



RESEARCH LETTER

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Key Points:

- Samples in diamond anvil cell experiments contract and expand in a strongly non-isotropic fashion upon compression and decompression
- Experimental reports on iron conductivity at high pressure contain errors due to the assumption of isotropic contraction or expansion
- Accurate *in situ* determination of sample geometry is necessary for thermal and electrical conductivity measurements at high pressure

Supporting Information:

Supporting Information may be found in the online version of this article.

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Non-Isotropic Contraction and Expansion of Samples in Diamond Anvil Cells: Implications for Thermal Conductivity at the Core-Mantle Boundary

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Abstract The thermal conductivities of mantle and core materials have a major impact on planetary evolution, but their experimental determination requires precise knowledge of sample thickness at high pressure. Despite its importance, thickness in most diamond anvil cell (DAC) experiments is not measured but inferred from equations of state, assuming isotropic contraction upon compression or assuming isotropic expansion upon decompression. Here we provide evidence that in DAC experiments both assumptions are invalid for a range of mechanically diverse materials (KCl, NaCl, Ar, MgO, silica glass, Al₂O₃). Upon compression, these samples are ~30–50% thinner than expected from isotropic contraction. Most surprisingly, all the studied samples continue to thin upon decompression to 10–20 GPa. Our results partially explain some discrepancies among the highly controversial thermal conductivity values of iron at Earth's core conditions. More generally, we suggest that *in situ* characterization of sample geometry is essential for conductivity measurements at high pressure.

Plain Language Summary The thermal and electrical conductivities of the materials making up Earth's core and lowermost mantle are crucial inputs for modeling Earth's interior and the geodynamo mechanism. Yet, large disagreements between published values of conductivity are common, including a factor-of-seven discrepancy in the thermal conductivity of iron at core-mantle boundary conditions. One possible source of systematic uncertainty is the estimate of sample thickness during high-pressure experiments. Here we show that common materials in compression experiments tend to thin by much more than previously assumed. Surprisingly, the thinning continues upon decompression. These thinning trends could lead to ~30–50% systematic error, partially explaining the discrepancy in iron conductivity. *In situ* thickness measurements are thus crucial for accurate determination of conductivities of Earth's mantle and core.

1. Introduction

The cooling rate of the core is controlled by the thermal conductivity of the mantle at the core-mantle boundary (CMB) (Lay et al., 2008). A thermally-stratified outermost core develops if the mantle is not able to remove the heat supplied by conduction at the top of the core (Lister & Buffett, 1998; Mound et al., 2019). This is a plausible scenario given the extant data on the thermal conductivities of the mantle and core materials (Williams, 2018). Crystallization of the inner core leaves lower density elements in the liquid, causing compositional buoyancy to be a major driver of present-day core convection (Driscoll & Du, 2019). Prior to inner core nucleation, however, it is less clear whether thermal or compositional convection alone could have driven the geodynamo (Davies, 2015; Du et al., 2019; Pozzo et al., 2012). And yet Earth's paleomagnetic record is much older (at least 3.4 Gy (Tarduno et al., 2010; Tarduno et al., 2020)) than current estimates of inner core age (~1 Gy (Bono et al., 2019; Davies, 2015; Driscoll & Bercovici, 2014; Pozzo et al., 2012)), so some combination of buoyancies certainly drove dynamo action. The plausibility of thermally driven convection in the core prior to inner core nucleation depends on many factors, notably thermal conductivity of iron alloys (Davies, 2015; Driscoll & Bercovici, 2014; Landeau et al., 2022). Recent experimental reports on the thermal conductivity of iron at the CMB differ by nearly a factor of seven, ranging from 33 to 226 W/m/K (Gomi et al., 2013; Konopkova et al., 2016; Ohta et al., 2016; Zhang et al., 2020). The source of this large discrepancy has not been identified.

High-pressure experiments in diamond anvil cells (DACs) allow measurements of thermal conductivity at CMB conditions, but accurate knowledge of the sample thickness is crucial in these experiments (Zhou et al., 2022). Despite their importance, the thicknesses of samples are almost never measured *in*

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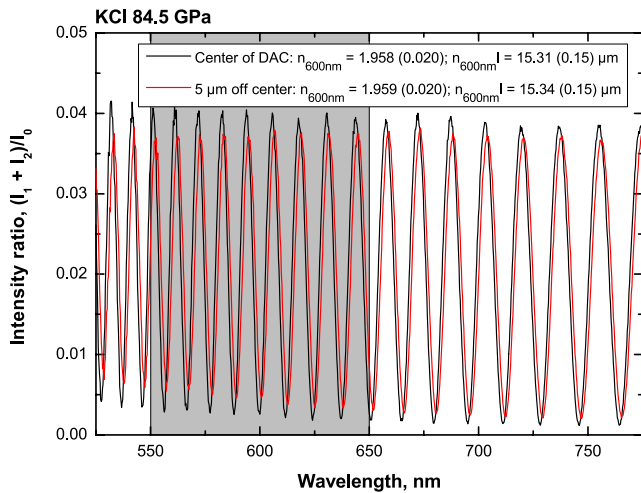


Figure 1. Intensity ratio spectra ($(I_1 + I_2)/I_0$) measured in KCl at 84.5 GPa at the center of the diamond anvil cell (DAC) cavity and $\sim 5 \mu\text{m}$ off the center. See Lobanov et al. (2022) for graphical definitions of the measured quantities. The gray box depicts the spectral range used for averaging the signal to obtain the refractive index at 600 nm ($n_{600\text{nm}}$). The different contrast of the two fringe patterns is due to diamond cupping (van Straaten & Silvera, 1988), which apparently has no effect on the inferred refractive index. The optical path length ($n_{600\text{nm}}l$) is $\sim 30 \text{ nm}$ longer when measured off center from the axis of the diamond anvils. This is not a significant source of error in sample thickness.

situ in DACs. Instead, the thickness of each sample at high pressure, l , is commonly inferred from the measured thickness prior to the experiment (Zhang et al., 2020) or after decompression (Geballe et al., 2020; Gomi et al., 2013), assuming isotropic contraction upon compression or isotropic expansion upon decompression: $l/l_0 = (V/V_0)^{1/3}$, where V_0 and V are the unit cell volumes at 1 atm and high pressure given by the room temperature equation of state. In this study, we employ direct optical measurements of the thickness of samples in DAC experiments and find that it evolves in a strongly non-isotropic fashion for a mechanically diverse set of samples. Using our measurements to correct the results of previous studies for non-isotropic behavior, we find that the discrepancy in iron conductivities is indeed smaller than the discrepancy without our correction. *In situ* measurements of sample thickness are thus crucial for accurate future measurements of thermal conductivity in DACs.

2. Materials and Methods

The flat tips of diamond anvils form an interferometer; thus, the distance between the anvils can be measured directly by analyzing the interference pattern in the spectrum of a reflected (or transmitted) white light if the refractive index of the sample is known (Dewaele et al., 2003; Kim et al., 2021). Here we apply a recently developed approach (Lobanov et al., 2022) to measure the diamond-to-diamond separation upon compression up to $\sim 135 \text{ GPa}$ (P at the CMB) and subsequent decompression. Briefly, our approach involves reflecting a broadband laser probe from the diamond-sample-diamond assemblage and recording the reflected signal on a charge-coupled device. The ratio of incoming (I_0) and reflected ($I_1 + I_2$) intensities (Figure 1)

averaged over 550–650 nm allows finding the refractive index at 600 nm ($n_{600\text{nm}}$). The sample thickness (l) is then obtained from the spectral separation of the observed extrema which yields the optical path length ($n_{600\text{nm}}l$) under the assumption that index dispersion in the measured spectral range is small. The overall uncertainty in l is $\sim 1\%$ (Lobanov et al., 2022).

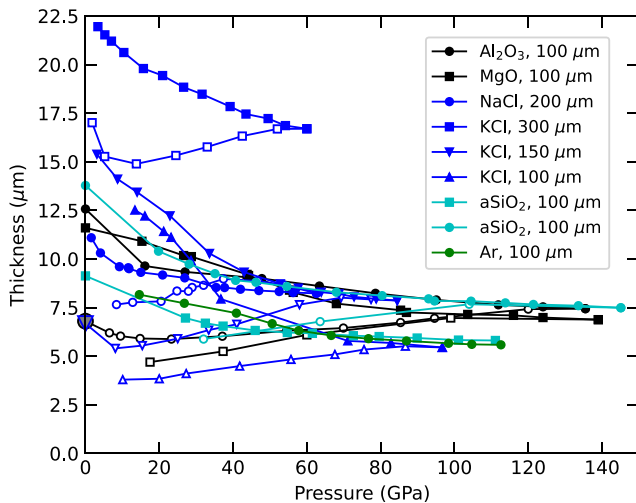


Figure 2. Diamond-diamond distance (sample thickness) in diamond anvil cell experiments with Al_2O_3 , MgO , NaCl , KCl , Ar , and silica glass (aSiO_2) as samples on compression (solid symbols) and decompression (hollow symbols). Gray shading inside symbols marks the thicknesses of fully decompressed samples measured with an SEM. The legend also indicates the diamond culet diameter. The uncertainty in sample thickness is $\sim 1\%$.

We prepared sample chambers by indenting 250 μm thick rhenium foils between 300 or 200 μm flat anvils or 300/150 μm or 300/100 μm beveled anvils and subsequently laser-drilling holes with diameters of ~ 100 or $\sim 35 \mu\text{m}$ at the center of the indentation. The sample chambers were filled entirely with either SiO_2 glass (aSiO_2), Al_2O_3 , MgO , KCl , NaCl , or Ar . These are common pressure media used in DAC experiments, and they have diverse mechanical properties ranging from highly incompressible Al_2O_3 ($K_0 = 255 \text{ GPa}$ (Oganov & Ono, 2005)) to highly compressible Ar ($K_0 \sim 2.65 \text{ GPa}$ (Dewaele et al., 2021)). The bulk moduli of B1 and B2 NaCl and KCl are intermediate ($K_0 \sim 20\text{--}30 \text{ GPa}$ (Dewaele et al., 2012; Dorogokupets & Dewaele, 2007)) while aSiO_2 is relatively compressible in the pressure range 0–40 GPa but more incompressible at pressures above 40 GPa (Petitgirard et al., 2017). Refractive indices of these materials will be reported elsewhere. Pressure was measured by the diamond Raman edge method with the relative uncertainty of $\pm 5\%$ (for 300/150 and 300/100 μm bevel/culet diameters) (Akahama & Kawamura, 2004) and by the spectral position of the ruby R1 line (for 200 and 300 μm culet diameters) (Syassen, 2008). The thicknesses of several fully decompressed samples were measured by cutting through the gaskets with a focused ion beam (FIB) and directly imaging the thickness in a scanning electron microscope (SEM).

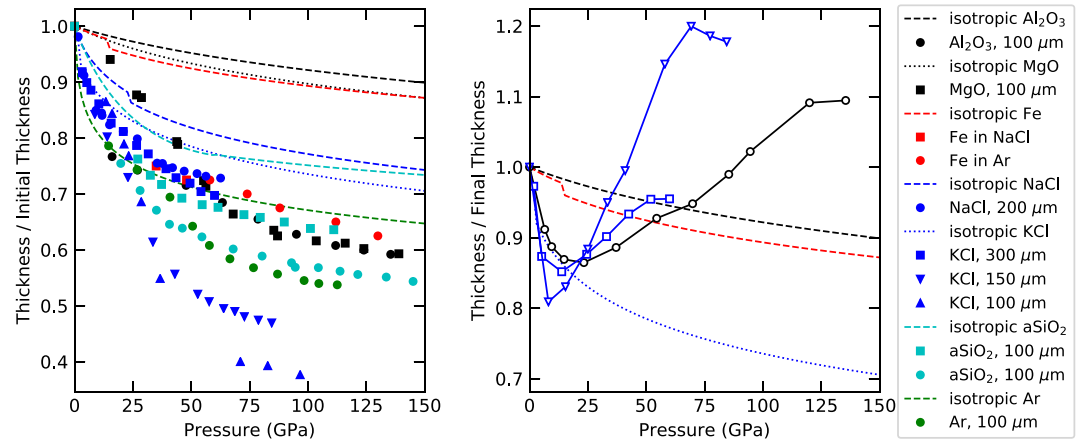


Figure 3. Diamond-diamond distance (sample thickness) normalized to its pre-compression value (LEFT) and after full decompression (RIGHT) as compared to models of isotropic contraction and expansion, following the equations of state in Refs. (Dewaele & Torrent, 2013; Dewaele et al., 2006, 2012; Petitgirard et al., 2017; Speziale et al., 2001). The thicknesses of Fe in NaCl and Ar media are from Konopkova et al. (2016).

3. Results

All absolute sample thicknesses recorded upon compression (solid symbols) and decompression (open symbols) are shown in Figure 2. Regardless of the sample, its thickness upon compression is always smaller than calculated assuming isotropic contraction (Figure 3 LEFT), likely due to uniaxial stress conditions in the sample cavity: $\sigma_{axial} > \sigma_{radial}$. The decrease in normalized thicknesses upon compression to ~ 100 GPa is larger for relatively compressible materials (e.g., KCl, Ar) than for incompressible ones (e.g., Al_2O_3 , MgO) (Figure 3 LEFT). This observation indicates that the thinning upon compression is material-dependent. However, other experimental parameters also affect the evolution of sample thickness. Notably, the diamond culet diameter, the starting gasket thickness, the initial sample chamber diameter, and the sample packing ratio (initial volume of sample/volume of sample chamber). The effect of diamond culet diameter is clear in the relative thickness data for KCl: smaller culets result in greater thinning (Figure 3 LEFT). For a fixed sample and culet diameter, the normalized thicknesses upon compression is reproducible within $\sim 10\%$ (see $aSiO_2$, $100\ \mu m$ culets). Upon decompression, isotropic expansion would predict that all samples thicken. Surprisingly, all samples in this study thin upon decompression from the highest pressure down to ~ 10 – 20 GPa. Upon final decompression from ~ 10 GPa to ambient pressure, all samples thicken, consistent with direct thickness measurements by SEM/FIB in select fully decompressed samples. These decompression trends are similar to that noted in helium by Dewaele et al. (2003). Sample thinning upon decompression is also evident from the radial stretching of the gasket holes filled with Al_2O_3 ($300/100\ \mu m$ bevel/culet diameter) or KCl ($300/150\ \mu m$ bevel/culet diameter). In the case of Al_2O_3 , the radial stretching upon decompression from 135.5 to 9.5 GPa is 33%, while the expected isotropic expansion is only 8% (Figure S1 TOP in Supporting Information S1). Similarly, decompressing KCl from 84.5 to 8 GPa results in a radial expansion of 40%, whereas the expected isotropic expansion is 16% (Figure S2 TOP in Supporting Information S1). Possible reasons for thinning upon decompression are related to forces at the gasket-diamond interface. The observed outward flow of the sample upon decompression could be triggered by the release of stresses on gasket material near the edge of the culet, due to elastic relaxation of the “cupped” diamond culet (Hemley et al., 1997). In addition, a decrease in static friction between diamond and gasket could allow sample material to flow along the diamond-gasket interface, as suggested by the observation of the recovered gasket-sample assemblage (Figure S1 BOTTOM in Supporting Information S1).

4. Discussion

Previous DAC studies of iron conductivity at 135 GPa relied on the assumption of isotropic contraction/expansion of iron samples upon compression/decompression in Al_2O_3 (Gomi et al., 2013) and $aSiO_2$ (Zhang et al., 2020) pressure media. The adequacy of this assumption depends on the extent to which the deformation of iron is affected by the uniaxial stresses developed in the pressure media. Because the yield strength of iron

is smaller than that of Al_2O_3 and of aSiO_2 (Dong et al., 2014; Gleason & Mao, 2013; Hemley et al., 1997; Lacroix et al., 2012; Mao et al., 2008; Singh et al., 1998; Wakabayashi et al., 2015), we assume the linear strain of iron in each dimension is equal to the linear strain of Al_2O_3 or aSiO_2 , and refer to this as the “matching strains assumption” hereafter. When iron is compressed in a pressure medium with lower yield strength, one might expect thickness variations that are relatively close to the isotropic model, at least compared to compression of iron in Al_2O_3 or aSiO_2 . Yet, direct thickness measurements by white-light interferometry reported by Konopkova et al. (2016) show that iron samples in NaCl and Ar media thin by $\sim 25\text{--}38\%$ upon compression to 35–130 GPa, whereas the expected isotropic thinning is only 5%–12%. (Figure 3 LEFT). These data show that the thinning of iron samples is nearly as great as that of NaCl and Ar (despite their relatively low yield strengths). We surmise, therefore, that the “matching strains assumption” may represent the maximum yet common departure from the isotropic thinning model, even for samples in relatively soft pressure media.

These observations motivate us to use our data on the variation of thicknesses of Al_2O_3 and aSiO_2 upon compression and decompression to approximate the error in the thickness estimates of iron samples at 135 GPa caused by the assumption of its isotropic contraction and expansion. Non-isotropic contraction of aSiO_2 to 135 GPa results in 45% thinning in our experiments, whereas the isotropic model for iron used in Zhang et al. (2020) predicts a mere 12% thinning (Figure 3 LEFT). Decompression of alumina from 135 GPa results in 9% thinning in our experiments, whereas the isotropic model for iron used in Gomi et al. (2013) predicts a 9% thickening (Figure 3 RIGHT). These discrepancies, combined with the matching strains assumption, suggest that systematic errors of several tens of percent may be present in the inferred values of conductivity in previous studies.

For metals, thermal conductivity (k) is related to electrical resistivity (ρ) through the Wiedemann-Franz Law:

$$k = \frac{1}{\rho} LT \quad (1)$$

where L is the Lorentz number and T is temperature. In the experiments of Zhang et al. (2020) and Gomi et al. (2013) the resistivity of iron samples is calculated by:

$$\rho = \rho_0 \frac{R}{R_0} \frac{l}{l_0} \quad (2)$$

where ρ_0 is the resistivity at ambient pressure, R_0 (R) and l_0 (l) are resistances and thickness at ambient (high) pressure. Combining this equation with the Wiedemann-Franz Law yields the proportionality, $k \propto \rho^{-1} \propto l_0/l$. Our results suggest that the models of isotropic contraction and expansion underestimate l_0/l and k in experiments where l_0 is measured prior to compression, but overestimate l_0/l and k in experiments where l_0 is measured after decompression.

Following this logic, we now estimate thickness-related errors in k reported by Zhang et al. (2020) and Ohta et al. (2016). Note that here we correct the high-temperature values reported by Ohta et al. (2016) who used the room-temperature conductivities of Gomi et al. (2013) for normalization. Assuming the errors in estimated sample thicknesses at 135 GPa discussed above, we obtain the following corrections to the thermal conductivity of iron at the pressure and temperature conditions near the CMB: $100 \rightarrow 133$ W/m/K (+33%) for Zhang et al. (2020) and $226 \rightarrow 185$ W/m/K (−18%) for Ohta et al. (2016). The proposed corrections bring the experimental values closer together, and closer to the computed conductivities of iron at similar pressure-temperature conditions from the studies of de Koker et al. (2012) and Pozzo et al. (2012). However, more recent computations of the thermal conductivity of hcp-Fe at CMB conditions that consider electron-electron and electron-phonon scattering point toward a lower value of ~ 100 W/m/K (Xu et al., 2018). Importantly, the proposed corrections are merely estimates, because the details of pressure-dependent changes in sample thickness depend on many factors, including the relative yield strength of sample versus pressure medium, the strength and compressibility of the gasket material, the shape and size of electrodes, and the gasket's starting thickness and hole diameter. In addition, samples may irreversibly thin at the laser-heated spot due to the release of stresses. Nonetheless, these corrections demonstrate that the controversy in the thermal conductivity of iron at CMB conditions may be partially reconciled by the revised estimates of sample thickness. Although the thicknesses of iron samples have been measured by white-light interferometry at high pressure in Konopkova et al. (2016), the low values of thermal conductivity of iron (33 W/m/K at the CMB) could be systematically biased due to complex, irreversible variations in samples thickness induced by laser heating.

More generally, a word of caution is in order for many other reports on thermal and electrical conductivity at high pressure. The electrical conductivities of Fe-Si-Ni alloys at CMB conditions reported in Refs. (Zhang et al., 2021, 2022) likely need an upward revision because of the assumption of isotropic contraction. Sample thickness at high pressure is also essential for the experimental techniques that determine lattice thermal conductivity in DACs. In these experiments the inferred values of thermal conductivity, k , depend on estimates of sample thickness, l , with an analytical relationship that can be described by $k \propto l^n$ with $n \geq 1.5$. Yagi et al. (2011) proposed $n = 2$ for the thermoreflectance method, which has subsequently been used to study lattice thermal conductivity of lower mantle minerals (Ohta et al., 2012; Okuda et al., 2017, 2020; Yagi et al., 2011). For the laser flash method, Geballe et al. (2020) suggests that $n = 1.5$. Similarly, the inferred thickness of ~ 100 nm-thick metallic films at high pressure is a crucial parameter for accurate determination of k during time-domain thermoreflectance measurements (Chen et al., 2011). *In situ* measurements of the thickness of these films could be important to test the model of thickness changes during compression used in such studies (Chen et al., 2011; Hsieh et al., 2017, 2018, 2020; Marzotto et al., 2020). Likewise, *in situ* measurement of thickness of metal films on substrates could be important to test the isotropic model of thickness change assumed in picosecond acoustics experiments (Decremps et al., 2014; Edmund et al., 2020). Sample thickness is also essential for the spectroscopic determination of optical absorption coefficients, which serve as primary input for estimates of radiative thermal conductivity (Goncharov et al., 2006, 2008, 2015; Keppler et al., 2008; Lobanov et al., 2017, 2020, 2021; Murakami et al., 2014, 2022; Thomas et al., 2012). Deviations from isotropic contraction and expansion may partially account for the large discrepancy in the estimates of radiative conductivity at the base of the mantle (~ 0.5 – 5 W/m/K). Nonetheless, further direct optical measurements of sample thickness in strong and weak pressure media are needed to test the isotropic contraction/expansion assumption and the matching strains assumption.

In closing, we demonstrated that samples contract and expand in a strongly non-isotropic way in DACs for a range of mechanically diverse samples. In all cases, the decrease in thickness is anomalously large upon both compression and decompression. Future measurements of mantle and core thermal conductivity will need to quantify sample thickness at high pressure.

Data Availability Statement

The data set used in this work is deposited at Mendeley Data, V4 (<https://doi.org/10.17632/wnvbty8y83.4>).

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