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X-ray diffraction and laser heating: application of a moissanite anvil cell

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Abstract

A moissanite anvil cell (MAC) has been used in research at high-pressure recently. In this paper, we describe its application in x-ray diffraction studies with laser heating. A high temperature of 3700 K has been achieved in a MAC; the lattice constants of graphite were determined by means of *in situ* x-ray diffraction at room temperature and 3700 K, and the results are in good agreement with reference data.

1. Introduction

A moissanite anvil cell (MAC) is suitable for use in x-ray diffraction experiments although the x-ray absorption of moissanite (single crystal hexagonal SiC) is greater than that of diamond. This makes it only workable with a synchrotron x-ray source [1]. Because a large-size MAC is available, it can be used for x-ray diffraction studies on a large amount of sample: it is especially useful for investigating light elements and their compounds.

Using diamond anvil cells, the high-pressure laser heating technique combines high-pressure and high-temperature conditions together [2]. Effective isolation is the key to achieving higher temperatures. Large-sized moissanite cells can be used in such circumstances.

For simplicity, the achievable temperature can be approximated as

$$cm(T - T_0) = tJ - [tks/h](T - T_0)$$

where c and m are the heat capacity and mass of the sample (graphite); T and T_0 are the temperatures of the sample and environment—the cell body; J is the energy density input from the laser; t is the total time for laser heating; s is the area of graphite; h is the thickness of the isolation layer; k is the heat conductivity of the isolation layer. The first term is the heat input and the second term is lost heat. Thus, the achievable T can be written as

$$T - T_0 = J/[(cm/t) + (ks/h)].$$

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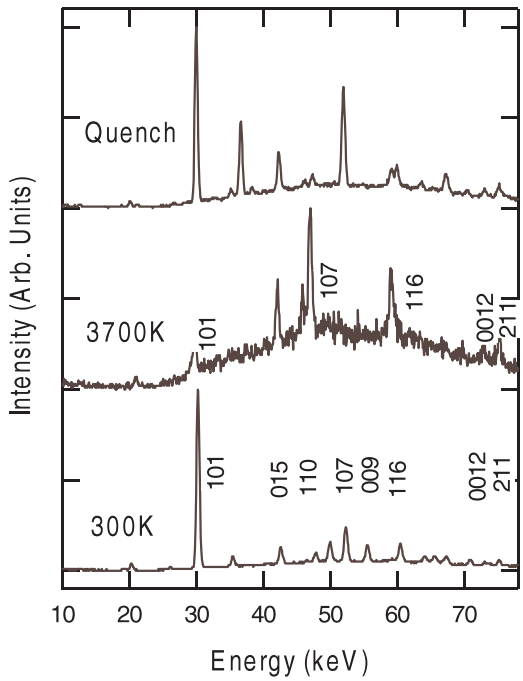

Figure 1. X-ray diffraction patterns of graphite at 3 GPa in a MAC at room temperature, 3700 K and after a quench.

In our experiment c is 0.7 J g^{-1} K^{-1}. The mass of the graphite sample m is around 1.4×10^{-5} g; t is about 300 s. The sample heating part cm/t is about 0.33 erg K^{-1} s^{-1}. However, on the heat loss side, k is 74 mW cm^{-1} K^{-1}; s is 3.14×10^{-4} cm^2; h is 0.015 cm. Hence ks/h is around 1.55 mW K^{-1} or 1.55×10^4 erg K^{-1} s^{-1}. Therefore, the term cm/t can be ignored. And at equilibrium, the input power is almost equal to the heat loss. Consequently, the equation can be simplified to

$$T - T_0 = (Jh)/(ks).$$

In order to reach higher T, we need larger J and h, and k and s as small as possible. However, here s is related to J. In order to keep enough energy input, s cannot be changed dramatically. The large moissanite anvil shows its advantage again in providing a large h-value.

2. Experiment

A special symmetrical Mao–Bell cell is used in this study. This kind of cell provides enough space to focus the laser beams from the front and back. A pair of 1400 μm culet moissanite anvils was mounted in this cell. A NaCl isolation body 500 μm in thickness and 600 μm in diameter (with a 200 μm graphite sample in the centre) was loaded in a MAC with several ruby chips on the top of the sample. The NaCl was also as the pressure medium. The pressure was determined by using the ruby scale. The sample was pressurized to 3.0 GPa. *In situ* synchrotron x-ray diffraction and double-sided Nd:yttrium–aluminium garnet laser heating were performed at the X17B beamline at the National Synchrotron Light Source, Brookhaven National Laboratory, USA. The experimental set-up is described elsewhere [2]. Because of

the large sample size, a 150 μm by 150 μm x-ray beam was used. The thermal body emission and x-ray diffraction spectra were collected every 3 min.

3. Results

According to the spectra of the thermal body emission obtained, the highest temperature achieved was 3700 K. Figure 1 shows the x-ray diffraction patterns of graphite at room temperature, 3700 K and after a quench. The 2θ diffraction angle is 7°. Compared to x-ray diffraction patterns obtained with a DAC using a synchrotron source, the onset energy in a MAC is shifted to 25 keV because of the stronger x-ray absorption of moissanite compared to diamond anvils. From these x-ray diffraction patterns, the lattice constants of graphite are determined as

Room temperature:

$a = 2.470 \pm 0.010$ Å; $c = 9.616 \pm 0.070$ Å; $V = 50.81 \pm 0.41$ Å^3

3700 K:

$a = 2.480 \pm 0.065$ Å; $c = 10.110 \pm 0.048$ Å; $V = 53.8 \pm 2.7$ Å^3.

Obviously, the average thermal expansion coefficients are thus determined as 1.18×10^{-6} for the parallel direction (a-axis), 15.1×10^{-6} for the vertical direction (c-axis) and 17.2×10^{-6} for the volume, respectively. They are basically in agreement with reference data: 0.66×10^{-6} and 29.2×10^{-6} along the a- and c-directions at 1500 K [3].

Acknowledgments

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References

[1] Xu J and Mao H K 2000 *Science* **290** 783

[2] Ma Y, Mao H K, Hemley R J, Gramsch S A, Shen G and Somayazulu M 2001 Two-dimensional dispersive x-ray diffraction at high pressures and temperatures *Rev. Sci. Instrum.* **72** 1302

[3] Gel'man E G 1997 *Handbook of Physical Quantities* ed I S Grigoriev, E Z Meilikhov and A A Radzig (Boca Raton, FL: Chemical Rubber Company Press) p 286