Hysteresis loops in measurements of the magnetic moment of hydrides under high pressure: implications for superconductivity

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Measurements of magnetic moments of hydride materials under high pressure have been claimed to prove the existence of superconductivity in these materials [1–6]. However, detection of the signal from the small sample requires subtraction of a large background contribution whose details are largely unknown. Here we analyze reported measurements and point out that the resulting hysteresis loops are incompatible with the conclusion that they result from superconductivity, independent of what assumptions are made about the background signal. We argue that this also invalidates the conclusion that the magnetic moment measured after the external magnetic field is turned off is evidence for trapped magnetic flux resulting from superconducting currents, as proposed in Ref. [6]. Our results imply that to date no magnetic evidence for the existence of high temperature superconductivity in hydrides under pressure exists, despite multiple claims to the contrary.

I. INTRODUCTION

In 2014 [7] and 2015 [1], A.P. Drozdov, Mikhail Eremets and coworkers reported the discovery of high temperature superconductivity in sulfur hydride under high pressure, confirming theoretical predictions [8, 9] and expectations [10] based on conventional BCS-electron-phonon theory [11]. This launched the development of a new highly active area of research. By now, more than two dozen hydrogen rich materials have been claimed to be high temperature superconductors based on experimental evidence [12], and hundreds such materials have been claimed to be high temperature superconductors based on theoretical evidence [13].

The experimental claims are mostly based on resistance measurements. However, as acknowledged by the creator of the field himself [14], *"To guarantee that superconductivity occurs, the most crucial signature that must be present is the expulsion of the magnetic field below T_c (the Meissner effect). Without these measurements, Science and Nature rejected our paper even if we had already substantial shreds of evidence of superconductivity"*.

Nature did accept the 2015 paper [1] even though it presented zero evidence for *"expulsion of the magnetic field below T_c"*. In fact, not a shred of evidence for expulsion of magnetic fields in these materials exists even today [15]. But the 2015 paper did present results of magnetic moment measurements using a SQUID magnetometer that appeared to show the development of a diamagnetic moment upon application of a magnetic field at low temperatures, and that was deemed sufficient [16] for publication in Nature at that time [1].

No new measurements of magnetic moments of these materials were reported for the ensuing 6 years. Then in 2021 and 2022, new measurements of magnetic moment of sulfur hydride again using a SQUID magnetometer were reported by V. S. Minkov, Mikhail Eremets and coworkers [2–6]. These measurements are the subject of this paper. No other magnetic moment measurements on these materials by other groups using SQUIDs have been reported to date.

II. MAGNETIC MEASUREMENTS IN 2022 VS IN 2015

It is to be expected that with the passage of time and improvements in the experimental equipment, sample preparation methods and experimental techniques, magnetic measurements should become more accurate and indicative of the physics of the samples under study. Indeed that seems to be the view of the authors of these papers [1–6], as illustrated in the following.

In Ref. [5] of 2022, the authors state that the superconducting transition detected through magnetic measurements in the 2015 work *"fully agrees'with the recent and more accurate measurements [2]"* of 2021. Furthermore, the authors in 2022 [5] state that *"Only recently, we succeeded in improving our measurements of the magnetic susceptibility significantly [2] (see Fig. 5c-h). For that we used a solid BH_3NH_3 as an alternative source of hydrogen instead of pure hydrogen. This allowed us to produce large samples with a diameter close to that of the culet of the diamond anvils, and to obtain a pronounced diamagnetic signal from superconducting phases under high pressures"*.

We start by comparing hysteresis loops without background subtraction measured in 2015 [5] and 2022 [3], shown in Fig. 1 at temperature T=100K and similar pressures (140 GPa and 155 GPa respectively). It is surprising that the background was paramagnetic in 2015 and is diamagnetic in 2022, as indicated by the sign of the slopes of the curves in Fig. 1, given that for both experiments the cell used was reported to be a non-magnetic cell made of Cu-Ti alloy [1, 3] *"in order to minimize the magnetic signal over a wide temperature range [3]"*. It is furthermore surprising that the background signal is four times *larger* in 2022 compared to 2015, given that it would be desirable to design the experiment so as to minimize the magnitude of the background signal that can mask the behavior of the sample under study.

Fig. 2 shows the hysteresis loop obtained from the 2015 measurements after subtraction of a background signal measured at 210K [5]. The behavior shown resembles

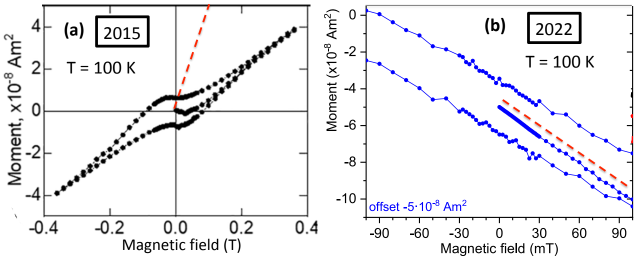

FIG. 1: Comparison of magnetic moment versus magnetic field at 100K measured in 2015 and 2022, without background subtraction, as reported in Refs. [5] and [3] respectively. The background is paramagnetic in 2015 and diamagnetic in 2022. The slopes of the curves are approximately 4 times larger in 2022 compared to 2015, as indicated by the dashed red lines that we added that have the same slope in both panels.

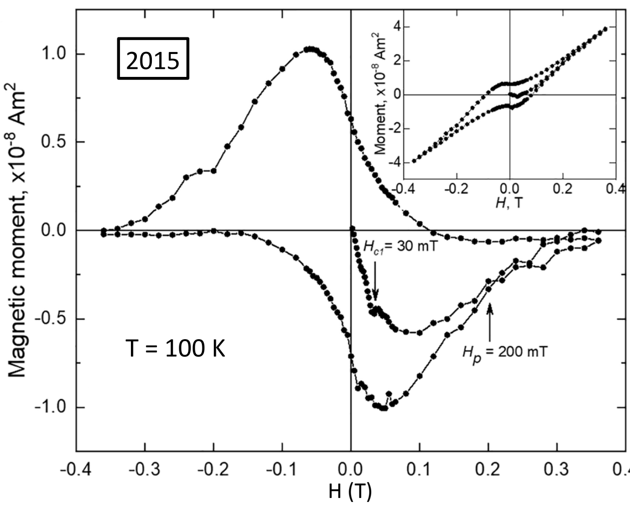

FIG. 2: Magnetic moment of sulfur hydride versus magnetic field measured in 2015, reported in 2022 [5]. The figure caption reads *"M(H) data measured at 100 K showing the initial virgin curve and hysteretic loop on alternating the magnetic field. Inset shows the original hysteretic loop before subtraction of the background signal originating from the DAC at 210 K"*.

what is seen in type II superconductors.

Fig. 3 shows the hysteresis loop measured in 2022 [3] for field between -1T and 1T at 100K. We would like to analyze its behavior by looking at the numerical values of the data. Unfortunately, the authors have declined to make those data available despite repeated requests and despite the paper's stated Data Availability statement [15, 17]. We discuss our analysis based on the published figures in the next sections.

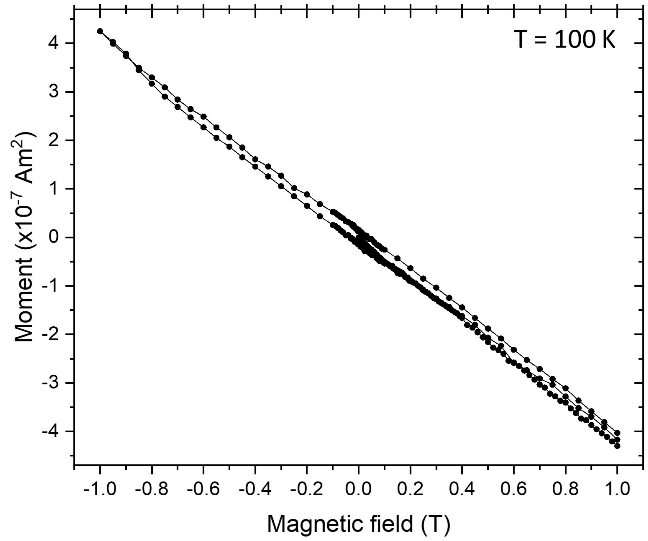

FIG. 3: Hysteresis loop for the magnetic moment of sulfur hydride versus magnetic field measured in 2022, reported in Fig. S10 of Ref. [3], for magnetic fields between -1T and 1T.

III. ANALYSIS OF EXTRACTION OF LOWER CRITICAL FIELD

Before analyzing the hysteresis loops we would like to discuss the extraction of lower critical field. In Ref. [5] of 2022, the authors state, referring to the 2015 work Ref. [1] *"A lower critical magnetic field H_{c1} of $\sim$ 30 mT was only roughly estimated as the point associated to the bending of the M(H) hysteretic loop"*, and that instead *"the correct value of the inflection point in the M(H) virgin curves was determined in the recent work and its value is higher $\sim$ 96 mT at 0 K for H_3S"*, with "recent work" referring to Ref. [3].

Fig. 4 shows on the left panel the magnetic moment versus field virgin curves reported in Fig. 3a of Ref. [3]. From the point where the curves start to deviate from linearity, the value of the lower critical field was extracted, which was then plotted in Fig. 3c of Ref. [3] versus temperature. That plot is shown on the inset of the right panel of Fig. 4.

Focusing on temperature T=100K, the light blue curve on the left panel of Fig. 4 starts to deviate from linearity around H=67T, and the corresponding point for the critical field versus temperature in the inset of Fig. 4 (from Fig. 3c of Ref. [3]) is indicated by an arrow. The curve in the inset reaches its maximum value 96mT at zero temperature.

However, we have recently pointed out that the provenance of the data shown in Fig. 3a of Ref. [3] is unknown [15], and that in fact the measured data shown in Fig. 3e of Ref. [3] (Fig. 1 right panel here) cannot give rise to the 100K curve shown on the left panel of Fig. 4 [18]. On the right panel of Fig. 4 we show what we obtained from digitizing the measured data shown on the right panel of Fig. 1 and subtracting a linear background, obtained by

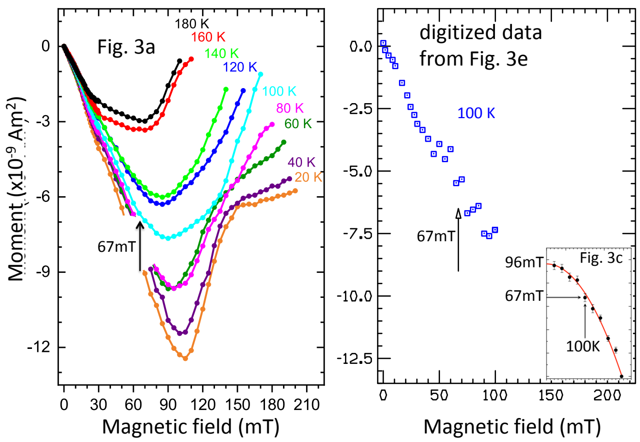

FIG. 4: Left panel: magnetic moment versus field reported in Fig. 3a of Ref. [3], reportedly obtained by subtraction of a linear background from the measured data reported in Fig. 3e of Ref. [3]. Right panel: results obtained by us by subtraction of a linear background from the digitized measured data reported in Fig. 3e of Ref. [3]. The linear background is a straight line connecting the points for H=0T and H=1T in Fig. 3. The inset on the right panel is Fig. 3c of Ref. [3] showing the behavior of critical field versus temperature extracted from the left panel, with critical field 96 mT at zero temperature and 67 mT at temperature 100K.

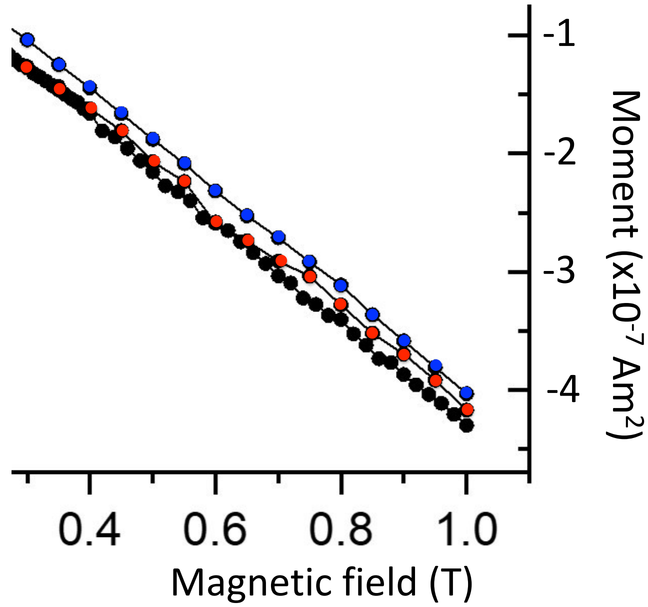

FIG. 5: Illustration of extraction of the signals from the hysteresis loop of Fig. 3. The blue points and red points are for the upper and lower branches of the hysteresis loop respectively. The spacing between the colored points is 0.05T. The black points are the virgin curve, with smaller spacing.

connecting the H=0 and H=1T values of magnetic moment shown in Fig. 3, which is the prescription given in Ref. [4] to subtract the background.

It can be seen in Fig. 4 that the points on the right panel and the light blue curve on the left panel look rather different. In particular, it is impossible to decide from the data on the right panel that a deviation from linearity sets in at H=67T, while it is plausible to do so from the data on the left panel. We don't know the detailed behavior of the measured data immediately above 100mT (the curves shown in Fig. 3 have very low resolution), but it would appear from the right panel of Fig. 4 that the data derived from the measured data would also be consistent with a lower critical field of 100mT or larger, which would be larger than the zero temperature value shown in the inset.

It is also apparent from Fig. 4 that the data on the right panel, that we extracted from the measured data shown in Fig. 3e of Ref. [3], have a lot more scatter than the data reported in Fig. 3a of Ref. [3] shown on the left panel. This indicates that, contrary to a recent suggestion [19], it is unlikely that the data shown in Fig. 3a of Ref. [3] originated from a different measurement run of similar nature to the measurements performed to obtain the reported measured data in Fig. 3e of Ref. [3].

IV. ANALYSIS OF 2022 HYSTERESIS LOOP

We extracted approximate numerical data for the 100K hysteresis loop from digitization of the images shown in Fig. 3 and in Fig. 1 right panel, which are reproductions of Figs. 3e and S10 of Ref. [3]. Fig. 5 shows for illustration the region for field $H \geq 0.3T$. Our task was facilitated by the observation that both for the upper and lower branches of the hysteresis loop it can be seen that the points are uniformly spaced, with spacing 0.05T, in the regions $0.1T \leq |H| \leq 1T$. This allows us to identify which are the virgin curve data in that range, with the exception of the range $0.1T < H < 0.3T$ where the points of the lower hysteresis branch overlap with the denser virgin curve points so that the distinction between the two is ambiguous. The identification of the upper hysteresis branch is unambiguous over the entire field range. For field in the range $0 \leq |H| \leq 0.1T$ we obtained the numerical values from digitization of Fig. 1 right panel.

Figure 6 shows our results. It can be seen that our digitized figures closely resemble the figures reproduced from Ref. [3], i.e. Fig. 1 right panel and Fig. 3. We have also added to both panels in Fig. 6 a red straight line connecting the H=0 and H=1T points of the virgin curve. According to Refs. [3, 4], this red line was assumed to be the background signal, and it was reportedly subtracted from the data to obtain the magnetic moment of the sample shown in Fig. 3a of Ref. [3], from which estimates for the values of the lower critical magnetic field and London penetration depth were derived [3], as discussed in the previous section.

Performing the subtraction of the red line from the digitized data shown in Fig. 6, we obtain the hysteresis loop and virgin curve shown in Fig. 7. Note that the

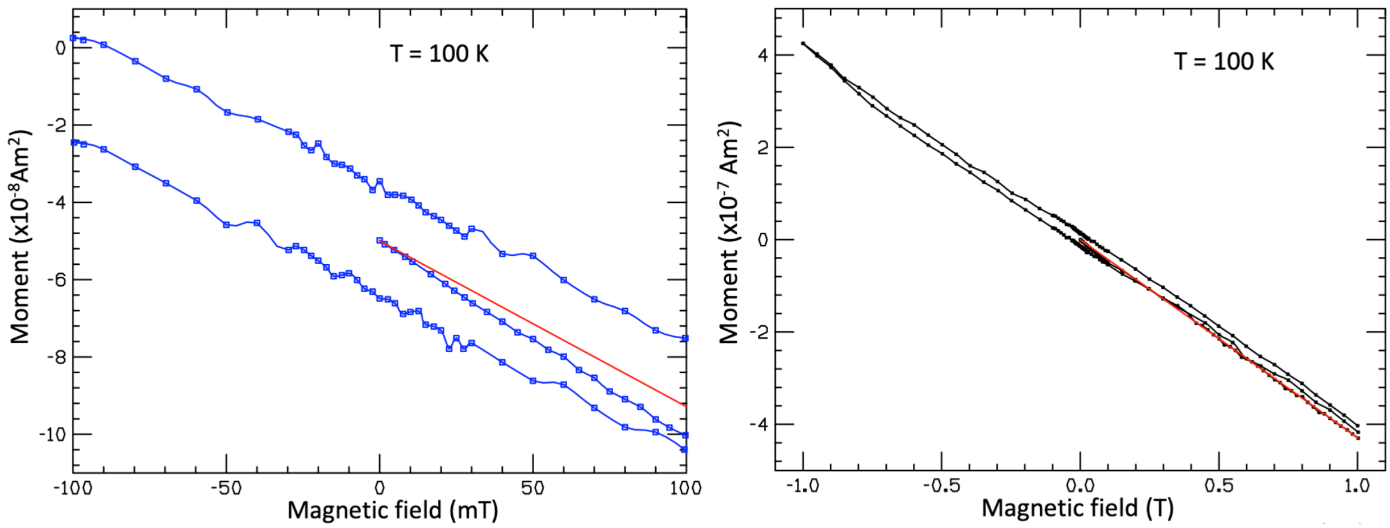

FIG. 6: Digitization of data for T=100K shown in Figs. 3e (left panel) and Fig. S10 (right panel) of Ref. [3]. The red lines connect the points for H=0 and H=1T, with slope $-4.30 \times 10^{-7} Am^2/T$.

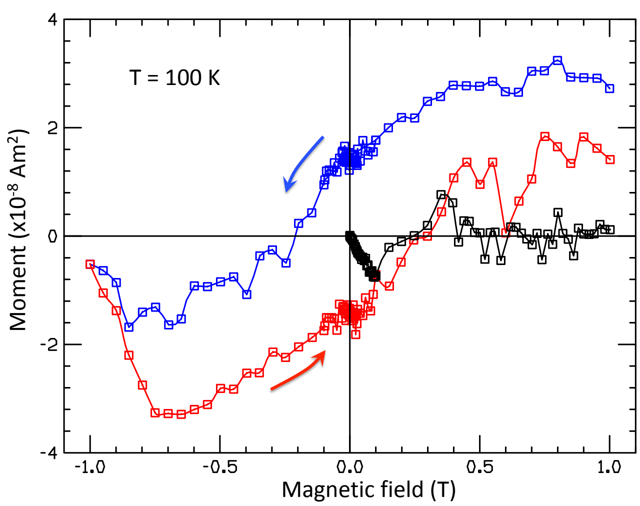

FIG. 7: Hysteresis loop resulting from subtraction of the linear background (red line in Fig. 4) from the digitized data in Fig. 4. The virgin curve are the black points.

three curves don't join at the highest field value 1T. This is surprising because the figure caption of Fig. S10 of Ref. [3] states that the field range (-1T, 1T) is "the full range of hysteresis". Perhaps this is a misprint, and the range of fields extended to larger positive values.

It is clear that the curves shown in Fig. 7 cannot reflect the behavior of the magnetic moment of a superconducting sample. To begin with, the virgin curve crosses zero for applied field around 0.3 T. For a superconducting sample with upper critical field approximately 90 T, as estimated for this material, the magnetic moment should remain negative for fields much larger than 0.3T. Furthermore, the red curve, denoting the magnetic moment in the return loop after having reduced the field to -1T, crosses the virgin data curve (black points) for magnetic field larger than 0.4T. This is impossible for a superconducting sample, the return hysteresis branch should always lie below or coincide with the virgin curve, contrary to what is seen in Fig. 7.

It may be thought that this anomalous behavior resulted from subtraction of an incorrect background. However, we point out that *any* assumed background that is subtracted from the data in Fig. 6, assuming the background is non-hysteretic, would necessarily yield the anomalous behavior seen in Fig. 7, i.e. that the data for the virgin curve (black points) are below the data from the return loop (red points), hence would be incompatible with superconductivity.

To illustrate this point, we show in Fig. 8 the result obtained by assuming as background signal the average of the forward and reverse M(H) field sweeps. This was the procedure reportedly used to obtain hysteresis loops shown in Fig. 4a of Ref. [2], which for some reason is no longer contained in the published version of the paper [3]. It can be seen in Fig. 8 that the virgin data obtained by subtraction of this assumed background are again incompatible with the assumption that they reflect the behavior of a superconducting sample since they fall below the red curve for fields above 0.4T.

Fig. 9 shows as the green curve and points the hysteresis loop reported in Fig. 4 of Ref. [2], obtained by subtracting from the measured loop the average of the forward and reverse M(H) sweeps as explained in Ref. [2]. It can be seen that it looks similar to the loop that we obtained in Fig. 8 by digitization of the data and following the same averaging and subtraction procedure. We also show in Fig. 9 the magnetic moment of the sample plotted in Fig. 3a of Refs. [2, 3] (Fig. 4 left panel here). As we already pointed out in Ref. [15], the fact that the moment from Fig. 3a does not join the hys-

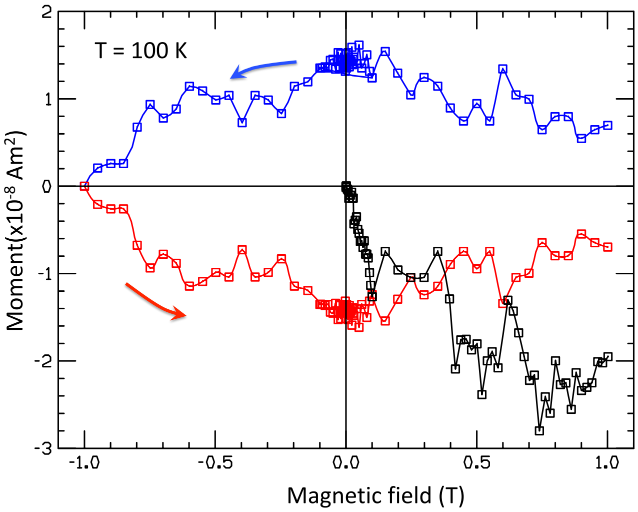

FIG. 8: Hysteresis loop resulting from subtraction of a background constructed from the average of the forward and reverse M(H) field sweeps as described in Ref. [2] from the digitized data in Fig. 4. The virgin curve are the black points.

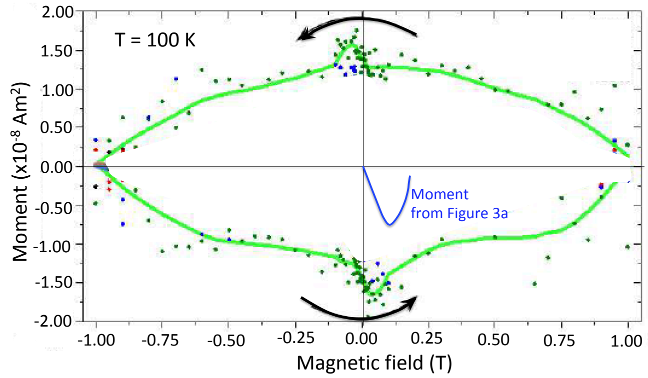

FIG. 9: Green curve and points show the hysteresis loop reported in Fig. 4 of Ref. [2] obtained according to Ref. [2] by using as background the average of the forward and reverse M(H) field sweeps. The blue curve shows the moment plotted in Fig. 3a of Refs. [2, 3] for T=100K. This figure is reproduced from Ref. [15].

teresis loop in Fig. 9 indicates that there is something very anomalous about these data. Now we understand the origin of this anomaly: the green loop in Fig. 9 was obtained from the measured data by assuming a certain background, namely the average of the forward and reverse M(H) sweeps [2], and the moment from Fig. 3a of Refs. [2, 3] was obtained from the measured virgin data by assuming a completely different background, namely the straight red line in Fig. 6. Ref. [2] contained both the hysteresis curve Fig. 6 and the magnetic moment curves shown in the left panel of Fig. 4, but did not explain how the magnetic moment curves were obtained from the measured data: that was only revealed in the Author Correction Ref. [4]. Of course it makes no sense to subtract different backgrounds from measured data in the same range of fields for different branches of the loop.

V. DISCUSSION

For reasons that have not been explained, certainly not superconductivity, the miniature high-pressure cell used in the magnetic measurements of 2022 [3, 6] shows a significant diamagnetic response [4]. The authors state that they were able *"to obtain a pronounced diamagnetic signal from superconducting phases under high pressures"* [3]. However the diamagnetic signal measured is the superposition of the signal attributed to the superconducting sample and a *much larger* diamagnetic signal from the background: the largest diamagnetic signal attributed to the sample is more than six times smaller than the background diamagnetic signal at the same value of magnetic field. To establish that the signal of the sample is diamagnetic requires to have confidence that the background signal has been properly identified: if the diamagnetic signal attributed to the background is underestimated by 20%, the resulting signal for the sample obtained by subtraction would be paramagnetic rather than diamagnetic. For that reason, there has to be complete transparency on what transformations were made to eliminate the background contribution from the signal. In our view that has not been accomplished, not by a long shot.

In this paper we have found that the hysteresis loop and virgin curve for the sample resulting from the measurements of magnetic moment for sulfur hydride reported in Refs. [2, 3] for temperature T=100K, under any assumption about the background signal, are incompatible with the conclusion of Ref. [3] that the measurements indicate that the sample is superconducting. It is difficult to understand how it is possible that in 2015 the hysteresis loop shown in Fig. 2 was measured, which appears to be compatible with superconductivity, and 7 years later the new results obtained [3] are incompatible with superconductivity. We have also found here and in Ref. [18] that the data plotted in Fig. 3a of Ref. [3] for 100K did not result from subtracting a linear background from the measured data, as Refs. [3, 4] states. In addition, in Ref. [17] we discussed anomalies in the reported data for 160K.

It would be of great interest to determine whether these problems occur for other temperatures for which results for magnetic moment of the sample were reported in Ref. [3]. In particular, no information on the measured hysteresis loops from which the published results were derived is given in Ref. [3] for temperatures T = 20K, 40K, 60K, 80K. We conjecture that the same or even more serious anomalies may be apparent in those undisclosed data at those lower temperatures. None of the data underlying the measurements reported in Ref. [3] have been released by the authors upon request, contrary to the Data Availability statement in the paper.

The authors of Refs. [3, 5, 6] have consistently assumed that the observed hysteresis is only due to the sample, and that it is in fact proof that the sample is superconducting. Experimental support for that assumption would be provided by performing the experiments under the same conditions of temperature and pressure with the non-superconducting precursor sample before the laser heating treatment that supposedly triggers the chemical reaction that forms the superconducting compound, and finding that no hysteresis occurs. Such an obvious control experiment has not been reported. Obvious questions arise: (i) If such experiments have not been performed, why not? (ii) If they have been performed, why haven't the results been reported?

Under the assumptions that the measured magnetic moments reported in Ref. [3] are the sum of the magnetic moment of the sulfur hydride sample under study and a non-hysteretic background signal, we argue that our results discussed in this paper unequivocally rule out that the sample is superconducting. If we relax the assumption that the background is non-hysteretic, the measured data would not rule out the possibility that the part of the signal from the sample originates in superconductivity, if it is assumed that in the M(H) return field sweep with the field increasing after the field reached large negative values the background is in a different state (less diamagnetic) than it was during the virgin curve measurements. But of course the experiment does not provide evidence in favor of such interpretation, the only justification that could be argued for that interpretation is that it would be in accordance with theoretical expectations based on BCS theory [11].

If the possibility that the background is hysteretic is allowed, it is of course no longer possible to argue that the observed hysteresis is proof of superconductivity. It is then also not possible to conclude that the sample is superconducting without additional information.

Concerning the reported measurement of trapped magnetic flux interpreted as arising from superconducting currents in Ref. [6], we argue that the results of this paper imply that the measurements of Ref. [6] provide no information on the superconductivity or non-superconductivity of the samples, contrary to what is argued in Ref. [6]. If the background is non-hysteretic, we have shown that the measured hysteresis loop cannot result from a superconducting sample. The sample could show hysteresis and trapped moments for other reasons unrelated to superconductivity [20]. And if the background is hysteretic, there is no way to know whether the trapped moments measured in Ref. [6] are due to the sample or the background.

It should also be noted that the experiments reporting trapped flux in Ref. [6] could easily have been done also with the non-superconducting sample before heat treatment, to verify that no trapped flux results in that case, consistent with the interpretation of Ref. [6] that the measured trapped flux is due to superconductivity. The same obvious questions arise. If such control experiments have not been performed, why not? If they have been performed, why haven't the results been reported?

In summary, the results reported in this paper indicate that the experimental results reported in Refs. [3] and [6] are not due to superconductivity. For the results reported in Ref. [3] and its conclusions, namely that they are evidence for superconductivity in sulfur hydride and lanthanum hydride under pressure, to have any claim to scientific validity, the measured data for hysteresis loops for the 15 temperature values for which magnetic moment data were reported in Fig. 3 a and b of Ref. [3] need to be released, so readers can analyze them and reach their own conclusions.

In conclusion we argue that our analysis here and in other papers on magnetic evidence for superconductivity in hydrides under pressure [15, 17, 20–22] shows that to date there is no such evidence, 9 years after the onset of research in this class of materials.

Acknowledgments

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