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High Pressure X-ray Diffraction Study of Nanocrystalline Iron

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Beamline(s): X17C

Introduction: Nanocrystalline iron (n-Fe) has been the subject of many experimental and theoretical studies. Investigations on n-Fe have reported magnetic, mechanical, thermal, and electrical properties, surface passivation, and mechanical properties such as ductility, hardness and phonon density-of-states. Here we report the results of high pressure x-ray diffraction experiments, performed to measure the equation of state of n-Fe. The α–ε phase transition has also been investigated.

Methods and Materials: Nanocrystalline iron was made by mechanical attrition of Fe of 99.995% purity. The material was milled in a Spex 8000 mixer/mill with a hardened steel vial, lid and balls and a ball-to-powder weight ratio of 20:1. Powder iron of 10nm were loaded in a Mao-Bell type diamond anvil cell. A small piece of gold was included to calibrate the pressure. A mixture of 4:1 methanol:ethanol was loaded into the sample compartment to reduce the nonhydrostatic stresses. X-ray diffraction data were measured from 0 to 46 GPa, using x-ray diffraction at beamline X17C of NSLS. The diffraction data was acquired in energy dispersive mode, using liquid nitrogen cooled Ge detectors positioned at $2\theta = 9.958 \pm 0.002^0$ and $2\theta = 9.893 \pm 0.003^0$.

Results: Three x-ray diffraction lines, 110, 200 and 211 for α-Fe and the lines, 100, 002, 101, 102 and 110 lines for ε-Fe were obtained at each pressure. (Fig. 1). The pressure dependencies of the unit cell volume of n-Fe from 0 to 46 GPa are summarized in Fig. 2. Fits to the data yield $K = 170.6 \pm 5.2$ GPa, and $K' = 2.93 \pm 0.83$ for quasi-hydrostatically compressed n-Fe of α phase. X-ray diffraction of n-Fe at ambient condition gives its zero pressure unit cell volume, $V_0 = 23.6$ Å^3 , which is the same as that of bulk α-Fe. For the ε phase, we get $K = 179.4 \pm 8.1$ GPa, and $K' = 3.57 \pm 0.65$. The extrapolated zero-pressure unit cell volume of nanocrystalline ε-Fe is 22.9± 0.2 Å^3, compared to 22.3±0.2 Å^3 for large-grained ε-Fe. It has been found that α-ε phase transition of n-Fe starts at ~18.8 GPa and completes at ~ 24.5 GPa. The ε-α phase transition happens at around 5 GPa.

Conclusions: X-ray diffraction measurements were performed on nanocrystalline iron up to 46 GPa. For nanocrystalline α- and ε-Fe, respectively, analysis of lattice parameter data provides a bulk modulus, K, of 171±5 GPa and 179±8 GPa, and a pressure derivative of the bulk modulus, K', of 2.9±0.8 and 3.6±0.7, similar to the large-grained control sample. It has been found that the α-ε phase transition pressure of n-Fe is higher than that of bulk iron while the ε-α phase transition happens at a lower pressure than that for its bulk counterpart.

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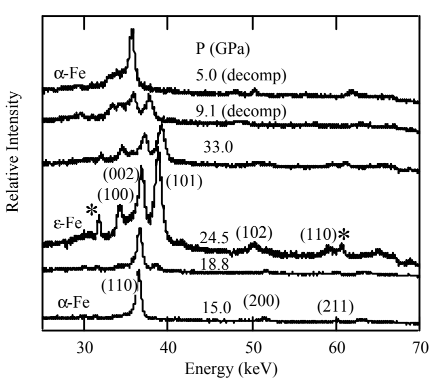

Fig. 1. Representative x-ray spectra. The peaks labeled with an asterisk are from gold.

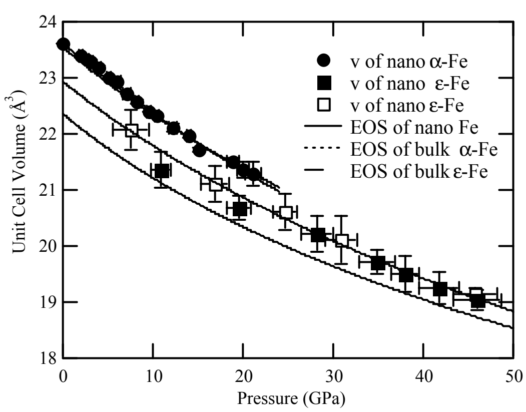

Fig. 2. Pressure dependence of the unit cell volume for n-Fe. Closed symbols represent compression data, while the open ones represent decompression data