Metallic Hydrogen

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Paul Loubeyre, Florent Occelli, and Paul Dumas, "Synchrotron Infrared Spectroscopic Evidence of the Probable Transition to Metal Hydrogen," *Nature* 577, no. 7,792 (2020): 631–35, doi:10.1038/s41586-019-1927-3.

In his 2003 Nobel lecture, Vitaly Ginzburg defined thirty problems for the twenty-first century.[1] The first three involved hydrogen and metallic hydrogen; and, indeed, the problem of producing metallic hydrogen in the laboratory has remained unsolved for over eighty years. Significant challenges are involved, not least of which is the generation of pressures greater than those at the center of the earth.

Atomic hydrogen is the first and simplest atom in the periodic table. It has a single electron bonded to a nucleus of a single proton: together they form a neutral atom. Though hydrogen is the most abundant element in the universe, the isolated hydrogen atom does not occur naturally on earth because it is chemically reactive. If hydrogen atoms are confined, they combine to form molecular hydrogen, H_2. Molecular hydrogen is the lightest of all molecules and comprises two protons and two electrons held together by covalent bonds. The protons are tightly bound to electrons due to attractive Coulomb forces, with a high density of the electrons distributed between the protons. If a gas of H_2 is cooled, it will liquefy at a temperature of 20.4 K and solidify at around 14 K.[2] Solid molecular hydrogen is known as a quantum solid due to its large zero-point energy and zero-point motion. Large zero-point motion is a property of light particles such as hydrogen and helium. As a result of zero-point motion, in the zero-temperature limit, the solid lattice is expanded in comparison to a classical solid, so that molecules have a low probability of overlapping. As a consequence, hydrogen is highly compressible at low temperature in a liquid or solid state. In a solid state, hydrogen molecules can translate, rotate, and vibrate, and these excitations are useful in studying the phases of solid hydrogen.

In condensed matter physics, an important goal is to understand the phase diagrams of materials as a function of temperature and pressure. As temperature and pressure are varied, properties such as lattice structure, electrical conductivity, and viscosity can change when new phases are entered. In 1935, little was known about the phase diagram of hydrogen, except that it was a molecular solid and a transparent insulator. The protons in the molecule were very close, 0.74 angstroms (Å) apart, the nearest neighbor molecules separated by about 3.8 Å. The lattice structure was not known. It was believed that, at a high enough pressure or density, all substances would become metallic.

Eugene Wigner and Hillard Huntington studied the theoretical possibility of a metallic phase of hydrogen in the high density, low temperature limit.[3] In this case, as pressure or density is increased, the nearest neighbor separation of molecules becomes smaller and smaller, so that the molecular separation becomes comparable to the atom–atom separation in a molecule. Eventually, the free energy of the solid can be lowered if the molecules dissociate to become an atomic solid. This solid has a half-filled energy band so that it becomes a metal—atomic metallic hydrogen. Since little was known of the equation of state regarding pressure versus density for hydrogen, Wigner and Huntington assumed the known compressibility at zero pressure to be the same for all pressures and arrived at a transition pressure of 25 GPa, or 0.25 megabar (100 GPa = 1 megabar). Modern theory predicts ~400 to 500 GPa.[4] In our experiment at Harvard, we found a transition at 495 GPa, or about twenty times the pressure predicted by Wigner and Huntington.[5] The Wigner and Huntington transition is expected to take place in both hydrogen and its isotope deuterium at around the same pressure. Wigner and Huntington had challenged the experimental world to produce metallic hydrogen. It would be eighty years before the challenge was met.

In 1968, Neil Ashcroft predicted that metallic hydrogen might be a high-temperature superconductor with a high critical temperature.[6] A pressure of 25 GPa represented a great experimental challenge, but in time, both experimental and theoretical methods advanced. Jeffrey McMahon and David Ceperley predicted a critical temperature above room temperature for atomic metallic hydrogen.[7] David Ramaker, Lalit Kumar, and Frank Harris then predicted that molecular hydrogen could ionize to

become metallic, even as Carlos Friedli and Neil Ashcroft found a transition to metallic molecular hydrogen at a compression 9.15 times the zero-pressure density and a comparable pressure.[8] Pier Cudazzo et al. predicted a high critical temperature, something like room temperature, for the molecular metal.[9] There were other interesting predictions: high-pressure atomic metallic hydrogen might be a liquid at zero temperature due to its zero-point energy, but a metastable atomic metal when the pressure is removed.[10] Ashcroft, Egor Babaev, and Asle Sudbø considered the possibility of both superfluidity and superconductivity in liquid metallic hydrogen.[11]

In addition to the low-temperature thermodynamic pathway to metallic hydrogen determined by Wigner and Huntington, there is also a high-temperature pathway. At intermediate high pressure on the order of 1 megabar, solid molecular hydrogen can be heated so that it first melts to liquid molecular hydrogen and then crosses a first-order phase transition line to become liquid atomic hydrogen. The high-temperature form is the liquid metallic hydrogen found on Jupiter.[12] This transition was considered on general theoretical grounds by Genri Norman and Andrey Starostin, who named it the plasma phase transition.[13] The liquid–liquid phase transition has been studied extensively in hydrogen.[14] Its phase line has a negative pressure–temperature slope and a critical point at lower pressures and higher temperatures. The metallic liquid phase was first observed using dynamic shock compression by Sam Weir, Arthur Mitchell, and William Nellis.[15] Shock experiments are very short, typically less than a microsecond, and the sample is heated to high temperatures. Weir et al. observed metallic hydrogen at high pressure and temperature, but did not observe the predicted phase transition line. They conjectured that metallic hydrogen was due to closing of the electronic band gap.[16] Later, in static measurements on hydrogen in a diamond anvil cell (DAC) and at temperatures in the range of 1,000 to 2,000 K, the phase line *was* observed[17] and shown to be a metal in both hydrogen and deuterium.[18] The phase diagram was further studied in dynamic experiments.[19]

Initial efforts to observe metallic hydrogen encountered phase transitions within the molecular crystal at pressures that exceeded Wigner and Huntington's prediction. These did not lead to metallic hydrogen or deuterium. Rather, they were structural changes of the molecular lattice that remained in the transparent insulating phase.[20] With increasing pressure in DACs, new phases were observed at low temperatures.[21] Since hydrogen is a very weak x-ray scatterer, these phases were observed by optical techniques, Raman scattering, or infrared absorption due to lattice excitations. In 1998, Chandrabhas Narayana et al. achieved the highest experimental pressure yet observed and noted that hydrogen was transparent at 342 GPa. It was not yet a metal.[22] In achieving this pressure, they broke fifteen sets of diamonds using Raman scattering for analysis: every time they shined a green laser beam onto the sample through the highly stressed diamonds, the diamonds failed. A few years later, Paul Loubeyre, Florent Occelli, and René LeToullec observed hydrogen become black in the visible region at a lower pressure of 320 GPa.[23] These contradictory results were discussed by Isaac Silvera as possible problems in pressure determination.[24]

In 2011, Mikhail Eremets and Ivan Troyan observed two new phases around room temperature and pressures of 220 and 270 GPa.[25] They claimed that the higher-pressure phase was liquid atomic metallic hydrogen. This claim was subsequently refuted.[26] A sample at these conditions was later shown to be transparent in the infrared by, among others, a team led by Eremets, confirming that it was not metallic but a transition within the molecular solid.[27] Ross Howie et al. determined the phase lines of these transitions.[28] The line had a shallow slope, so that the phase line existed in the region of room temperature. Although there were several other experimental studies, observation of metallic hydrogen seemed to have hit an experimental barrier in achieving higher pressures: diamond anvils failed at pressures on the order of 300–350 GPa, and most studies used Raman scattering, which were shown by Narayana et al. to be destructive of diamond anvils.[29]

At Harvard, we developed techniques or protocols to achieve higher pressures. Most of these were based on our experience: we developed a good idea of what caused diamond anvils to fail, and started using synthetic instead of natural diamonds. Natural diamonds contain inhomogeneities—impurities that are invisible to the eye. Diamond culet flats were etched to remove polishing defects in the culet region, where the diamond contacts the sample. It is known that hydrogen can diffuse into diamond and embrittle the anvils, so the diamonds were coated with a thin film of alumina acting as a diffusion barrier. To further inhibit diffusion, DACs were cryogenically loaded with hydrogen and maintained at liquid nitrogen temperatures of ~77 K and lower. Rhenium gaskets were specially designed to achieve the higher pressures and were also coated with alumina. Finally, infrared spectroscopy was used rather than Raman scattering, to avoid shining high-power laser light through the stressed diamonds. Pressures greater than 400 GPa were achieved and a new quantum phase transition of molecular hydrogen named H_2PRE was observed at around 360 GPa.[30]

In 2017, we observed the Wigner–Huntington phase of atomic metallic hydrogen at 495 GPa at temperatures between 5 and 83 K.[31] The disc-shaped sample had diametrical dimensions that varied between ~20 microns to 10 microns as pressure increased. At 205 GPa, the sample was transparent, as expected. With increasing pressure, hydrogen enters the H2PRE phase around 360 GPa and darkens until it is opaque and black in reflectance at 415 GPa. When the pressure reached 495 GPa, the sample turned from black to shiny and highly reflective.

This is what we expected for the transition to metallic hydrogen.

To show that it was, indeed, atomic metallic hydrogen, we measured the reflectance as a function of wavelength and fitted this to a Drude free-electron model of a metal to determine the plasma frequency. From the plasma frequency and the molar volume of the sample, we determined that the density of the electrons corresponded to one electron per atom—the mark of atomic metallic hydrogen.[32] The Wigner and Huntington form of atomic metallic hydrogen was produced for the first time in the laboratory—and for the first time in the universe.

The dark hydrogen observed at high pressures, we speculated, came about when the electronic bandgap was closed, so that hydrogen became semiconducting and absorbed light via valence band transitions.[33] We measured the integrated transmission in the infrared and showed that the sample was opaque in this region for pressures above 420 GPa. To determine the pressure, we used calibrated absorption lines in the infrared up to about 335 GPa. Thereafter, infrared absorption was difficult to measure as the sample darkened. Rather than use the shift of the diamond phonon in the stressed part of the diamond, which required laser power, we used an inhouse technique that we have maintained for years. Our DACs are equipped with strain gauges that measure the force or load applied to the gasket and sample. We have found that pressure is proportional to load, which in our case is proportional to the turn of a screw. In this way, we achieved 495 GPa. At this pressure, we measured the pressure using the diamond phonon scale: it agreed with our earlier determined pressure, and the diamonds survived.[34]

Our techniques did provoke a few critiques in the literature, all of them answered in detail.[35] None challenged our claim to have observed the reflectance of metallic hydrogen. Some questions were raised with respect to our claim to have achieved pressures of ~500 GPa where others had failed. It is entirely reasonable that critics asked that our observation should be reproduced. Eremets et al. almost achieved this, attaining a pressure of 480 GPa and measuring electrical conductivity up to 440 GPa.[36] We have analyzed their data and shown that the resistance of their sample was approaching metallic values as the pressure approached 500 GPa.[37]

We stand behind our experimental claim.

Recently, Loubeyre, Occelli, and Paul Dumas (LOD) studied a sample of hydrogen in the infrared using synchrotron radiation; they claim that, in all likelihood, they produced metallic hydrogen.[38] LOD did not actually measure any metallic properties of their sample, such as reflectance and conductivity, and they reported a maximum pressure of 427 GPa. At this pressure, the integrated intensity of the infrared transmission goes to zero. Without further evidence, LOD claim that their sample is metallic molecular hydrogen due to the closure of the bandgap; thus not the Wigner–Huntington transition to atomic metallic, but molecular metallic. Blackening in the infrared is the same observation that we made earlier in our infrared measurements at around 420 GPa,[39] and we speculated that the hydrogen was semiconducting. LOD's observation is not a new result, but a confirmation of our own, albeit with a different interpretation. In their paper, LOD also state that the sample turns black in the visible region at 310 GPa, whereas in earlier papers they reported the same observation at 320 GPa and 300 GPa—a rather large variation of pressure for the blackening in different samples.[40]

A common practice in physics is to submit a paper to a journal and simultaneously post it on *arXiv*.[41] In 2019, LOD posted an article on *arXiv* claiming to observe metallic hydrogen at a pressure of 427 GPa.[42] We found this paper flawed and posted a comment to that effect.[43] LOD's paper went through peer review and was eventually published with a change in the title to "...*Probable* [emphasis added] Transition to Metal Hydrogen."[44] In analyzing their revised paper, we still encountered problems. LOD neglect to measure any properties of hydrogen, such as reflectance, that could be interpreted in terms of metallization. Their samples are quite small, around 5 microns across. They observe hydrogen to become black in the visible region at 310 GPa and nontransmitting in the near-infrared region at 427 GPa: it is this they claim as metallic molecular hydrogen. Using data from Eremets et al. who claim hydrogen may be semimetallic,[45] LOD estimate the plasma frequency to be smaller than 0.1 eV, which is in the infrared (wavelength ~12 microns). According to the Drude free-electron model, a metal becomes transparent at frequencies higher than the plasma frequency.[46] Transmission through metals above the plasma frequency is not unusual: years ago high-pressure xenon was observed to be semimetallic and it transmitted light in the visible.[47]

If LOD's interpretation is correct, their sample should be transparent in the visible region.[48] Their sample is black and nontransparent, even at lower pressures. There are further problems. It is remarkable that LOD did not measure the reflectance of their sample in the visible and infrared regions, since this could establish their case. They base their claim of metallization on someone else's work: the assertion of hydrogen being a semimetal by Eremets et al. who measured the electrical resistivity—that is, the inverse of conductivity—of their sample at high pressure and down to low temperature.[49] In an earlier study, Eremets et al. studied metallization of xenon down to temperatures of 27 mK.[50] They correctly stated that the condition for metallization is $dR/dT > 0$ in the limit $T \rightarrow 0$ K, where R is resistivity and T is temperature.[51] In Eremets et al.'s study of hydrogen, the resistance has a negative slope: $dR/dT < 0$ as $T \rightarrow 0$ K,[52] yet they claim that hydrogen is semimetallic in this regime.

There are other problems with LOD's work. Their pressure determination does not seem to be reliable. They concede that their pressure scale differs from other calibrations for higher pressures where the diamond scale is used.[53] Earlier, it was shown that their pressure scale, based on the hydrogen vibron frequency, differed from all others.[54] Our group and the Eremets group have observed a new phase at pressures of ~360 GPa; LOD do not see this phase. In order to present their observation as unique, they claim that a conventional DAC is limited to pressures of 400 GPa, a statement contradicted by observations of the Eremets group and the Harvard group that achieved pressures approaching 500 GPa with conventional DACs. Perhaps LOD's most puzzling observation is that hydrogen becomes black in the visible region at intermediate pressures of 300, 310, and 320 GPa in different experiments. Almost all other researchers have reported that hydrogen transmits light in the visible region up to pressures of around 350–360 GPa.[55]

How can these varying observations be explained? We propose that LOD's samples may be contaminated with metallic impurities that can change the properties of pure molecular hydrogen. Some years ago, Anders Carlsson and Neil Ashcroft proposed to lower the metallization pressure of hydrogen through doping with metallic impurities.[56] It is not difficult to produce doped samples, as hydrogen itself is very reactive and diffusive. In fact, recent experiments to produce hydrogen-rich compounds at high pressure to study high-temperature superconductivity create samples by heating metals embedded in hydrogen-filled cells.[57] Diffusion is temperature dependent and scales exponentially with temperature. At Harvard, to avoid this problem we cryogenically load samples and hold temperatures to between ~4.2 and 80 K. LOD load their samples at room temperature in a high-pressure metallic chamber. The molecular hydrogen is in contact with chamber metals and the gasket, for lengths of time that are not reported. In the period before being further pressurized and cooled, it is possible for the metals to dissolve into the hydrogen. Years ago, Hiroyasu Shimizu et al. studied Brillouin scattering in hydrogen.[58] Shimizu found that his room-temperature Brillouin scattering signal varied in time; a later study attributed this to the gasket slowly dissolving into the hydrogen so the sample was contaminated.[59] Varying impurity densities might explain why LOD observe hydrogen to blacken at 300, 310, and 320 GPa. It is significant that LOD report the observation of impurity modes in their infrared spectra.[60]

The presence of metallic impurities in hydrogen can impact the phase diagram of hydrogen so that the 360 GPa phase transition to H2PRE may not exist in the contaminated samples. Impurities can also cause a shift in the pressure-dependent frequencies of the vibron modes, explaining why the LOD pressure scale deviates from others. These impurities can cause hydrogen to blacken at pressures that others have not observed. Darkening due to impurities could also explain why their sample with the so-called plasma frequency in the infrared is not transparent in the visible region. We conclude that LOD may have made proper measurements, not on hydrogen, but on a sample of hydrogen heavily doped with metallic particles. The metallic molecular phase may exist, but currently there is no evidence of this. The only observation of atomic metallic hydrogen in a pure sample is the one reported by Harvard at 495 GPa.[61]

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