

Dynamic compression and pressure cycling in dynamic diamond anvil cells: MgO and neon studied by time-resolved x-ray diffraction

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Static compression experiments using diamond anvil cells (DAC), combined with x-ray diffraction (XRD) are widely used to study material properties at extreme pressures. Recent advances enable time-resolved XRD during dynamic compression along tailored loading paths. However, the influence of compression rate on derived properties remains poorly understood, and the use of tailored compression paths—particularly pressure cycling—requires further investigation. Here, we employed time-resolved XRD in a dynamic DAC to compress MgO in a Ne pressure medium along a tailored compression path consisting of a ramp, plateau, and cyclic loading. Pressure-volume relations derived from ramp compressions at average compression rates of 0.22 and 0.46 GPa s^{−1} agree closely with previous static experiments, with minor but detectable systematic deviations in the faster compression run. Stress analysis suggests that this deviation might be related to an increased flow strength of Ne under fast compression. During the postramp plateaus, MgO pressures continued to increase, highlighting the effect of compression rate on the time required for the coupled mechanical DAC system to reach equilibrium. Under cyclic loading, we observe phase shifts between oscillating differential stresses and volume strains in Ne, possibly indicating that viscoelastic/plastic behavior of either Ne or the gasket material contributed to the overall response. We found this effect more pronounced for lower frequencies and larger cycling amplitudes. The findings presented in this paper improve our understanding of material behavior during dynamic loading in the DAC and provide reference data and methodology for studies of complex materials and loading paths.

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I. INTRODUCTION

The development of time-resolved x-ray diffraction (XRD) in dynamic diamond anvil cells (dDAC) represents a major advance in probing material properties across a range of compression rates that bridge static and shock experiments [1–4]. The technique enables investigations of compression-rate effects on phase transitions [5,6], elasticity [7–10], and plasticity [11–13]. The ability to apply cyclic loading at high pressures allows systematic probing of frequency and amplitude dependencies [7,11], opening new opportunities to study time-dependent processes relevant to Earth and materials sciences, possibly including seismic attenuation, strain-rate-dependent yield strength, and work hardening.

These processes span a range of timescales and stresses. For example, seismic periods are on the order of ~ 0.1 –100 s, whereas microstructural evolution takes place over microseconds to hours. Dynamic seismic stresses are ~ 0.1 –10 MPa, while the high-pressure strength of materials is on the order of MPa to GPa.

Time-resolved XRD in the dDAC has been performed both at synchrotron facilities [2,4], and at the European X-Ray Free-Electron Laser Facility [14,15]. Despite these developments, the effects of dynamic compression at intermediate rates and cyclic loading, particularly in the presence of pressure-transmitting media (PTM), remain understudied.

Here, we performed two dDAC experiments on magnesium oxide (MgO) in a neon (Ne) PTM, using gold (Au) and platinum (Pt) as pressure calibrants. MgO was selected because it is a well-characterized reference material for high-pressure studies [16,17] and represents the Mg-rich end member of (Mg_{1−x}Fe_x)O ferropericlase, the second most abundant mineral in the Earth's lower mantle [18,19]. Ne was chosen as the PTM because it has been extensively used in high-pressure and high-temperature experiments owing to its chemical inertness, low x-ray scattering background, and ability to

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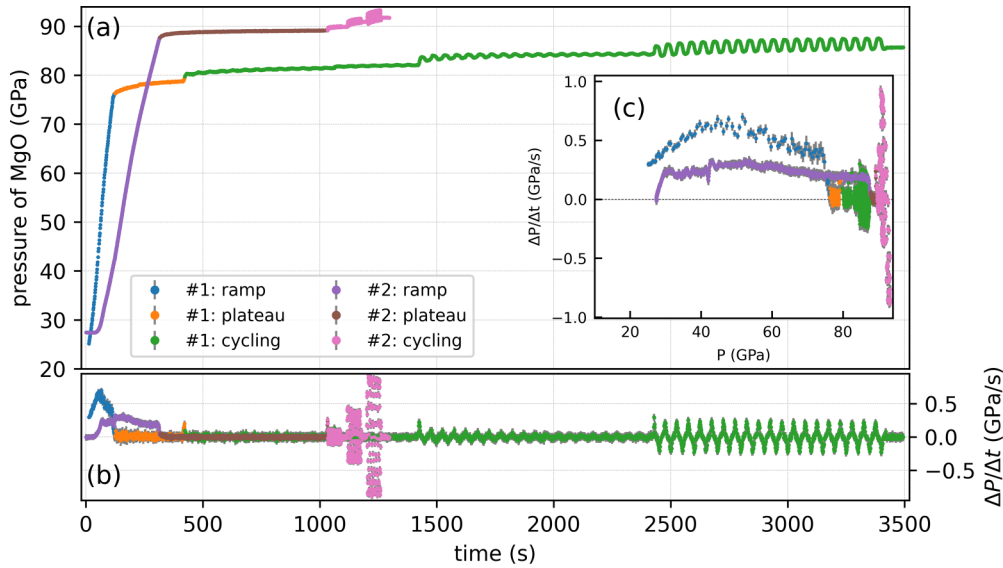


FIG. 1. Pressure evolution of MgO in two dDAC runs. (a) Pressure-time relationship, (b) compression rate ($\Delta P/\Delta t$) as a function of time, and (c) $\Delta P/\Delta t$ as a function of pressure. Pressures were derived from MgO volumes obtained from the 200 reflection using the third-order Birch-Murnaghan EoS with parameters listed in Table I. Error bars represent uncertainties propagated from peak fitting; in cases where they are not visible, they are smaller than the symbol size. The color version of this figure is available online.

maintain quasi-hydrostatic conditions to relatively high pressures [20–22].

In both experiments, the compression sequence consisted of three segments, with diffraction patterns continuously collected throughout. First, the dDAC was used to rapidly compress the sample to high pressures to assess the effect of compression rate on the derived pressure-volume (P - V) relationship. Second, the sample was held at a constant load to monitor the postcompression behavior. Finally, loading cycles were applied to investigate the effect of cyclic loading at high pressures.

II. METHODS

Two dDAC experiments were conducted at the Extreme Conditions Beamline P02.2, PETRA III, Hamburg, Germany [23]. The beam was focused to 8 μm (horizontal) $\times$ 3 μm (vertical) full width at half maximum (FWHM) intensity at the sample position using compound refractive lenses.

For DAC preparation, a rhenium (Re) gasket was preindented to $\sim 40\ \mu\text{m}$ thickness in a symmetric DAC using 200 μm culet diamonds. A 150 μm diameter hole was first laser-cut at the center of the indentation. To minimize Re diffraction peaks, a ring of amorphous metallic glass (outer diameter 130 μm , inner diameter 80 μm) was inserted into the Re gasket hole, following the procedure described in Refs. [3,24].

Pure, dried MgO, Pt, and Au powders were precompressed into $\sim 10\ \mu\text{m}$ thick flakes before loading. In both runs, a MgO flake ($\sim 40\ \mu\text{m}$ diameter) was vertically stacked on top of a Pt flake (run #1) or an Au flake (run #2) of $\sim 30\ \mu\text{m}$ diameter at the cell center. Ne was gas-loaded into the chamber as the PTM using the gas-loading system at P02.2 [23]. A sketch of the DAC loading as well as an optical image of run #2 is provided in Supplemental Material Fig. S1 [25].

To achieve the tailored compression paths, the symmetric DAC was coupled to a high-voltage piezoelectric actuator.

Voltage-time profiles defining the compression path were generated using Benchlink Waveform Builder software (Keysight Technologies) and loaded onto a waveform generator (Agilent 33522B). The output from the waveform generator was routed through a delay generator (SRS DG645/5 with Rb clock) to an amplifier (Piezosystem Jena GmbH), which drove the piezoelectric actuator to compress the DAC. More details about the dDAC setup can be found in Ref. [2]. In both dDAC runs, the compression sequence comprised three segments: a ramp compression, a postramp plateau without changing the applied load, and three groups of loading cycles with increasing amplitudes (Fig. 1).

The dDAC can be viewed as a coupled mechanical system consisting of (1) a pair of opposing diamond anvils acting as pistons, (2) a metal gasket between the anvils, (3) a soft Ne PTM that isolates the sample from the gasket/diamond surfaces, and (4) the sample together with the pressure standard (see Supplemental Material Fig. S1 [25]). In general, the response of the sample to the applied voltage signal to the piezoelectric actuator will be modulated by the entire assemblage.

Under applied force by the piezoelectric actuator, the diamond pistons drive gasket deformation (elastic and plastic), reducing the sample-chamber volume. The soft Ne PTM responds to this volume reduction by building up stress,

TABLE I. Internally consistent third-order Birch-Murnaghan EoS parameters for MgO, Pt, Au, and Ne at 300 K.

	V_0 ($\text{\AA}^3$)	K (GPa)	K'_0
MgO	74.70	160.6(2)	4.22(1)
Ne	88.97	1.4(1)	8.27(18)
Pt	60.38	274.7(20)	5.51(10)
Au	67.85	171.1(14)	5.81(7)

decreasing its unit-cell volume and transferring stress to the sample and pressure standard. Under experimental “isostress” conditions as usually expected in static DAC experiments using a PTM, the stress and pressure experienced by the sample and pressure standard should be equal to that transmitted by the PTM.

In run #1, 2800 diffraction patterns were collected at 1-s intervals using a PerkinElmer XRD 1621 flat panel detector. The ramp compression lasted 2 min (piezo voltage linearly increased from 0 to 850 V), followed by a 5-min plateau (piezo voltage kept at 850 V), and then three groups of loading cycles with increasing voltage amplitudes (50, 100, and 150 V) over 50 min, each consisting of 20 cycles at 0.020 Hz. In run #2, 2700 diffraction patterns were collected at 0.5-s intervals using a Lambda detector [2,26]. The ramp compression lasted 4.5 min (piezo voltage increased from 0 to 850 V), followed by a 12-min plateau (piezo voltage kept at 850 V). Three groups of loading cycles with increasing voltage amplitudes (50, 100, and 150 V) span 4 min in total and were separated by 0.5-min intervals, with each group lasting 1 min with 10 cycles, corresponding to an oscillation frequency of 0.167 Hz. For the PerkinElmer detector, the sample-detector distance and detector tilt were calibrated using CeO₂ (NIST 674b), while for the Lambda detector, calibration was performed with Cr₂O₃ (NIST 674b).

Two-dimensional diffraction patterns were first integrated into 2θ -intensity profiles using *DIOPTAS* [27]. A Python-based batch peak-fitting script was then used to analyze the diffraction patterns [10,28]. Unit cell volumes of MgO, Pt, Au, and Ne were obtained from the fitted peak positions. For MgO in run #1, for example, volumes were determined from the 111, 200, and 220 diffraction peaks, yielding $V_{\text{MgO}111}$, $V_{\text{MgO}200}$, and $V_{\text{MgO}220}$ with corresponding uncertainties $\sigma_{\text{MgO}111}$, $\sigma_{\text{MgO}200}$, and $\sigma_{\text{MgO}220}$. The final MgO unit cell volume was calculated as the uncertainty-weighted average using

$$V_{\text{MgO}} = \frac{\frac{V_{\text{MgO}111}}{\sigma_{\text{MgO}111}^2} + \frac{V_{\text{MgO}200}}{\sigma_{\text{MgO}200}^2} + \frac{V_{\text{MgO}220}}{\sigma_{\text{MgO}220}^2}}{\frac{1}{\sigma_{\text{MgO}111}^2} + \frac{1}{\sigma_{\text{MgO}200}^2} + \frac{1}{\sigma_{\text{MgO}220}^2}}. \quad (1)$$

Overlapping peaks and those with weak intensity were excluded from the volume calculations. In run #1, the 111, 200, and 220 peaks were used to determine the MgO volume, the 111 and 220 peaks for Pt, and the 111, 200, and 220 peaks for Ne. In run #2, the 111, 200, and 220 peaks were used for both MgO and Au volume determination, while the 111 and 200 peaks were used for Ne.

To evaluate the macroscopic stress in the DAC sample chamber, the stress in Ne was estimated using the line-shift method [29,30]. This method analyzes the relative shifts of diffraction peaks to derive the differential stress, i.e., the difference between the maximum stress direction (typically along the compression axis) and the minimum stress direction (typically along the radial axis) within the DAC sample chamber. The elastic constants of Ne used in the stress calculations were taken from Ref. [31] (see also Supplemental Material Fig. S2 [25]). Because the 220 reflection was weak in run #1 and absent in run #2, only the 111 and 200 reflections of Ne were used for the stress estimations, ensuring consistency between the two runs and with previous static studies [21,22].

Although the EoS for MgO, Pt, Au, and Ne have been widely studied, calculated pressure differences between EoS from different studies can exceed 5 GPa in the 50–100 GPa range [32]. Even within the same study, interphase pressure differences can exceed 5 GPa at 150 GPa [33]. Such inconsistencies hinder the comparison of subtle pressure differences between phases during fast compression. To minimize discrepancies arising from variations in the EoS, we derived internally consistent EoS for MgO, Ne, Pt, and Au (Table I) from previously reported static DAC experiments in which high-temperature annealing was used to reduce nonhydrostatic stresses [22,33–36]. Details of the procedure used to derive this internally consistent EoS are provided in the Supplemental Material [25] (and references therein [17,22,32–36]). We followed the approach that was previously applied to MgO and Au by Ref. [35].

III. RESULTS AND DISCUSSION

Figure 1 shows the pressure profiles of the two dDAC runs. Both begin with a ramp compression, followed by a pressure plateau without further compression or decompression, and end with three groups of loading cycles with increasing amplitudes, reflecting the programmed compression paths described in the Methods section. Supplemental Material Fig. S4 [25] shows contour plots of 2800 integrated diffraction patterns from run #1 and 2700 integrated diffraction patterns from run #2. In run #1, pressure cycling is clearly visible as oscillations of the diffraction peak in the contour plot.

A. Ramp compression

During ramp compression, the pressure of MgO in run #1 increased from 20 to 75 GPa over 120 s, corresponding to an average compression rate of 0.46 GPa s^{−1}. In run #2, the MgO pressure increased from 28 to 88 GPa over 270 s, at an average compression rate of 0.22 GPa s^{−1}. Although the piezo voltage was programmed to increase linearly with time, the compression rate ($\Delta P/\Delta t$) was not constant during the ramps, particularly in run #1 [see Figs. 1(b) and 1(c)]. The compression rate increased from ~ 0.25 to 0.7 GPa s^{−1} in the first half of the ramp and then decreased to ~ 0.4 GPa s^{−1} in the second half. In contrast, the compression rate was relatively stable in run #2. These changes in compression rates, as typically observed in dDAC experiments [2], reflect the response of the entire mechanical system, i.e., changes in resistance of components of the DAC, such as the gasket and pressure medium. In our study, this variation enables the investigation of the effect of compression rate on the derived pressure-volume relationships.

Figure 2 shows the pressure-volume (P - V) and pressure-pressure (P - P) relationships among MgO, Ne, Pt, and Au obtained from the two dDAC runs. Overall, the derived curves, particularly those from run #2 (the slow compression run with little changes in compression rates), agree well with results from previous static compression experiments [22,32,33]. The close agreement of run #2 with previous static compression experiments indicate that continuous compression experiments with moderate compression rates are well suited to study the elastic behavior of materials under pressure (e.g., Ref. [37]).

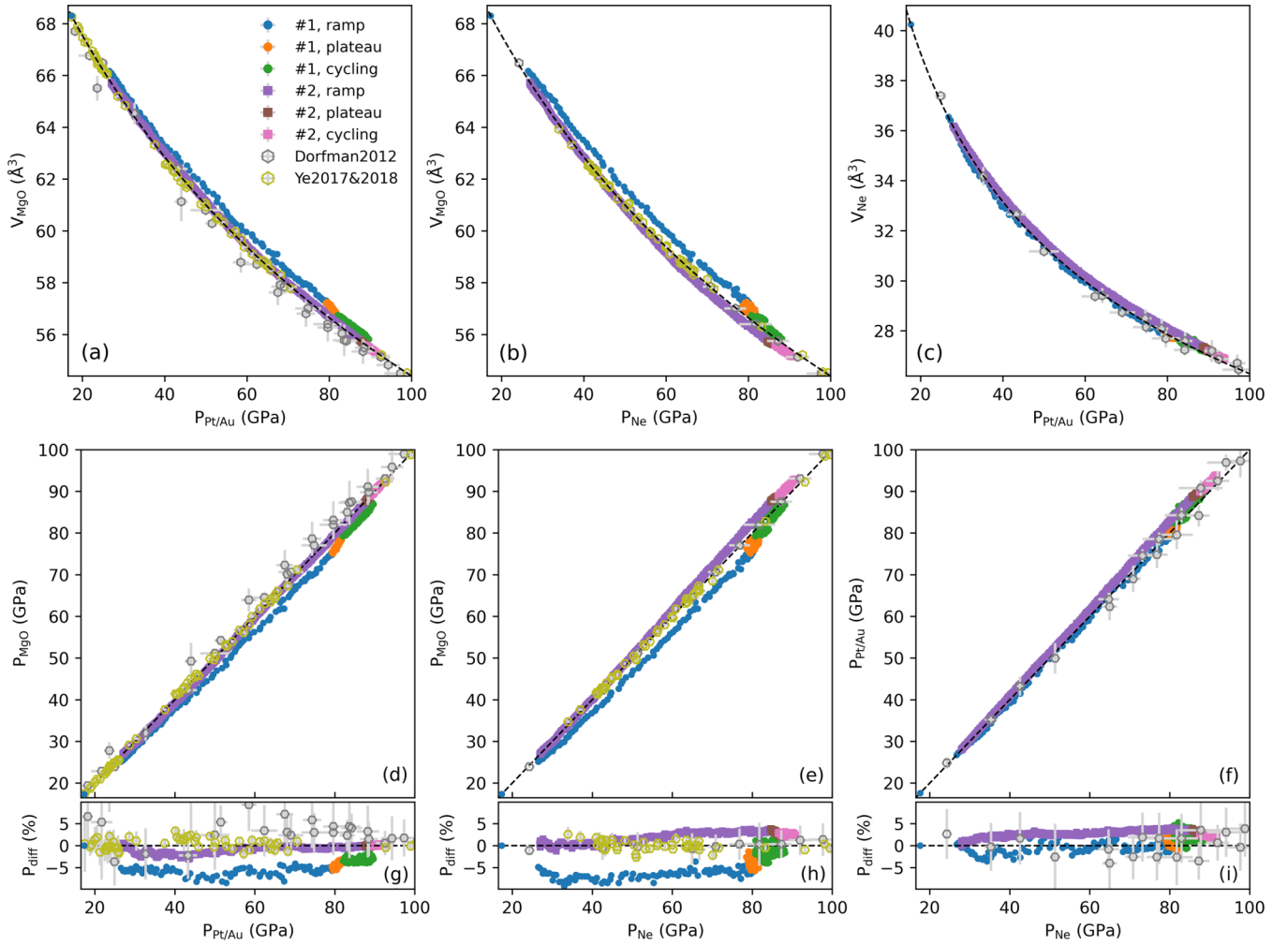


FIG. 2. Pressure-volume (P - V) and pressure-pressure (P - P) relationships between phases in two dDAC runs, compared with previous studies. (a) $P_{\text{Pt/Au}}-V_{\text{MgO}}$, (b) $P_{\text{Ne}}-V_{\text{MgO}}$, (c) $P_{\text{Pt/Au}}-V_{\text{Ne}}$, (d) $P_{\text{Pt/Au}}-P_{\text{MgO}}$, (e) $P_{\text{Ne}}-P_{\text{MgO}}$, and (f) $P_{\text{Ne}}-P_{\text{Pt/Au}}$. Panels (g), (h), and (i) show the relative pressure differences (in percent) between the phases in panels (d), (e), and (f), respectively. Dashed black lines in panels (a), (b), and (c) represent the expected P - V curves based on the internally consistent EoS (Table I). Dashed black lines in panels (d), (e), and (f) indicate the 1:1 line. For comparison, previous high-temperature annealed experimental data [22,33] and cold compression data [32] are also plotted. Pressures of previous studies are recalculated using the internally consistent EoS. Error bars represent uncertainties propagated from peak fitting only, which are smaller than the symbol size in most cases. The color version of this figure is available online.

A systematic pressure difference of $\sim 5\%$ between P_{MgO} and P_{Pt} [Fig. 2(g)], and $>5\%$ between P_{MgO} and P_{Ne} [Fig. 2(h)], is observed in run #1. The offset between P_{MgO} and P_{Pt} is similar to the differences observed in a previous static experiment without high-temperature annealing to minimize nonhydrostatic stress [32]. In contrast, the larger discrepancy between P_{MgO} and P_{Ne} exceeds those observed in earlier studies [22,32]. Since all pressures were recalculated using internally consistent EoS parameters (Table I), we interpret these systematic differences to be caused by rapid compression, possibly resulting from deviatoric stresses. We note that in our experiments, both the sample and the pressure standard are surrounded by the PTM (Supplemental Material Fig. S1 [25]). We thus expect any differences in the behavior of the PTM, sample, and pressure standard to be related to differences in their (time-dependent) physical behavior.

In this study, we focus on the comparison of MgO and the Ne PTM, between which the largest pressure difference

is observed. To test whether the systematic deviations from the expected compression behavior in run #1 (Fig. 2) are related to deviatoric stress during dynamic compression, we calculated the differential stress of Ne using the line-shift method [29,30] as shown in Fig. 3 and Supplemental Material Fig. S6 [25]. Because the Ne PTM is the softest material in the sample chamber of the DAC and surrounds the sample and the pressure calibrant, the differential stress in Ne determines the overall stress within the sample chamber assuming isostress conditions.

Overall, the Ne differential stresses in the two dDAC runs are comparable to those reported for traditional cold-compression DAC experiments (Fig. 3 and Supplemental Material Fig. S6 [25]). However, in run #1—the fastest compression run—the derived Ne differential stress increases with compression rate [compare Figs. 3(a) and 1(c)]. The transition from ramp to plateau is accompanied by a decrease of the differential stress by ~ 0.5 GPa. In static compression exper-

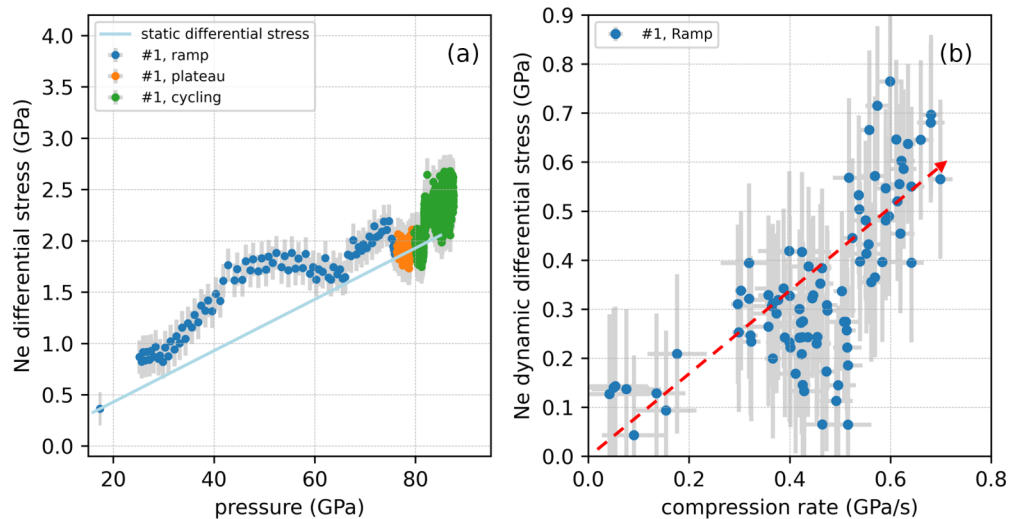


FIG. 3. Ne differential stress in run #1: (a) Ne differential stress as a function of pressure and (b) “dynamic differential stress” as a function of compression rate. In panel (a), the light blue line represents the “static differential stress,” estimated by linearly interpolating between a measurement taken under static conditions before the ramp compression and data collected during the plateau after the ramp. In panel (b), the dynamic differential stress is obtained by subtracting the static differential stress (light blue line) from the measured Ne differential stress (solid blue points). The red dashed line serves as a visual guide to illustrate the compression rate dependence of the dynamic differential stress. Three diffraction peaks (111, 200, and 220) were used for stress determination before the ramp (17.3 GPa), with the error bar reflecting the fitting uncertainty (Supplemental Material Fig. S7 [25]). Stress values during the dDAC compression experiment are based on only two peaks (111 and 200) and we assumed the uncertainty to mirror that at 17.3 GPa. The color version of this figure is available online.

iments, it is generally assumed that the soft PTM reaches its static flow stress (usually assumed to be a proxy for the yield strength), thereby limiting the maximum supported differential stress. For dynamic compression, however, there might be insufficient time to relax differential stresses by plastic flow of the PTM, allowing it to support larger differential stresses suggesting a larger flow stress.

To estimate the excess stress supported by neon during our dynamic compression experiment, we assume that the static differential stress in Ne increases linearly with pressure and estimate its pressure dependence by interpolating between the measurement taken under static conditions before the ramp compression and data collected during the plateau after the ramp [Fig. 3(a)]. We then attribute the excess Ne differential stress to dynamic compression and refer to it as “dynamic differential stress.” We find this component to increase with compression rate reaching 0.5–0.6 GPa at a compression rate of 0.6 GPa s^{-1} [Fig. 3(b)]. Although the dynamic differential stress in our experiments is smaller than the static component, its compression rate dependence suggests that it may reach substantially higher values in faster compression experiments. Notably, compression rates realized in dDAC experiments are routinely on the order of 100 GPa s^{-1} , with reported maxima up to $160\,000 \text{ GPa s}^{-1}$ [2,4,6].

B. Postramp plateau

During the postramp plateau, the MgO pressure increased by 3.30(5) GPa over 5 min in run #1 and by 1.50(5) GPa over 12 min in run #2 (Supplemental Material Fig. S5 [25]). Relaxation of the entire mechanical system likely contributes to this time-dependent pressure increase and the plastic deformation of the gasket and Ne PTM continues after the diamonds stop

compressing. The evolution is nonlinear, with $\sim 80\%$ of the total increase occurring within the first 200 s.

Another observation during the pressure plateau is the development of pressure differences between the Ne PTM, MgO, Pt, and Au. In run #1, the Pt pressure increased by 3.20(10) GPa in 5 min, closely matching the 3.30(5) GPa increase in MgO, while the Ne pressure increased by only 1.90(5) GPa. The MgO pressure increase is about 74% greater than the Ne pressure increase. In run #2, the Au pressure increased by 1.40(15) GPa in 12 min, close to the 1.50(5) GPa increase in MgO, whereas the Ne pressure increased by 2.05(20) GPa. The MgO pressure increased 26.8% less than Ne.

The close agreement in pressure changes for MgO, Pt, and Au suggests that these materials were under near-isostress elastic conditions in the Ne PTM. In contrast, the different behavior of the Ne PTM might indicate time-dependent relaxation of remnant deviatoric stress in Ne and the surrounding gasket material. Ne releases some accumulated stress to the sample, causing a larger pressure increase during the plateau in the sample and pressure standard than in Ne in the fast compression run.

C. Cyclic loading

Run #1 had three groups of loading cycles with pressure amplitudes of approximately 0.1, 0.2, and 1.3 GPa, each consisting of 20 cycles at 0.020 Hz. Run #2 also had three groups of loading cycles with pressure amplitudes of approximately 0.1, 0.5, and 1.2 GPa, each consisting of 10 cycles at 0.167 Hz (Fig. 1). The cyclic loading segments were quantitatively evaluated by fitting the volume strain (f)-time and pressure-time data with a function composed of a sinusoidal and a poly-

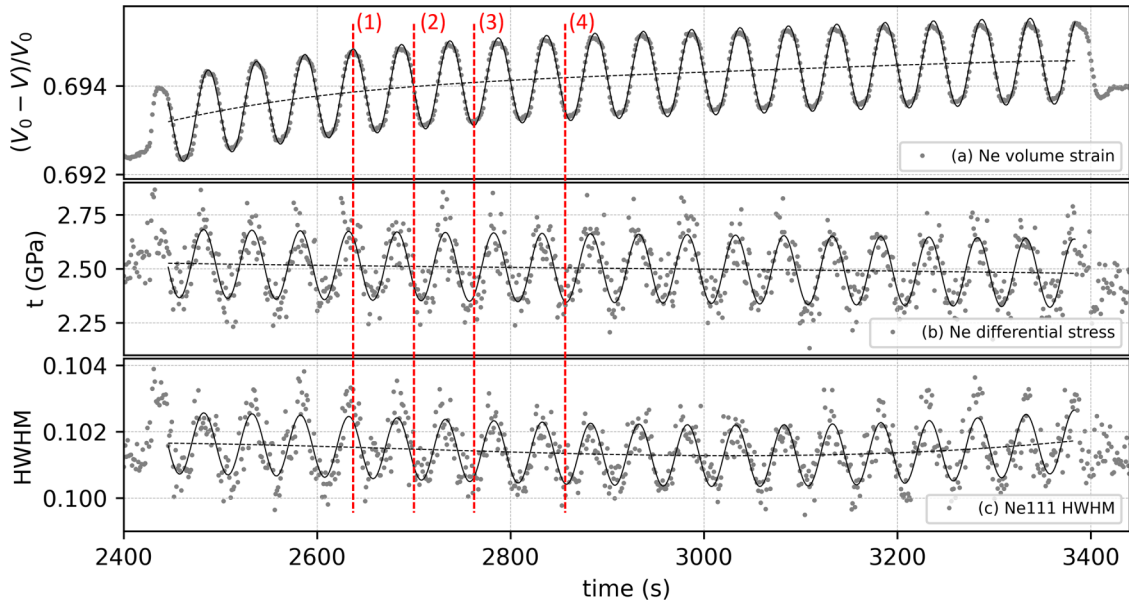


FIG. 4. Magnified view of the third cyclic loading segment in run #1. (a) Volume strain $(V_0 - V)/V_0$ of Ne, calculated using the 111 reflection, which should be least sensitive to differential stress [21]. (b) Differential stress (t) in Ne calculated from the reflections 111 and 200. (c) Half-width at half-maximum (HWHM) of the 111 diffraction peak of Ne. Solid lines represent fits to the data using a function composed of a sine term and a polynomial term as explained in the text. The nonoscillating component of the fit is shown as the dashed lines. The red vertical dashed lines [aligned with the (1) maximum, (2) average, and (3) minimum volume strain, and (4) minimum of differential stress and HWHM] are reference lines to show the phase shift between the volume strain, the differential stress, and the peak width. The color version of this figure is available online.

nomial term: $A \sin(2\pi t/T + \varphi) + (\alpha_1 + \alpha_2 t + \alpha_3 t^2 + \alpha_4 t^3)$, where t is time, A the amplitude, T the period, φ the phase shift, and α_{1-4} the polynomial coefficients. The sinusoidal term captures the cyclic loading component. The polynomial term accounts for the nonoscillating component.

Supplemental Material Table S2 [25] summarizes the fitting results. In run #1, the amplitudes of pressure (P) oscillations differ among the three materials mostly in the order $\Delta P_{\text{MgO}} > \Delta P_{\text{Ne}} > \Delta P_{\text{Pt}}$, while the volume strain (f) amplitudes follow the order $\Delta f_{\text{Ne}} > \Delta f_{\text{MgO}} > \Delta f_{\text{Pt}}$. In run #2, the pressure amplitudes ranked $\Delta P_{\text{Au}} > \Delta P_{\text{MgO}} > \Delta P_{\text{Ne}}$, and the strain amplitudes ranked $\Delta f_{\text{Ne}} > \Delta f_{\text{MgO}} > \Delta f_{\text{Au}}$. At the pressures of cyclic loading, the elastic bulk moduli (K) of the four materials decrease in the order of $K_{\text{Pt}} > K_{\text{Au}} > K_{\text{MgO}} > K_{\text{Ne}}$ (Supplemental Material Fig. S8 [25]). Overall, samples with bulk modulus (K) exhibit larger pressure amplitudes and smaller volume strain amplitudes, with the only exception of Pt in run #1, possibly as a result of its high bulk modulus that leads to small peak shifts under cyclic loading that are difficult to resolve. We note that pressures derived from different diffraction lines of the same material significantly differ because of stresses present at high pressure.

The observed differences in pressure and volume strain amplitudes among the different materials generally fall in between experimental isostress and isostrain conditions during cyclic loading.

The results of cyclic loading experiments provide further insight into the time dependence of Ne flow strength during dynamic loading. Figure 4 shows the third cyclic loading segment of run #1, where oscillatory loading produces a measurable change in the differential stress of Ne [Fig. 4(b)]. Before the onset of oscillations, the measured Ne stress was

~ 2.5 GPa. During the loading cycles, the Ne differential stress varied periodically around this value with an amplitude of ~ 0.25 GPa. This variation is about an order of magnitude larger than expected from the pressure dependence of Ne flow strength, i.e., a pressure change of ~ 1 GPa would be expected to change the static flow strength of Ne by only ~ 0.025 GPa (Fig. 3).

The Ne differential stress and the peak width of the 111 reflection of Ne (the strongest Ne reflection, where the width change was resolvable) are in phase, i.e., the maximum of differential stress and peak width occur at the same time. The Ne differential stress reflects the difference between the maximum stress (typically along the compression axis) and the minimum stress (typically along the radial direction) within the DAC. Varying peak width indicates changes in microstress between grains under the assumption of constant grain size, with larger widths corresponding to higher microstress [29].

Interestingly, there is a resolvable time offset between the maximum Ne differential stress and the maximum Ne volume strain $(V_0 - V)/V_0$ during the cycles: the maximum differential stress is reached between the points of maximum (de)compression rate and the maximum or minimum pressure. This behavior suggests that the observed Ne differential stress is sensitive to both compression rate and pressure, consistent with conclusions drawn from the ramp compression section, i.e., that there is a “dynamic differential stress” component that depends on compression rate in addition to the static differential stress component, which is a function of pressure.

Figure 5 shows magnified views of the first two cycles of the largest amplitude oscillations in each run [Figs. 5(a)–5(c) for run #1 and Figs. 5(d)–5(f) for run #2]. In run #1, two cycles lasted 100 s, corresponding to a frequency of 0.02 Hz

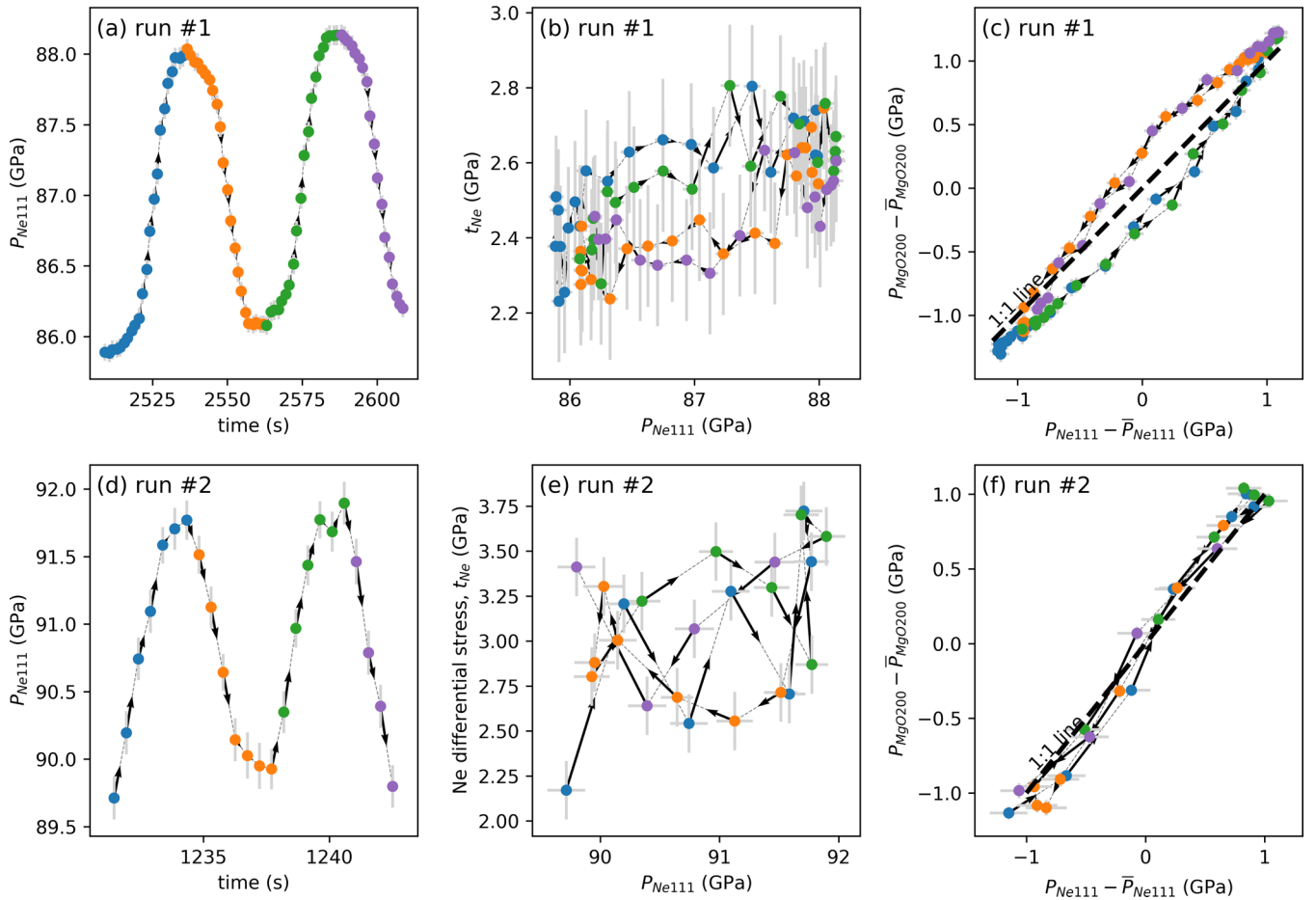


FIG. 5. Magnified view of the first two cycles in the third cyclic loading segments of run #1 (a)–(c) and run #2 (d)–(f). Compression segments are shown with blue and green symbols, decompression segments with orange and purple symbols. (a, d) Ne pressure as a function of time. (b, e) Ne differential stress versus pressure. (c, f) Relative pressure changes in MgO and Ne. Pressures P_{Ne111} and P_{MgO200} were derived from the 111 reflection of Ne and the 200 reflection of MgO, respectively, which are the strongest diffraction reflections of each material and should be least sensitive to stress. $\bar{P}_{\text{Ne111}}$ and $\bar{P}_{\text{MgO111}}$ denote the average pressures of Ne and MgO over the two selected cycles, respectively. Grey error bars indicate uncertainties. The color version of this figure is available online.

[Fig. 5(a)], whereas in run #2, two cycles lasted 12 s, corresponding to 0.167 Hz.

In run #1, a clear hysteresis between Ne differential stress and pressure is observed (Fig. 5(b) and Supplemental Material Fig. S9b [25]). Compression segments have higher differential stress than the decompression segments at the same pressure, indicating a time-dependent but recoverable response of Ne differential stress to cycling loading.

Hysteresis is also observed when looking at the relative pressure changes in MgO and Ne (Fig. 5(c) and Supplemental Material Fig. S9c [25]). In the first half of the compression segment (increasing compression rate), the Ne pressure increases more rapidly than the MgO pressure, causing a deviation from the 1:1 line, whereas in the second half, MgO pressure increases more rapidly. A similar pattern is observed during decompression: the Ne pressure initially decreases faster than the MgO pressure, but later in the cycle the MgO pressure decreases more rapidly. Mirroring observations during the postramp plateau, we interpret these observations as a viscoelastic/plastic response of Ne and/or the gasket material. We note that the yield strength of rhenium at 80 GPa is >10 GPa [38], which is about four times that of neon (Fig. 5).

The observed behavior might thus be more likely attributed to plastic flow in the Ne pressure medium.

Figures 5(d)–5(f) show the third cyclic loading segment of run #2 with loading cycles at a frequency ~ 10 times higher but with comparable pressure amplitude to those in run #1. In this case, no measurable time lag was observed in differential stress [Fig. 5(e)] or relative pressure change [Fig. 5(f)], likely because the higher cycling frequency leads to predominantly elastic behavior of samples, pressure medium and gasket material. We note that comparing the first two cycles (Fig. 5) and the last two cycles (Supplemental Material Fig. S9 [25]) does not indicate changes in Ne or MgO's behavior over time during the cycles performed here.

In addition to the effect of frequency, the response of Ne to cyclic loading is also affected by the amplitude of loading cycles. Figure 6 compares the pressure evolution of Ne and MgO during three groups of cyclic loading at differing amplitudes in run #1. Hysteresis is evident only in the third group, with a MgO pressure cycling amplitude of approximately 1.3 GPa [Fig. 6(c)] but is absent in the first and second groups with amplitudes below 0.2 GPa [Figs. 6(a) and 6(b)]. This indicates that Ne and/or the gasket material act elastically to loading

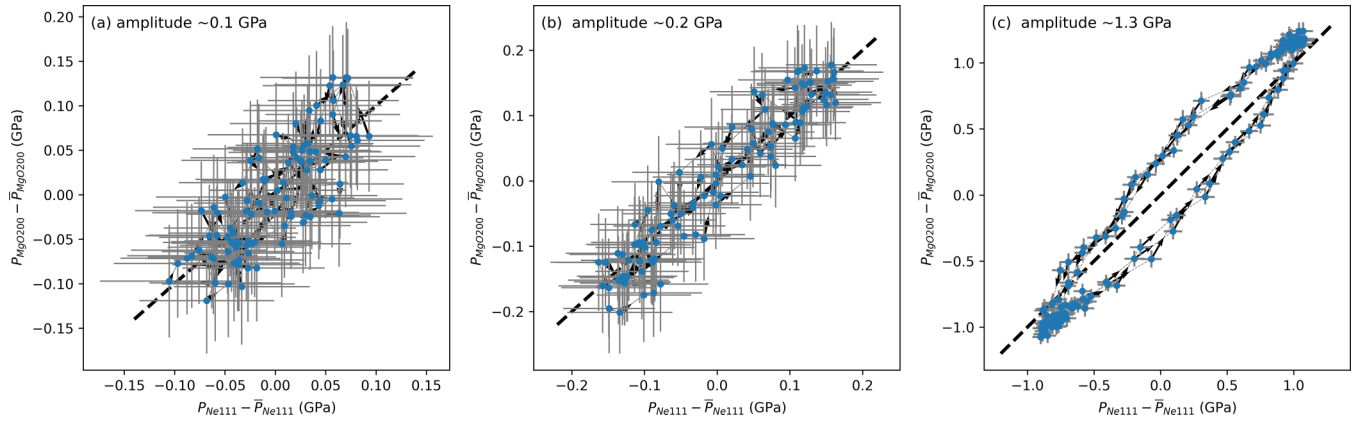


FIG. 6. Magnified view of the relative pressure changes during the three different cyclic loading segments of run #1 (last three cycles for each). (a) First segment with amplitude ~ 0.1 GPa, (b) second segment with amplitude ~ 0.2 GPa, and (c) third segment with amplitude around 1.3 GPa, all at a frequency of 0.02 Hz. Black arrows indicate the progression of time. No clear hysteresis is observed in panels (a) and (b), whereas hysteresis is evident in panel (c). The progression of time indicated by the arrows is most apparent in panel (c). Pressures P_{Ne111} and P_{MgO200} were derived from the 111 reflection of Ne and the 200 reflection of MgO, respectively, which are the strongest diffraction reflections of each material and those least sensitive to stress. Grey error bars indicate uncertainties propagated from peak fitting. The color version of this figure is available online.

cycles with low amplitudes, but start to show plastic behavior when the loading amplitude increases to a certain value, in our case, about 1.3 GPa.

IV. CONCLUSIONS AND IMPLICATIONS

We conducted two continuous compression experiments on MgO in a Ne PTM using time-resolved XRD in a dynamic DAC. Tailored compression paths consisting of a ramp compression, a postramp plateau, and three segments of cyclic loading with increasing amplitudes were applied. Time-resolved XRD enabled monitoring of changes in the diffraction patterns throughout the loading path.

The pressure-volume relations derived from ramp compression closely match those from previous static DAC studies; however, small systematic deviations appear in the fast-ramp run, which had an average compression rate of 0.46 GPa s^{-1} . While the differential stress in Ne during both dDAC runs is overall comparable to earlier cold-compression DAC experiments, compression-rate dependent effects were detected.

Pressure during the postramp plateau increased by up to 2.6 GPa, where $\sim 80\%$ of the pressure increase occurred within the first 200 s, likely due to relaxation of the entire coupled mechanical DAC system. Pressure increases in MgO, Pt, and Au followed near-isostress conditions, whereas the pressure evolution of Ne deviated, possibly because of remnant differential stresses in the Ne PTM that need time to relax.

The cyclic loading also shows evidence of viscoelastic/plastic behavior at low frequencies and large pressure amplitudes. During cyclic loadings, the response of the PTM and the sample appears to be governed by different relaxation times. Early in a ramp, neon takes up more of the load (pressure rises faster in neon). As the maximum compression rate is surpassed, the pressure observed in MgO catches up. At fast cycling frequency, the composite system behaves like a purely elastic assemblage and differences in apparent

pressure between PTM and MgO cannot be resolved. Our findings suggest that time-resolved XRD in the dynamic DAC might be a feasible way to study viscoelasticity/plasticity, as well as its amplitude- and frequency-dependence. Future work, however, is needed to more confidently separate the sample response from that of the coupled mechanical system dDAC.

Future work should also aim to connect observations to the underlying physical principles, which are beyond the scope of this study. This may be achieved through (1) advanced analysis of pressure/strain-time relationships, e.g., using Fourier transform analysis, and (2) integration with numerical modelling, e.g., elastic-viscoplastic modelling [39,40]. The recently developed resistively heated dynamic diamond anvil cell [3] or laser-heating in the dDAC could facilitate similar experiments at elevated temperatures where viscoplastic processes are expected to be enhanced. Moreover, combining pressure cycling in the time-resolved XRD in dDAC with radial scattering geometry could provide a powerful approach for further probing the time-dependence of viscoelastic/plastic processes [11,12].

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DATA AVAILABILITY

The data that support the findings of this article are openly available [41].

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