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Report:

As iron is the dominant component of the Earth's core, information on its behavior at high-pressure and temperature is fundamental to Earth sciences. However, despite numerous study on this subject (see Anderson, 1996 for a review), there is still uncertainty about the crystallographic structure it adopts at the relevant pressures and temperatures. The accurate determination of the phase diagram of this element has remained an experimental challenge because of the extreme conditions involved. Shock-wave (Brown and McQueen, 1986) and diamond anvil cell (Boehler, 1993) techniques have both indicated that iron undergo phase transformation at high pressures and temperature, but stability field of the new phases are not well defined. In fact, recent reports are still conflicting about the 40-100 GPa region of the phase diagram, and it clearly needs clarification.

We present angle dispersive diffraction data on iron up to 60 GPa and 2500 K. We thus revisited the iron P-T phase diagram in this moderate pressure range with the best X-ray diffraction environment available at this time. Problems of recrystallization generally encountered at high-temperature have been largely avoided with the use of monochromatic radiation and imaging plates, These techniques represent a real improvement of the resolution of the measurements, making possible investigation of subtle structural details of the iron structure.

One of the principal difficulty of the study remains the pressure and temperature measurements in the laser hot spot. For this purpose, we use an optical system for on-line measurements during the X-ray diffraction experiments (see Fiquet et al., 1996). It is achieved with optical fibers guiding signals to spectrometer placed outside the hutch. The angle dispersive X-ray diffraction measurements used the high-pressure dedicated ID30 line at ESRF (Grenoble, France). The monochromatic X-ray beam (λ = 0.4245 A) was focused using two single-clcctrode bimorph mirrors to FWHM of about than 15*8 μm^2 (see Haüsermann et al., 1996). It is a most convenient size to study the central part of the thermal gradients produced in laser hot spots. Full reciprocal angle was collected using image plates located at 400 mm from the sample, acquisition time was between 10 to 15 minutes. Imaging plates were integrated along the circular symmetry using the usual precautions needed for this calculation.

We present in figure 1 diffraction patterns recorded before, during, and after the laser heating of iron at nominal pressures of 44.6 GPa. We reported expected positions for the hcp e-iron 100, 002, 101, and 102

dhkl Bragg lines. The high temperature spectra show significant deviations from the hcp lattice, with mostevident feature being a strong diffraction line located at around 2.35 A. This feature is clearly reversible after the laser heating. This effect shows that these new lines are not due to reaction with transmitting medium or iron oxidation, The deviations are due to change of the e-iron structure to an high-pressure high-temperature polymorph. It is most probably same phase than that previously reported by Saxena et al. (1995). However, our spectra do not correspond to D-hcp lattice previously proposed. We instead propose that iron adopts an orthorhombic unit cell in these P-T conditions (see Andrault et al., 1997).

The quality of the data was sufficient to Rietveld refine the high-pressure high-temperature spectra. Indeed, peaks intensities could be used as a quantitative information, since spectra were recorded in an angle dispersive mode. The full structure determination of iron at 44.6 GPa and 1964 K was successful. Fits are presented in figure 2. This determination is a first step in opening a new field for the extreme condition crystallography. Fine determination of structural changes in extended P-T field is now possible.

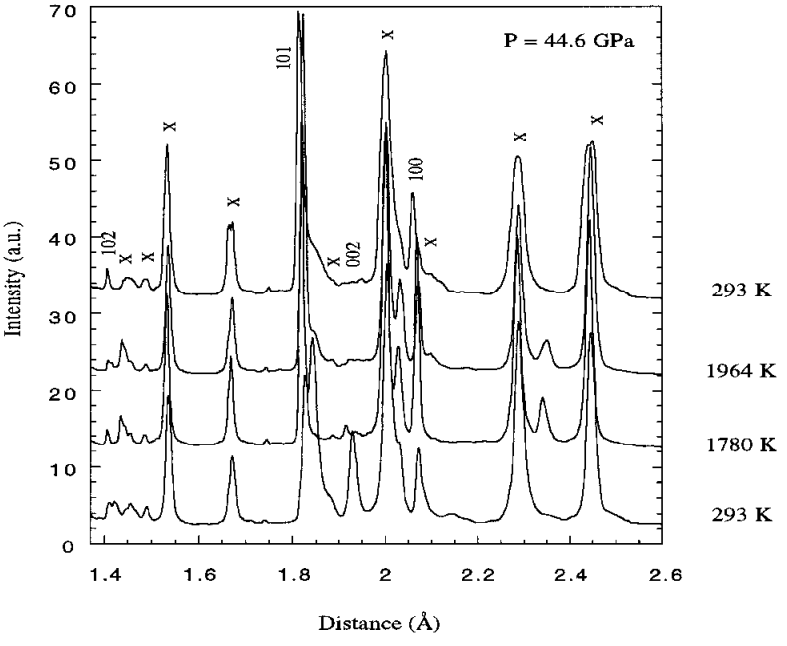

Figure 1 : Typical laser heating sequence of angle dispersive diffraction spectra recorded at 44.6 GPa. Diffraction peaks referenced as X correspond to the corundum pressure transmitting medium. Bottom and top spectra correspond to e-iron after slight laser heating and quenched spectra respectively. New features which do not correspond to e-iron clearly appear during laser heating. Among them the occurrence of strong Bragg lines at *2.03* and 2.35 Å, or the disappearance of the hcp 002 line. This changes cannot be preserved through quenching.

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