Article ID:1000-5773(2012)06-0608-09

A Revised Ruby Pressure Scale up to 160 GPa*

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Abstract: The ruby pressure scale is revised using available static compression data of eleven markers at pressures up to 160 GPa, which includes Al, Cu, W, Au, Pt, Ta, Ag, Mo, Ni, Co, and Zn. The shock wave reduced isotherms of these markers are derived from shock Hugoniot and thermodynamic data using the same theoretical framework. These isotherms are then used to calibrate the ruby pressure scale, which shows good consistency based on simultaneous volume measurements. The calibration was processed by fitting all of these data to two ruby pressure scale forms, and gives the parameters $A=1\,923.4$ GPa, $B=9.75$, and $m=1\,889.0$ GPa, $n=5.48$, respectively. These two sets of ruby pressure scale differ by less than 2.1 GPa in the pressure range of 200 GPa. Making use of our ruby pressure scale in latter form, the static compression data of Au are reassessed. When the bulk modulus is fixed at 167 GPa, fitting the reassessed pressure-volume data to Vinet equation of state gives the first order pressure derivative of bulk modulus as 5.95, which has an excellent agreement with ultrasonic measurements.

Key words: equation of state; pressure scale; ruby

CLC number: O521.2 **Document code**: A

1 Introduction

The static diamond anvil cell (DAC) technique has been widely used to generate extremely high pressure in materials[1-3]. Accurate determination of pressure in DAC experiments is important for characterizing a range of high-pressure phenomena such as equation of state (EOS) and phase transition. Unfortunately, the exact pressure achieved in DAC experiments cannot be directly measured, and a secondary pressure scale must be used[4-6]. The ruby pressure scale firstly introduced by Forman *et al*.[7] is a simple and accurate method of measuring pressure in DAC experiments especially at room temperature. With the ruby pressure scale, pressure is derived from the previously calibrated R_1 line shift of ruby fluorescence with pressure. Piermarini *et al*.[8] obtained the pressure dependence of R_1 line shift up to 19.5 GPa at room temperature by simultaneously measuring the R_1 line shift of ruby fluorescence and specific volumes of NaCl[9] as a function of pressure. Mao *et al*.[10] extended the marker method under non-hydrostatic condition to the megabars pressure range using shock wave reduced isotherms[11] of Cu, Mo, Pd, and Ag. Following these works, the ruby pressure scale under quasi-hydrostatic compression up to 80 GPa was developed by Mao *et al*.[12] using shock wave reduced isotherms of Cu and Ag, which is expressed as

* **Received date**: 2012-02-17; **Revised date**: 2012-10-23

Foundation Item: Defense Industrial Technology Development Program (B1520110001); Science and Technology Foundation of the Chinese Academy of Engineering Physics (2010A0101001)

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$$p = \frac{A}{B}\left[\left(\frac{\lambda}{\lambda_0}\right)^B - 1\right] \tag{1}$$

where p is the pressure in a unit of GPa, λ is the measured wavelength of the ruby R_1 line, and $\lambda_0 = 694.24$ nm. The free parameters are given by $A = 1\,904$ GPa and $B = 7.665$.

Based on a combination of X-ray and Brillouin scattering measurements on MgO up to 55 GPa, Zha *et al*.[13] developed a ruby pressure scale with $A = 1\,904$ GPa and $B = 7.715$ as expressed in Eq. (1), which is very close to that of Mao *et al*.[12]. However, subsequent works[14-23] indicated that Mao ruby pressure scale might underestimate the pressure at higher pressure range. The pressure difference among previously published ruby pressure scales might reach 10-15 GPa at 150 GPa[20,22].

Dewaele *et al*.[23] measured 300 K isotherms of Fe, Co, Ni, Zn, Mo, and Ag up to 200 GPa using helium as the pressure transmitting medium in DAC experiments. A new ruby pressure scale was proposed by the comparison of the static measurements with shock wave reduced isotherms. However, we noticed that following shock wave reduced isotherms were used by Dewaele: Co, Ni, and Ag[24], Mo[25], Al[22], and Zn[24,26-28]. Similarly, the previous calibration of the ruby pressure scale proposed by Dewaele *et al*.[19] was on the basis of measured 300 K isotherms of Al, Cu, W, Au, Pt, and Ta under quasi-hydrostatic condition. The shock wave reduced isotherms used for this calibration is Cu, Au, Pt, and Ta[29], W[25], and Al[30], respectively. In fact, the key issue for a successful calibration of the ruby pressure scale is the accuracy and consistency of the involved shock wave reduced isotherms. Dewaele *et al*.[23] also pointed out that the disagreement between two previously published calibrations of the ruby pressure scale[19-20] is duo to a different choice of the shock wave reduced isotherm of Al.

Recently, we proposed a p-V-T EOS model for MgO, Au, and Pt at pressures up to 300-550 GPa and temperatures up to 3 000 K based on shock Hugoniot and thermodynamic data using a Mie-Grüneisen type analysis framework.[31-33] The derived EOSs are consistent with all available experimental volume compression data in a widen range of pressure and temperature. Particularly, the calculated p-V-T EOSs of Au, Pt and MgO were found mutually consistent if checked using independent simultaneous volume measurements of Au, Pt, and MgO at various temperatures. This mutual consistency shows the reliability of our EOS model. Comparison of our calculated 300 K isotherms of Al, Cu, W, Au, Pt, Ta, Ag, Mo, Co, Ni, and Zn with corresponding quasi-hydrostatic data of Dewaele *et al*.[19,23] and Chijioke *et al*.[20] lend us much credit to calibrate a new ruby pressure scale.

2　The Consistency of Calibrating Data

The method of using markers to calibrate ruby pressure scale was introduced by Piermarini *et al*.[8] with NaCl as a pressure standard. In this method, markers and ruby chips are loaded in a DAC with pressure transmitting medium. The R_1 line shifts of ruby fluorescence are measured together with the simultaneous X-ray determination of the markers' volumes to yield calibration points. We will use this method since it is the most reliable method of producing a calibration. The most extensive data with high quality available in literatures for calibrating ruby pressure scale are from Dewaele *et al*.[19,23], where 300 K isotherms under quasi-hydrostatic condition had been measured for Al, Cu, and W to 153 GPa, for Au, Pt, and Ta to 94 GPa, for Ag and Mo to 122 GPa, and for Co, Ni and Zn to 156 GPa, respectively. Another set of Au compression data up to 90 GPa[20] is also used. We first calculated the 300 K isotherms of Al, Cu, W, Ta, Ag, Mo, Co, Ni, and Zn using the method described in detail elsewhere[31-33] with parameters listed in Table 1. The 300 K isotherms of Au and Pt can be found in Ref. [33].

Before calibrating the ruby pressure scale, it is important to examine the consistency of the calcu-

lated 300 K isotherms of markers. To do this, pressures are extracted from the calculated 300 K isotherms using simultaneously measured volumes of two or three markers[19,23].

Based on the simultaneous volume measurements of Dewaele *et al.*[19], the difference in pressure calculated with our 300 K isotherms of Al, Cu, and W up to 153 GPa and Au, Pt, and Ta up to 94 GPa is plotted in Fig. 1 and Fig. 2, respectively. As Shown in Fig. 1, the discrepancy between 300 K isotherms of Cu and W is less than 0.5 GPa below 100 GPa, and gradually increases to 2.6 GPa (1.7%) at 156 GPa. The agreement between our calculated 300 K isotherms of Al and Cu is also good. The discrepancy is less than 2.2 GPa within the entire studied pressure range. Fig. 2 shows that the difference between the 300 K isotherms of Pt and Au ranges from −0.8 GPa to 1.5 GPa within a pressure of 100 GPa. Similarly, the pressure difference between our calculated 300 K isotherms of Pt and Ta is less than 2.1 GPa. The comparison demonstrates that our calculated 300 K isotherms of these materials have good self-consistency at room temperature and pressure up to 160 GPa if they are used as internal pressure scales in DAC experiments.

Table 1 The material parameters used in present calculations and the calculated 0 K EOS parameters

Material	ρ_0/(g/cm^3)	c/(km/s)	S	γ_0[34]	θ_D[35]/(K)	ρ_{0K}(g/cm^3)	B_{0K}/(GPa)	B'_{0K}
Al	2.703	5.349[26]	1.338[26]	2.19	423	2.735	79.3	4.34
Cu	8.924	3.981[36-37]	1.479[36-37]	2.02	342	9.008	145.3	4.92
W	19.264	4.029[26,26]	1.237[26,26]	1.67	388	19.312	314.5	3.95
Ta	16.654	3.310[36-37]	1.294[36-37]	1.82	247	16.736	184.6	4.17
Ag	10.490	3.264[27,38]	1.572[27,38]	2.40	228	10.610	115.5	5.29
Mo	10.220	5.143[25,27,38]	1.248[25,27,38]	1.59	459	10.246	271.9	3.99
Co	8.820	4.772[26]	1.285[26]	2.22	452	8.879	203.4	4.13
Ni	8.875	4.594[26]	1.440[26]	1.93	427	8.933	190.8	4.76
Zn	7.139	3.072[26]	1.498[26]	2.24	316	7.264	70.7	5.00

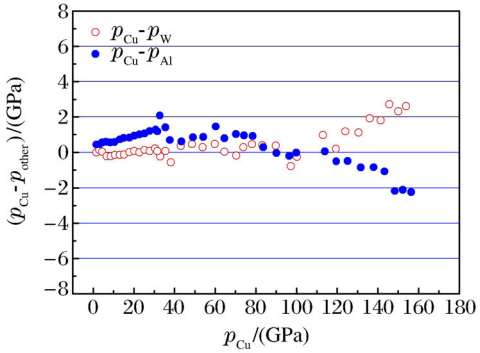


Fig. 1 Comparison of our shock wave reduced isotherms of Al, Cu and W based on simultaneous volume measurements of Ref. [19]

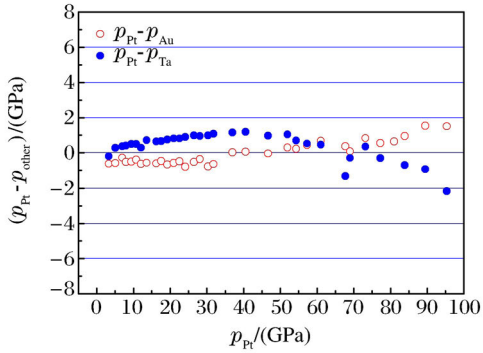


Fig. 2 Comparison of our shock wave reduced isotherms of Au, Pt and Ta based on simultaneous volume measurements of Ref. [19]

Recent simultaneous volume measurements on Ag, Mo, Ni, Zn, and Co[23] at room temperature can also be used to examine the self-consistency of our EOS model. The comparisons of 300 K isotherms of these materials are plotted in Fig. 3 and Fig. 4. As shown in Fig. 3, the agreement between the calculat-

ed 300 K isotherms of Ni and Co is excellent within the studied pressure range. Furthermore, deviations from our calculated 300 K isotherms of Ni and Zn only slightly exceeds 2.0 GPa in the pressure range of 160 GPa. It can be seen from Fig. 4 that the calculated pressures of Ag and Mo differ by less than 1.5 GPa except for the experimental point at 125 GPa. The self-consistency confirms the reliability of our EOS model for calibrating ruby pressure scale using the high quality experimental data of Dewaele *et al*.[19,23] and Chijioke *et al*.[20].

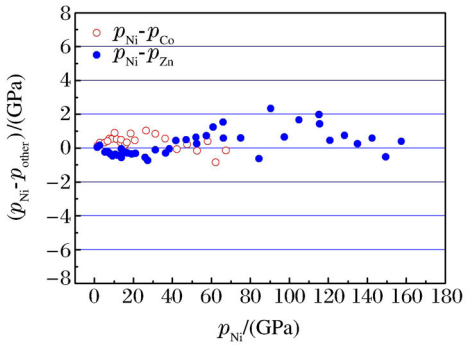


Fig. 3　Comparison of our shock wave reduced isotherms of Ni, Co and Zn based on simultaneous volume measurements of Ref. [23]

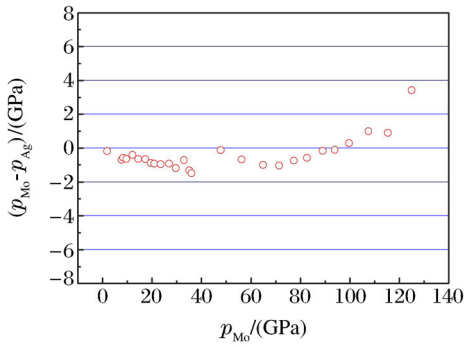


Fig. 4　Comparison of our shock wave reduced isotherms of Ag and Mo based on simultaneous volume measurements of Ref. [23]

3　Comparison with Other Ruby Pressure Scales and Discussion

In order to obtain a reliable ruby pressure scale, various formulae[10,15-16,18] have been proposed to describle the pressure dependence of R_1 line shift of ruby fluorescence. Based on the published experimental data[19-20,23], we extracted the ruby R_1 line shift as a function of the compression ratio of markers, and then calculated the pressure of the corresponding markers at the same compression ratio using our EOS model. We performed a non-linear least squares fit to these data. As a result, a new pressure scale was obtained with parameters $A=(1\,923.4\pm4.5)$GPa and $B=9.75\pm0.10$ in the form of Eq. (1) (we call it scale Form-1), where errors are the standard deviations from the fit. Since Dorogokupets *et al*.[22] preferred to using a form of Eq. (2) proposed by Aleksandrov *et al*.[15], we also fitted all of the data to Eq. (2) and obtained another ruby pressure scale with parameters $m=(1\,889.0\pm4.3)$GPa and $n=5.48\pm0.08$ (we call it scale Form-2), as shown in Fig. 5 together with Form-1. It should be noted that in the fitting we allowed both A and B or m and n to vary, and gave an equal weighting factor to all of the data.

$$p = m\left(\frac{\lambda-\lambda_0}{\lambda_0}\right)\left[1+n\left(\frac{\lambda-\lambda_0}{\lambda_0}\right)\right] \qquad (2)$$

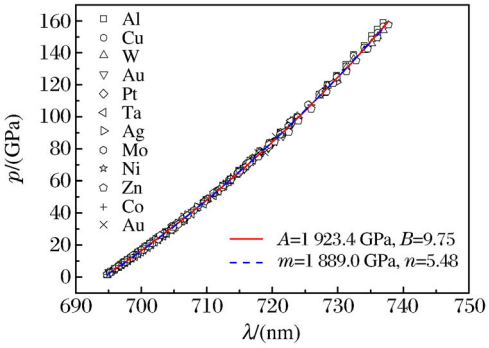


Fig. 5　Calibration curve of the ruby R_1 line shift with pressure. The compression data of Al, Cu, W, Au, Pt, and Ta are taken from Ref. [19], Ag, Mo, Ni, Co, and Zn are taken from Ref. [23], and Au is taken from Ref. [20]. The solid curve and dash curve are almost indistinguishable.

Although the Mao ruby pressure scale has been widely used in DAC experiments, recently proposed ruby pressure scales with the same form of Eq. (1)[19-23] indicated that Mao's scale might underestimate the pressure, especially above 100 GPa. Dewaele *et al*.[19] proposed a ruby pressure scale with

parameters $A=1\,904$ GPa and $B=9.5$ in the form of Eq. (1) based on the 300 K isotherms of six selected metals. Dewaele *et al*.[23] later revised his ruby pressure scale with $A=1\,920$ GPa and $B=9.61$ based on the 300 K isotherms of other five selected metals. Another important ruby pressure scale was recommended by Dorogokupets *et al*.[22] in the form of Eq. (2) with parameters $m=1884$ GPa and $n=5.5$. The comparison of these ruby pressure scales[12,19-20,22-23] is plotted in Fig. 6. We can see that these ruby pressure scales have good consistency only within a pressure range up to 80 GPa, beyond that they begin to diverge. Our scales have good agreement with the proposals of Ref. [22] and Ref. [23]. The pressure calibration done by Dewaele *et al*.[19] using different sources of shock wave reduced isotherms[25,29-30] of the six markers might underestimate the pressure by about 2.0 GPa at 200 GPa. On the contrary, the ruby pressure scale proposed by Chijioke *et al*.[20] with parameters $A=1\,876$ GPa and $B=10.71$ might overestimate the pressure at pressures above 120 GPa. Dorogokupets *et al*.[22] pointed out that this deviation was largely related to the different forms of the pressure dependence of ruby R_1 line shift. As mentioned above, we have proposed two ruby pressure scales in the forms of both Eq. (1) and Eq. (2), as well as done by Dorogokupets *et al*.. Our two pressure scales differ by less than 2.1 GPa in a pressure range up to 200 GPa. Comparing the two ruby pressure scales proposed by Dorogokupets *et al*., one can see that the pressure difference reaches 3.6 GPa at 200 GPa. Therefore, the Dorogokupets scale should be revised by combining previous 300 K isotherms measurements[19] with new experimental data[23].

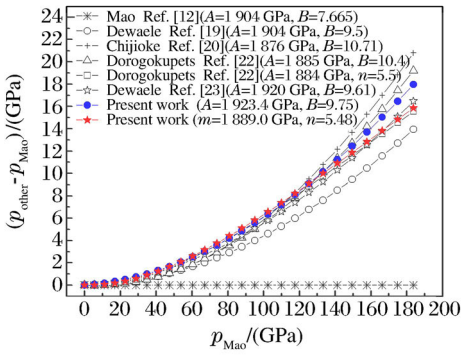


Fig. 6 Difference between recently proposed ruby pressure scales and Mao ruby pressure scale

Our calibration includes eleven markers, and the volumes of these markers and the ruby R_1 line shift were measured using identical techniques with the same accuracy. The possible errors in the calculated 300 K isotherms will be statistically reduced. More importantly, the 300 K isotherms of markers used in our calibration are calculated with the same theoretical framework and have good self-consistency at room temperature and pressures up to 160 GPa. Furthermore, the quality of our calibration can be checked by plotting the difference between the pressure calculated from our EOS model and from our ruby pressure scale (Form-2 as example), as shown in Fig. 7. The deviation is very small, only slightly in excess of 2 GPa in the pressure range of 160 GPa.

Au is an important standard material in both static DAC and dynamic shock wave experiments. Much effort towards establishing its EOS has been and is currently being expended[19,39-50]. In order to check the validity of our new ruby pressure scale, the pressures of Au in DAC experiments of Takemura[47], Dewaele *et al*.[19], and Takemura and Dewaele[48] are re-determined with our Form-2 ruby pressure scale, and are used to reassess the bulk modulus B_0 and its first order pressure derivative B_0' at ambient condition. B_0 of Au was measured by ultrasonic experiments[40-42]. The results have excellent agreement with each other and span slightly from 166.1 GPa to 166.8 GPa. For this reason, B_0 is usually fixed at 167 GPa when the compression data of DAC experiments[19,46-48] (determined with different ruby pressure scales[12-13,19,22]) are fitted to Vinet[51] or Birch-Murnaghan[52] EOS to yield the value of B_0'. However, large discrepancy still exists in B_0' ranging from 5.01 to 6.00. We have noticed that the large discrepancy is duo to the choice of different ruby pressure scales. For example, Takemura, Dewaele *et al*., and Takemura and Dewaele constructed the pressure-volume relation of Au based on

Zha[13], Dewaele[19], and Dorogokupets[22] ruby pressure scale, respectively. By fitting these data to the Vinet EOS and fixed B_0 at 167 GPa, B_0' value is of 5.46, 6.00, and 5.90, respectively. In constrast, our re-determined pressure data gives B_0' of 5.68, 6.14, and 5.95, respectively, when fitting to the Vinet EOS. It can be seen that the value of B_0' becomes better agreement with ultrasonic measurements, and the disperstiveness of B_0' decreases from 0.54(9.0%) to 0.46(7.5%).

Fig. 8 shows the plot of relative volume of Au as a function of pressure based on the measurements of[19,47-48] where the pressures are determined with our Form-2 ruby pressure scale. The best-fitted B_0' value is 5.95 for these data when B_0 is fixed at 167 GPa. Moreover, the same data set yields B_0' =5.99 and B_0 =166.2 GPa when B_0 is allowed to vary. These two fitted results have very good consistency and show excellent agreement with the ultrasonic measurements[40-42]. The X-ray diffraction data on Au in NaCl pressure medium by Hirose *et al*.[49] are also plotted in Fig. 8 for a comparison, where the pressures are determined with our MgO pressure scale[32]. It is worth emphasizing that our MgO and ruby pressure scales are constructed within the same theoretical framework. As shown in Fig. 8, the four re-determined data sets[19,47-49] are in excellent agreement, and can be well reproduced by Vinet EOS with parameters B_0 =167 GPa and B_0' =5.95 in the pressure range of 150 GPa. A very close value of 5.9 for B_0' was obtained[48], where the pressures are determined by the Dorogokupets ruby pressure scale[22]. The EOS parameters of Au obtained here should be considered as an improvement since they are not only based on large data sets[19,47-48], but also reduce the disperstiveness in the pressures calculated with different ruby pressure scales.

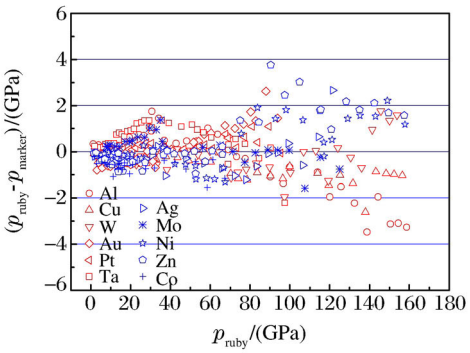


Fig. 7　Difference between the pressures determined by our ruby pressure scale with parameters m=1 889.0 GPa and n=5.48 in the form of Eq. (2) and our calculated shock wave reduced isotherms of eleven markers based on the measurements of Dewaele *et al*. (Refs. [19,23]).

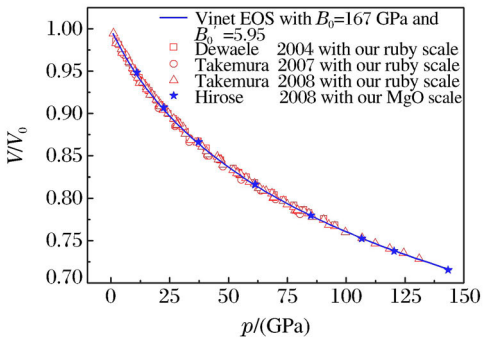


Fig. 8　Experimental compression data of Au at room temperature (Refs. [19,47-48]), where the pressures are calculated from our ruby pressure scale or MgO pressure scale. The solid line are calculated by the Vinet EOS with B_0 =167 GPa and B_0' =5.95.

4　Summary

In summary, we have calibrated the ruby pressure scale up to 160 GPa by comparing the precisely measured volumes of eleven markers and ruby R_1 line shift with the corresponding shock wave reduced isotherms of these markers, which are formulated within the same theoretical framework. The possible statistical errors in calibration process can be remarkably reduced because of the good consistency among our calculated shock wave reduced isotherms. Our calibrated results have excellent agreement with the ruby pressure scales recently proposed, and imply that the ruby calibration of Dewaele *et al*. might underestimate the pressure by about 2.0 GPa at 200 GPa. The pressures of Au in DAC experi-

ments are re-determined with our ruby pressure scale. The fitting of these data to the Vinet EOS gives $B_0'=5.95$ with B_0 fixed at 167 GPa, which is in excellent agreement with ultrasonic measurements.

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160 GPa 压力范围内重新标定的红宝石压标

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摘要: 在同一理论框架内, 基于冲击 Hugoniot 和热力学参数计算了 Al、Cu、W、Au、Pt、Ta、Ag、Mo、Ni、Co 和 Zn 的 300 K 等温压缩线, 并结合现有静高压实验数据, 在 160 GPa 压力范围内重新标定了红宝石压标。所采用的两种红宝石压标形式的标定结果分别为 $A=1\,923.4$ GPa、$B=9.75$ 和 $m=1\,889.0$ GPa、$n=5.48$, 两者具有非常好的自洽性, 200 GPa 压力范围内确定的压力偏差小于 2.1 GPa。基于提出的红宝石压标, 重新计算了 3 组 Au 等温压缩实验的加载压力。固定等温体模量为 167 GPa, 重算的实验数据拟合至 Vinet 物态方程所得等温体模量对压力的一阶偏导为 5.95, 与超声实验数据非常吻合。

关键词: 物态方程; 压标; 红宝石

中图分类号: O521.2 **文献标识码:** A