Melting of Troilite at High Pressure in a Diamond Cell by Laser Heating

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We have assembled a system which allows us to measure melting temperatures at high pressures. Figure 1 is a schematic diagram of our apparatus. The sample is heated with radiation from a YAG laser (λ = 1.06 um), either continuously or in a Q-switched mode. The Q-switch can be used to produce individual pulses of 100-200 nsec duration. The power of each pulse is of the order of 1,000 watts. The laser can be triggered by the computer (Fig. 1) at the same time that the photodiode array starts to collect a series of spectra. The beam is reflected downward through a microscope objective, through the upper diamond anvil, and focused onto the sample. The laser light is thereby strongly converging onto the sample, producing intense heating only at the sample and not within the diamond anvils. The heated area is ~10 um in diameter. A vidicon system is used to observe the sample during heating.

Incandescent light from the heated sample passes back through the objective lens into a grating spectrometer. The spectrum of the incandescent light is received by the photodiode array and stored in the multichannel analyzer. These data can then be transferred to floppy disk for analysis. A curve-fitting program is used to compare the spectra with standard blackbody curves and to determine the temperature.

Pressure is measured by the ruby fluorescence method. A frequency-doubling crystal is placed in the laser beam to produce green light having a wavelength of 530 nm. This light, when focused onto particles of ruby inside the diamond cell, excite fluorescence. The wavelength of the emitted red light can then be used to measure the pressure. The pressure is measured at room temperature by this method.

We have tested our apparatus by melting several different materials. These materials provide various criteria for determining whether or not melting has occurred. Our technique in these experiments is to bracket the melting temperatures of the materials. During each laser pulse a spectrum of the emitted incandescent light is collected and stored. It is necessary to remove the sample and look for evidence of melting using optical and electron microscopy. We have used various criteria in different samples to establish whether or not melting has occurred, including the formation of droplets from elongate fibers of platinum, formation of droplets from angular particles of silicon and diamond, formation of a groove with ridges on a diamond face, encapsulation of droplets of the pressure medium within diamond (Gold et al, 1984), and holes melted through tungsten foil. We have also inferred melting from electron diffraction patterns of samples (Weathers and Bassett, 1986).

The samples which are loaded into the diamond anvil cells consist of a mixture of small grains (<10 microns) of the actual sample (e.g., graphite, troilite, diamond, iron, etc.) and a pressure medium (e.g., LiF, NaCl, or KBr). Usually between 20 and 50 grains in each sample are heated using the laser radiation. The position of each heated grain is recorded on a photograph. When the sample is removed from the diamond cell, the pressure medium is slowly dissolved in alcohol so that the grains maintain their relative positions and can be identified from the earlier photographs. The dissolution of the pressure medium is done so that the sample is deposited on a grid which can be put directly into the electron microscope.

We are studying the melting behavior of natural troilite (FeS). Powdered troilite mixed with NaCl at ~200 kbar was laser-heated. Our initial transmission electron microscopy study showed that we successfully melted grains of the troilite, converting angular particles into spherical grains (Fig. 2). An example of a preliminary temperature determination is shown in Figure 3. A blackbody curve for 2800 K was fitted to the processed spectrum which represents a grain of troilite that melted at 5 kbar. Further analyses of temperatures obtained at various pressures, combined with determinations of whether or not the heated grains melted will allow us to bracket the melt curve for troilite.

Gold, J.S., Bassett, W.A., Weathers, M.S., and Bird, J.M. (1984) Melting of diamond, Science 225, 921-922.

Weathers, M.S., and Bassett, W.A., Melting of carbon at 300 kbar, submitted, Phys. Chem. Min.

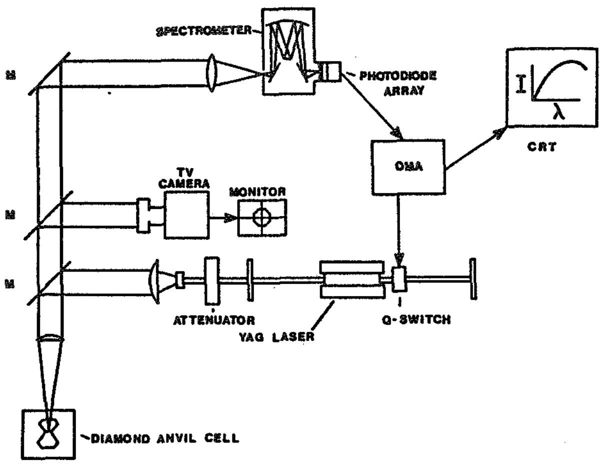

Figure 1. Diagram showing our experimental apparatus for melting materials at high pressures and determining temperatures. The YAG laser can be rapidly pulsed using the Q-switch. The intensity of the laser light is controlled by means of an attenuator. The laser beam is focused onto the sample in the diamond cell. A beam-splitter mirror allows a portion of the light to be used to monitor the sample during the experiment. The incandescent light given off by a heated sample is directed to the grating spectrometer and photodiode array. Spectra collected by the photodiode array are stored in the computer controlled optical multichannel anlyzer and are later processed to determine temperature.

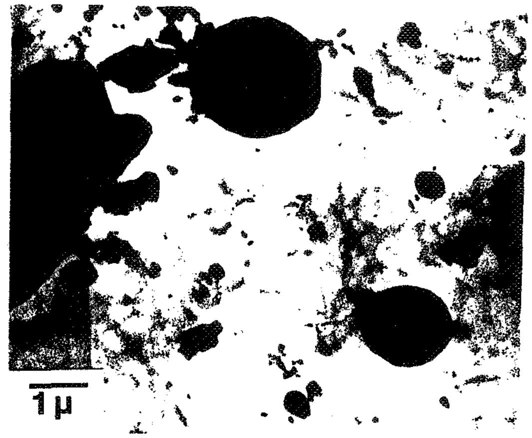

Figure 2. Transmission electron image of spherical grains of troilite (FeS). These grains, which were originally angular, were melted by laser-heating.

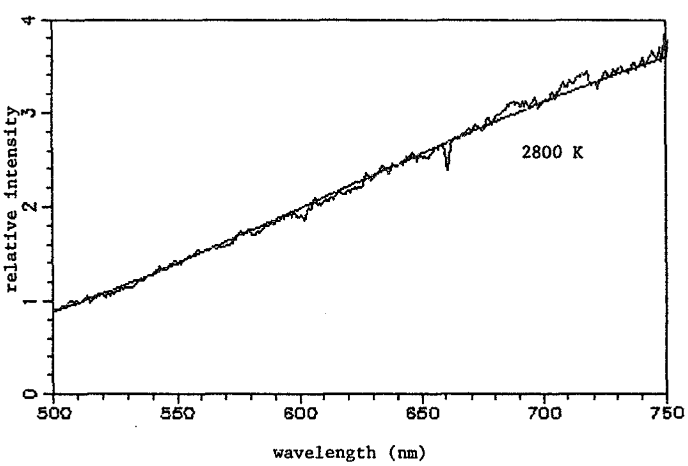

Figure 3. Processed spectrum from a heated grain of troilite fit to a blackbody curve of 2800 K. The pressure at the time of heating was 5 kbar. This spectrum is from a grain that showed evidence of melting.