



To study extreme conditions of pressure and temperature using Raman spectroscopy

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Abstract

The primary advantage of Raman spectroscopy is its capacity to deliver a vast amount of quickly and readily analyzed information. Observations of the associated elementary excitations can be used to characterize the electronic, magnetic, vibrational, and elastic subsystems using the Raman spectra. Because of their extreme selectivity, the Raman spectra of phonons-lattice and molecule vibrations-allow for finger-printing study of a material's phase, including information about its composition and condition. Vibrational selection criteria, which control the Raman activity of phonon modes based on their symmetry and the wave vector, reveal information about the crystal structure. By coupling to the vibrational modes and observing the spectra of the electronic and magnetic excitations, one can gain access to electronic and magnetic states.

Keywords: Raman, spectroscopy, magnetic, vibrational, elastic

1. Introduction

In situ Raman spectroscopy is a useful tool for studying related phenomena since it may observe changes in the elemental excitation spectra with pressure. These consist of phase transitions (including melting), chemical reactivity, magnetic and electronic transitions, and variations in the energy of vibrational excitations. These transitions are of relevance for earth and planetary research, materials science, and fundamental physics and chemistry, thus this information can be applied in a variety of ways. Finally, but just as importantly, Raman spectroscopy is an excellent instrument for measuring pressure at both very high and very low temperatures.

Prior thorough analyses of Raman spectroscopy at high pressures have mostly covered applications related to environment and mineralogy. Since freshly developed methods and recently achieved, ground-breaking experimental results have frequently led to new scientific understanding, other reviews need to be updated. Furthermore, the subject of Raman research at high pressure and high temperature employing laser heating in diamond anvil cells (DACs) is very new. This, in turn, made a new review paper appealing. The author reviews high-pressure

Raman spectroscopy techniques and applications here, including these recent advances.

2. Development of Raman Techniques

When using high-pressure Raman instrumentation, the DAC interface needs to be used with extra caution. Samples in the DAC typically have dimensions of several tens of micrometers and a thickness of a few micrometers; if measurements to ultrahigh pressures (>100 GPa) are anticipated, these dimensions will be much smaller. Large geometric and chromatic aberrations are caused by an optically thick diamond window (2 mm) in the optical path, which compromises depth selectivity, signal collection efficiency, and image quality. Large numerical aperture optics were not practical until recently because to their incompatibility with the stability requirements of supporting backing plates and diamond anvils. Large working distance objective lenses (typically 10 mm) are also required. Furthermore, Raman studies at low frequencies are challenging due to parasitic back reflections caused by diamond windows in the optical channel. The reason for this is that the laser light's elastically scattered and specular reflection raises the background level, which lowers the

signal-to-noise ratio. The background level is also influenced by the fluorescence of diamond anvils in the laser beam. Due to all of these constraints, standard laser microscopes usually do not offer enough flexibility to solve all of the above-described concerns. Therefore, custom-made laser microscopes are required to connect with the DAC.

Our earlier articles provide a description of the bespoke Raman microscope for DAC. Here, the author will highlight three key features: the flexibility in selecting the scattering geometry, the presence of the intermediate field aperture (spatial filter) to reduce spurious background, and the ease with which the excitation wavelength can be changed to provide the best excitation conditions and reduce fluorescence.

Much of the spurious elastically dispersed laser radiation is attenuated by a confocal field aperture whose width matches the size of the laser point. Additionally, it reduces the depth of focus, significantly reducing the diamond fluorescence backdrop. The confocal geometry can be realized by narrowing the detector readout in the nonspectral direction and using the spectrometer's entrance slit, which makes the intermediate field apertures redundant. However, our experience indicates that the latter strategy is insufficient for the DAC operation due to a significantly higher level of spurious radiation than that of free-standing samples.

When compared to pure backscattering geometry, the employment of angular excitation geometry (Figure 1) provides for a drastic reduction in background spurious radiation, boosting the signal-to-noise ratio and simplifying access to the low-frequency spectra. Furthermore, this method permits a significant reduction in the background signals of Raman and fluorescence from the diamond anvils since the associated signals, which are mostly excited off-axis, are suppressed more effectively than in the case of axial laser illumination (pure backscattering geometry). Later on, research of metals and high-temperature superconductors will be used as examples to demonstrate this. By adding a beamsplitter (neutral optical density or dichroic), the system shown in Figure 1 can also employ a traditional backscattering geometry.

The DAC needs to be built or altered to permit angular light access in order to achieve the angular excitation geometry. For this aim, previously, tungsten carbide seats with angular conical holes that were specially constructed have been employed^[5, 9]. One needs to utilize an objective lens or separate lens to concentrate the laser energy on the sample. This lens's focal length is longer than the lens that gathers the dispersed radiation because of geometrical factors. As a result, the minimum laser spot dimension at the sample is effectively increased. Furthermore, because off-axis geometric aberrations (such as astigmatism) alter the size and form of the focused laser beam in this instance, the employment of angular laser incidence geometry causes the laser spot to enlarge even more. The usefulness of this geometry is limited by both of these variables (e.g., for heterogeneous samples). However, in the case of metals, it was discovered that the use of the angular geometry was essential for getting high-quality spectra.

The development of very sensitive and low-background array detectors (CCD), holographic transmission optics, and fast imaging spectrometers has allowed Raman

spectroscopy to make measurements much more quickly. New hard-coated optical filters have entered the market more recently, and they have similar transmission band transition widths to those made with holographic technology. With this technology, unwanted Rayleigh-scattered light is effectively blocked by notch, bandpass, and dichroic filters/beamsplitters, eliminating the need for laborious low-transmission double subtractive monochromator-filter systems for routine measurements above 50 cm⁻¹. Furthermore, a new generation of solid-state technology-based notch and bandpass filters has recently been developed, with a transition width as tiny as 10 cm⁻¹. It is anticipated that these filters will not deteriorate over time and will be far more environmentally stable.

Rapid wavelength changes are frequently necessary when using Raman spectroscopy for research at extremely high pressures. In order to offer the best excitation (for example, ruby fluorescence), identify the nature of the bands observed (e.g., Raman versus fluorescence), and investigate resonance phenomena, this is necessary. In comparison to traditional triple-stage spectrometers, modern Raman spectrometers, which employ the very light-efficient and narrow transition width filter technologies (see above), are less versatile when it comes to excitation wavelength adjusting. However, the micro-Raman system presented here (Figure 1) can quickly switch the excitation wavelength over the spectrum range up to 100 nm without requiring the employment of additional holographic filters. This is accomplished by adjusting the filters' angular positions with regard to the directions of the scattered beam and the laser. The system alignment is unaffected by these filter rotations. Confocal Raman imaging (e.g., in the DAC) would be a crucial advancement in high pressure Raman techniques. Using objective lenses with a significantly higher numerical aperture (e.g., NA 0.7) is possible with newly constructed laterally supported diamond anvils than with anvils of the usual design. This allows for 3D Raman mapping in theory. Large optical distortions caused by diamond windows remain a barrier. The wave front is distorted by these aberrations, which makes it challenging to make depth-selective observations. Specialized compensating optics can be used to solve this issue; work in this direction is ongoing. Recent developments have produced in situ Raman measurements in laser-heated DAC. When compared to the prior CO₂ laser heating method, the employment of near-infrared (1060 nm) lasers as a heating source allows for the reduction of the hot spot and the production of more steady heating conditions. Since near-infrared fiber lasers with enhanced mode quality and stability went on sale, these benefits have been much more apparent. The possibility of using the method at very high pressures is made possible by the enhanced laser beam quality, which permits very localized heating of the materials inside the DAC cavity. The invention of laser beam shapers, which enable control over the distribution of laser power in a focused plane, has recently allowed for significant improvements to the technology. Radial temperature gradients are lessened and heating conditions are more consistent when a flat top or bimodal power distribution is used.

It is difficult to heat materials that do not absorb laser energy. As a laser beam absorber (coupler), a thin foil with one or more tiny holes, or a powder composed of chemically

stable metals, can be used to provide high-temperature conditions for the surrounding sample material. Using couplers with tiny holes has perhaps produced the greatest results for in situ Raman spectroscopy. An IR heating laser spot larger than the hole heats the coupler, while another tightly focused visible laser stimulates the material in the hole for Raman scattering. The temperature gradients through the material being probed are reduced by this shape. Additionally, by applying thermal insulation layers close to the diamond anvil tips—which stay cool throughout the experiment because of the diamond's high thermal conductivity—the sample is removed from these regions, further lowering the temperature gradients across the sample that is being probed. The temperature variations across the sample inside the coupler hole are lessened with the use of a double-sided laser heating. With the use of a series of wave plates and polarization cubes, the laser strength may be divided into two halves and separately controlled at each. Data acquisition for this system has recently been automated, and as part of that process, all spectral measurements (radiative and Raman temperature measurements) at varied heating laser power have been carried out. By using this upgrade instead of manual data collecting, you can save up to 90% of the time. This enhancement is crucial for research involving extremely chemically reactive materials, like hydrogen, for instance. Raman measurements at high temperatures are hard due to the heat radiation released by the sample and the coupler.

By distinguishing the heat radiation both geographically and spectrally, a significant portion of it can be avoided. This can be accomplished by employing a confocal backscattering geometry in conjunction with a blue excitation wavelength (such as 458 nm from a solid-state laser). In the experimental setting, high-quality Raman spectra can be produced up to about 2000 K. A pulsed Raman system with 532 nm excitation synchronized with the gated intensified CCD detector can be used in conjunction with continuous or pulsed laser heating at higher temperatures when thermal radiation rises above a critical threshold and obstructs the Raman signal. Any continuous (or longer-lasting) spurious radiation is suppressed by a factor that is proportional to the ratio of the time interval between pulses to the gate width, as the detector records the entire Raman signal (the Raman processes are very short, typically <1 ps) during the relatively short gate time and is closed between pulses. It has been stated that suppression factors of up to 50,000 are possible, meaning that Raman signals can be obtained at temperatures higher than 5,000 K. The previous introduction of innovations in Raman instrumentation has opened up several new possibilities for high-pressure Raman experiments, which will be discussed here. The capacity of contemporary Raman systems to do measurements at very high pressures and temperatures (P-T) with an acquisition time of less than one second is arguably the most notable feat.

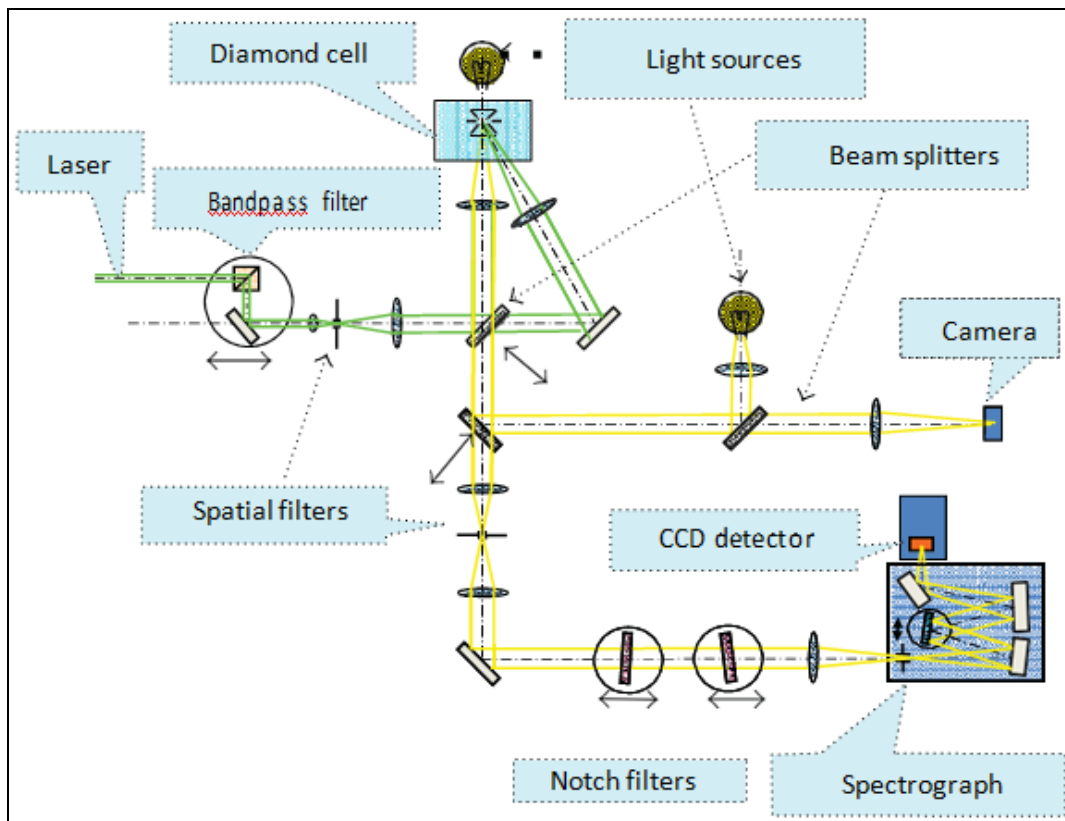


Fig 1: Layout of the Raman optical microscope system

3. Raman studies at high pressures

Diatomic Molecule Phase Diagrams. Because vibrational property measurements offer a potent diagnostic of this process, Raman spectroscopy is perfectly suited for

analyzing the evolution of the bond character in diatomic molecules under compression. Compression modifies the electrical density function in order to reduce the system's overall energy. Since the kinetic energy of the involved

electrons rises sharply with compression, pressure tends to disrupt the intramolecular bonds in molecular materials. Other phenomena that impact molecule stability, like the development of certain intermediate states, could complicate this process. Investigating basic molecules like diatoms can be a useful strategy in resolving this issue.

Because hydrogen has a simple electrical structure and is widely distributed across the universe, where it is a key component of stars and planets, its behavior under pressure is of particular interest. The Raman spectroscopy technique is still quite limited today, able to examine hydrogen under circumstances of molecular dissociation down to about below 2000 K and 150 GPa. Since hydrogen can be regarded as a group VII element, the author will start by discussing the behavior of the halogens, which can be thought of as analogous to hydrogen in electronic structure. The phenomena of interest in these materials occur at much lower pressures than in hydrogen (<100 GPa). Iodine (I₂) and bromine (Br₂) have been the subject of much research recently. The presence of an intermediate charge modulated phase as a function of pressure, the insulator-metal transition, and the change in bonding character were all made possible by the use of Raman spectroscopy. The Raman spectra did, in fact, show several changes with pressure that are consistent with the findings from other methods, including electrical conductivity, X-ray absorption spectroscopy, and X-ray diffraction. Discontinuous frequency shifts and the emergence of new bands that may be connected to metallization are seen in Raman experiments. Theoretical calculations have revealed that these observations may also be linked to more modest phase shifts, or both occurrences may occur at the same time. As the molecular dissociation transition approaches, the Raman spectra exhibit significant changes. Just prior to the changeover

A new low-frequency soft mode linked to the incommensurate structural and electrical modulation emerges in relation to the monatomic phase. I₂ and Br₂ exhibit highly comparable behaviors, and the ability to scale vibrational frequencies suggests that the observed consecutive phase transition mechanism for molecule dissociation is generally applicable.

Nitrogen serves as an example of a material with more complex high-pressure behavior because it presents an extra option for the creation of a material with covalent bonding—a single-bonded polymeric phase. Raman spectroscopy has revealed the extremely rich polymorphic behavior of molecular nitrogen, which contributes to the additional complexity. Expected to destabilize at pressures above 50 GPa, the triply bound N₂ molecule exhibits tenacious stability up to 170 GPa at 300 K (200 GPa at 80 K) in Raman and infrared absorption measurements. The kinetic limits between several types of molecular and nonmolecular nitrogen phases are the cause of this phenomenon. Because Raman spectroscopy is extremely sensitive to even small changes in the crystal structure, it is an extremely useful technique to study several solid-solid phase transitions and metastability phenomena in nitrogen (and other molecular materials, see below). This is due to the Raman selection criteria' sensitivity to the presence of atomic sites with distinct symmetry, as well as to orientational ordering and vibrational coupling. X-ray

diffraction and Raman spectroscopy data finally helped identify the monatomic polymeric phase, which is nitrogen. Based on the combined results of several studies, it was discovered that this change happened abruptly at 110 GPa. This unusual dependence on pressure demonstrates that molecular dissociation is a volume-driven process that causes the molecular orbitals to become unstable. The fluid-fluid transition associated with molecular dissociation was reported based on shock-wave experiment results and theoretical calculations. Static high-pressure experiments are needed to study this phenomenon in order to gain a better understanding of its nature, as these experiments can yield more informative diagnostic tools. In the upcoming years, this topic will present a challenge to the traditional Raman spectroscopy instruments.

Another great example of how Raman spectroscopy may be used to determine the melting line is nitrogen. A modification of the vibrational selection rules in a fluid state serves as the foundation for this. In fact, because the molecular centers of gravity lack translational order, one might anticipate that the phonon modes—translational intermolecular vibrations—should manifest in the fluid molecular phase. These observations, however, can be mistaken for phenomena that occur in conjunction with the transition from a solid phase that is orientationally disordered to a flexible phase. Thankfully, there are concurrent changes in the Raman spectra of molecular vibrons (intramolecular vibration) associated with the melting of nitrogen, which enable more accurate diagnostics to be made. Specifically, two major vibron modes (ν_1 and ν_2) are present in solid molecular nitrogen phases, which correspond to different site symmetries found in these structures. These vibron modes result from slightly different intramolecular bond lengths and intermolecular arrangement, which in turn results in different vibrational frequencies. This difference naturally disappears in the fluid phase, giving rise to a single vibron mode (almost at the frequency of ν_2) and a sudden slight frequency shift of ν_2 . One common occurrence linked to phase transitions in molecular materials is a discontinuity in the vibron frequency, which is typically caused by modifications in the intermolecular coupling. An further diagnostic for melting is a broadening of the vibron mode. This extra widening results from sudden changes in the surrounding environment causing time-dependent thermally-induced bond length fluctuations in the molecules in the fluid phase. Other experimental methods, such as X-ray diffraction measurements of the diffuse scattering ring, should give the final test for the Raman melting diagnostics.

For the past few decades, the phase diagram of the hydrogen isotopes (H₂ and D₂) has been extensively studied at very high pressures using Raman spectroscopy of an ensemble of vibrational modes in these compounds. Three main solid molecular phases can be seen in the experimentally accessible region of the hydrogen phase diagram (<310 GPa at 100 K): phase I, which is orientationally disordered, and phases II and III, which are partially or fully ordered high-pressure low-temperature phases. The appearance of the fluid or solid phase with freely rotating molecules can be seen in the Raman spectra as a sequence of rotational transitions, or rotons. J , the total angular quantum momentum number, governs quantum statistics that explain

the Raman rotational transitions. With compression, the deviations from this straightforward model get progressively larger. An increase in anisotropic intermolecular contact causes the phase II transition, also known as the broken-symmetry phase, or BSP, and alters the rotational ground state, making it non-spherical. Significant alterations in roton Raman spectra and vibron-roton linked modes (vibron sidebands) as well as the emergence of restricted rotational motion modes (librons) are indicative of this quantum ordering transition. Phase III is characterized by additional changes in the low-frequency Raman spectra, as several narrow libron/phonon modes replace the remaining roton mode remnants, indicating a shift towards a classically orientationally organized phase. In terms of phase transitions, the behavior of the phonon mode—which is associated with the out-of-phase lateral translational intermolecular vibrations—is less instructive; yet, as this mode steadily increases, it indicates that intermolecular coupling increases with compression.

These sharp shifts in the rotational Raman spectra with pressure are extremely informative for molecular rotational and orientational state diagnostics, but their findings are quite difficult to make experimentally. Thus, phase boundaries were mapped out in most investigations using Raman (and also IR) spectroscopy of the molecular vibron (which is simpler to discern). Between phases II and III as well as at the transition to phases II and III, the vibron frequency exhibits an abrupt discontinuity. The remaining elusive are the observations of extra solid phases of hydrogen, which were first proposed theoretically and empirically. Although this regime has not yet been investigated, the transition to phase III at 300 K is predicted to be seen at 180 GPa. For the I-II and II-III transitions, respectively, the vibron frequency discontinuity ranges in value from a few wavenumbers ($<15\text{ cm}^{-1}$) to over 100 cm^{-1} (Figure 3). A alteration of intra- and intermolecular interactions associated to orientational ordering and structural changes during the transitions is the cause of the elevated value of the vibron discontinuity for the phase III transition. Additionally, phase III is marked by significantly higher IR activity, providing evidence for asymmetric atomic site symmetries and orientational ordering.

Up to megabar (100 GPa) pressures, vibrational spectroscopy is still the primary diagnostic tool for determining the state and structure of dense hydrogen. Phases II and III structures still needed to be definitively defined because the X-ray and neutron diffraction data that are currently available are insufficient for this purpose. At 100 K , measurements of hydrogen's optical and vibrational characteristics have been expanded to 315 GPa , while research at higher temperatures is still extremely difficult technically. In fact, no report of hydrogen above 200 GPa at ambient temperature has been reported.

Since this phenomenon is related to a partial charge transfer due to an increased coupling of the molecular orbitals through the intermolecular bonds, a steady decrease of both Raman (Figure 3) and IR active vibrons, which indicate the approach of molecular dissociation, is an important feature of the vibron behavior at high pressures. It's interesting to note that I_2 and Br_2 do not fundamentally disclose the vibron softening, which was proposed as a "harbinger" of the molecular dissociation. Therefore, this phenomena is not

widespread. Alternatively, it has been demonstrated that other modes become softer close to the monatomic state transition. Subsequent investigations into hydrogen (H_2 and D_2) will reveal whether the low-frequency phonon softening is a typical aspect of the shift to a monatomic state or if alternative, more diffusive phase transition mechanisms are also possible.

One might anticipate that Raman spectroscopy can be successfully applied for studies of hydrogen at high pressures and temperatures approaching the fluid phase and the plasma transition given its great success in mapping the phase diagram and unraveling the bond properties of hydrogen at high pressures and low temperatures. It has been demonstrated that the hydrogen vibron softens significantly at high temperatures, and the melting transition can be identified from a tiny vibron discontinuity. A significant negative vibron frequency discontinuity was found close to the melting limit in a recent Raman analysis of hydrogen in the laser-heated DAC. The roton bands have also seen significant broadening and intensity drop in tandem with this. These findings imply that the fluid is undergoing a pressure-induced transition, which may be connected to the melting line's temperature maximum as a function of pressure. Such a fluid with altered intramolecular interactions has been postulated by earlier Raman studies in the laser-heated DAC. It has also been hypothesized that pairing fluctuations of nuclei may be relevant in such fluid states. Prospective investigations within the elevated P-T regime that examine these states could necessitate enhanced diagnostic methods and possibly an alternative experimental approach, such as the pump-probe methodology. It's true that dynamical features appear to be more significant in this situation than time-averaged material properties, which classical Raman spectroscopy can only reveal.

4. Conclusion

In the DAC, Raman spectroscopy has established itself as one of the most effective and widely utilized techniques for high-pressure research. Recent advancements have increased its sensitivity and spatial resolution, enabling it to be used as a portable system, like online systems at X-ray synchrotron beamlines, and as a quick diagnostic tool for in situ laser heating measurements. The Raman systems used for high-pressure studies have the potential to significantly enhance their temporal and spatial resolution. Furthermore, Raman systems with very high throughput and low frequency ($<10\text{ cm}^{-1}$) will soon be widely available.

Numerous interdisciplinary investigations, such as material science, planetary interior physics and chemistry, search for other energy sources and carriers, detonation physics and chemistry, and many more, have been made possible by these technological advancements. The study of chemical reactivity in heterogeneous samples (including mineral surfaces) and other quick processes will be made possible by the higher spatial and temporal resolutions. This development is closely linked to the undeveloped new capabilities of surface enhanced Raman spectroscopy. Furthermore, the potential of time-resolved and pump-probe methods has to be further explored; the integration of dynamic high-pressure techniques with coherent anti-Stokes Raman spectroscopy (CARS) will expand the applications

of high-pressure and geoplanetary science.

New advancements in Raman techniques that enable direct probing of these features in situ statically or dynamically employing sophisticated laser sources and detection techniques will be necessary for future potential scientific endeavors. These include time-resolved detectors with enhanced spatial and temporal resolution (such as streak-cameras) and ultrafast lasers. Developing new tools to investigate material properties in extreme P-T environments and very small quantities is of particular interest. These tools may be beyond current capabilities, but they may offer new insights into the synthesis of materials under nonequilibrium conditions as well as the structure and history of planets.

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