Structure of β-iron at High Temperature and Pressure

D. Andrault *et al.* (*1*) conducted an in situ x-ray study of heated iron at high pressure (*P*) and found a structural transformation of ε (hexagonal close packed, hcp) iron to another phase, which they assign to an orthorhombic lattice. There are two problems with this conclusion. First, their method, applying structural refinement for the purpose of "quantitative assessment of a structural model," is invalid. Second, we question whether phase analysis of collected x-ray patterns can be interpreted as a mixture of known iron phases [ε (hcp), or β (dhcp, double hexagonal close packed), or both], iron oxide, and pressure medium.

X-ray data (*1*) are unsuitable for quantitative structural refinement because of (i) preferred orientation effects, (ii) large temperature (*T*) gradients in the sample, and (iii) stress effects. One condition for a successful application of the Rietveld refinement of crystal structures using powder x-ray diffraction data is the random distribution of the fine (small) crystallites in the sample (*2*). In other words, the Debye rings collected on imaging plate detector must be clear and smooth. The patterns collected at high *T* and *P* [figure 1 in (*1*), for example] demonstrate highly spotty discontinuous lines; many spots on those lines have their own shape and substructure, which means that crystallites in such samples are not small enough. The crystallites have uneven distribution, different shapes, and different orientation with respect to stress axes in the diamond anvil cell (DAC).

In diamond-anvil cell (DAC) experiments, especially with solid mediums such as those described in the report (*1*), samples have preferred orientations. Andrault *et al.* (*1*) do not describe how they take this effect into account (*3*). The preferred orientation not only decreases quality of powder diffraction data and increases uncertainty in the results of structural refinement, but also makes the reliability of the structural model doubtful. For example, they (*1*) stress the point that (002) ε-Fe (hcp-Fe) lines disappeared after heating (for example, when conditions for recrystallisation were created). Such behavior of hcp metals is common and can be explained by an alignment of crystallites with the *c* axis parallel to the load direction (*4–7*). Therefore, "the absence of the 010, 001, and 011 reflections, and the presence of the 100 reflection" could reflect the existence of strong preferred orientation effects and does not justify a selection of possible space groups for structural models (*8*).

Andrault *et al.* (*1*) state in their report, "artifacts due to pressure or temperature gradient (spatial or temporal) are excluded." Note that with Nd:YAG laser and 15*8 μ^2 FWHM X-ray beam, radial *T* gradients of 100 to 200 K are difficult to avoid (*9*). But more important is the vertical (axial) *T* gradient. Nd:YAG laser radiation is completely absorbed in the first several dozen nanometers of iron, and the rest of the metal (>99%) is heated only by thermal conduction (*10*). Andrault *et al.* (*1*) state that, as a result of heating diffraction, peak widths increase 1.4 to 3.5 times as compared with those of ambient conditions for Si-standard. According to the equation of state of iron (*11*), this finding could be a result of a significant *T* gradient (400 to 500 K). Moreover, the *P* medium (corundum) next to the diamond-sample interface is cold, and *T* gradient within corundum could be as high as 1500 to 1800 K at ∼2100 K. Most of the iron reflections partially overlap with corundum reflections and, as a result, structural refinement of powder data should be done for metal and *P* medium simultaneously. The GSAS program (*12*) used for structural refinement in the report (*1*) does not include options to take into account high *T* gradients within the samples. Resulting orthorhombic symmetry of iron obtained in the report (*1*) could be an artifact resulting from significant *T* gradients in the sample.

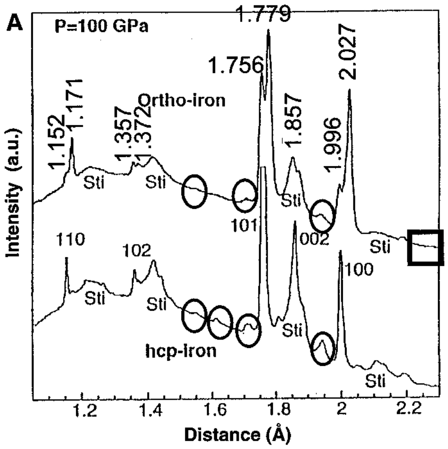
It is important to consider deviatoric stress in interpretation of all results of DAC experiments. It was shown that in iron, the uniaxial stress component *t* reaches a value ∼10 GPa at a *P* range of 50 GPa (*5, 6, 13*). With the use of recently developed theory of diffraction from specimen compressed nonhydrostatically in an opposed anvil device (*14*) and elastic moduli (*15*), we calculated the positions of the diffraction lines of ε-Fe at 50 GPa and various *t*, from 5 to 10 GPa. We found that, as a result of deviatoric stress, ideal hexagonal hcp lattice of ε-Fe looked like orthorhombic, with a *b*/*a* ratio of 1.74 to 1.745 (the ideal ratio for hexagonal lattice is 1.732; Andrault *et al.* obtained a value of 1.766). Therefore, the iron orthorhombic lattice found in the report (*1*) could be a result of the application of an incorrect fitting procedure for the samples under stress conditions (*16–18*).

Turning now to the second problem with this report, Andrault *et al.* (*1*) do not present clear hcp-Fe patterns. Bottom lines in figure 2 in the report (*1*) already contain at least two additional features at 1.85 and 2.03 Å. They mention that those reflections "are due to initiation of the transformation of ε hcp iron toward a high-temperature polymorph" and incomplete transition to a new phase. At 100 GPa (Fig. 1A), there are even more unexplained features on the pattern that Andrault *et al.* have marked as "hcp-iron" (*19, 20*), which raises the question whether Andrault *et al.* (*1*) had pure hcp-Fe at any *P*; they do not present any data on the unheated samples. The additional features are quenchable at high *P* [see figures 2 and 4 in (*1*)]. The corundum lines, for example, (104) (∼2.44 Å), (110) (∼2.27 Å), and (113) (∼1.99 Å), are much broader after heating and look almost like doublets. Moreover, according to Andrault *et al.* (*1*) "the features are mostly unquenchable." We would not expect the high-*P*,*T* phase to remain the same after decompression. So, if the new features on diffraction patterns are only "mostly" unquenchable (meaning that some high-*P*,*T* reflections continue to show after quenching), we have to question whether the sample has changed by possible chemical reactions.

Three of four new lines (1.44, 2.03, and 2.35 Å) appeared at high *T* (Table 1) at 44.6 GPa [cold *P* according to the report (*1*)] correspond to iron oxide FeO with B1 structure [lattice parameter *a* = 4.070 Å (*3*) corresponds to 48 GPa cold *P*, according to (*11*)]. At *P* higher than 16 GPa and room temperature, wustite transforms to a phase with rhombohedral lattice (*21*). The reflections of this rhombohedral phase (Fig. 1B) shows almost completely overlap with corundum (104), (110), (113), and (108) lines. Therefore, appearance and disappearance of lines at 1.44, 2.03, and 2.35 Å during heating and cooling at 44.6 GPa [figure 2 in (*1*)] is just a result of the transformation of rhombohedral FeO to cubic and vice versa (*22*) and not a result of the presence of an orthorhombic iron phase. That leaves us with only one reflection (1.85 Å) (or, equally, shoulder [see Figure 2 and 5 in (*1*)], as it was observed in our laboratory (*23*)] unexplained in terms of

Table 1. Possible indexing of the diffraction lines recorded at 44.6 GPa and 2125(70) K and described as iron lines by Andrault *et al.* [table 1 in (*1*)]. Columns with ε-Fe and dhcp-Fe taken from the report (*1*) and with the sign "+" indicate some experimental features which cannot be explained by the occurrence of ε-Fe.

Experiment (*1*)	ε-Fe (hcp)	β-Fe (dhcp)	FeO (B1)
2.347	+	+	111
2.072	100	100	
2.031	+	004	200
1.846	+	102	
1.824	101	+	
1.440	+	104	220
a, Å	2.393	2.393	4.070(3)
c, Å	3.845	8.126	



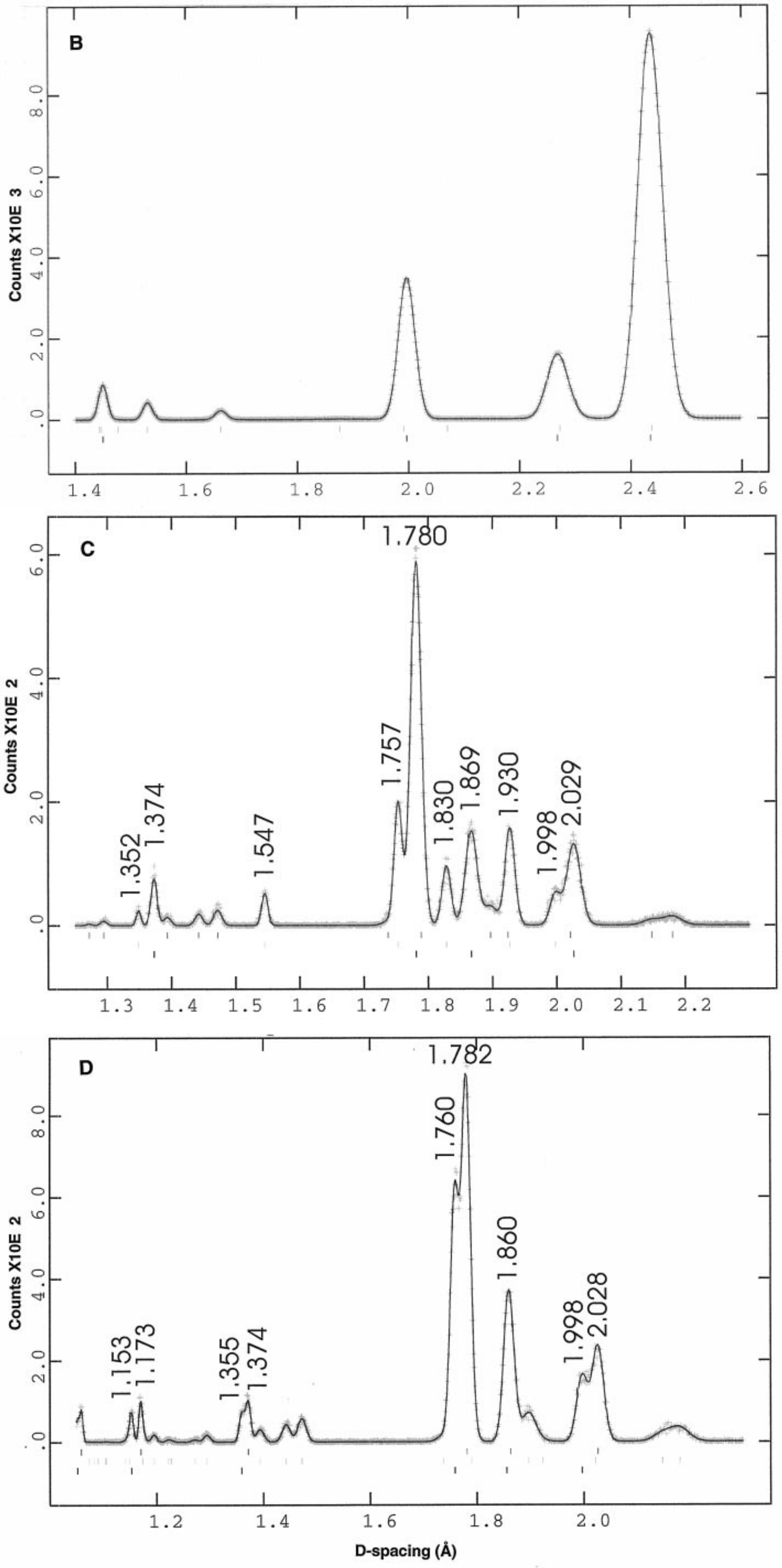


Fig. 1. (**A**) Diffraction spectra of iron and silica collected before (bottom line) and after laser heating [reproduced from figure 4 in (*1*)]. Values of d-spacing taken from table 2 in (*1*). Square marks the area where one could expect the exclusive (100) orthorhombic iron reflection. Circles denote features that were not explained by Andrault *et al*. (*1*). Silica reflections (for example, around 2.1 Å) are quite broad and appear not to be single peaks. Arbitrary units, a.u. (**B**) Calculated diffraction pattern of a mixture of corundum at 45 GPa (upper marks, $a = 4.5453$ Å, $c = 12.4197$ Å) and rhombohedral FeO ($a = 2.7545$, $c = 7.3045$ Å). Positions of corundum and FeO reflections are close; they are broad and may even be split reflections as seen on the top line in figure 2 of (*1*) (iron and corundum sample after laser heating at 44.6 GPa), which could be a result of partial oxidation of iron and presence of rhombohedral FeO in the sample. (**C**) Calculated diffraction pattern of a mixture of hcp-Fe (bottom marks), dhcp-Fe (middle marks), and silica with CaCl$_2$-like structure (top marks) (*19*). Major features in Fig. 1A, including small reflections ~1.93 Å and ~1.53 Å, could be explained by this model. (**D**) Calculated diffraction patterns of mixture of ε-Fe (hcp) at $P = 98$ (bottom marks) and 72 (top marks) GPa (*11*), and silica with CaCl$_2$-like structure (middle marks) (*19*). Difference in P could be a result of a drop in P locally at the laser heated spot, or a result of phase transition in silica, or both. This model explains major lines presented in figure 1 and Table 2 in (*1*), but cannot explain, for example, small lines ~1.93 Å and ~1.53 Å.

mixture of corundum, ε-Fe and iron oxide. This line was observed in our previous experiments with iron (*23–25*) and has been explained as the most intensive (102) dhcp-Fe (double hexagonal close packed) line, which could appear alone in the case of an incomplete transformation from hcp to dhcp structures, or which could be a result of effects of preferred orientation (*26*).

An analysis of the pattern collected at 100 GPa (*1*) is more difficult because (i) the quality of the data from the quenched sample is not precise; (ii) the description of the data in the report is schematic [for example, see the position of the silica reflections; some reflections on the pattern are not described or not explained, see figure 1a in (*1*)]; and (iii) crystal chemistry of silica and Fe-O system at extremely high-*P*,*T* is poorly known. But the "exclusive orthorhombic" (100) reflection (near $\sim$2.3 Å) is absent (Fig. 1A) (*27*), and most of the features could be qualitatively explained by the mixing of hcp-Fe, dhcp-Fe, and silica with $CaCl_2$-like structure (Fig. 1C). A less preferred alternative is that *P* dropped on the locally heated spot and the x-ray pattern is for hcp-Fe at different *P* in the sample (Fig. 1D) (*28*). Note that the model with a mixture of hcp- and dhcp-Fe could explain, for example, the reflection $\sim$1.93 Å [dhcp-Fe (101)], which was not explained by orthorhombic iron structural model.

On the basis of discussion above and our interpretation of the data, we conclude that data of Andrault *et al*. (*1*) do not provide evidence of the existence of iron with orthorhombic structure, but instead support the existing data on the transition from ε-Fe to β-Fe, which probably has a dhcp structure (*23–25, 29*).

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References and Notes

1. D. Andrault, G. Fiquet, M. Kunz, F. Visocekas, D. Häusermann, *Science* **278**, 831 (1997).
2. J. E. Post and D. L. Bish, *Rev. Mineral.* **20**, 227 (1989).
3. We were not able to reproduce, for example, relative intensities of reflections for orthorhombic iron shown on figure 5 in (*1*) without assuming strong preferred orientation in *c* direction.
4. L. S. Dubrovinsky, S. K. Saxena, P. Lazor, *Geophys. Res. Lett.* **24**, 1835 (1997).
5. H. K. Mao, Y. Wu, L. C. Chen, J. F. Shu, *J. Geophys. Res.* **95**, 21737 (1990).
6. R. J. Hemley *et al.*, *Science* **276**, 1242 (1997).
7. Figure 4a in (*6*) shows that while (002) hcp-Fe reflection is absent in the pattern obtained by axial diffraction [such type of set up was used in (*1*)], it becomes the strongest peak in the radial measurements, with $\psi = 0^o$ indicating a strong preferred orientation effect on hcp-Fe in the load direction.
8. Figure 1 in (*1*) shows several spots at 2θ-angles lower than those of first corundum line. Andrault *et al*. (*1*) do not describe and do not discuss them.
9. P. Lazor, G. Shen, S. K. Saxena, *Phys. Chem. Min.* **20**, 91 (1993); S. K. Saxena and L. S. Dubrovinsky, in *Advanced Materials '96, Proceedings of the 3rd NIRIM ISAM '96*, Tsukuba, Japan, March 4–8, 1996, pp. 137–142.
10. M. Manga and R. Jeanloz, *J. Geophys. Res.* **102**, 2999 (1997).
11. S. K. Saxena and L. S. Dubrovinsky, *U.S. Japan Seminar on High Pressure-Temperature Research: Properties of Earth and Planetary Materials, Jan. 22–26, 1996* (American Geophysical Union monograph), in press.
12. A. C. Larson and R. B. Von Dreele, Los Alamos National Laboratory LAUR pub. no. 87545 (1994).
13. According to Andrault *et al*. (*1*), (002) line of ε-Fe at 44.6 GPa and 293 K in their experiment with corundum *P* medium located at 1.93 Å, which corresponds the *P* 34 GPa [4,5,11].
14. A. K. Singh, *J. Appl. Phys.* **73**, 4278 (1993); ______ and C. Balasingh, *ibid.* **75**, 4956 (1994).
15. H. Mao, J. Shu, R. J. Hemley, A. K. Singh, *National Synchrotron Light Source Activity Report*, B-148 (1997); P. Söderlind, J. A. Moriarty, J. M. Wills, *Phys. Rev. B* **53**, 14063 (1996).
16. The GSAS program does not have options to describe the effects of deviatoric stress on the powder diffraction data (9).
17. While the heating of the sample could release the stresses, this would not happen with laser heating as used in (*1*) because (i) the heating is not homogeneous, and (ii) with increasing temperature, shear modulus of iron decreases and the mechanical and thermal stresses transmitted from corundum would produce higher strains. For more discussion of stresses in laser-heated DAC, see D. L. Hainz, *Geophys. Res. Lett.* **17**, 1161 (1990); A. B. Belonoshko and L. S. Dubrovinsky, *Am. Mineral.* **82**, 441 (1997). Figure 6 in (*1*) shows "an estimate of the pressure-temperature path of iron during laser heating under pressure" (the basis of such an estimation is not clear from the report). According to that estimation, hydrostatic component of *P* increases by $\sim$10 GPa, while *T* increases to 2000 K. In term of stresses, it means an increase of *t* of $\sim$30 GPa as a result of laser heating.
18. Figure 5 in (*1*) contains at least three not explained small reflections at $\sim$14° and 17.5°, and orthorhombic iron structural model predicts incorrect positions for reflections $\sim$17°, 21.5° and 24°.
19. Andrault *et al.* (*1*) marked silica reflections at 100 GPa as stishovite. This value is incorrect because at pressure higher than 50 to 70 GPa at room temperature, stishovite transforms to $CaCl_2$-structure and could transform to α-PbO_2-like or some unknown structures at high temperatures. See, for example, Y. Tsuchida and T. Yagi, *Nature* **340**, 217 (1989); K. J. Kingma, R. E. Cohen, R. J. Hemley, H. K. Mao, *ibid.* **374**, 243 (1995); K. J. Kingma, H. K. Mao, R. J. Hemley, *High Pres. Res.* **14**, 363 (1996); L. S. Dubrovinsky *Nature* **388**, 362 (1997). But even if the possible reflections of silica with $CaCl_2$- or α-PbO_2-like structures are taken into account, it is not clear how to explain some features in fig. 4 in (*1*), for example, the reflection at $\sim$1.93 Å. The use of a material with unknown phase diagram as a *P* medium in an experimental study of another material with unknown phase diagram seems an inaccurate method.
20. Andrault *et al*. (*1*) mention that they studied phase transitions in Fe in *P* between 30 and 100 GPa. Their report does not contain any pattern of γ-Fe that might be expected at 30 GPa.
21. H. K. Mao, J. Shu, Y. Fei, J. Hu, R. Hemley, *Phys. Earth Planetary Inter.* **96**, 135 (1996).
22. In our experiments with laser-heated DAC on Fe and MgO as *P* medium, we noticed, for example, that iron could form the oxide FeO if periclase was not sufficiently dried before experiments, or was exposed on open air for a long time (several hours), or both.
23. S. K. Saxena *et al.*, *Science* **269**, 1703 (1995).
24. S. K. Saxena, L. S. Dubrovinsky, P. Häggkvist, *Geophys. Res. Lett.* **23**, 2441 (1996).
25. L. S. Dubrovinsky, S. K. Saxena, P. Lazor, *Geophys. Res. Lett.* **24**, 1835 (1997).
26. Reflections 2.35 and 2.03 Å are close to (100) and (101) reflections of Re gaskets, respectively. Andrault *et al.* (*1*) repeated the experiment with W gasket and report that they record "the same new peaks at high pressure and temperature." It is not clear what *P* medium was used in the experiments and which new peaks were found.
27. We call reflection of orthorhombic iron (100) around 2.3 Å "exclusive" for orthorhombic iron, because this reflection was not observed in any previous study and it is the only reflection out of those observed (*1*) that cannot be explained as a reflection of dhcp-Fe. Andrault *et al.* (*1*) mention the presence of the reflection 2.28 Å on the pattern of heated sample in table 2 in the report, but in figure 4 of the report, it appears to be absent.
28. In one of our experiments, we pressurized 5 μm thin Pt foil surrounded by enstatite $MgSiO_3$ to 75(2) GPa and then heated it by Nd:YAG laser radiation to 2200(150) K. In the heated spot, $MgSiO_3$ transforms to perovskite, *P* drops to 54(2) GPa, and with x-ray diffraction we observed splitting of all platinum reflections.
29. R. Boehler, *Nature* **363**, 534 (1993); C. S. Yoo, J. Akella, A. J. Campbell, H.-K. Mao, R. J. Hemley, *Science* **270**, 1473 (1995); C. S. Yoo, P. A. Söderlind, J. Campbell, *Phys. Rev. Lett A* **214** 65 (1996).

13 January 1998; accepted 6 April 1998

Response: Dubrovinsky *et al.* do not agree with our interpretations (*1*) of iron at high *P* and *T*. We favor an orthorhombic-iron explanation of the experimental features, which seems to us the most parsimonious. Dubrovinsky *et al.* state that we encountered several artifacts including: (i) stress at 2125 K that would make ε-iron look like an orthorhombic lattice; (ii) *P* as different as 72 and 96 GPa in the same 15 to 8 μm^2 area after the sample annealing; and (iii) a severe oxidation in a new unquenchable FeO-polymorph (*2*). Their criticism might be viewed as a defense of the "d-hcp" model of iron at high *P* and *T* (*3*). We answer each of their criticisms in turn.

1) Powder statistics. Dubrovinsky *et al.* state that with reduced powder statistics the reliability of the observed intensities is not sufficient for an assessment of a crystallographic model. The data show, however, that even if our iron diffraction lines are somehow spotty, they do correspond to hundreds of crystallites over the 2Θ rings (Fig. 1) [figure 1 in (*1*)]. It is because there is a reduced number of iron grains in the x-ray spot that angle dispersive diffraction with use of a 2-dimensional detector is required. For such small samples, the use of energy dispersive diffraction is prohibited because of the limited reciprocal space covered by the 0-dimensional Ge-detector. In an energy-dispersive experiment, the occurrence or absence of particular diffraction peaks is often not reproducible (*4*).

2) Preferred orientation. Preferred orientations of the crystallites are likely to happen on compression, especially for anisotropic structures such as ε-hcp. In our

report (*1*), we state that the refinement revealed a preferred orientation of the c_{hex} (or c_{ortho}) along the compression axis. The use of such a parameter while one is computing calculated intensities is common, and it is available in the GSAS program package. This parameter corresponds to a statistical effect that is apparently sufficiently small that all diffraction lines of ε-iron are observed [figure 4 in (*1*)]. In any case, we do not agree that a c_{hex} lattice preferred orientation can explain the absence of 010 and the presence of 100.

3) Thermal gradient. A pressure medium is essential for thermal insulation between the hot sample and the diamonds. In our study, the good insulation of the iron-sample was demonstrated by the high intensity of the pressure-medium diffraction [lines in figure 5 in (*1*)]. According to calculations of the phase content in our diffraction spectra, the iron thickness was less than that of the Al_2O_3 medium on each side of the sample along the 15*8 μm^2 x-ray spot. Furthermore, if our sample had encountered large *T* gradients, we would have observed a broadening of the diffraction peaks at high *T*, which was not the case (*5*). We therefore exclude artifacts resulting from large *T* gradients (*6*), as a possible source of our data.

4) Deviatoric stress in the pressure chamber. Deviatoric stress is well known to be much more severe in cold samples than in annealed or hot samples (*7*). It is possible, however, that some stress can be built up on *T* quenching, as illustrated by the slight broadening of the Al_2O_3 diffraction peaks on the top spectrum of figure 2 of (*1*). This result is probably arises from the fact that corundum undergoes the highest thermal gradient, because it is located between the laser-heated iron and the cold diamonds. The generation of stress during either cold compression (*8*) or *T* quenching is the main reason why we used high-*T* spectra to test our structural model. Stress is lowest at high *T* because the iron shear modulus decreases with increasing *T*. Dubrovinsky *et al.* appear to agree with this concept, but do not question the previous d-hcp iron determined with the use of quenched spectra (*3*).

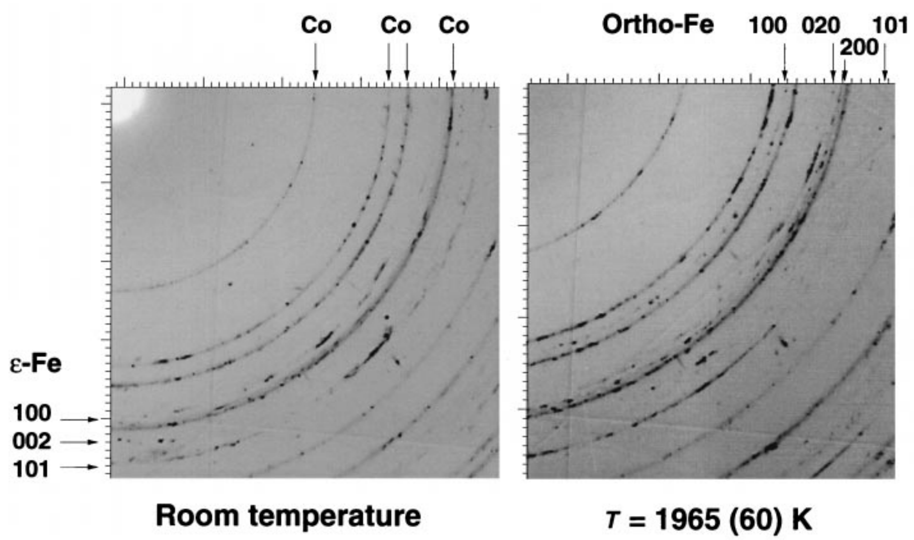

Fig. 1. Two dimensional diffraction patterns of iron (Fe) recorded at 44.6 GPa at room *T* (left) and 1965 (60) K (right) in corundum (Co). Presence of the ε-polymorph (left) is evidenced by the 100, 002, and 101 d_{hkl} lines. The 002 line corresponds to a reduced number of crystallites because of a c_{hex} preferred orientation along the compressional axis. High-*T* spectrum shows new lines, with a sufficient statistic, that do not correspond to an intermediate metastable polymorph. Four iron lines indexed in this pattern, as well as others found at higher 2Θ angle, correspond to the high-*P* and -*T* orthorhombic polymorph of iron.

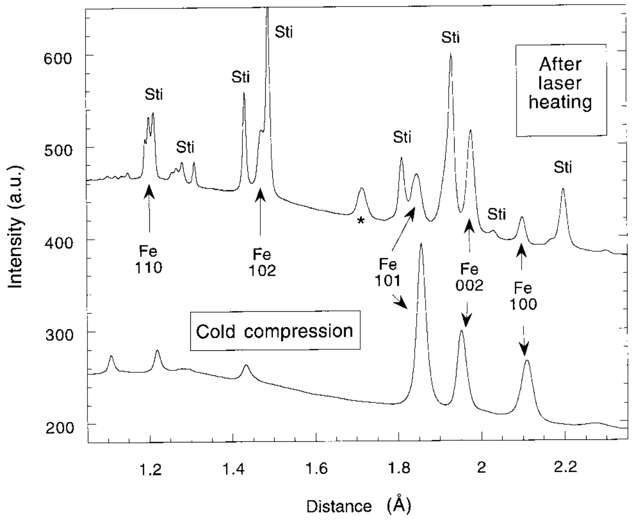

Fig. 2. Integrated diffraction patterns of Fe in SiO_2 pressure medium at 35 GPa, using a W-gasket. On cold compression (bottom spectrum), all ε-hcp iron diffraction lines are visible showing the high-purity of our starting material. In this spectrum, there is no SiO_2-feature because quartz has become amorphous. Top spectrum was recorded after several laser-heating sequences. No oxidation of the iron-sample was encountered, because all diffraction peaks can be indexed as a mixture of iron and stishovite. In this experiment, a reduced portion of γ-fcc iron has been quenched. Two main γ-iron Bragg lines are found at 1.714 Å (γ-002 indicated as *) and 1.977 Å (γ-111 superimposed with ε-002). Arbitrary units, a.u.

5) Purity of our starting material. Dubrovinsky *et al.* state that the new peaks observed at high *T* for iron [figure 2 of (*1*)] are those of a hypothetical B1-cubic high-*T* polymorph of FeO. This polymorph would (in their opinion) be unquenchable, but if it were, we would not have observed the diffraction lines of the low *T* FeO rhombohedral phase (*9*), which overlap with the corundum spectrum. Their criticism is answered by the fact that we did not observe the FeO-rhombohedral lines at any *P* performed in our study (*1*) with the use of Al_2O_3 [*10*] and with SiO_2 as the *P* medium. The SiO_2 diffraction pattern does not overlap with that of FeO (Fig. 2).

Still, the quality of diffraction spectra recorded at extreme conditions of *P* and *T* might not be sufficient for a full Rietveld structure refinement. We used the GSAS package to test our structural model by comparing calculated with observed intensities. The fact that we obtained such a good agreement [figure 5 of (*1*)] is a strong corroboration of the validity of the Pbnm-model for iron.

It is this agreement between experiments and a structural model that makes the difference between the Pbnm and d-hcp models for iron at high *P* and *T*. The d-hcp model does not provide a definite crystallographical model, and previous studies did not propose a space group related to an atomic topology. Also, there is no Lebail refinement available that would support the validity of the d-hcp model to explain experimental features (*11*). It seems that the occurrence of the d-hcp Bragg-lines has not been reproduced, although attempts have been made, and thus these lines should not be used to determine extinction rules (*12*). We suggest that the d-hcp structure corresponds to an intermediate iron structure that occurs at moderate *T*.

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References and Notes

1. D. Andrault, G. Fiquet, M. Kunz, F. Visocekas, D. Häusermann, *Science* **278**, 831 (1997).
2. The only data we know of for a new FeO polymorph at high *P* and *T* is S. K. Saxena and L. S. Dubrovinsky, presentation at the U.S.-Japan Seminar on High Pressure-Temperature Research: Properties of Earth and Planetary Materials, Kyoto, Japan, 22–26 January 1996.
3. S. K. Saxena *et al*., *Science* **269**, 1703 (1995); S. K. Saxena, L. S. Dubrowinski, P. Häggkvist, *Geophys. Res. Lett*. **23**, 2441 (1996).
4. The d-hcp lattice was proposed on the basis of results using energy dispersive diffraction [see (*3*), and L. S. Dubrovinsky, S. K. Saxena, P. Lazor, *Eur. J. Mineral*. **10**, 43 (1998)].
5. At a nominal pressure of 44.6 GPa [Figure 2 of (*1*)], the iron peak located around 2.06 Å show a FWHM of 6.6, 5.3, and 5.6 10^{-3} for temperature of 300, 1965, and 2125 K, respectively.
6. In a previous study in 1996, Saxena *et al*. deliberately produced a huge thermal gradient and argued for the occurrence of d-hcp lattice [fig 2 in (*3*)]. This is the best way to develop strong thermal stresses, thus promoting formation of nonequilibrium phases.
7. D. J. Weidner, Y. Wang, M. T. Vaughan, *Science* **266**, 419 (1994).
8. A. K. Singh, *J. Appl. Phys*. **73**, 4278 (1993).
9. H. K. Mao, J. Shu, Y. Fei, J. Hu, R. Hemley, *Phys. Earth Planet. Inter*. **96**, 135 (1996).
10. Corundum peaks cannot mask the FeO features from 30 to 65 GPa because FeO (K_T = 142 to 182 GPa [9]) is significantly more compressible than corundum [K_T = 253 GPa; P. Richet, J. A. Xu, H. K. Mao, *Phys. Chem. Minerals* **16**, 207 (1988)].
11. L. S. Dubrovinsky, presentation at a meeting of the American Geophysical Union, San Francisco, USA, 8–12 December 1998.
12. Dubrovinsky *et al*. recently reported a first quantitative assessment of the experimental lines using the d-hcp lattice [table 1 in (*13*)]. Two characteristic lines (004 and 103) are single spots on the reported pattern [fig. 2 in (*13*)] that disappear on prolonged heating. The occurrence of the 100, 102, and 104 d-hcp lines could correspond to that of ε-hcp (100, 101, 102). Thus, they report only 1 stable new line located at 2.0009 Å, a line that is expected for orthorhombic iron [110-line; see table 2 of (*1*)].
13. L. S. Dubrovinsky, S. K. Saxena, P. Lazor, *Geophys. Res. Lett*. **24**, 1835 (1997).

9 March 1998; accepted 6 April 1998