TECHNICAL RESPONSE

HIGH-PRESSURE PHYSICS

Response to Comment on "Observation of the Wigner-Huntington transition to metallic hydrogen"

Isaac F. Silvera* **and Ranga Dias**

Liu *et al*. present negative comments on our observation of the Wigner-Huntington transition to metallic hydrogen (MH). Earlier attempts to produce MH were unsuccessful due to diamond failure before the required pressures were achieved. We produced the highest static pressures (495 gigapascals) ever on hydrogen at low temperatures. Here, we respond to their objections.

Liu *et al.* (*1*) start their Comment by revealing their failed attempts to achieve high pressures in diamond anvil cells (DACs) using the same procedure, stating that they have a 96% failure rate in attempting to reach pressures exceeding 350 GPa. In our experiments, we have eliminated all procedures known to lead to diamond failure, as described in our paper (*2*), enabling us to achieve higher pressures. Liu *et al.* violate some of our procedures for attaining higher pressure. All of their studies use laser Raman scattering; laser illumination is known to weaken or cause failure of stressed diamonds (*3*). They use room-temperature gas loading of hydrogen, allowing hydrogen diffusion and embrittlement of the diamond anvils. In our procedure, the diamonds and gasket are covered with an alumina layer that acts as a diffusion barrier, and the temperature in which hydrogen is in contact with diamond anvils never exceeds ~80 K. In our past three experimental runs on hydrogen and its isotopes, we have achieved pressures of 340, 420, and 495 GPa (*2*, *4*, *5*). Higher pressures using beveled diamond anvils like ours (560 GPa) had been achieved 25 years ago (*6*), but not on hydrogen.

Liu *et al.* criticize our method of determining the pressure. We plot measured pressure versus load to show that our load curve was linear in our region of study. Our DACs are equipped with calibrated strain gauges to measure the load. In Fig. 1, we show our load curves for a few runs using the same DAC [similar to the design in (*7*), but improved and made of Vascomax]. Pressure was measured for all the indicated data points by probing the sample or the diamond, and we observe a broad range of linear behavior. For the run on MH, an extrapolation is made to the highest pressure point that was determined by the shift of the Raman active phonon in the stressed region of the diamond. Liu *et al.* give three distinct regions where pressure-versus-load curves develop their shape; they omit an important case. Load curves depend on the type of DAC that is used and are unique to each DAC. We have monitored our DACs for years and find shapes similar to those exhibited in Fig. 1, which is quite different for their DAC load curves shown in figure 1 in (*1*). They also state that the nonlinear part of the curve is due to cupping of the diamond. In our case, the cupping is minimized. The gasket is very thin (~1 μm) at high pressure. The deviation of the culet from flatness across the culet flat cannot be more than ½ μm and is probably much less. For thicker gaskets, it has been shown by analysis that at high pressure the hydrogen becomes very incompressible and the gasket hole expands with increasing load, so that the sample volume does not decrease (*8*). Their load curves may suffer from this. They suggest that the sample may have diffused out of the gasket. In this case, the gasket hole would close up. It is clear from Fig. 2 and figure S6 in (*2*) that this is not the case: The sample of hydrogen and the gasket edges remain well defined at the highest pressures. In Fig. 2, we show the measured reflectance of the gasket and sample of MH across a diameter of the culet. Our diamond Raman phonon is no different than spectra observed by other researchers (*9*), including spectra from the Gregoryanz group. We do not use "four additional meaningless points of 'visual observation'" to determine pressure, as they state; we use the determined load curve, which has an end point determined by Raman scattering.

Liu *et al.* make unsupported statements such as "The IR absorption data are consistent with the loss of the sample and contradict the authors' own previous claims" in (*5*). Clearly, Liu *et al.* are not familiar with infrared (IR) spectroscopy, as they plot transmission rather than absorption. Liu *et al.* interpret the broadening and weakening of the line strength by observing the transmission increase as loss of sample; they should compare the integrated intensity of the absorption coefficient (proportional to the line strength) as a function of pressure. In both our data and that of Zha *et al.* (*10*), the vibron line is observed to weaken and broaden with increasing pressure, shown here in Fig. 3 for our data. Thus, there is no basis to suggest that we lost

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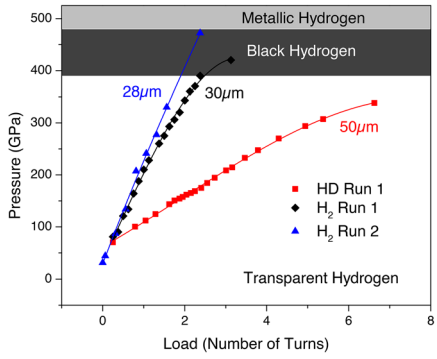

Fig. 1. The load versus pressure for three different runs with our DAC. The data points are measured pressures. The curves show plateauing at the highest loads, but the curve in which MH was made has not yet plateaued, indicating that higher pressures were accessible. The culet diameters are indicated for each curve.

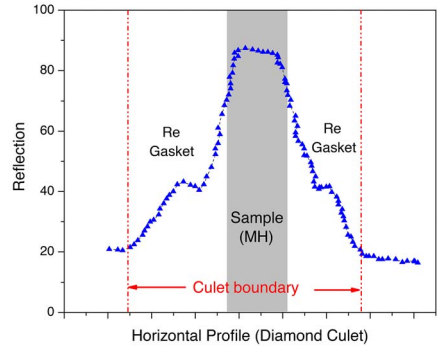

Fig. 2. Reflectance of the gasket and MH across a culet diameter.

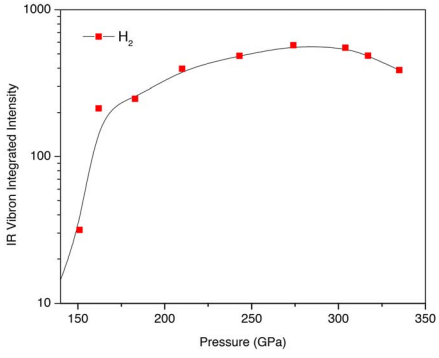

Fig. 3. The integrated absorption of the vibron line in phase III as a function of pressure. The scale is logarithmic. Note the weakening of the line at the highest pressures.

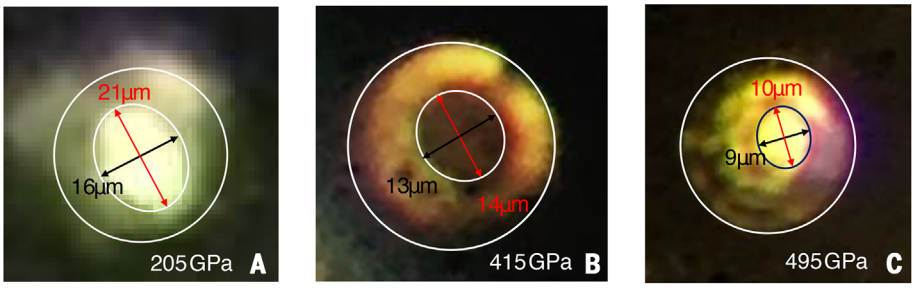

Fig. 4. iPhone photos and the sample dimensions. In (**B**) and (**C**), we can use the known dimension of the diamond culet to set the scale. The culet is observable and has no important change in dimension with increasing pressure. The culet is not clearly observable in (A) because the sample was back illuminated. In (**A**), we estimate the dimensions from knowledge of the starting dimensions of the gasket hole; this has a larger uncertainty of a few μm.

sample. The results in our MH paper and the other paper (*5*) where we observed the H_2-PRE phase are the same where there is overlap. In the other paper, as the sample turned black, we performed IR measurements. Because we use a weak thermal source for IR radiation, in order to achieve sufficient signal-to-noise it was necessary to integrate and average the signal for up to 40 min per spectrum. Weaker signals at higher pressures become prohibitive in measurement time, with little gain in signal-to-noise ratio. In the MH experiment, we did not repeat these measurements when hydrogen became black. They are concerned that the sample was black in an earlier experiment and transparent for the same pressure in the MH experiment. In (*5*), we used the Akahama-Kawamura 2010 pressure scale, whereas in the MH paper we use their 2006 linear scale. Using the same scale, there is no inconsistency.

Liu *et al.* incorrectly analyze the photos that we took using an iPhone camera to imply loss of sample. What they do not realize is that the iPhone magnification was not the same for each photo, but they tacitly make this assumption and find specious results. We show the correct analysis in Fig. 4. They also refer to photos in (*11*), figure 3 in (*1*), and figure 3D in (*12*); we find no photos at all in (*11*), and figure 3D in (*12*) does not seem to be relevant to the gasket flow scenario. In the case of gasket flow with a collapsing hole, the gasket edges and hydrogen sample will not remain well defined, but the gasket-MH interface is distinct, clearly shown in Fig. 2.

Liu *et al.* seem to assail our observation of H_2-PRE that we presented in (*5*). They do not realize that, after entering this new phase, the sample is still transparent and darkens or becomes black as the pressure is increased. We hope that our response has clarified the conditions and procedures under which MH was experimentally produced in our laboratory.

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