DIA-type devices with a single-stage cubic anvil system, capable of generating pressures up to 13 GPa using tungsten carbide anvils. These two facilities (SMAP1, SMAP2; SPring-8 multi-anvil press) are installed on the JAERI (Japan Atomic Energy Research Institute) beamlines. The JAERI beamlines were originally constructed for exclusive use by JAERI researchers; however, 20% of the total machine time has been made available for public use. SMAP1 is installed on beamline BL11XU, where high-flux monochromatic x-rays from an undulator device are available (20–40 keV). Using monochromatic x-rays, angle dispersive x-ray diffraction experiments (ADX) can be performed. Either an imaging plate or an x-ray CCD camera may be selected as a detector. In this experiment, a radial slit (multi-channel collimator) is indispensable for reducing the contamination signal from surrounding materials such as a pressure transmitting medium [2]. On the other hand, SMAP2 on beamline BL14B1 enables energy dispersive x-ray diffraction studies (EDX) using white x-rays (20–200 keV) from a bending magnet source. By switching the optics of the beamline, monochromatic x-rays are also available, so XAFS experiments under high pressure may be performed.

2. Research activities

Over 40 proposals employing multi-anvil experiments were accepted in the period between September 2000 and June 2001. The acceptance rate was about 65%. Here, we briefly summarize the categories of research activities. For further information, please visit the SPring-8 web site [3], where all user reports on the high-pressure experiments which were carried out at SPring-8 may be obtained.

2.1. Phase relation and equations of state

One of the most popular research topics using the current multi-anvil system is related to phase relations or equations of state involving geophysical materials. Since the initial excellent study which determined the post-spinel phase boundary in Mg_2SiO_4 [4], many experiments have continuously been carried out on various materials such as $MgSiO_3$ [5–7], $CaMgSi_2O_6$ [8], $Mg_3Al_2Si_3O_{12}$ [9], FeS [10], C_{60} [11], ZrO_2 [12], and SnO_2 [13]. These experiments have taken advantage of the stability and controllability of pressure–temperature conditions and of the ability to record multiple data in only a single experimental run. Recently, it has been pointed out that experimental results on the post-spinel phase boundary obtained when using the multi-anvil and diamond anvil cells are not consistent. This problem will be discussed in the next section.

2.2. Kinetics

The combination of a large-volume press and energy dispersive x-ray diffraction is especially suitable for the study of transformation kinetics at high pressure, as it is possible to collect a large amount of kinetic data efficiently. Several studies which elucidated the kinetics of the phase transformation of Mg_2SiO_4 have been performed [14, 15]. On the basis of these kinetic data, transformation–temperature–time diagrams were constructed which may be useful in evaluating whether or not the post-spinel transformation occurs within the timescale for subduction (106 years) in the cold interior (700 °C) of descending oceanic plates. Recently, a similar experiment investigating the graphite–diamond transformation has been carried out [16]. A powder mixture of graphite and brucite ($Mg(OH)_2$) was used as a starting material, and water formed by the dehydration of brucite played the role of catalyst for the graphite–diamond conversion. Kinetics data for this transition at various conditions were accumulated (figure 2), and the pressure–temperature effect of the incubation time for nucleation of diamond has been clarified.

2.3. Liquid

Characteristics of the white x-rays at the SPring-8 (high flux, wide energy range) are also very effective in obtaining good diffraction profiles of liquids. By intensive studies on liquid phosphorus, we have succeeded in making the first direct observation of the first-order structural phase transition of a pure liquid in a pure substance [17]. This reversible transition is caused by an abrupt structural change between a molecular liquid form and a polymeric form at a pressure around 1 GPa. Further investigations for other materials have been executed [18, 19]. Most previous experiments were made using the energy dispersive method; a new study employing the angle dispersive method has recently begun.

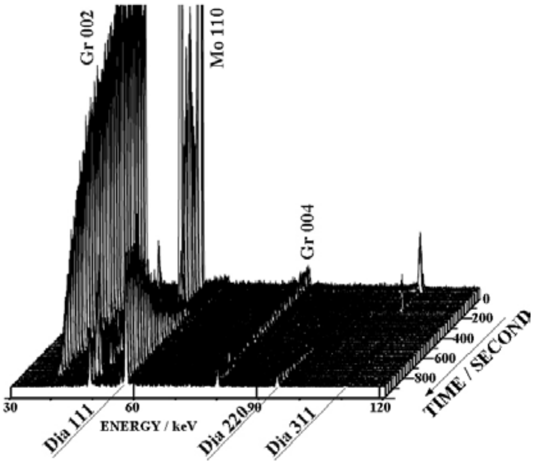

Figure 2. The time-resolved x-ray diffraction profile of the graphite–diamond conversion with an aqueous fluid catalyst [16].

2.4. Materials science

One of the important applications of the SPring-8 is in the field of materials science. Many studies of diamonds and related super-hard materials have been made. Also, a series of high-pressure synthesis experiments concerning the complex transition metal oxides ($(VO)_2P_2O_7$, $Ca_{2-x}Na_xCuO_2Cl_2$, etc) have been performed [20]. In these experiments, Pt or Au capsules are commonly used to control oxygen fugacity or to avoid chemical reactions with surrounding materials. The high-energy and high-flux x-rays of SPring-8 have made it possible to observe the diffraction profile of samples placed in a capsule made of such heavy metals. Formation of the high-pressure phase and its melting behaviour at high temperatures were clearly observed. The information thus obtained by *in situ* observation was fed back to a synthesis experiment in the laboratory and a large single crystal of the high-pressure phase was successfully synthesized.

2.5. XAFS

Several XAFS experiments under high pressure have been made using SMAP2 combined with monochromatic x-rays of the BL14B1 beamline. Structural changes in crystalline and vitreous GeO_2 under high pressures were investigated and the difference between the changes in their coordination numbers has been clarified [21]. In this experiment, the pressure was calculated from Au–Au distances determined by Au L-edge EXAFS measurement.

2.6. Radiography and viscosity measurement

Using the combination of a fluorescence screen and a CCD camera, radiographic images of samples in the high-pressure cell can be observed with a resolution of 5 μm. This technique has proven to be very useful for deformation or rheology studies, and has also been applied to the measurement of inter-diffusion in melts and to the determination of melting temperatures. Its most important application, however, is in viscosity measurements. Real-time images of the sinking process of Pt spheres in the melt are recorded, enabling calculation of sphere terminal velocities by analysing the captured images. The viscosity is then calculated from the velocity value using Stokes's equation. Viscosities of albite ($NaAlSi_3O_8$) melts [22] and Fe–FeS melts [23, 24] have previously been measured at various pressures.

3. The post-spinel phase boundary in Mg_2SiO_4

In this section, we introduce the post-spinel experiment as an example of a phase relation study, and discuss potential reasons for its deviation from recent results employing a diamond anvil experiment.

The first experiment which used the SPEED-1500 system, *in situ* observation of the post-spinel transition in Mg_2SiO_4, was a collaboration between Japanese high-pressure groups [4]. Change of the x-ray diffraction profiles from the spinel phase to the perovskite phase was clearly observed, and its phase boundary was determined. However, this result stands in contrast to previous studies; the transition pressure based on *in situ* observation was 21 GPa at 1600 °C, which is about 2 GPa lower than that obtained by earlier quench experiments [25]. Since the pressures corresponding to the 660 km seismic discontinuity are believed to range between 22.5 and 24.5 GPa, the *in situ* data have introduced a serious issue in that the inferred phase transformation at the 660 km discontinuity and its experimentally determined P–T conditions do not match. Recently, a similar *in situ* experimental study of this phase transition in Mg_2SiO_4 using a diamond anvil cell was reported [26]. In contrast to the case for the multi-anvil study, the pressure and temperature of the post-spinel transition obtained by the diamond anvil experiment were consistent with the 660 km discontinuity; the transition pressure was about 23.5 GPa at 1600 °C, which was about 2.5 GPa higher than that based on the *in situ* multi-anvil study (see figure 2 in [26]).

One possible explanation for this significant discrepancy may concern the difference between the pressure standard materials used in the relevant experiments. In the multi-anvil experiment, Au was used as the pressure marker, while Pt was used in the diamond anvil study. Moreover, there is a critical question of which equation of state should be used. In the *in situ* multi-anvil experiment, Anderson's equation of state for Au was used to determine the pressure [27]—which has been widely used in recent *in situ* experiments. However, there is another famous equation of state for Au (Jamieson's scale [28]) that yields a different pressure for a given volume at high temperature. Figure 3 shows the pressure differences between these two equations of state of Au at different pressure and temperature conditions. The pressure derived from Jamieson's scale is about 2.5 GPa higher than that from Anderson's at 22 GPa and 1600 °C. This suggests that if the pressures in the multi-anvil experiment were recalculated on the basis of Jamieson's scale, the post-spinel phase boundary would shift in the higher-pressure direction by 2.5 GPa, and consequently the two experiments would become consistent. This result seems to support the idea that Jamieson's equation of state for Au should be viewed as the best one for estimating pressures and should be used instead of Anderson's scale. However, this idea is not easily accepted because of the following experimental results.

In order to compare various pressure standard materials, a series of *in situ* high P–T experiments using SPEED-1500 have been carried out in which plural pressure calibrants, such as NaCl and Au, Au and Pt, and Au and W, were compressed together and the pressures derived from various equations of state of materials were calculated [7, 9]. The results are summarized in figure 3 of [9]. This experiment demonstrated that Jamieson's Au scale deviated greatly from other pressure scales such as those for NaCl, Pt, W, and Mo, while pressures determined by Anderson's Au scale were almost consistent with those based on the other calibrant materials. The Pt scale used in the *in situ* diamond anvil experiment predicts even lower pressures (by 0.5–1.0 GPa) than Anderson's Au scale (more than 3 GPa lower if compared with Jamieson's scale). Therefore, neither the difference of the pressure standard material nor the choice of

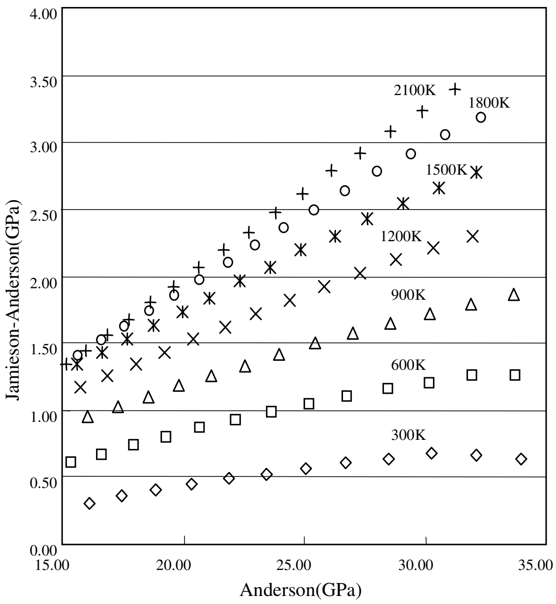

Figure 3. The pressure difference between the two equations of state for Au (Jamieson's scale and Anderson's scale).

equation of state can explain the differences between the post-spinel boundaries obtained by the multi-anvil and diamond anvil experiments.

For further discussion of this discrepancy, potential problems both in diamond anvil and multi-anvil experiments must be checked carefully. Here, we summarize factors that may lead to errors in the *in situ* x-ray experiment employing the multi-anvil apparatus. A similar consideration for the diamond anvil experiments is required.

3.1. Temperature measurements

Since pressures are calculated on the basis of the P–V–T relationship of standard materials, precise temperature measurement is very critical. Temperatures are usually monitored by a thermocouple embedded in the high-pressure cell. However, owing to a temperature gradient in the cell, temperatures at the sample position may be somewhat different from those indicated by the thermocouple. Also, the effect of pressure on the emfs of the thermocouple may cause a significant difference in the temperature measurement, which is usually ignored in multi-anvil experiments. Nevertheless, for the post-spinel experiment mentioned above, the temperature had to be underestimated by nearly 400 K to shift the boundary toward higher pressures by 2.5 GPa, which was very unlikely even if both effects are considered.

3.2. Effect of non-hydrostatic stress

Even in the multi-anvil device, considerable uniaxial stress exists which depends on the cell design. This results in systematical variation of d-values with hkl Miller indices [29, 30]. Sometimes this leads to a serious error in the calculation of pressures. The variation of the d-value is a function of the angle between the major compression axis and the direction of detector, which complicates the situation. Thus, the uniaxial stress effect is large for materials with a small G/K ratio, where G and K are the shear and bulk moduli, respectively. Unfortunately, Au is known to be one of those materials. In addition, inhomogeneity in a two-material mixture potentially introduces non-hydrostatic stress. The stress–strain state of such mixture materials should be intermediate between the Reuss and Voigt limits; however, quantitative measurements in the high-pressure cell have not been made. Although these effects are very complex and are difficult to discuss quantitatively, they are expected to become very small at temperatures greater than 1000 °C. In fact, most phase relation experiments using SPEED-1500 revealed diffraction peaks of Au with different *hkl* Miller indices which were consistent with each other at high temperatures. Thus, evidence of anisotropy was not observed.

3.3. Energy dispersive method

Diffraction experiments using SPEED-1500 are performed by the energy dispersive method. The diffraction angle, 2θ, is fixed in this method and the present system has no sample oscillation mechanism. Thus, only diffracted x-rays from the lattice planes that happen to lie in a certain direction can be observed. As long as the particle size remains small compared with the x-ray beam size, the 'powder condition' is satisfied. When substantial grain growth in the sample occurs at high temperature, however, observed peak intensities become very anomalous, which could lead to misidentification of high-pressure phases or to inaccuracy in pressure determinations.

3.4. Kinetic effect

The most common procedure in which SPEED-1500 is used to determine the phase boundary is by observing changes of the diffraction profile during variation of P–T conditions. Many data points could be obtained in a single experimental run. In some cases, however, results were inconsistent and seemed to depend on various factors such as the holding time or the experimental P–T path. In order to minimize this kinetic problem, the 'one-shot method' has recently been attempted [7, 9]. In this method, the pressure and temperature are directly increased to target conditions and are then maintained for a certain time, taking diffraction profiles repeatedly to observe their variation with time. Although one experimental run provides only one data point, this method is very useful for determining the precise phase boundary, particularly for transitions in which the reaction occurs very slowly indicating a significant kinetic effect. The phase boundary in $MgSiO_3$ thus determined [7] was also several GPa lower than that observed in previous quench experiments.

The real situation must be a combination of all the factors listed above. Current multi-anvil experiments have not reached the stage at which enough precise quantitative data have been obtained for one to discuss these effects with accuracy. Further continuous studies are necessary, including an effort to establish the absolute pressure standard. Nevertheless, we would like to emphasize that it is very unlikely that these effects (except for the pressure standard issue) are large enough to explain the underestimation of pressure by 2.5 GPa. Therefore, we have so far no good reason to suspect the reliability of the post-spinel boundary based on the *in situ* multi-anvil experiment. Many other studies related to phase relation experiments using the multi-anvil apparatus at SPring-8 have been published [4–13]. Although high-pressure cell designs and experimental procedures differed between experiments, their final results were fairly consistent each other. This fact also supports the reliability of the *in situ* multi-anvil experiment. For details, please refer to the original papers.

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