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Equation of state of rhenium and application for ultra high pressure calibration

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The isothermal equation of state of rhenium has been measured by powder X-ray diffraction experiments up to 144 GPa at room temperature in a diamond anvil cell. A helium pressure transmitting medium was used to minimize the non-hydrostatic stress on the sample. The fit of pressure-volume data yields a bulk modulus $K_0 = 352.6$ GPa and a pressure derivative of the bulk modulus $K'_0 = 4.56$. This equation of state differs significantly from a recent determination [Dubrovinsky *et al.*, Nat. Commun. **3**, 1163 (2012)], giving here a lower pressure at a given volume. The possibility of using rhenium gasket X-ray diffraction signal, with the present equation of state, to evaluate multi-Mbar pressures in the chamber of diamond anvil cells is discussed. © *2014 AIP Publishing LLC*. [http://dx.doi.org/10.1063/1.4863300]

I. INTRODUCTION

Rhenium (Re) belongs to the group VII of transition metals with a half filled *d* band. The crystalline structure observed for this metal is hexagonal close packed (hcp) up to 660 GPa.[1–4] First principle calculations[5] predict that hcp is more stable against other simple structures up to 1000 GPa at least. Surprisingly, the equation of state (EOS) of hcp rhenium measured in diamond anvil cells under non-hydrostatic compression[3] does not significantly deviate from the recent measurements under quasi-hydrostatic compression in helium pressure transmitting medium.[4] This is in contrast to what has been reported for other metals, such as Ta.[6] Also, there is a disagreement between the EOS of rhenium measured using shock wave experiments, reduced to ambient temperature[7] and the most recent static measurement under quasi-hydrostatic conditions.[4] The two compression curves, which should be identical, differ by 15% in pressure, for the same compression, around 150 GPa.

Rhenium is ductile but has high bulk and shear moduli,[8] a high strength,[9,10] and a high melting point—3453 K, the second of all metals. For these reasons, rhenium has been used as a gasket material for studies under ultra-high pressures in diamond anvil cell. The fact that a rhenium gasket is present in the vast majority of diamond anvil cell experiments performed above 100 GPa makes it a convenient potential pressure X-ray calibrant. In fact, adding new materials in a few micrometers-sized pressure chamber of multi-Mbar experiments[4] is not always possible. For that purpose, a reference EOS has to be established. In addition, it should be determined whether the specific volume of the gasket measured by X-ray diffraction (XRD) can be used for pressure determination without any bias. In fact, the gasket being directly compressed between the anvils, its stress state is complex and probably different from one experiment to another.

We present here data taken under quasi-hydrostatic compression, which reconcile static and dynamic measurements of the EOS of rhenium. We also analyze the XRD signal of rhenium gaskets recorded during several ultra-high pressure experiments, and we evaluate to which extent this signal can be used with the present EOS for an estimation of the sample pressure.

II. MEASUREMENTS WITH HELIUM PRESSURE MEDIUM

Three different experimental runs were carried on the ID27 and ID09 beamlines of the European Synchrotron Radiation Facility. For each run, a sample composed of a grain of powder of Re (Alpha Aesar product, 99.99% purity) was embedded in helium pressure transmitting medium and put a few micrometers away from the pressure gauge in the high pressure chamber of a diamond anvil cell. Re was also used as the gasket material, but thanks to the dimension of the X-ray spot size ($2 \times 3\ \mu$m), much smaller than the high pressure chamber diameter (20 μm minimum), we were able to collect only the signal scattered by the rhenium grain. The pressure was measured from the luminescence of a ruby gauge or from the measurement of the atomic volume of helium pressure medium or tungsten X-ray gauge (see Table I). Tungsten has been chosen as an X-ray pressure gauge because of its high X-ray scattering power and the accuracy of its EOS, attested by the consistency between static, dynamic, and ultrasonic measurements.[11,12] The calibration of ruby and tungsten gauges has been taken from Ref. 13. The calibration of helium gauge is from Ref. 14, with a modification that takes into account the update of the ruby pressure scale.[11,13]

The monochromatic XRD signal has been collected on image plates. The diffraction geometry was determined using LaB_6 and Si reference samples. The signal was circularly integrated using the FIT2D[15] software. Fig. 1 shows the raw and integrated angle-dispersive XRD pattern of Re at different pressures. All the diffraction peaks belong to the hcp structure. The diffraction lines (100), (002), (101), (102), (110), (103), and (112) have been used for the measurement

TABLE I. Conditions of each experimental run. Sizes are in μm; PTM: pressure transmitting medium.

Run	P range (GPa)	P gauge	PTM	Diamond's culet size	Sample size
1	0.64–36.9	Ruby	He	300	4
2	14.0–64.8	He	He	150	3
3	1.45–144	W + Ruby	He	100	3

of the lattice parameters. The raw images of the powder sample show diffraction rings with some texturing, which was already present in the starting sample. With increasing pressure, the diffraction peaks become larger, which is a sign of an increase of the micro-stress in the sample.[16] This micro-stress is different from the macroscopic non-hydrostatic stress, which is qualitatively discussed in the next section.

A. Macroscopic non-hydrostatic stress

It is desirable to achieve purely hydrostatic conditions in the sample chamber of a diamond anvil cell, which would be provided by a liquid pressure transmitting medium. However, even helium becomes solid above about 12 GPa at room temperature and is therefore prone to sustain a non-hydrostatic stress in the sample chamber. In addition, even at lower pressure, the stress on the sample can become non-hydrostatic if it bridges the anvils due to excessive thinning of the gasket or due to a large initial thickness of the sample. The macroscopic stress in a sample can be determined by an analysis of the measured XRD spectra,[16] if the diffraction lines exhibit a measurable shift from hydrostatic compression. Table II shows the interplanar spacings measured at the highest pressure reached (144 GPa) in our experiments. They deviate from the spacings calculated using the lattice parameters obtained by a refinement of the whole spectrum by less than 0.06%. This deviation corresponds to the uncertainty on the measurement of each diffraction peak. We can thus conclude that the non-hydrostatic stress is below the detection limit of our measurements. That is in agreement with the quantification of the macroscopic non-hydrostatic stress on metals embedded in helium that reaches 0.5 GPa at 150 GPa.[17]

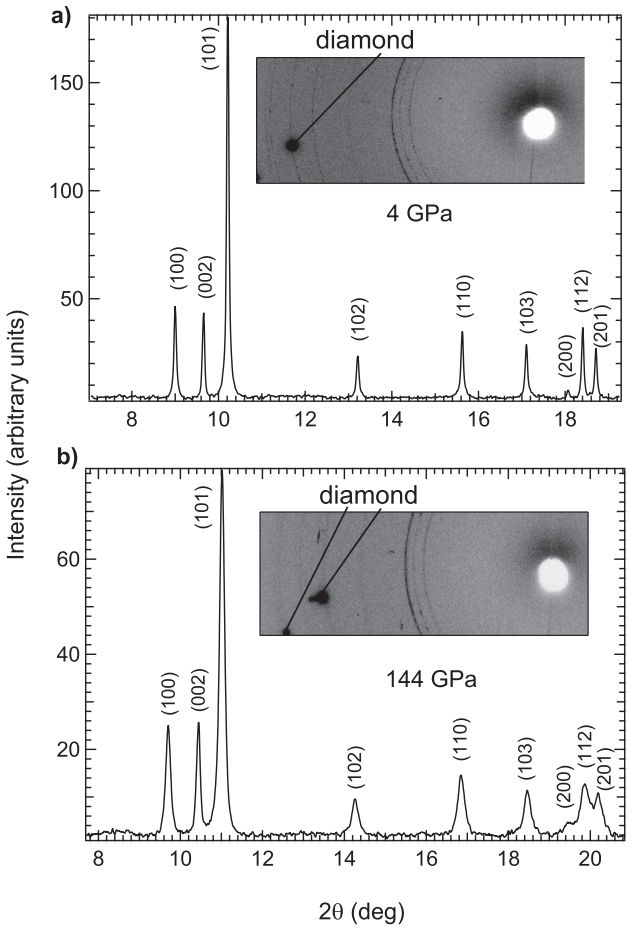

FIG. 1. Integrated powder X-ray diffraction patterns of Re at low (a) and high (b) pressure. The insets show a part of the recorded raw diffraction images. The diamond contribution to the X-ray raw spectra are masked during the integration.

B. Evaluation of the micro-stress

From the analysis of the width of the XRD lines, it is possible to estimate the micro-stress in a powder material, i.e., the difference in stress between the crystallites that constitute the powder.[16] This difference causes a broadening of each XRD peak identified by its Miller indices (*hkl*), which can be expressed as[17]

$$(2w_{hkl}\cos\theta_{hkl})^2 = (2w^0_{hkl}\cos\theta_{hkl})^2 + \eta^2_{hkl}\sin^2\theta_{hkl}, \quad (1)$$

where $2w_{hkl}$ is the full-width at half maximum (FWHM) of the diffraction angle $2\theta_{hkl}$, $2w^0_{hkl}$ its value at $P=0$, η_{hkl} the micro-strain on the (*hkl*) plane related to the micro-stress σ via

$$\sigma = \eta_{hkl}E(hkl)/2, \quad (2)$$

where $E(hkl)$ is the single crystal Young modulus along the direction (*hkl*). An approximate expression for the average micro-stress is

$$\sigma = \langle\eta_{hkl}\rangle E/2, \quad (3)$$

where $\langle\eta_{hkl}\rangle$ represents the average value of η_{hkl} over measured diffraction peaks and E the Young modulus of the

TABLE II. Measured reflections for rhenium at 144 GPa. (*hkl*) are the Miller indices of the reflection. d_m is the corresponding interreticular distance measured by individual peak fitting. d_{calc} is the interreticular distance calculated by a fit of the whole spectrum.

(*hkl*)	d_m (Å)	d_{calc} (Å)	d_m-d_{calc} (Å)
(100)	2.2116	2.2103	0.0013
(002)	2.0550	2.0561	−0.0011
(101)	1.9480	1.9469	0.0011
(102)	1.5056	1.5054	0.0001
(110)	1.2765	1.2761	−0.0004
(103)	1.1650	1.1649	0.0001
(112)	1.0834	1.0843	−0.0009

polycrystalline aggregate.[18] E is related to the bulk and shear moduli K and G of the aggregate

$$E = \frac{9KG}{3K+G}. \quad (4)$$

The variation of E with pressure have been computed using one Re EOS,[7] and the extrapolation of ultrasonic data[8] using the relation[10]

$$G = G_0 x^{-\frac{5}{3}}\left[1+\frac{1}{2}\left(\frac{3K_0G_0'}{G_0}-5\right)\left(x^{-\frac{2}{3}}-1\right)\right], \quad (5)$$

with $x = V/V_0$, $G_0 = 179$ GPa, and $G_0' = 1.8$.[8] The micro-stress σ has been estimated using Eqs. (1) and (3) for seven diffraction peaks and is plotted on Fig. 2. It increases from 0 at the beginning of the experiment to $\simeq$7 GPa at 144 GPa. These values are smaller than the macroscopic uniaxial stress sustained by a rhenium sample (yield strength) compressed directly between the anvils, measured in two studies.[3,9] In Ref. 3, the measured yield strength of Re exceeded 20 GPa at 120 GPa. It is expected that the micro-stress is upper bounded by the yield strength: our measurements are thus compatible with earlier yield strength estimates.[3,9] This micro-stress is isotropic and so does not affect the EOS measurements whereas the macroscopic stress leads to measurable effects.

C. Equation of state

The bulk modulus K_0, its pressure derivative K_0', and the volume V_0 at ambient pressure are determined by a least-squares fit of the present pressure-volume data (Table III) to the Rydberg-Vinet[20] EOS formulation ($x = V/V_0$):

$$P(V) = 3K_0 x^{-\frac{2}{3}}(1-x^{\frac{1}{3}})\exp\left\{1.5\left(K_0'-1\right)\times\left(1-x^{\frac{1}{3}}\right)\right\}. \quad (6)$$

The resulting values are as follows: $V_0 = 8.8726 \pm 0.01$ cm^3/mol, $K_0 = 352.6 \pm 8$ GPa and $K_0' = 4.56 \pm 0.17$. The current and literature EOS parameters are listed in Table IV. The error bars correspond to the 95% confidence interval of the fitted values. The current EOS agrees with the EOS based on shock experiments reduced to ambient temperature[7] (Fig. 3) and with earlier measurements performed in a moderate pressure range.[1,9] It also agrees with a recent density-functional theory calculation within generalized gradient approximation.[21] However, it progressively diverges from the EOS recently measured[4] using diamond anvil cell and helium pressure transmitting medium as the pressure increases. For the same volume, the pressure is higher by up to 13% in Ref. 4 measurements than in our measurements. Only a very small part of this difference can be attributed to pressure gauges calibration issues: if the calibration of Ref. 22 is used for gold X-ray gauge, instead of the calibration of Ref. 23 used in work of Dubrovinsky *et al.*, the pressures differ by less than 2% (see Fig. 3(a), open circles). The calibration of Ref. 22 has been shown to be compatible with the current gauges calibration.[11] The difference between the two sets of data can only be explained by a non-hydrostatic compression of the rhenium sample in Ref. 4 experiments, for instance, by a direct compression of the sample between the diamond anvils. In fact, in the conventional diffraction geometry in diamond anvil cells, the volume is overestimated under non-hydrostatic compression and Ref. 4 set of data agree with the measurements of Jeanloz *et al.*,[3] where the sample was non-hydrostatically compressed. The large difference between the yield strength of

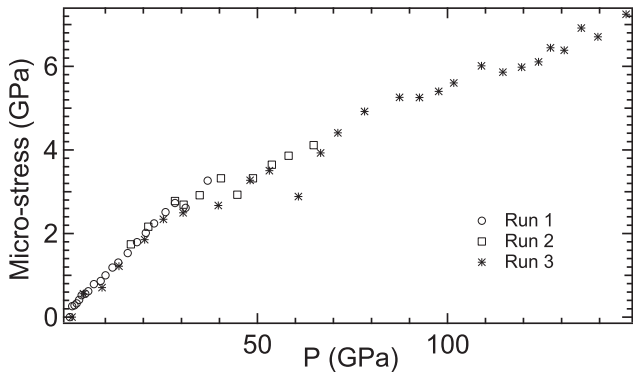

FIG. 2. Micro-stress in the rhenium sample as a function of pressure.

TABLE III. Measured pressures in GPa (P_R: ruby pressure, P_W: tungsten pressure, and P_{He}: helium pressure) and lattice parameters of tungsten (a_W) and rhenium (a and c) for the three experimental runs. The data are listed in the order they have been taken. Experimental uncertainty on lattice parameters is lower than 0.003 Å. Uncertainty on pressure measurement increases from 0.05 GPa at 1 GPa to $\simeq$2 GPa at 150 GPa.[19]

Run	P_R	P_{He}	a (Å)	c (Å)	Run	P_W	a_W (Å)	a (Å)	c (Å)
1	0.65		2.7619	4.4590	3	1.42	3.1599	2.7587	4.4536
	1.26		2.7598	4.4557		4.43	3.1500	2.7501	4.4416
	1.88		2.7579	4.4526		9.31	3.1347	2.7388	4.4223
	2.43		2.7561	4.4499		13.8	3.1216	2.7275	4.4039
	3.07		2.7546	4.4472		20.5	3.1031	2.7131	4.384
	3.74		2.7531	4.4446		25.3	3.0904	2.7035	4.3668
	4.69		2.7506	4.4410		30.5	3.0777	2.6958	4.3514
	5.48		2.7485	4.4372		39.6	3.0566	2.6791	4.3245
	6.96		2.7449	4.4310		47.8	3.0390	2.6670	4.3016
	8.77		2.7407	4.4247		52.8	3.0289	2.6577	4.2916
	10.0		2.7372	4.4196		60.3	3.0143	2.6478	4.2706
	11.9		2.7331	4.4134		66.1	3.0038	2.6395	4.2590
	13.4		2.7295	4.4063		70.5	2.9959	2.6308	4.2476
	15.9		2.7240	4.3994		77.4	2.9841	2.6238	4.2350
	18.3		2.7183	4.3901		86.3	2.9694	2.6112	4.2117
	20.6		2.7137	4.3815		91.5	2.9614	2.6063	4.2013
	22.8		2.7088	4.3739		96.4	2.9540	2.6003	4.1896
	25.8		2.7033	4.3637		100	2.9482	2.5947	4.1847
	28.3		2.6989	4.3574		107	2.9380	2.5872	4.1709
	31.1		2.6943	4.3475		113	2.9304	2.5819	4.1622
	36.9		2.6847	4.3322		118	2.9238	2.5770	4.1549
2		16.7	2.7225	4.3943		122	2.9182	2.5719	4.1472
		21.3	2.7140	4.3802		125	2.9141	2.5696	4.1429
		28.3	2.6995	4.3588		129	2.9096	2.5649	4.1358
		30.6	2.6958	4.3511		133	2.9041	2.5620	4.1308
		34.8	2.6883	4.3400		137	2.8989	2.5582	4.1226
		40.4	2.6797	4.3241		144	2.8902	2.5537	4.1147
		44.7	2.6740	4.3132					
		48.8	2.6643	4.3002					
		53.8	2.6580	4.2880					
		58.2	2.6505	4.2779					
		64.8	2.6422	4.2636					

TABLE IV. EOS parameters of rhenium measured in different experiments. The volume V_0, bulk modulus K_0 and its pressure derivative K'_0 are listed. Experimental methods and EOS formulation are specified. PTM: Pressure transmitting medium; XRD: X-ray diffraction; DAC: diamond anvil cell; BM: Birch-Murnaghan; GGA-PBE: generalized gradient approximation—Perdew Burke Ernzerhof.

Reference	V_0 (cm^3/mol)	K_0, K'_0 (GPa, no unit)	P range (GPa)	PTM	Pressure gauge	EOS	Method
This study	8.8726	352.6, 4.56	0.64–144	He	Ruby, W, He	Vinet	XRD in DAC
Ref. 1		336, 4[a]	0–35	NaCl	NaCl	BM	XRD in DAC
Ref. 7		365.2, 4.35	0–280			Vinet	Reduced Shock
Ref. 8		360.3, 5.43	0.00–0.42				Ultrasound
Ref. 4	8.87	348, 6.07[b]	0–165	He	Au[23]	Vinet	XRD in DAC
Ref. 4	8.87	353, 5.80	0–165	He	Au[23]	BM3	XRD in DAC
Ref. 21	8.842	376, 4.58	0–200			Vinet	GGA-PBE

[a]Fixed parameter.
[b]The published value $K'_0 = 7.57$ has been modified because it was incompatible with P-V data points.

rhenium[3,9] and gold (which is much lower[17]) would imply that the overestimate of sample and pressure marker volume under non-hydrostatic compression do not compensate. The compression curve measured in Ref. 4 becomes so stiff with increasing pressure that a fourth-order Birch-Murnaghan EOS is needed to fit the P-V data. It is not needed with the current measurements. Our data suggest that if the Re EOS established by Dubrovinsky *et al.*[4] is used to calibrate the pressure in a diamond anvil cell, it will overestimate P by already 40 GPa around 200 GPa, an overestimation which will further increase with increasing pressure: a real pressure of 450 GPa would correspond to a pressure of 600 GPa estimated using the calibration of Dubrovinsky *et al.*

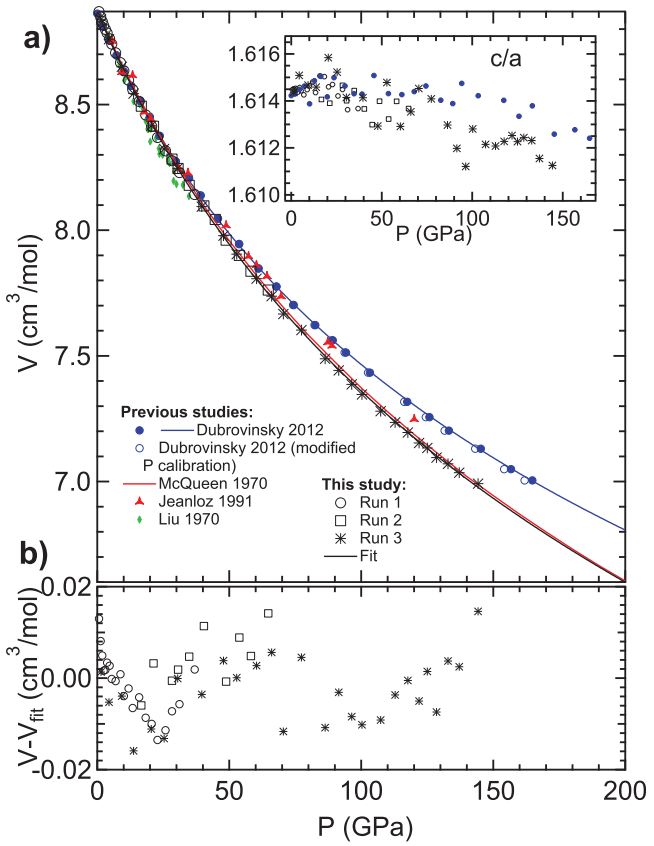

FIG. 3. (a) Measured volume of rhenium as a function of the pressure, compared with literature data. Inset: evolution of the c/a ratio. (b) Difference between measured and fitted volume, Vinet formulation with $V_0 = 8.8726$ cm^3/mol, $K_0 = 352.6$ GPa, and $K'_0 = 4.56$.

III. USE OF Re GASKET AS AN X-RAY PRESSURE CALIBRANT

In this section, we phenomenologically discuss whether the Re gasket XRD signal can be used to estimate the pressure in the sample chamber of a diamond anvil cell. For that purpose, we have analyzed the XRD signal from the gasket collected in five experiments, where the sample pressure had been accurately measured using a pre-calibrated luminescence or X-ray marker. The conditions of these experiments are summarized in Table V. In all cases, the X-ray spot was located $\simeq 5\ \mu$m away from the visual edge of the gasket, a distance slightly larger than the FWHM of the X-ray spot (2–3 μm). The collected signal was therefore scattered by the most inner part of the gasket; i.e., very close to its edge with the pressure chamber. Runs with pressure media considered as soft (He, Ne) or harder (KCl, Xe), with (runs 4 and 5) or without (runs 1–3) thermal annealing, and with a large (runs 1–3) or small (runs 4 and 5) gasket deformation during loading have been considered, so that these data represent the various gasket stress conditions in diamond anvil cell experiments.

Fig. 4(a) compares the measured volume of rhenium gasket with the volume of rhenium calculated at the sample pressure (estimated with the gauges summarized in Table IV), using the current quasi-hydrostatic EOS. The agreement between the two values is surprisingly good, the difference being smaller than 1.2%. In other words, the measured volume of Re gasket is close to what would have been expected

TABLE V. Conditions of the runs for which the X-ray diffraction signal of the rhenium gasket has been analyzed here. PTM: pressure transmitting medium.

Run	P range (GPa)	P gauge	PTM	Published reference
1	28–131	Ruby[13]	He	22
2	107–205	W[11]	Ne	24
3	141–259	Au[22]	Xe	25
4	64–204	Fe[24]	KCl	26
5	107–174	Fe[24]	KCl	26

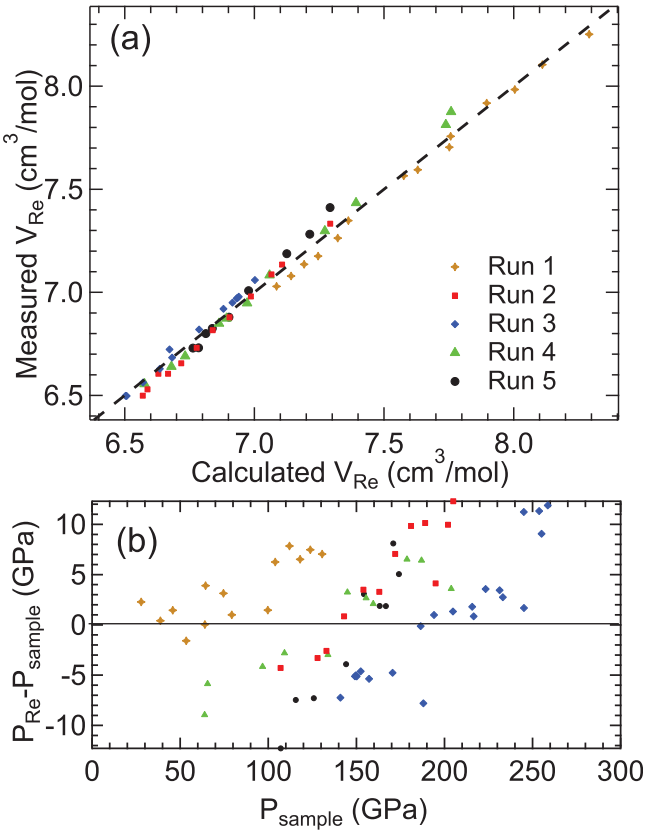

FIG. 4. (a) Volume of rhenium gasket measured by X-ray diffraction at the edge with the sample, as a function of the volume of rhenium calculated using the current quasi-hydrostatic EOS (Table IV, first line) at the sample pressure P_{sample}. The dotted line corresponds to measured volume = calculated volume. (b) Difference between P_{Re} (calculated using its quasi-hydrostatic EOS) and P_{sample}.

if Re had been compressed quasi-hydrostatically in the sample chamber. This contradicts the general view of an overestimate of volume in a material directly compressed between the anvils[17] discussed above. This view is too simplistic here: it is possible that the innermost part of the gasket does not sustain a large non-hydrostatic stress because it is in contact with a relatively soft pressure medium. Indeed, preferred orientation, which is the signature of a large plastic train under uniaxial loading, could not be detected in the XRD signal of the gaskets. However, we note that at very high pressure (low volume), the average measured volume becomes slightly smaller than the calculated volume (Fig. 4(a)), which means that a pressure from the gasket X-ray signal will be overestimated.

Fig. 4(b) shows the difference between the pressure which would have been estimated using the gasket EOS (P_{Re}) and the real sample pressure (P_{sample}). In practice, this would have been the error in pressure measurement if the gasket was used as a pressure gauge. The scatter of the measurements is large but does not drastically increase with pressure, even if above $\simeq$200 GPa, the gasket has a trend to overestimate the pressure (by $\simeq$5% at most). We therefore propose that the use of rhenium gasket as pressure gauge can be done in a confidence interval of 5% up to 260 GPa if the XRD signal is taken at the edge between the gasket and the high pressure chamber and the present quasi-hydrostatic rhenium EOS used. This proposal, which is based on a phenomenological approach, should be confirmed by the collection of similar data during future experiments, especially at higher pressure. We also stress that the gasket data have been collected with a micro-focused X-ray spot, and this method might not be applicable for larger beam sizes which would collect information from a large gasket volume with a heterogeneous stress state.

IV. CONCLUSION

The quasi-hydrostatic EOS of Re has been determined from a powder XRD experiment using He as a pressure transmitting medium up to 144 GPa. The XRD lines measured for rhenium show no evidence of non-hydrostatic compression. The volume, bulk modulus, and its pressure derivative at ambient pressure and temperature have been obtained with a Rydberg-Vinet EOS: $V_0 = 8.8726 \pm 0.01$ cm^3/mol, $K_0 = 352.6 \pm 8$ GPa, and $K_0' = 4.56 \pm 0.17$. This EOS agrees with shock compression data[7] but contradicts a recent study carried out in diamond anvil cell.[4] This suggests that the use of rhenium as an X-ray pressure gauge with the calibration using Ref. 4 data will lead to a large pressure overestimate (for instance, 600 GPa instead of 450 GPa). Using XRD, data collected on the rhenium gasket in various configurations of compression, we propose that this information and the current EOS could be used for pressure estimation at multi-Mbar pressures (within $\simeq$5% of the real pressure above 1 Mbar), if the diffraction signal is taken at the sample/gasket interface.

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