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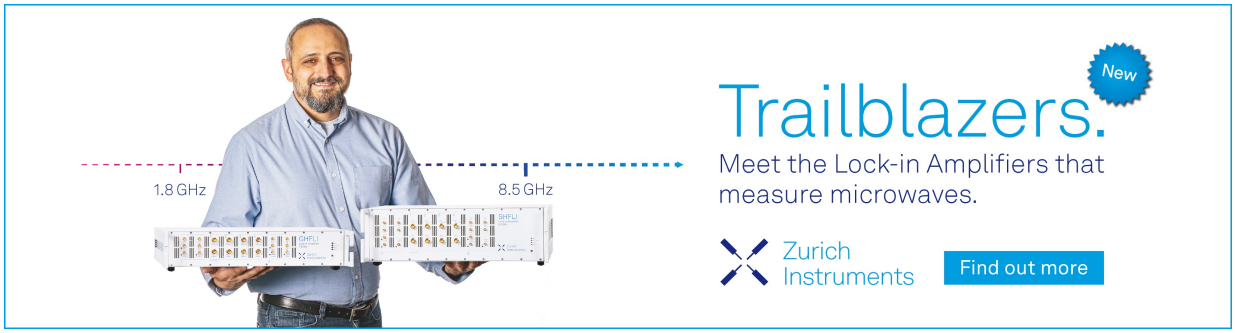

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Femtosecond laser quenching of the ϵ phase of iron

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The quenching of the ϵ phase of iron, which has not been observed under a conventional shock compression, was attained using a femtosecond laser. The crystalline structure in a recovered iron sample was determined using an electron backscatter diffraction pattern system. The femtosecond laser driven shock may have the potential to quench high-pressure phases of other materials.
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Iron is one of the most important materials in the field of both industry and geophysics because iron is considered the principal element in the Earth's core.[1] The lower pressure and temperature α phase of the bcc structure of iron transforms at higher temperatures to the γ phase of the fcc structure and at higher pressures to the ϵ phase of the hcp structure. The existence of the β phase of iron at higher pressures and higher temperatures and its crystalline structure have been discussed in this decade.[2–5] Another α' phase was also suggested above 200 GPa in shock experiments.[6,7]

A shock induced $\alpha \leftrightarrow \epsilon$ phase transition in iron is one of the most famous transitions under high pressure.[8] It was first reported by Minshall,[9] and the Hugoniots in these two phases were measured first by Bancroft *et al.*[10] Shock-wave temporal profiles have been measured which show that approximately 180 ns are required to complete the shock-induced transition from α to ϵ iron.[11] This phase transition is very sluggish, especially when compared to the few-nanoseconds pulse duration of the laser-driven shock, and suggests that the α–ϵ transition might be quenched. This phase transition was argued martensitic because this transition is an athermal process and the quantity of the phase does not vary as a function of time but pressure, although the question of the mechanism of this transition is still open.[12] In addition, the quenched ϵ phase after shock unloading has not been observed, so that the shock-induced α–ϵ phase transition in iron has been considered diffusionless and completely reversible. For an irreversible type of phase transition, however, a high pressure phase can be quenched as a metastable state after unloading. For instance, diamond is synthesized from graphite using the shock compression and rapid quenching method.[13,14]

Laser is absorbed, and thermal conduction occurs into the sample at supersonic velocities before mass has time to ablate. Eventually ablation occurs and drives a mechanical shock into the material.[15–17] Eventually the thermal front cools and its velocity drops below that of the following ablation shock. The shock front passes the thermal front and the shock then traverses unheated material. The amplitude of the shock pressure decreases continuously to zero with increasing propagation distance. However, the region behind the shock front remains in thermal equilibrium.[18,19] Thus, ϵ-iron is made by such a conventional shock wave.

The pulse width of the laser used in the present study is 120 fs. We illustrate how short the laser pulse is by relating the 120 fs time width to the vibrational period of a phonon at the Debye temperature. The Debye temperature of ϵ-iron at high pressures is approximately 700 K.[20] The vibrational period of this phonon is 7×10^{-14} s, which means that 120 fs is the time it takes for only two phonon vibrations. This is not expected to be long enough to drive a phase transition, which is evidence that the femtosecond laser pulse itself does not synthesize ϵ-iron. As described earlier, on the other hand, this implies that the α to ϵ transition is driven by the conventional shock wave that forms.

Even though high pressure and/or high temperature states are anticipated to occur in a region which is influenced by the femtosecond laser irradiation, phase transitions induced by the irradiation have never been investigated. The purposes of this letter are, first, to observe the femtosecond laser induced phase transition in a recovered iron sample and, second, to determine the high pressure and/or high temperature phases crystallographically. The electron backscatter diffraction pattern (EBSP)[21,22] system was used to determine the crystalline structure in a recovered sample.

Polycrystalline iron (purity: 99.99%) was annealed at 1200 K under a low pressure of 10^{-3} Pa for 72 h to increase the size of the crystalline grains. The obtained grain sizes were on the order of a millimeter. The laser pulses can be irradiated on the single crystalline grain surface because the spot diameter of the laser beam of $\sim$50 μm is small enough compared to the grain sizes. The irradiation on the mirror polished surface was performed in an argon atmosphere (the purity of the argon gas used was 99.99%) using the femtosecond laser (Spectra-Physics Inc., Spitfire; wavelength: 800 nm, pulse width: 120 fs). A groove ($\sim$50 μm width and $\sim$100 μm depth) was generated on the surface by scanning the laser beam with a Gaussian profile of the intensity of 8.0×10^{13} W/cm^2, of which 2000 pulses were directed to a point on the groove surface. A cross section of the sample perpendicular to the groove was polished after having been embedded in resin. FE-SEMs with minimum spatial resolution of 10 nm (Japan Electron Optics Laboratory Co., Ltd., JSM-6500F) and equipped with EBSP systems (Oxford Instruments plc and TexSEM Laboratories Inc.) were used to determine the crystalline structure in a recovered iron sample. The electron beam was focused on a relevant part of the polished surface, and the orientations of the backscat-

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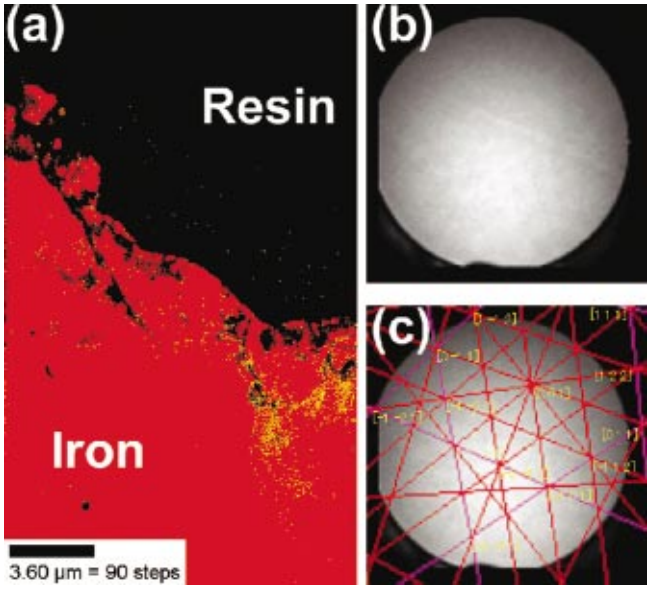

FIG. 1. (Color) (a) EBSP mapping data of iron sample, where red, yellow, and green colors indicate the α, ϵ, and γ phases, respectively. (b) The diffracted channeling patterns in a region where the grains of the ϵ phase exist. (c) The determined orientation for each pattern. Red- and purple-colored lines indicate patterns originated from the α and ϵ phases, respectively.

tered diffracted channeling patterns were determined using the EBSP system.

The results of the EBSP analysis are shown in Fig. 1. Figure 1(a) illustrates the EBSP phase mapping data, where red, yellow, and green colors indicate α, ϵ, and γ phase, respectively. The black area indicates the resin or the area where any patterns were not detected. Crystalline grains of the ϵ phase, whose typical size is several nm, are scattered around 4 μm deeper than the bottom surface of the groove. There are no indications of the presence of any γ phase. The diffracted channeling patterns in an area including the ϵ-phase grains are shown in Fig. 1(b). The orientation was determined for each pattern as shown in Fig. 1(c), where red- and purple-colored lines indicate patterns originated from the α and ϵ phases, respectively. Patterns originated from the ϵ phase are confirmed in the figure. This result also contributes to the evidence for the quenching of the ϵ phase and the absence of the γ phase, indicating that this α to ϵ transition was attained by a shock compression.

Since states on the Hugoniot are in thermodynamically equilibrium, temperatures along the Hugoniot are calculated from the result of Walsh and Christian[23–26]

$$C_V\frac{dT}{dV}+\frac{\gamma}{V}C_VT=\frac{1}{2}\frac{dp}{dV}(V_0-V)+\frac{1}{2}(p-p_0), \qquad (1)$$

where p is pressure, V is specific volume, T is the shock temperature behind the shock front, C_V is constant-volume specific heat, γ is the Grüneisen parameter, and the subscript 0 refers to an ambient state. The shock temperature T can be accurately calculated using this equation. We use the refined Hugoniot data of Brown *et al.*[27] and $C_V=3Nk_B+\Gamma_0(V/V_0)^g$, where N is the number of atoms per unit mass, k_B is the Boltzmann constant, $\Gamma_0=0.091$ J/kg K^2, and $g=1.34$, of Brown and McQueen,[6] and assume that γ/V is constant to calculate the shock temperature T.

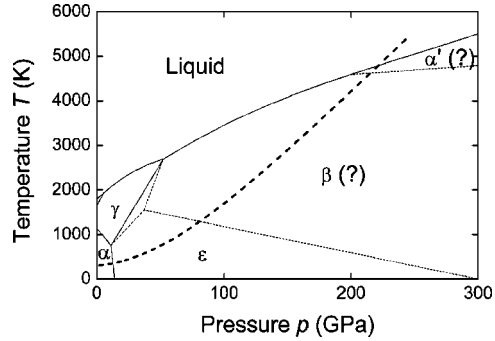

FIG. 2. Calculated temperature Hugoniot $T(p)$ (heavy dashed curve). Dashed lines indicate the γ–ϵ, γ–β, ϵ–β, and β–α' boundaries, assuming the β and α' phases exist.

The obtained temperature Hugoniot $T(p)$ is shown as the heavy dashed curve in Fig. 2, where we apply the data of Bundy[28] to the triple point of α–γ–ϵ phases, Anderson and Isaak[29] to the γ–ϵ-liquid triple point and melting curves, and Saxena and Dubrovinsky[30] and Brown[7] to the γ–ϵ–β and β–α'-liquid triple points, respectively, assuming that the β and α' phases exist. The temperature Hugoniot $T(p)$ is below the transition point to the γ phase. Therefore, the shock-induced temperature-rise does not drive the α to γ transition, which is consistent with the experimental observations. The temperature Hugoniot passes through the ϵ–β and β–α' boundaries, assuming the β and α' phases exist. This means that the femtosecond laser driven shock may possibly quench the β and α' phases as well as the ϵ phase.

In conclusion, the ϵ phase of iron was quenched using the femtosecond laser. The calculated temperature Hugoniot indicated that the femtosecond laser driven shock might also quench the β and α' phases, assuming they exist. The femtosecond laser driven shock may have the potential to quench the high-pressure phases of other materials.

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