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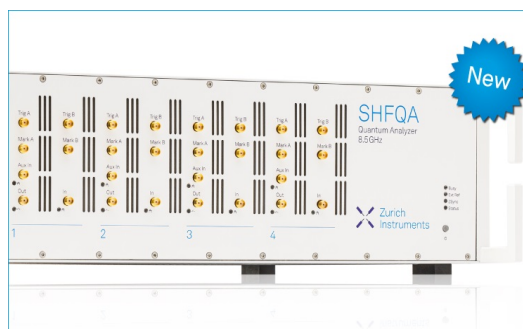
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Calibration of the ruby R_1 and R_2 fluorescence shifts as a function of temperature from 0 to 600 K

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Recent work by Gupta and Shen [Appl. Phys. Lett. **58**, 583 (1991)] has shown that in a nonhydrostatic environment, the frequency of the ruby R_2 line provides a reliable measure of the mean stress or pressure. When using the frequency of either the R_1 or R_2 line to measure pressure at nonambient temperature, it is necessary to know the temperature dependence of the line shift. Unfortunately, the shift of the R_2 line with temperature has not been reported. The ruby R_1 and R_2 fluorescence shifts have been determined as a function of temperature from 15 to 600 K. Both can be fitted very well to the simple cubic forms $R_1(T) = 14\,423 + 4.49 \times 10^{-2}T - 4.81 \times 10^{-4}T^2 + 3.71 \times 10^{-7}T^3 \text{ cm}^{-1}$ and $R_2(T) = 14\,452 + 3.00 \times 10^{-2}T - 3.88 \times 10^{-4}T^2 + 2.55 \times 10^{-7}T^3 \text{ cm}^{-1}$. From 300 to 600 K the shifts fit well to linear functions of temperature. In addition, it is found that the R_1 - R_2 splitting changes by about 3 cm^{-1} over the 600 K temperature range. Linewidths were found to vary both with temperature and from sample to sample.

I. INTRODUCTION

Since its introduction in 1972,¹ the ruby R -line fluorescence has provided the most convenient technique for determining pressures in diamond-anvil cells (DACs). Much effort has gone into calibrating the R -line shift as a function of pressure P .²⁻⁷ At room temperature under hydrostatic conditions, the R_1 and R_2 lines both shift nearly linearly with pressure by the same amount (about $-7.57 \text{ cm}^{-1}/\text{GPa}$) and do not change shape. Pressures have usually been determined from the frequency of the R_1 line, however either line can be used.

When the ruby is subject to small nonhydrostatic stresses, the R -line splitting changes by several wave numbers.^{4,8} Whether the splitting increases or decreases depends on the orientation of the stress with respect to the crystallographic orientation of the ruby. Whether the change in splitting is due to shifts in R_1 or R_2 , or both, has not been known until recently.^{9,10} (Additional complications occur in DACs because it is not possible to determine the entire stress tensor. Furthermore, nonhydrostatic stresses are generally accompanied by stress gradients. A stress gradient across a ruby chip results in broadened R_1 and R_2 lines. Broadened lines have been used as an indicator of nonhydrostatic stresses in DACs,^{2,11} and this effect was used to explore the hydrostatic pressure limits of the 4:1 methanol/ethanol pressure medium¹¹ that is now widely used.)

The effect of nonhydrostatic stresses on the R lines has been shown by the very nice uniaxial-strain shock-wave experiments and analysis reported by Gupta *et al.*^{9,10,12,13} Shock loading results in very nonhydrostatic—but well defined—stresses in ruby; for a 2.4% compression along the c axis, the c -axis stress is 124 kbar while the stress perpendicular to the c axis is only 29 kbar.¹² R -line measurements on shocked rubies show that the R_1 -line shift versus volume change (or mean stress) is greater for compression along the a axis than for compression along the c

axis. By contrast, the R_2 -line shift is the same for compression along either axis, and furthermore is the same as the line shift obtained under hydrostatic conditions. The R_1 line is sensitive to the nonhydrostatic nature of the stress but the R_2 line is not. Thus, for nonhydrostatic experiments, Gupta and Shen⁹ concluded that pressure (mean stress) can be determined more accurately using the R_2 line.

For experiments at nonambient temperatures T it is necessary to know the shifts of the R lines with temperature, as well as with pressure. With the usual assumption that the pressure derivatives are independent of T (and there is some evidence to support this conjecture¹³⁻¹⁵) only the zero-pressure R -line frequencies need to be known. Several workers over the last several decades have measured the zero-pressure R -line frequencies as a function of temperature.^{2,4,16-20} Curiously, only Powell *et al.*²⁰ have reported the R_2 line shift versus T for the full ~ 0 –600 K temperature range. Unfortunately, their result is reported by way of an inconvenient expression, which poorly fits their data at temperatures above 350 K.

McCumber and Sturge¹⁶ have shown on theoretical grounds that the R -line shift with temperature is described by the equation

$$R_i(T) - R_i(0) = \alpha \left(\frac{T}{T_d} \right)^4 \int_0^{T_d/T} \frac{x^3 dx}{e^x - 1}, \quad (1)$$

which describes the electronic energy-level shift which is expected when the electronic state interacts with acoustic phonons. α is the electron-phonon coupling constant and arises from the scattering of phonons by the impurity Cr^{3+} ion. T_d is a Debye temperature, although, since all phonons are not coupled to the electronic state this T_d is not necessarily the same as that which determines the heat capacity. For the temperature range 0–700 K, McCumber and Sturge¹⁶ found that Eq. (1) fit $R_1(T)$ very well using

$\alpha = -400 \text{ cm}^{-1}$ and $T_d = 760 \text{ K}$. The value of $R_1(0)$ depends on the specific sample of ruby.

Powell *et al.*²⁰ have used the theory of McCumber and Sturge¹⁶ to fit the R_1 and R_2 line shifts in heavily doped rubies (2.1 at. % Cr and 0.94 at. % Cr). In the heavily doped rubies they found $\alpha = -177 \text{ cm}^{-1}$ and $T_d = 450 \text{ K}$ for both the R_1 and R_2 lines. The R_1 shift for the lightly doped rubies used by McCumber and Sturge¹⁶ is similar in magnitude to that measured by Powell *et al.*,²⁰ hence the reason for the large difference in fitting parameters is not known. As mentioned above, Powell *et al.*'s²⁰ expression fits their data poorly above 350 K.

Over more limited temperature ranges other authors have provided empirical fits for the R_1 line shift with temperature. Buchsbaum and co-workers¹⁷ were able to obtain a close fit to their $R_1(T)$ frequencies over the range 15–300 K with a polynomial expression. It could not, however, be extended to higher temperatures. In the 77–300 K range, Adams and co-workers⁴ found that both their R_1 and R_2 data lay on curves given by Eq. (1) with the same parameters specified by McCumber and Sturge.¹⁶ Barnett and co-workers² and Yamaoka and co-workers¹⁵ reported that the R_1 shift above room temperature was linear with a wavelength coefficient of 0.0068 nm/K (which corresponds to $-0.141 \text{ cm}^{-1}/\text{K}$). Munro *et al.*¹⁸ fitted the R_1 frequencies with the form

$$R_1(T) = R_1(T_0) - 3.551 \times 10^8 [\ln(T/T_0)]^{11.54}, \quad (2)$$

where T_0 is not 0 K.

Recently, Yen and Nicol¹⁹ measured $R_1(T)$ from 120 to 603 K. They found that Eq. (1) fit their data very well with $\alpha = -419 \text{ cm}^{-1}$ and $T_d = 760 \text{ K}$. Neither a straight line nor Eq. (2) fit their data in a satisfactory way. Over the limited temperature range 393–620 K, they found the wavelength of the R_1 line to vary almost linearly with temperature by the factor 0.0074 nm/K ($-0.153 \text{ cm}^{-1}/\text{K}$).

We began this work simply to determine the temperature dependence of the R_2 line so that we could do nonhydrostatic experiments with pressures determined by the more reliable R_2 line.⁹ As described below, we found that over the temperature range 15–600 K the R_1 and R_2 frequencies fit extremely well to a simple cubic equation in T , thereby avoiding the cumbersome Eq. (1) (Ref. 16) and the inadequate Eq. (2).^{18,19} For the temperature range 300–600 K, linear relations in T also work well. In the course of this work we also discovered several other interesting features of the R -line spectra that have not been previously reported.

II. EXPERIMENTAL METHOD

Initial testing was conducted on an unoriented ruby (approximately $2 \times 3 \times 5 \text{ mm}^3$), containing an unknown amount of Cr_2O_3 . To determine the effect of crystal orientation on R_1 and R_2 peak positions, oriented samples (Union Carbide, 0.5% Cr_2O_3 by weight) were obtained from Gupta of Washington State University. X-ray precession photos were used to confirm the orientation of the $\sim 3 \text{ mm}^2$ by $250\text{-}\mu\text{m}$ -thick a -axis and $\sim 3 \text{ mm}^2$ by $50\text{-}\mu\text{m}$ -thick

c -axis ruby samples. The oriented samples had the defined crystal axis (a or c) parallel to the thin dimension, (or normal to the plane of the crystal plate). Laser light was delivered and fluorescence collected in a direction within 5° of parallel to the defined axis.

A Merrill-Bassett DAC²¹ was used to hold the ruby while luminescence measurements were made. Since all the work was conducted at $P=0$, the diamonds were removed and the ruby was placed between the plates. A hollow cylinder of annealed gold was placed over the ruby and between the two plates. The pressure bolts were then tightened until the plates made contact with the gold, exerting just enough pressure on the ruby to establish good thermal contact between cell and sample, but not enough to cause any detectable changes in the R -line spectrum.

For work at room temperature and above, the loaded DAC was placed in a vacuum oven.²² To measure the temperature of the ruby, three $75\text{-}\mu\text{m}$ -diam tungsten rhenium [W-Re(5%)/W-Re(26%)] thermocouples by Omega Engineering were positioned around the DAC. One thermocouple was placed between the cell holder and the bottom plate; another thermocouple was placed adjacent to the sample; the third thermocouple was placed in a bolt hole on the top plate. Because the temperature sometimes drifted slightly, we recorded the temperature at the beginning and end of every luminescence scan. The six temperatures were averaged and the largest deviation from this average was taken as the error (0.7–11 K).

For the low-temperature studies (from $\sim 15 \text{ K}$ to room temperature) the DAC was placed in a Model DE-202 Displex refrigerator with Chromel-gold thermocouples placed in a similar configuration as for the high-temperature measurements. A Scientific Systems model 5500-5 temperature controller was used to regulate the temperature. Temperature errors, calculated by the above method, were generally less than 2 K.

Ruby luminescence was excited by the 488 nm line of a Spectra-Physics model 164 argon-ion laser operating at a power of 40 mW; 60% of this power was lost through the optics, yielding a power of 16 mW on the ruby sample. For some measurements, the power on the sample was increased to 200 mW; no change in the spectrum was observed, indicating that the laser did not appreciably heat the sample at either power. A backscattering geometry was used to collect the fluorescence. For the oriented samples, laser light was delivered and fluorescence collected in a direction within 5° of parallel to a crystallographic axis.

The ruby fluorescence was collected into a Spex model 1403, 0.85 m double monochromator. Slit widths were typically $200 \mu\text{m}$ or less, resulting in R lines with apparent linewidths [full width at half-maximum (FWHM)] less than 0.8 cm^{-1} at 15 K. Signal was detected using an RCA C31034 low-dark-count photomultiplier tube refrigerated by a Products for Research thermoelectric cooler. An IBM PS-2 model 50Z with a data-collection and analysis system prepared by Lowe, Kutt, and Blumenroeder²³ was used for monochromator and detector control. The refrigerator and oven were mounted on separate optical tables containing identical lasers, monochromators, photomultipliers, etc.

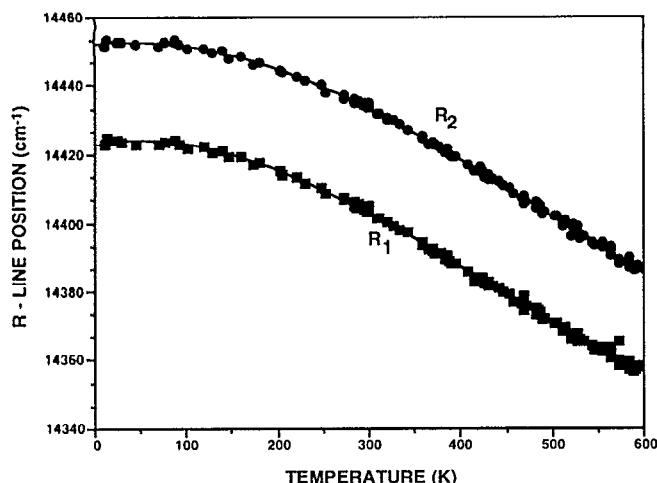


FIG. 1. R -line position vs temperature. Data is shown for both a - and c -axis rubies and for the ruby of unknown origin. The smooth curves drawn through the data are given by Eqs. (3) and (4).

The $\sim 1 \text{ cm}^{-1}$ scatter seen in the data arises because the monochromators were only calibrated to an absolute accuracy of $\sim 1 \text{ cm}^{-1}$.

To determine the polarization of the fluorescence, an a -axis ruby was placed in the DAC with the c axis running vertically. A polarizing filter was placed in the collection beam allowing us to separate the fluorescence into components with electric vector parallel or perpendicular to the ruby c axis. To verify the orientation of the polarization analyzer to the crystal, we rotated the analyzer to find the minimum ratio of peak intensities (R_1/R_2). Nelson and Sturge²⁴ have shown that the c axis and fluorescence electric vector are parallel when R_1/R_2 is minimum. Within experimental error, the c axis found in this way was oriented in the same direction as the c axis found by x-ray diffraction. Polarized laser excitation was also used in some scans, but the effect on the R -line spectrum was indiscernible.

R -line frequencies, linewidths, and intensities were obtained by fitting the measured luminescence spectrum to a double Lorentzian.^{25,26} The background resulting from detector dark count and the tails of other fluorescence lines was assumed to be a linear function of frequency and was added, as an adjustable fitting parameter, to the double Lorentzian.

III. RESULTS

A plot of the R -line positions as a function of temperature is shown in Fig. 1. Positions from all oriented and unoriented samples are included, as there was no detectable difference in line positions between samples. From 15 to 600 K the R_1 - and R_2 -line positions can be fit to similar cubic equations,

$$R_1(T) = 14\,423 + 4.49 \times 10^{-2}T - 4.81 \times 10^{-4}T^2 + 3.71 \times 10^{-7}T^3 \text{ cm}^{-1}, \quad (3)$$

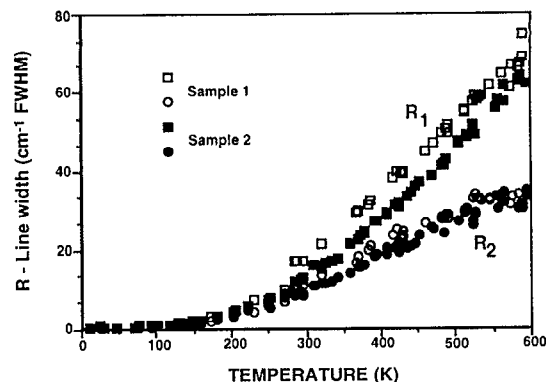


FIG. 2. Linewidths [full width at half-maximum or (FWHM)] of the R lines vs temperature. The open symbols are for the ruby with unknown Cr^{3+} concentration and the closed symbols are for the oriented ruby samples with 0.5% Cr_2O_3 .

$$R_2(T) = 14\,452 + 3.00 \times 10^{-2}T - 3.88 \times 10^{-4}T^2 + 2.55 \times 10^{-7}T^3 \text{ cm}^{-1}. \quad (4)$$

The coefficient of determination²⁷ for the fits given in Eqs. (3) and (4) is $r^2 \geq 0.990$ indicating that these fits can be used with very high confidence. From 300 to 600 K the line positions fit well, though more poorly than to the cubic equations, to linear functions of T ,

$$R_1(T) = 14\,450 - 0.158T \text{ cm}^{-1}, \quad (5)$$

$$R_2(T) = 14\,483 - 0.162T \text{ cm}^{-1}. \quad (6)$$

Line positions given by the linear fits of Eqs. (5) and (6) deviate by as much as 1.7 cm^{-1} from the line positions given by the cubic fits of Eqs. (3) and (4). This deviation is likely correlated with the increased splitting which is discussed below. In Eqs. (3)–(6) we have only given line positions to within 1.0 cm^{-1} , as this is the absolute accuracy to which our monochromators were calibrated, and is indicative of the scatter in the data. The shifts, however, are independent of the absolute spectrometer calibration, and therefore much more accurate.

A plot of the R_1 and R_2 linewidths (FWHM) versus temperature is shown in Fig. 2. The width of both lines is seen to increase with temperature. The width of R_2 however, increases more slowly and always remains smaller than that of R_1 . It is interesting to note that the R_1 linewidths are different for the ruby with unknown Cr_2O_3 concentration and the ruby with 0.5% Cr_2O_3 . Initially we believed that this difference was due to the different chromium concentrations. To test this assumption, we measured the linewidths of a 0.12% Cr_2O_3 ruby at room temperature, but found them to be the same as those of the 0.5% Cr_2O_3 ruby, indicating that other factors may also be involved. In contrast to the R_1 linewidth, the R_2 linewidth shows little difference between samples.

Figure 3 shows the R_1 - R_2 splitting with temperature. The splitting increases very slowly from 15 to 250 K, then increases quite rapidly reaching a maximum at about 450 K. At temperatures above 500 K, the splitting appears to

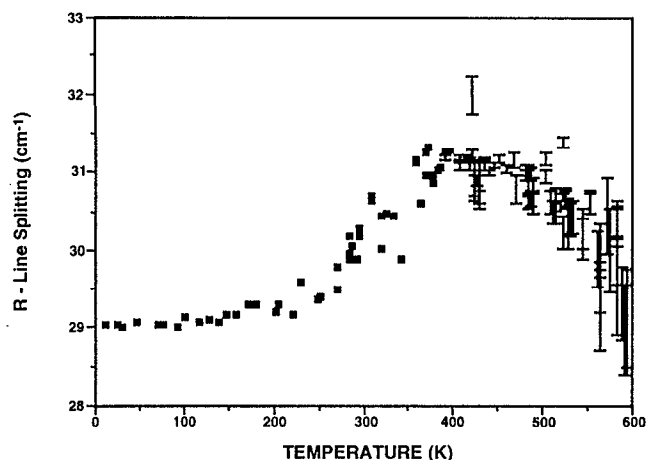


FIG. 3. R -line splitting vs temperature. Error bars were obtained from the fitting procedure and are based on the statistics of the spectral data. For temperatures below 400 K, the error bars were typically less than 0.2 cm^{-1} and were not put on the plot.

decrease. Because the error bars above 500 K get rather large ($\sim 1.5 \text{ cm}^{-1}$) the splitting decrease is somewhat uncertain. For temperatures below 400 K, error bars were typically less than 0.2 cm^{-1} and were not put on the plot. (Error bars were determined by the fitting algorithm and are based on the statistics of the data.)

The polarization measurements showed two main features. First, comparison of the intensities in the two polarizations, indicates that the fluorescence is highly polarized with its electric vector perpendicular to the c axis. This result is in agreement with the work of Nelson and Sturge.²⁴ Second, the R_1/R_2 intensity ratio changes markedly with fluorescence polarization. When the analyzer is rotated so that the fluorescence component with E parallel to c is collected, the R_1/R_2 intensity ratio is minimum; at 300 K, R_1 is about half as intense as R_2 . When the analyzer is rotated so that the fluorescence component with E perpendicular to c is collected R_1/R_2 is maximum; R_1 is about 1.5 times as intense as R_2 and the spectrum appears approximately the same as the unpolarized fluorescence spectrum. (This again indicates that the fluorescence is highly polarized with E perpendicular to c .) Finally, between polarizations the intensity of R_1 changes a lot while the intensity of R_2 is more constant, indicating R_1 is more polarized than R_2 .

The polarization measurements also produced a confusing result. At higher temperatures ($>450 \text{ K}$), the line positions seemed to depend on the fluorescence polarization. For example, at 470 K in the E perpendicular to c and E parallel to c polarizations, the fitted R_1 -line positions differed by 4.0 cm^{-1} and the fitted R_2 positions differed by 2.5 cm^{-1} . Because the fitted R_1 linewidths were different by 4.1 cm^{-1} and the fitted R_2 linewidths were different by 2.7 cm^{-1} for the two polarizations, we believe that this effect is not real but an artifact of the fitting process. The fitting difficulties may arise because of the broad overlapping lines and because the relative intensities are different in the two polarizations.

IV. DISCUSSION

A. Line shifts

Our line shifts for the temperature range 300–600 K are considerably larger than those previously reported. Equations (5) and (6) give the shifts as $\Delta R_1/\Delta T = -0.158 \text{ cm}^{-1}/\text{K}$ and $\Delta R_2/\Delta T = -0.162 \text{ cm}^{-1}/\text{K}$, while Refs. 2 and 19 list the shift for R_1 as $\sim -0.14 \text{ cm}^{-1}/\text{K}$. Yen and Nicol's¹⁹ R_1 shift of $-0.153 \text{ cm}^{-1}/\text{K}$ is closer to our value. The likely reason for the difference in line shifts is that we have used line positions obtained from fits to the spectrum, whereas, the authors of Refs. 2, 15, and 19 obtained line positions by eye. Fitting the spectrum to a double Lorentzian allows us to separate the R_1 and R_2 components, thereby obtaining accurate line positions. Peak positions obtained by eye will give R_1 positions that do not shift as fast because, at high temperature, the R_1 and R_2 lines overlap and the apparent R_1 peak is at higher frequency than the line's true resonant frequency. Use of the $-0.14 \text{ cm}^{-1}/\text{K}$ shift^{2,15} will result in an R_1 line position error of 4 cm^{-1} (0.5 GPa) at 500 K.

While the present work was not intended to test the assumption that the R -line shifts have independent temperature and pressure components, there is evidence for the validity of this assumption which deserves comment.^{12–15} For pressures of a few kbar, Noack and Holzapfel¹⁴ measured the R -line pressure shift at room and liquid-He temperatures. They found the pressure derivative to be the same at both temperatures. Yamaoka and co-workers¹⁵ have also inferred the independence of the pressure and temperature shifts from their experiments. Better evidence comes from Horn and Gupta,¹² who have made R -line measurements on shocked ruby initially at room temperature, and Burt,¹³ who has made identical measurements except that the ruby was initially at 77 K. Both authors corrected for the small shock temperature increase (about 10 K for a 125 kbar shock¹²) using the ambient pressure temperature shift [Eq. (1), or Eqs. (3) and 4)]. When this correction was applied, the R -line shifts versus volume change were identical at both temperatures.¹³ Thus, at these two temperatures, the R -line shifts clearly have independent pressure and temperature components. A weaker argument is that the temperature and pressure shifts are caused by different mechanisms. McCumber and Sturge showed that the temperature shift is caused by the interaction of acoustic phonons with the electronic energy levels.¹⁶ By contrast, Sharma and Gupta have shown that the pressure shift is caused by the strain induced change in the potential surrounding the Cr^{3+} ion.¹⁰ Because of this evidence, we believe the assumption, that the R -line shifts have independent temperature and pressure components, is reasonable.

B. R -Line splitting

R_1 and R_2 do not shift exactly together with increasing temperature. This is indicated by the different fitting coefficients for the two lines [Eqs. (3)–(6)] and also in the changing separation seen in Fig. 3. Errors as large as 3

cm^{-1} (0.4 GPa) can be introduced by assuming that R_2 shifts by the same amount as R_1 .

The change in splitting with temperature is previously unreported and its origin is unknown. Although the thermal expansion of ruby is slightly anisotropic, calculations using the symmetry adapted strain theory of Sharma and Gupta,¹⁰ indicate that the c/a ratio does not change in such a way as to account for more than 0.2 cm^{-1} of splitting change. We think that it is likely that, with increasing temperature, the Cr^{3+} ion shifts slightly in relation to the surrounding oxygens. This conjecture could be confirmed by x-ray measurements at elevated temperatures. If the chromium ion does shift with increasing temperature, it may also contribute to the temperature induced line shifts.

C. Linewidths

The width of the R_1 line is strongly dependent on temperature, but also depends on chromium concentration (Fig. 2). Although Powell *et al.*²⁰ do not point this out, their linewidth versus temperature plots also show dependence on chromium concentration. Yamaoka and co-workers¹⁵ have recommended use of the R_1 linewidth to estimate temperatures. Because the R_1 linewidth is so strongly dependent on Cr_2O_3 concentration, this practice can result in substantial errors unless a temperature calibrated ruby is used. Our results, as well as those of Powell *et al.*²⁰ indicate that the width of the R_2 line is much less dependent on the chromium concentration. If the ruby is not previously calibrated, using the width of the R_2 line would lead to smaller errors.

Line broadening in rubies is caused primarily by the interaction of the R -line electronic levels with acoustic phonons;^{8,16} however, it can also be caused by direct processes within the ground-state manifold and microscopic strains within the crystal. These factors have been carefully discussed by Schawlow,⁸ and by McCumber and Sturge.¹⁶ Differing chromium concentrations could conceivably result in differing levels of microscopic strain. Furthermore, microscopic strain levels might be different for crystals grown under nominally identical conditions. (The reason that the linewidths are equal for the 0.5% and 0.12% Cr_2O_3 samples may be because these two samples have the same levels of microscopic strain.) Thus, not all line broadening is caused by temperature, and temperatures estimated from linewidths¹⁵ should be used only with knowledge of the other factors involved.

D. Recommendations

(i) Especially at high temperatures or when using the less intense R_2 line it is advisable to obtain peak positions, linewidths, and intensities by fitting the spectrum to a double Lorentzian. More reliable peak positions are obtained by this procedure than through choosing the peak position by eye. If line positions are chosen by eye, the R -line positions, and thus shifts, will be different than those obtained from fits. In this case the temperature shift given by Yen and Nicol¹⁹ should be used rather than Eqs. (3) or (4).

(ii) If the measured splitting is different than that given in Fig. 3 the ruby sample is subject to nonhydrostatic stress and, as suggested by Gupta and Shen,⁹ it is advisable to use the R_2 line to measure the mean stress or pressure.

(iii) Use separate functions for the R_1 and R_2 shifts with temperature.

(iv) When using the ruby linewidth to estimate temperature, use a temperature-calibrated sample. If this is not possible, use the width of the R_2 line as it is not as sensitive to Cr^{3+} concentration. Be certain that there are not stress gradients across your ruby chip (this also causes line broadening), and obtain the linewidths by fitting.

V. CONCLUSIONS

Gupta and co-workers^{9,10} have recommended the use of the R_2 line for pressure calibration in DACs when the pressures are nonhydrostatic and the orientation of the stress tensor relative to the ruby crystal orientation is unknown. Our results indicate that there are reasons for preferring R_2 at high temperatures as well: Its width is insensitive to Cr^{3+} concentration, it is slower to broaden with temperature, and it is not as highly polarized as R_1 .

In practice, the R_2 line is relatively easy to use. At low temperatures, the intensity of R_2 is weak, however it is sharp and well separated from R_1 . At higher temperatures, the intensity of R_2 increases so that it is nearly as intense as R_1 . A good double-Lorentzian fitting algorithm, which is necessary to get accurate R_1 line positions anyway, will also give accurate R_2 line positions.

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