

In situ x-ray diffraction of the α - ε phase transition in iron at intermediate strain rates

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The α (bcc)- ε (hcp) phase transition in polycrystalline Fe has been investigated under fast quasihydrostatic compression using piezoelectric actuator driven dynamic diamond anvil cells. X-ray diffraction data from Fe under intermediate strain rate compression were collected at megahertz repetition rates. Our results demonstrate, for the first time, the full evolution of the α - ε phase transition in powder and foil samples at strain rates of $\sim 10^2 \text{ s}^{-1}$. Under fast compression, the majority of the α phase transforms to ε -Fe in a timescale of tens of microseconds, with a corresponding coexistence pressure of $\sim 2 \text{ GPa}$. The observed onset phase transition pressure of 13.4–14.4 GPa is in good agreement with the results of previous quasihydrostatic compression studies, suggesting that the transition is not influenced by compression rates up to $\sim 80 \text{ TPa s}^{-1}$.

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I. INTRODUCTION

The structural response of materials subjected to high pressures and strain rates has attracted widespread interest [1–8] due to the increasing complex and extreme environments in which materials are employed in modern society, including applications such as land, aircraft, and space travel [9,10]. A number of different dynamic drivers have been developed and utilized to recreate these conditions of different strain rates [Fig. 1(a)], ranging from quasistatic compression methods such as diamond anvil cells (DACs) [10–12] to highly dynamic methods such as gas guns [13–15] and high power lasers [16–19] for quasiisentropic and shock compression. The majority of work was performed at one of the strain rate extremes though, i.e., under quasistatic ($< 10^{-3} \text{ s}^{-1}$) or shock ($> 10^4 \text{ s}^{-1}$) compression, and exhibits remarkable discrepancies in material behavior depending on methods; e.g., phase transition boundaries may be shifted to higher pressures due to kinetic effects [20], or shifted to lower pressures due to the uniaxial nature of shock compression [21,22]. However, due to the complex states (i.e., high shear stresses and extreme temperatures) experienced under shock compression, it can be difficult to make direct comparisons with static compression experiments. Consequently, kinetic (i.e., timescale and strain

rate) and stress-induced effects cannot easily be decoupled without additional data.

Despite the interest in rate-dependent behavior, material behavior at intermediate strain rates (10^1 – 10^3 s^{-1}) remains relatively unexplored. This regime has typically been accessed using Hopkinson Bar techniques [23]; however, these experiments are limited to low pressures determined by the material strength, and the uniaxial stress state of these experiments cannot be compared with DAC experiments. Recent work has shown that this intermediate strain rate regime can also be accessed via the rapid compression in a DAC, either through the use of a gas membrane (mDAC) or a piezoelectric actuator [dynamic diamond anvil cell (dDAC)], which can generate strain rates up to 10^1 s^{-1} [24] and 10^3 s^{-1} [25,26], respectively. One advantage of dynamic DACs is their versatility, as experimental parameters can be tailored in a similar way to conventional DAC experiments; the stress state can be controlled by the use/absence of a pressure-transmitting medium (PTM) and, as dDAC compression is not fundamentally/intrinsically coupled to temperature generation, there are no inherent limitations on the experimental P - T path. Effects of strain rate can therefore be decoupled from those induced by shear stress and temperature.

The pressure-induced α (bcc)- ε (hcp) phase transition in iron (Fe) is one of the most extensively studied pressure-induced polymorphic transformations, having been the focus of an extensive number of static [27–43] and dynamic compression [44–51] experiments since the early days of high-pressure science [27,44]. This transition is nondiffusive and martensitic in nature, proceeding via a Burgers transformation path [41], and is associated with a change in magnetic properties [52]. Consequently, hysteresis is observed between the α - ε and reverse ε - α transitions [29,40,41], which is characteristic of martensitic transitions, and orientation

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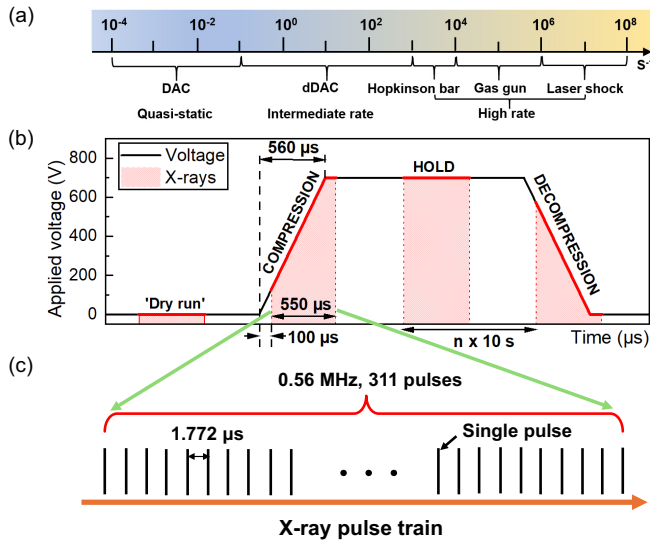


FIG. 1. (a) Approximate division of strain rate regimes and the possible methods used to explore these regimes, where the dDAC fills the strain rate gap between the quasistatic or low strain rate and high strain rate loading in high-pressure research. (b) Schematic diagram of the loading cycle of Fe under compression and decompression with a dDAC and the acquisition window for each stage with x-rays is also indicated. (c) Schematic diagram of the temporal structure of the individual long X-ray pulse train used for pulsed-resolved XRD. The total train duration is ~ 550 μ s, and the interval between each pulse is 1.772 μ s (0.56 MHz).

relationships exist between the parent and daughter phases [40].

The evolution of Fe across the α - ϵ phase transition has attracted extensive interest [27–43,46–49], in particular, the onset transition pressure (P_t) and the coexistence pressure range (P_c). Previous DAC experiments [28–43] investigated the α - ϵ transition under quasihydrostatic (with a PTM) and nonhydrostatic (without a PTM) conditions, and reported values of P_t ranging from 8.0 to 14.9 GPa, while P_c was observed from 0.5 to 14.8 GPa (Table S1 in the Supplemental Material [53]). Both P_t and P_c have been found to be strongly dependent on the PTM, which influences the stress state of the sample. Early work found that the use of a PTM with a high shear strength resulted in a lower onset transition pressure but a larger transition width, ranging from 0.5(5) GPa for argon to 14.8(10) GPa for Al_2O_3 [32]. This hypothesis is confirmed by reports of P_t ranging from 8 to 13 GPa and P_c from 5.1 to 10.65 GPa in samples compressed without a PTM [29,30,34,35,38,39], compared to a P_t of 13–14 GPa and P_c of 2–6.6 GPa in quasihydrostatic samples compressed in a He or Ne PTM [37,40–42]. The α - ϵ phase boundary has a negative P - T slope, terminating at the α - ϵ - γ triple point at ~ 9.4 GPa and ~ 730 K [54]. Early studies investigated the effect of temperature on the α - ϵ phase transition in isothermally compressed nonhydrostatic samples and found no correlation between temperature and P_c [30,31]. With respect to compression rate, an mDAC study reported $P_t \sim 12$ GPa and $P_c \sim 8$ GPa in the iron sample loaded without a PTM at a relatively low compression rate (3.4 GPa s $^{-1}$) [39]; however, due to the large spread in P_t and P_c reported for nonhydrostatic

samples, it is not possible to draw any conclusions on the effect of compression rate from a single mDAC run. Plückerthun has investigated the α - ϵ transition of Fe under nonhydrostatic conditions at different strain rates (10 GPa s $^{-1}$ to 1 TPa s $^{-1}$) with dDAC compression and reported P_t changing from 12 to 16 GPa and P_c from 5 to 11 GPa in his thesis [55]. However, limited to the pressure resolution of consecutive x-ray diffraction (XRD) images, he cannot resolve a strain-rate-dependent change in P_t and P_c . More recently, dDAC experiments on samples compressed in a KCl PTM reported P_t to shift to higher pressures with increasing compression rate (13.6 GPa at 360 GPa s $^{-1}$ compared with 10.8 GPa at 2.5 GPa s $^{-1}$), which was interpreted to be due to kinetic hindrance associated with the reconstructive nature of the α - ϵ transition [43]. However, the effect of compression rate on P_c was not reported.

When it comes to high strain rate shock experiments, it is well accepted that the transition occurs at around 13–14 GPa [50–52,56], which is similar to the transition pressure reported for quasihydrostatic compression experiments. Ramp compression experiments [57] reported an increase in the onset stress at the phase transition at strain rates above 10^6 s $^{-1}$, which was thought to originate from a phonon drag mechanism [49]. Later work also observed an overpressurization at strain rates above 3.2×10^7 s $^{-1}$ alongside a decrease in the α/ϵ coexistence region, which they modeled using an Avrami-type kinetics model of nucleation and growth with a constant completion time and no direct dependence on strain rate [58]. However, these studies were performed using VISAR as a primary diagnostic, lacking direct lattice information using *in situ* XRD measurements. In addition, due to the complex shock state as well as the temperature rise, it is difficult to determine whether strain rate is the only factor that influences the transition onset and coexistence pressures.

In this work, the effect of strain rates of $\sim 10^2$ s $^{-1}$ on the α - ϵ phase transition in Fe has been explored by compressing samples in ~ 560 μ s using piezo-driven dDACs at the High Energy Density Instrument (HED) at the European X-ray Free Electron Laser (European XFEL) [26,59]. megahertz pulse trains of up to 310 pulses were used to collect time-resolved XRD data with microsecond temporal resolution, which enabled us to map out the structural evolution of the sample across the α - ϵ phase transition and to accurately determine the onset transition pressure and coexistence region of the two phases. The dDAC platform was recently used to capture structural dynamics at compression rates up to ~ 100 TPa s $^{-1}$ [24]. However, data collection in these experiments was limited to a ~ 200 μ s time window, using a standard operation mode of the facility. This work utilizes a uniquely commissioned long-pulse train mode that can cover a time window up to 550 μ s, which is well matched to the dDAC compression timescales in this experiment. The results of this study allow us to understand the kinetic effects of the α - ϵ phase transition in the intermediate strain rate regime.

II. METHODS

Samples: Two types of Fe samples were investigated in this experiment: (1) Fe spherical powder with a grain size of < 10 μ m (Thermo Scientific, LOT:62000952) and (2) Fe foil

with a thickness of 5 μm (Goodfellow, FE000100). Samples were loaded into symmetric DACs equipped with 300 μm culet diamonds and ~ 40 μm thick stainless-steel gaskets with a 150 μm sample chamber. Ne was used as a PTM, and a ruby pressure gauge was included for pressure determination after loading. Samples were precompressed to a pressure below the α - ϵ phase transition pressure before the XFEL experiment.

dDAC experiments: Rapid compression experiments were conducted using the dDAC setup in Interaction Chamber 2 at the HED instrument at European XFEL (for full details, see Ref. [26]). Samples were compressed/decompressed using the DESY-type dDAC [26] by applying a time-dependent voltage profile to the piezoelectric actuator [Fig. 1(b)]. Each compression cycle consisted of compression, hold, and decompression segments that were individually synchronized with a single XFEL pulse train. The compressed/decompression voltage profiles had a rise/fall time of 560 μs and reached a maximum of 700 V, and the time between data collection from individual segments was typically on the order of several minutes. In this dDAC design, decompression of the sample is dependent on the rebound response and relaxation of the gasket after the removal of the force from the piezo. Therefore, the sample pressure either did not decompress below the ϵ - α transition pressure on release, or the process was too slow to be captured by the x-ray pulse train with the chosen timing. Consequently, the reverse ϵ - α phase transition was not observed during the XRD measurement and the work is entirely focused on the behavior of Fe on compression.

XRD measurements: X-rays with an energy of 18 keV were focused with compound refractive lenses to a focal spot size of ~ 40 μm (h) $\times$ 40 μm (v), which was determined by round edge scans using a polished W wire. The beam size was chosen to be large enough to minimize x-ray heating, while still avoiding unwanted parasitic XRD peaks from the surrounding gasket material. XRD images for each run were recorded with an 500 kpx adaptive gain integrating pixel detector (AGIPD) [60] running at the same repetition rate as the x-ray source. Data were collected using a total of 310 pulses at a repetition rate of 0.56 MHz, to provide the maximum coverage of the compression ramp (550 μs) as a trade-off between the maximum number of 352 frames that can be collected using AGIPD detector per pulse train and the maximum length of the latter. Cr_2O_3 powder was used as a diffraction standard to calibrate the sample-to-detector distance and orientation of the AGIPD detector, which was placed outside the vacuum chamber at a sample-to-detector distance of ~ 441 mm, providing reciprocal access up to $q = 4$ $\text{\AA}^{-1}$.

During the x-ray measurement, the sample can potentially be heated due to the high pulse energies of the XFEL pulses [61]. Therefore, in order to independently explore the influence of compression rate on the pressure-induced phase transition, it is crucial to minimize heating caused by XFEL irradiation. This was achieved by using a relatively large x-ray focal spot size (compared to typical DAC experiments), and by reducing the x-ray fluence using a series of solid attenuators. To evaluate the extent of x-ray heating, an x-ray only measurement with 310 pulses (“dry run”) was collected before each compression cycle [Fig. 1(b)], which was used to determine the optimal x-ray transmission ($\sim 1\%$), which balanced diffraction quality and temperature rise in the sample during

the run (Sec. 2 in the Supplemental Material [53]). Previous studies [62] have shown that within the x-ray pulse train, the sample temperature increases stepwise in the DAC, with each x-ray pulse first probing the state of the sample cooled from the previous pulse and subsequently increasing its temperature due to absorption. Simulations [62] have also shown much less x-ray heating at 455 kHz repetition rate than 2.2 MHz. With larger x-ray beam size (40 $\times$ 40 μm vs 20 $\times$ 20 μm) and higher x-ray photon energy (18 keV vs 13 keV), we expect less heating in our case. More details about the x-ray-induced heating in a DAC could be found elsewhere [63]. The measured maximum increase in temperature during all the dry runs did not detect more than 140 K in subsequent pulses. During the compression ramp, XRD images were collected 100 μs after the start of the 560 μs drive voltage ramp, providing data coverage of the remainder of the compression and the first ~ 90 μs of the plateau in the voltage waveform [Fig. 1(b)]. However, due to the time lag between the application of the voltage waveform and the pressure response of the DAC, the sample pressure continued to increase, while the piezo actuator waveform already reached a plateau.

Data analysis: Radial integration of the 2D XRD images was performed using the Dioptas software [64], and the diffraction intensity was normalized using pulse-resolved signal from the downstream diodes of Instrument BeamStop [59] to account for pulse intensity fluctuations during the XFEL pulse train. Peak positions were determined by fitting individual reflections in the one-dimensional (1D) XRD profile with a pseudo-Voigt function, and the unit-cell parameters were obtained from the refined peak positions and the corresponding Miller indices by least squares fitting. The (110) α -Fe and (0002) ϵ -Fe reflections overlapped during the mixed-phase region, and so the unit-cell volume and c/a ratio of ϵ -Fe were determined from the peak position of the (10-11) and (10-10) reflections. These reflections were also used to determine the evolution of the phase fraction of ϵ -Fe in the mixed-phase region, based on the change in their intensity as a function of pressure (Sec. 3 in the Supplemental Material [53]).

A thermal Birch-Murnaghan (BM) equation of state (EoS) of α -Fe [65] was used to evaluate x-ray heating in the dry runs, assuming that heating was isobaric (i.e., thermal pressure was neglected). The evolution of the sample pressure on compression was estimated from the unit-cell volume of Fe using a previously reported BM EoS for each of the Fe phases: $V_0 = 23.56$, $K = 166.6$ GPa, and $K' = 5.97$ [66] for α -Fe, and $V_0 = 22.47$, $K = 165$ GPa, and $K' = 4.97$ for ϵ -Fe [37]. This approach was chosen to avoid the potential introduction of systematic uncertainties due to the use of an independent pressure marker and choice of EoS when making comparisons with previous work. In addition, previous work found the use of internal XRD standard, other than the sample itself, to be an unreliable method of pressure determination for rapid compression experiments on nonhydrostatic samples [50]. The reported transition pressure (P_t) is based on the unit-cell volume of α -Fe when the first evidence of ϵ -Fe was observed. This approach was chosen as it allowed for pressure determination when only one peak ϵ -Fe was observed, which is not sufficient to determine the unit-cell parameters of this phase.

Precise determination of the α/ϵ mixed-phase region was complicated by the overlap of the (0002) ϵ -Fe and (110) α -Fe

TABLE I. Summary of the four compression runs conducted in this study. The initial and final pressures correspond to the pressure at the start and end of the section of the compression ramp that was captured by the XRD measurement, respectively, which were used to determine the average compression rate. The onset phase transition pressure corresponds to the Fe pressure at the start of the x-ray data collection, which was determined using the EoS of α -Fe [37]. The error comes from the pressure resolution of the XRD measurement (<0.2 GPa). The coexistence pressure was determined from the fitting (Sec. 3 in the Supplemental Material [53]). The temperature increase was estimated from the thermal expansion of α -Fe during the “dry run” collected before compression, in which the sample was irradiated with 310 XFEL pulses (Sec. 2 in the Supplemental Material [53]).

Run	Sample type	Initial P (GPa)	Final P (GPa)	Average compression rate (TPa s ⁻¹)	Onset transition $V\text{\AA}^3$ bcc	Onset transition P (GPa)	Coexistence region (GPa)	T increase (K)
21 ^a	Powder	10.89 ± 0.2	36.28 ± 0.2	86	22.02	13.75 ± 0.2	2.0	54.3
46	Powder	5.65 ± 0.2	41.51 ± 0.2	77	22.05	13.43 ± 0.2	2.5	81.8
41	Foil	9.37 ± 0.2	47.70 ± 0.2	83	21.94	14.59 ± 0.2	1.1	36.6
51	Foil	4.14 ± 0.2	29.99 ± 0.2	50	21.96	14.37 ± 0.2	0.6	55.9

^aOnly the first 239 frames were captured for run 21 due to a drop in the intensity of the XFEL pulse train.

reflections, which meant that it was not possible to directly monitor the disappearance of the α -Fe signal. However, a continuous increase in the integrated intensity of the (10-10) and (10-11) ε -Fe reflections, combined with a simultaneous decrease in the intensity of the (110) α -Fe reflection, suggested a region of coexistence of the α and ε phases in all runs. This compression section was followed by no significant changes in the intensity in the subsequent images, suggesting that only ε -Fe remained. The progression of the phase transition was therefore evaluated based on the evolution of ε -Fe phase fraction as a function of time (i.e., the phase fraction), based on the intensity evolution of the (10-10) and (10-11) reflections (Sec. 3 in the Supplemental Material [53]).

III. RESULTS AND DISCUSSION

A total of four compression cycles on Fe powder and foil samples were performed using the dDAC, details of which are given in Table I. Representative XRD data collected during the compression segment of run 46, in which the α - ε phase transition was observed, are shown in Fig. 2. Only the (110) Debye Scherrer ring from the α -Fe phase was observed at low pressures due to the limited angular coverage of the AGIPD detector, whereas three diffraction rings were observed from ε -Fe [Fig. 2(a)]. The temporal evolution of the 310 integrated XRD patterns collected during compression, corresponding to the 550 μ s x-ray acquisition window, is shown in Fig. 2(b) to give a full view of the entire compression process. The pressure at the start in this run was ~ 5.6 GPa, which is in good agreement with the pressure determined in the dry run (~ 5.8 GPa). The little difference might come from the temperature annealing induced by x-ray heating. Compression is easily identified by a shift of diffraction peaks to higher angles (smaller d spacing) due to an increase in the sample pressure. Despite the 100 μ s delay between the onset of the voltage ramp and the start of data collection, no obvious compression was observed during the first ~ 50 frames due to the delayed mechanical response of the dDAC assembly. Similar behavior has previously been reported for this dDAC design, where the sample pressure did not react until the voltage reached more than ~ 100 V during quasistatic compression [25]. Consequently, the actual compression lasted for the remaining 260 frames of the XRD measurement window.

The appearance of two additional diffraction peaks from Ne was observed at $\sim 108 \mu$ s at a pressure of 6.4 GPa, which shifted faster than those from Fe due to its higher compressibility. This pressure is slightly higher than the Ne solidification pressure reported in Ref. [67], i.e., 4.8 GPa at 300 K, most likely because the single-crystal like Ne grains did not initially fulfill the Bragg condition but later became polycrystalline during compression. This assumption is validated by the fact that the Ne reflections on the 2D XRD images consisted of single-crystal like arcs. On further compression, the first appearance of ε -Fe was identified based on the appearance of the (10-11) reflection at 206 μ s (pulse 115), which was followed by the appearance of the (10-10) reflection at 218 μ s (pulse 123), as shown in Fig. 2(c). However, the first appearance of the (0002) ε -Fe reflection could not be determined due to its overlap with the (110) α -Fe reflection. The experimentally observed onset phase transition pressure of 13.43 GPa is consistent with previous quasihydrostatic, low strain rate and high strain rate compression experiments [39,40,51].

The pressure evolution during the entire x-ray measurement window (550 μ s) is illustrated in Fig. 3(a) for run 46 (see Fig. S6(a) in the Supplemental Material [53] for all runs). The apparent decrease in sample pressure prior to the compression onset ($\sim 88 \mu$ s after the start of the X-ray measurement) is most likely due to a slight thermal expansion of the lattice due to X-ray induced heating (Sec. 2 in the Supplemental Material [53]). During compression, the pressure increased from ~ 5.6 to 41.5 GPa in $\sim 460 \mu$ s (~ 260 frames), which determines an average pressurization rate of ~ 77 TPa s⁻¹ and a strain rate of ~ 360 s⁻¹ for Fe. Note that the compression rate was determined by linearly fitting the slope of the BCC Fe in the pressure-time curve [Fig. 3(a)].

The pressure dependence of the c/a ratio of ε -Fe is shown for run 46 in Fig. 3(b) (see Fig. S6(b) in the Supplemental Material [53] for all runs), where its value decreased almost linearly with increasing pressure during the x-ray measurement window. The c/a ratio was previously found to be sensitive to the stress state of the sample, and it was usually used to evaluate the hydrostaticity of samples [43]. The c/a ratio in our experiments was either found to be in good agreement with, or slightly higher than, values reported in earlier quasihydrostatic compression experiments [37,39,40], suggesting

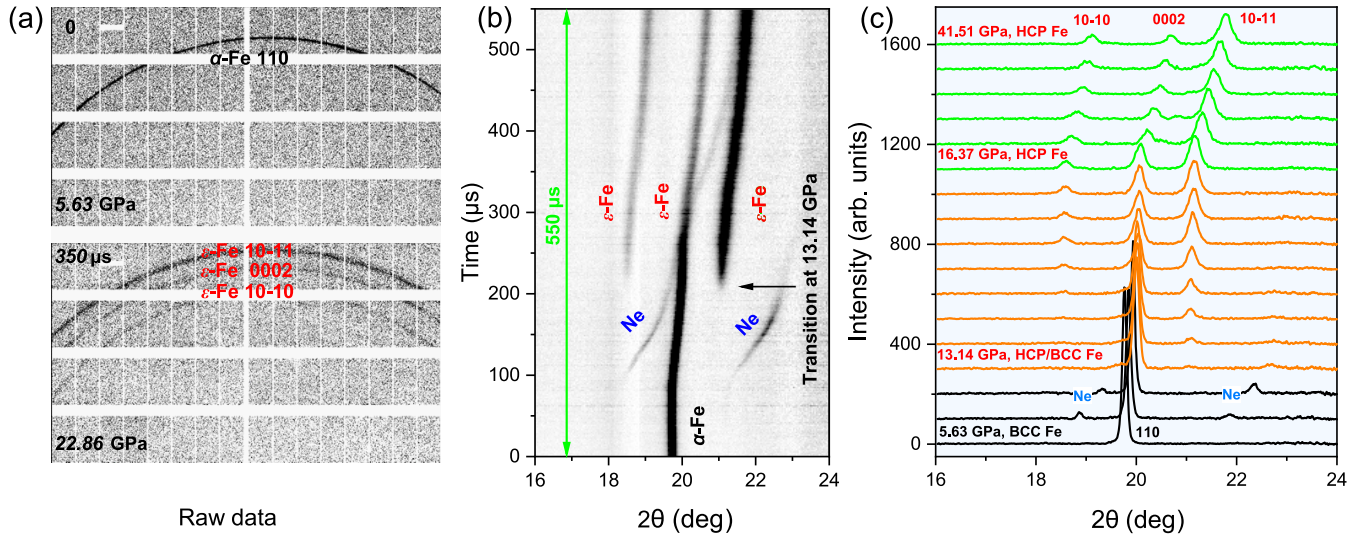


FIG. 2. Single pulse XRD data collected during run 46. (a) Selected 2D XRD images of Fe that were collected at 0 and 350 μ s after the start of the x-ray measurement window, where the XFEL pulse train arrived 100 μ s after start of the 560 μ s voltage drive. (b) Temporal evolution of the 1D integrated XRD patterns collected during compression, depicting the α - ϵ phase transition in Fe and the appearance of reflections from the Ne PTM. (c) Selected integrated XRD patterns illustrating the three stages of Fe under compression: (1) α -Fe before the phase transition (black), (2) α - and ϵ -Fe in the mixed-phase region as the phase transition progresses (orange), and (c) ϵ -Fe after the phase transition (green).

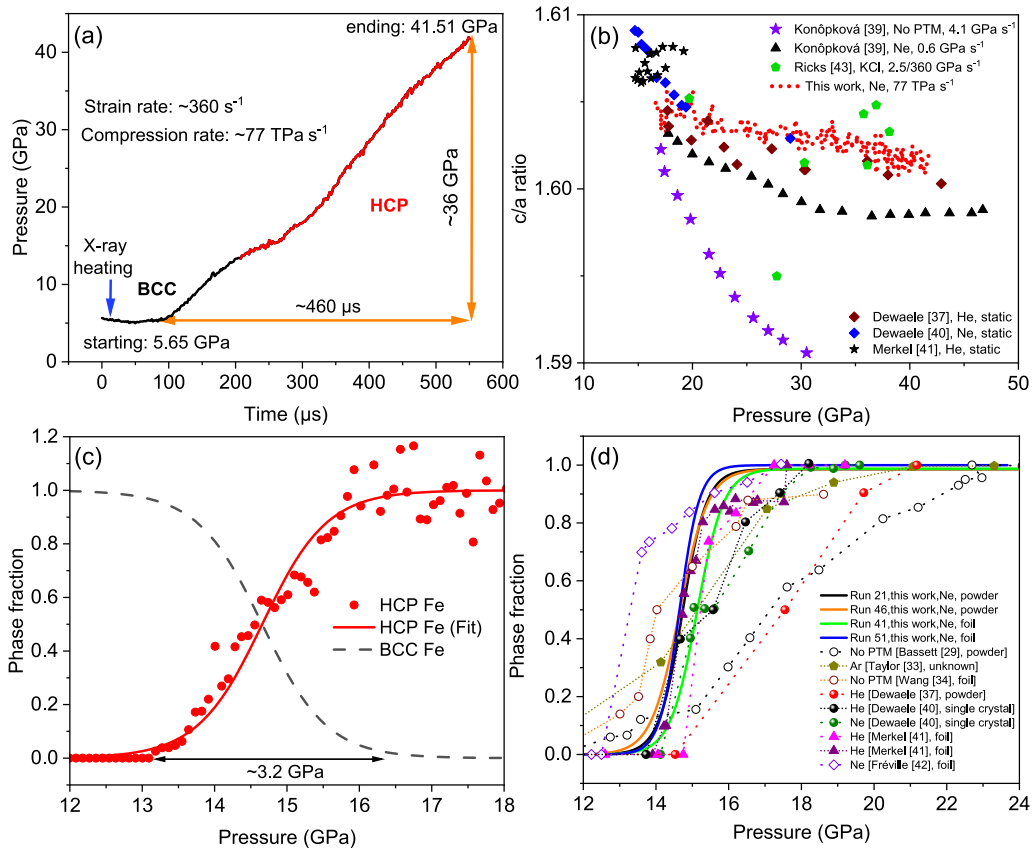


FIG. 3. (a) The pressure evolution of Fe in run 46, where the sample was compressed at an average compression rate of 77 TPa s^{-1} using a ddAC. The pressure was determined based on the unit-cell volume of Fe. (b) The corresponding c/a ratio of ϵ -Fe in run 46, in comparison to previous experiments [37,39–41,43]. (c) The phase fraction evolution during the α - ϵ phase transition, as determined from the intensity of the (10-10) and (10-11) ϵ -Fe reflections. The scatter in the data is primarily due to fluctuations in the x-ray pulse energy in the train. (d) Comparison of the phase fraction evolution of ϵ -Fe under compression with previous experiments [29,33,34,37,40–42].

that the samples in our experiment remained hydrostatic during rapid compression, despite the fast compression rate.

The phase fraction evolution is also examined to understand the effect of kinetics on the α - ε phase transition. For run 46, integrated intensity of the (10-10) and (10-11) ε -Fe reflections increased with increased pressure, which was accompanied by a simultaneous decrease in the intensity of the (110) α -Fe reflection over the same pressure range, as indicated by the arrow in Fig. 3(c). This was followed by no significant changes in the intensity of the patterns observed during the last ~ 150 images, suggesting that only ε -Fe remained. The intensity evolution of peaks from ε -Fe [Fig. 3(c)] suggests that the transition was complete at ~ 16 GPa, which determines a coexistence pressure range of ~ 2.5 GPa.

The phase fraction evolution for all runs are shown in Fig. 3(d). Although the width of the transition is similar in all runs, there is a small difference in the onset transition pressure for different sample types, with the transition starting at lower pressures in powder samples compared to foils. Our phase fraction results are also compared to those from previous studies [29,33,39–42] which were performed at slower compression rates [Fig. 3(d)]. As the stress state of the sample is known to play a crucial role in determining both P_t and P_c , the effect of strain rate can only be evaluated for samples compressed using the same PTM. P_c in our samples appears to be the smallest among studies performed using a Ne or He PTM, in good agreement with the work of Merkel *et al.* [41], in which foil samples were compressed in He, but narrower than the work of Dewaele *et al.* [37] that was performed using the same sample type and pressurization conditions. The coexistence pressure in our samples is also smaller than P_c reported by Dewaele *et al.* for single crystals compressed in Ne [40]. However, we note that the limited number of reflections used to determine the phase fraction in this work and the underlying assumptions due to the overlap of α - and ε -Fe reflections—in particular, the fact that the disappearance of α -Fe could not be directly measured—means that the transition width is possibly underestimated.

Figure 4 shows the P - T phase diagram of Fe determined during compression at different strain rates, with various loading methods including static or mDAC, dDAC, gas gun, and laser shock compression. The onset transition pressure determined in this work is in good agreement with the results from previous static compression experiments on quasihydrostatic samples and shock compression experiments. The agreement between different studies in this temperature regime is indicated by the gray dashed circle in Fig. 4, suggesting that the phase transition pressure is not influenced by compression rate.

A recent dDAC study by Ricks *et al.* [43] found a marked compression-rate-dependent shift of the α - ε phase boundary at intermediate strain rates, reporting onset transition pressures of 13.6 GPa for a sample compressed at 360 GPa s^{-1} compared to 10.8 GPa for a sample compressed at 2.5 GPa s^{-1} , where the increase in the observed transition pressure as a function of compression rate was attributed to kinetic hindrance due to the reconstructive nature of the phase transformation mechanism [68]. However, this study used pressed flakes of KCl both as an internal diffraction standard and PTM, where the pressure was determined based

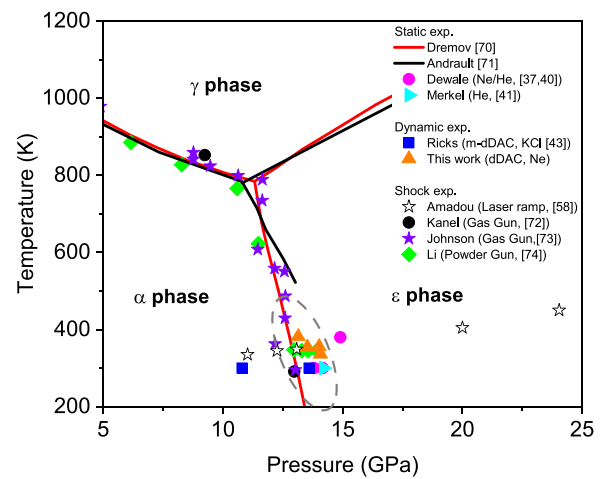


FIG. 4. Temperature-pressure phase diagram of Fe. The data points from this work show the onset transition pressure of the α - ε phase transition and the average temperature estimated from the dry run performed before compression. Our results are compared to phase lines determined in previous experiments conducted at different strain rates using various loading methods including static DAC, mDAC, dDAC, gas gun, and laser-induced ramp compression [37,40,41,43,58,70–74]. Note that for ramp compression, the temperature is taken from the principle hugoniot of Fe [45] as a reference since the author did not report the temperature in the paper [58].

on the previously determined EoS of KCl. The use of an internal pressure standard was previously found to be unreliable method of pressure determination in dDAC studies on non-hydrostatic samples due to the rate-dependent response of the sample/pressure marker assembly, which can potentially result in an overestimation in the extent of overpressurization of the phase transition boundary [69]. To avoid artifacts relating to the relative pressures of the sample/pressure marker during rapid compression, it is possible to compare the unit-cell volume of the sample material at the onset of the transition, which provides a true determination of the sample state and allows for a direct comparison with our data. Using this approach, the Fe volumes determined from the XRD patterns reported by Ricks *et al.* are $21.83 \text{ \AA}^3$ at 360 GPa s^{-1} and $21.84 \text{ \AA}^3$ at 2.5 GPa s^{-1} for bcc Fe, which corresponds to an onset transition pressure of 15.83 GPa at 360 GPa s^{-1} and 15.76 GPa at 2.5 GPa s^{-1} . The discrepancy between the KCl volume at the α - ε transition at different compression rates may arise from the sluggish compression of KCl at low compression rates, leading to a pressure mismatch between the Fe sample and KCl PTM. Overall, this suggests that the onset transition pressure is similar at these two strain rates, which is also in good agreement with the results of the present study (Table I) in which Fe was compressed at much higher compression rates.

In conclusion, this study utilized the x-ray pulse trains produced at the European XFEL to investigate the α - ε phase transition in Fe samples that were dynamically compressed using a dDAC using megahertz XRD. We provide, for the first time, *in situ* XRD measurements of the complete evolution of the α - ε phase transition under intermediated strain rate ($\sim 100 \text{ s}^{-1}$ or $\sim 100 \text{ TPa s}^{-1}$) compression with microsecond temporal resolution. The onset phase transition pressure and

phase fraction evolution of the transition were investigated and compared with the previous studies. The onset transition pressure is in good agreement with results from previous studies of samples compressed at vastly different strain rates using different loading methods, and it seems it is independent of strain rate. However, the coexistence pressure is slightly smaller at intermediate strain rate compared with static compression. Shear stress or hydrostaticity appears to play a much larger role.

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DATA AVAILABILITY

These data were collected as part of commissioning proposal p003246 and are available at [75]. The data that support the findings of this article are openly available [75].

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