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An assesment of high pressure thermal measurement using synchrotron radiation

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Sample will be heated by absorption of strong X-ray from synchrotron. Such heating would certainly happen in a sample under pressure during high pressure synchrotron X-ray diffraction experiments. However, we have not notticed such temperature change, and this is likely due to very small change of temperature for such sample. In our previous high pressure X-ray radiographic study reported before, the author noted temperature jump of the sample under pressure when the main beam shutter is opened. Encouraged by this observation, we have conducted a preliminary experiment to sudy temperature change of metal samples during synchrotron irradiation under pressure, and the result is reported here.

The experiment was conducted at beamline BL04B1 at SPring-8. SPEED1500 multi-anvil press was used to generate high pressure and temperature. Experimental details are similar to our X-ray radiography studies. We chose two samples, Pt wire and Fe rod. For Pt, 0.2mm diameter Pt wire which is welded to a thermocouple junction. For iron, 2mm diameter iron rod with 2mm height is used. Temperature is measured by 0.2mm diameter R-type thermocouple. The thermocouple emf is monitored by a digital multi-meter, and the data are stored to PC.

We have conducted two experiments. One experiment in which iron rod was used as a sample, was done first at about 1GPa and room temperature. An emf change was about 5 microvolts when 1mm height slit was used. We could observe change as small as 0.25mm slit, however, the height of emf jump decrease rapidly. In order to try similar measurement at high temperature, temperature was increased up to 1000 C at 3GPa. We could not identify the signal at high temperature. This is because thermal fluctuation due to the heater and/or heating system was about 10 microvolts, an order of magnitude grater than that of the jump by synchrotron radiation. For Pt wire, we obtained similar results.

Present study demonstrated that temperature change due to irradiation of synchrotron radiation to metal samples is large enough to be observed under pressure. This might open the possibility of thermal physical properties measurements at high pressure. However, we also noted that such signal would be buried in thermal fluctuation at high temperature. In order to avoid this, more sophisticated temperature measurement is necessary. Adding to this, sample geometry should be optimized for better heat absorption. Once such improvement is done, the technique can be applied to thermal conductivity measurement, for example. Ordinary measurement involves second heater for thermal modulation of the sample. However, second heater is not easy to incorporate in high pressure cell, especially for very high pressure and temperature conditions. If additional heat can be given to the sample by means of synchrotron radiation, second heater is no longer necessary, and the cell would be simplified. The slits before the press and press height displacement can be used to change the shape and the place of the heat source, respectively. Adding to this, heat source to thermocouple distance can be accurately determined by means of X-ray radiograph

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