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The present status of high-pressure research at Beijing Synchrotron Radiation Facility

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Abstract

The present status of high-pressure research at Beijing Synchrotron Radiation Facility is reported. A ten-poles wiggler beamline provides a white beam for investigating samples using a diamond anvil cell. *In situ* energy-dispersive diffraction is used to determine the pressure-induced phase transitions and equations of state. High pressure can be stably applied by a stepper-motorized loading system with a strain sensor. Some megabar experiments have been carried out without damage on diamonds. Improved beam collimation reduces the background and eliminates gasket scatter. Some research and future developments are also presented.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

The use of a diamond anvil cell (DAC) in energy-dispersive x-ray diffraction (EDXD) investigations has proved a powerful tool in the study of phase transitions and equations of state at high pressure. Because of the extreme spectral brilliance, small divergence, and wide range of wavelengths, synchrotron radiation (SR) has come to be considered the ideal x-ray source for EDXD. Since the first experiment by Buras [1] at DESY, several kinds of EDXD apparatus for DACs have been developed at SR facilities, e.g., CHESS at Cornell University (Baublitz [2], Bassett [3]), SPEAR at Stanford University (Skelton [4, 5]), and NSLS at Brookhaven National Laboratory (Jephcoat [6], Hu [7]). Recently, great developments have been achieved by combining a laser heated DAC with *in situ* x-ray diffraction at third-generation SR sources [8–12], which expanded the range of research under high pressure and high temperature.

The first EDXD apparatus with a DAC was developed at Beijing Synchrotron Radiation Facility (BSRF) in 1993 [13]. A single-pole wiggler, 4W1, of Beijing Electron–Positron Collider (BEPC) provides a white beam and is available part of the time for high-pressure research. After about three years of preliminary operation, the high-pressure station was moved to a new beamline, 3W1A of BSRF, in 1997 [14]. The ten-poles permanent magnetic wiggler

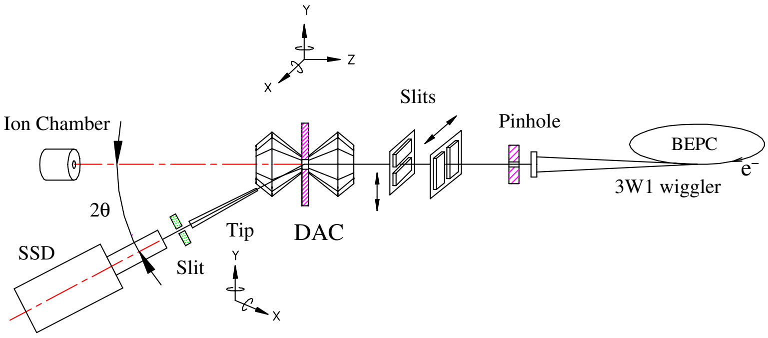

Figure 1. A schematic diagram of the high-pressure EDXD at BSRF.

provided about ten times the flux of x-rays in the 3.5–40 keV range compared to 4W1 [15]. The new station started operation in June 1997. Some megabar pressure EDXD experiments have been successfully carried out [16, 17]. In recent experiments the new diffracted beam collimator, designed to reduce the overall background, has led to improved diffraction patterns being obtained for some dilute samples.

2. EDXD apparatus at BSRF

A schematic diagram of the experimental arrangement for high-pressure EDXD at BSRF is illustrated in figure 1. 3W1A is a central branch of the ten-poles wiggler beamline, with an acceptance of 1 mrad in horizontal divergence. The polychromatic radiation passes through the pinhole and primary slits before entering the DAC. An ion chamber and remote scanning system are used to collimate the radiation beam at the electron orbit plane and ensure that the micro-beam only strikes the specimen.

X-rays diffracted from specimens are collected by an energy-selective solid-state detector and in a multi-channel analyser. A vertical slit of adjustable width in front of the detector defines a constant diffraction angle, 2θ. Due to the geometrical constraints of the DAC, 2θ must always be limited to within 0–35°. There is a collimation pipe (the so-called tip) between the pressure cell and detector, which can reduce the background radiation due to the scattering of x-rays by air and diamond.

3. Loading system

A novel high-pressure apparatus was designed for the 3W1A station (figure 2). A modified Mao–Bell DAC, which can generate and keep pressure higher than 100 GPa, is mounted upon a hardened support. The axial force is applied to the diamond anvils through the lever arm which is driven by the pull of the worm. The displacement of the worm is controlled by a stepping motor outside the radiation hutch, through the worm-gearing reducer. We attached two strain sensors on the lever arm. The strain of the lever can indirectly indicate the axial stress of the samples. In this way we can quantitatively control the pressure. In particular, the ability to decompress very slowly was effective in avoiding damaging the diamonds.

Figure 3 shows the relationship of the lever strain and pressure inside the gasket hole. The curve consists of four regions: (i) 0a: system relaxation; (ii) ab: linear region; (iii) bc: plastic deformation; (iv) d: breaking point. We always control the pressure in the linear region. One

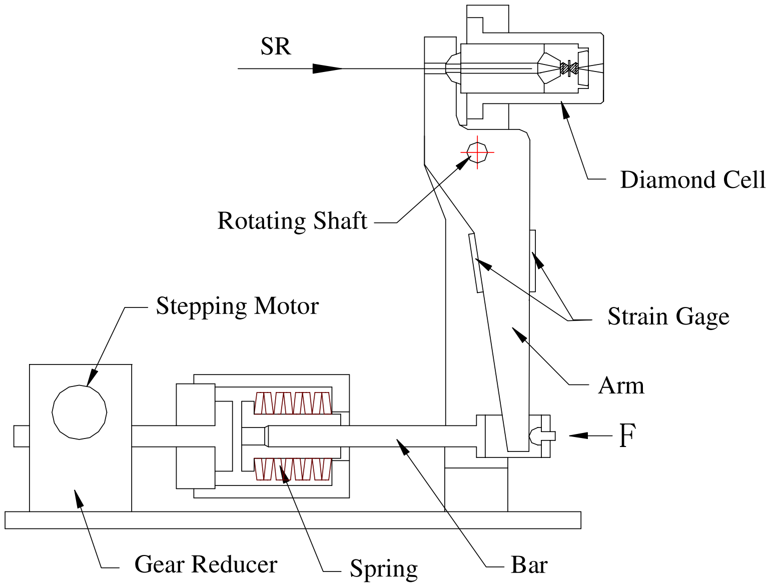

Figure 2. The DAC and loading structure.

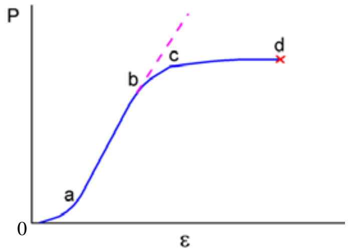

Figure 3. The relationship of lever strain and pressure.

never hesitates to stop compression if the nonlinear region appears. In this way experiments under pressure up to 103 GPa can be completed without damaging the diamond.

4. Research

During the last three years, some *in situ* measurements of phase transitions and equations of state have been carried out. The experiments were performed in the SR-dedicated mode of the BEPC. The electron energy was 2.2 GeV and the beam current was above 100 mA with a lifetime of about 20 h. This gives a sufficient intensity of x-rays for photon energy from a few keV to 40 keV.

Megabar pressure EDXD experiments on CsBr were carried out at 3W1A of BSRF by Wang *et al* [16]. The results indicated a phase transition from a simple cubic phase to a tetragonal phase at a pressure of about 53 GPa. Above 98 GPa, a colour change of CsBr from transparent to red–brown was observed. No pressure-induced transition from insulator to metal was found at higher pressure up to 115 GPa.

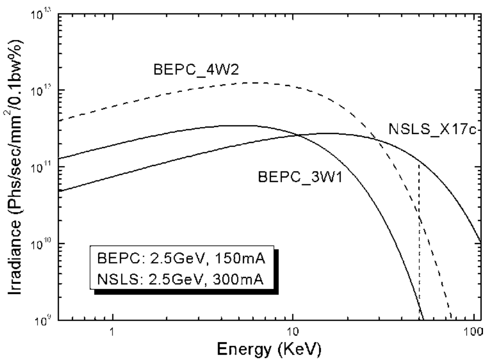

Figure 4. The spectral distribution for BEPC wigglers.

Zhao *et al* [18] have studied γ-Fe_2O_3 nanocrystals synthesized chemically by the BSRF nanomaterial research group. Two kinds of nanocrystal were coated with different stronger polar molecules on their surface. The diffraction by DBS-coated γ-Fe_2O_3, with grain size 10 nm, was studied under high pressure up to 85 GPa. A phase transition from γ-Fe_2O_3 to α-Fe_2O_3 was observed at 25 GPa. Measurements on CTAB-coated γ-Fe_2O_3 with grain size 6 nm indicated that the phase transition occurred at about 20 GPa. It is indicated that the transition pressure of nanocrystal γ-Fe_2O_3 is lower than that of the bulk. The bulk modulus of γ-Fe_2O_3 nanocrystal is 374 GPa [18], which is larger than that (203 GPa) for the bulk material.

The group from Superhard Materials Key Laboratory (SHMKL), Jilin University, has studied various nanomaterials at BSRF. Cui *et al* [19] determined the phase transition in anatase TiO_2 with average grain size 10 nm under pressure at room temperature. The results indicate that there is a phase transition from anatase to α-PbO_2 (TiO_2-II) at 16.3 GPa and it is irreversible. Comparing with the results on bulk material, the phase transition pressure for nanocrystal is higher. EDXD measurements on nanocrystal iron silicide have been performed using a DAC with SR by Liu *et al* [20]. Some new phenomena were found in the range of 27.1–31.5 GPa. Other nanomaterials, such as ZnO, GaN, ZrO_2, FeS, have been investigated by the same group. Some different pressure-induced behaviours, compared to bulk material, were observed.

Additionally, some high-temperature superconductors, amorphous bulk metallic glasses [21, 22], perovskite manganese oxides [23, 24] and novel optical function materials [25] have been investigated at the station.

5. Future

Recently, a new in-vacuum wiggler, 4W2, has been under design which will be installed in the quadrant section 4 of BEPC storage ring. The new wiggler will provide a one-order-higher flux of x-rays in the 10–50 keV energy region compared to 3W1. Figure 4 shows the spectral distribution of SR from BEPC wigglers. The curves indicate photons per unit area at the sample position. The irradiance from the X17C of NSLS at 2.5 GeV, 300 mA is also presented for comparison.

A new beamline will be built using the radiation from the 4W2 wiggler. It will be dedicated to high-pressure diamond cell research. In the new beamline we will arrange a K–B micro-focus system and monochromator. Angle-dispersive diffraction will be realizable. In the project of upgrading BSRF, the laser heating DAC technology will be developed. With these improvements a new level of high-pressure experiments will become possible at BSRF.

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