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ORIGINAL PAPER

On Magnetic Field Screening and Trapping in Hydrogen-Rich High-Temperature Superconductors: Unpulling the Wool Over Readers' Eyes

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Abstract

In Minkov et al. (Nat. Commun. 13:3194, 2022), Minkov et al. reported magnetization measurements on hydrides under pressure that claimed to find a diamagnetic signal below a critical temperature demonstrating the existence of superconductivity. Here, we present an analysis of raw data recently released (Minkov et al. Nat. Commun. 14:5322, 2023) by the authors of Minkov et al. (Nat. Commun. 13:3194, 2022) that shows that the measured data do not support their claim that the samples exhibit a diamagnetic response indicative of superconductivity. We also point out that Minkov et al. (Nat. Commun. 13:3194, 2022) in its original form omitted essential information that resulted in presentation of a distorted picture of reality, and that important information on transformations performed on measured data remains undisclosed. Our analysis also calls into question the conclusions of Minkov et al.'s trapped flux experiments reported in Minkov et al. (Nat. Phys. 19:1293–1300, 2023) as supporting superconductivity in these materials. This work together with earlier work implies that there is no magnetic evidence for the existence of high temperature superconductivity in hydrides under pressure.

1 Introduction

While abundant evidence from resistance measurements has been put forth claiming that hydrides under high pressure are high temperature superconductors [4], magnetic evidence presented in favor of superconductivity in hydrides remains scarce [5]. Minkov et al. have recently claimed that magnetization measurements [1] and trapped flux measurements [3] provide clear evidence in favor of superconductivity in these materials. In this paper, we show that their claims are not supported by underlying raw data recently released [2]. Instead, we argue that they are based on the preconceived assumption that the materials are superconductors rooted in BCS-Eliashberg theory [6, 7], and the subsequent interpretation of ambiguous experimental measurements biased by such prejudice.

Figure 1 shows magnetization measurements reported by Minkov et al. in Ref. [1]. The caption of the figure read, when the paper was published on June 9, 2022 and for 450 days thereafter [2, 8]: *M(H) magnetization data for Im-3 m-H3S at high pressure. Virgin curves of the M(H) magnetization data for the Im-3 m-H3S phase at PS* = 155 ± 5 *GPa at selected temperatures. The curves were superimposed for a better representation; so the linear trend of M(H) dependences coincides for measurements at different temperatures."* The associated text in the paper read: *"**M(H) magnetization measurements**. Measurements of the magnetic field dependence of magnetization allow us to estimate the characteristic superconducting parameters* H_{c1}, λ_L, κ *and* j_c. *The value of* H_p, *at which the applied magnetic field starts to penetrate the sample, was determined from the onset of the deviation of M(H) from the linear dependence (see Fig. 3)".*

These statements informed the reader that the magnetization curves shown in Fig. 1 were measured in a laboratory. However, they did not reflect reality. Indeed if the sample showed such behavior, it would be clear evidence that it is diamagnetic, and the magnitude of the signal and the fact that there is a clear break for a critical magnetic field suggest that it is superconductivity. However, what is shown in Fig. 1 were not the measured data, and this fact was not disclosed to readers until September 1, 2023 when an "Author Correction" was published [2].

The correction [2] was prompted by emails from one of us (JEH) to the authors of Ref. [1] beginning in October 2022

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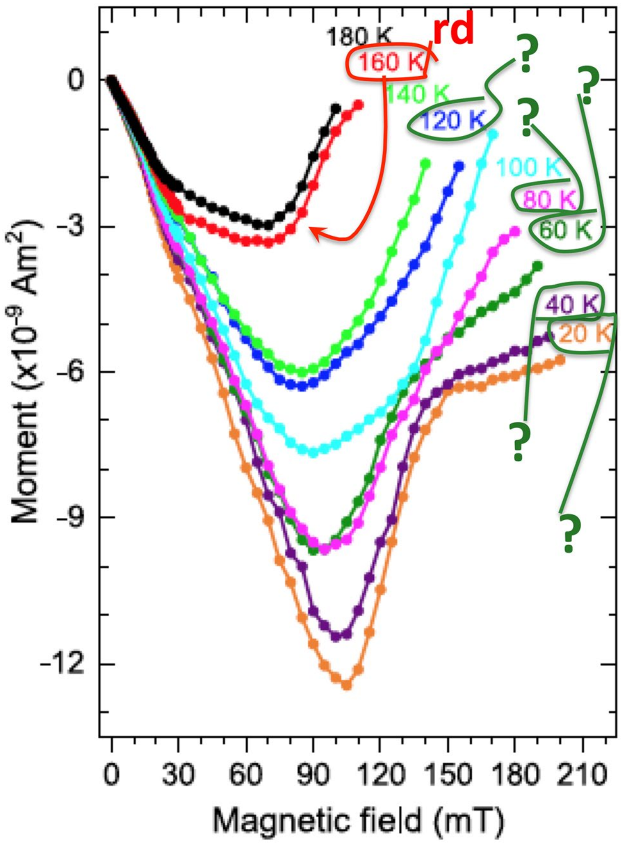

Fig. 1 Magnetization of H_3S under pressure reported in Ref. [1]. The question marks label curves for which no information exists on what their relation with measured data is. For the single curve labeled "rd," some information on underlying raw data has recently been disclosed [2]

[9], asking the authors to clarify an apparent inconsistency between Figures 3a and 3e of the original version of Ref. [1], i.e., Ref. [8]. The authors did not provide a response to these emails. This was followed by a Matters Arising manuscript by the present authors submitted to Nature Comm in November 2022 and declined by the journal in April 2023, that was subsequently published in another journal, Ref. [10]. In that paper, we argued that the data published in Ref. [8] were inconsistent with one another and with the expected behavior of superconductors.

The recently published correction [2] clarifies that the inconsistency between Figures 3a and 3e of Refs. [1, 8] pointed out in ref. [10] was only apparent, not real. The perception of inconsistency originated in the fact that the authors had failed to disclose to readers, and to one of us (JEH) in multiple private communications to all the authors where clarification was requested [9], that a variety of transformations had been performed in obtaining the curves shown in Fig. 1 (Fig. 3a of Refs. [1, 8]) from measured data (Fig. 3e of Refs. [1, 8]). In this paper, we analyze the significance of this disclosure to the interpretation of the experimental results of Ref. [1] and its implications for Ref. [3].

2 Raw Data and Transformations for T = 160K

All the figures in this section refer to the curve labeled "rd" in Fig. 1, for T = 160K. The Author Correction Ref. [2] explains that what was actually measured to infer the curve labeled "rd" in Fig. 1 was what is shown in Fig. 2: a diamagnetic signal, that was non-zero even for zero applied field, that showed some hysteresis when cycling the field between -1T and 1T, starting with the virgin data with no field. In fact, Fig. 2 already included a transformation that was not disclosed in the original publication: a vertical shift of the data, since the measured signal was not zero for zero field as Fig. 2 shows, rather it was negative. Figure 3 shows the actual measured data as reported in "Supplementary Figure S12b" of [1], that was added to the paper when the correction [2] was published.

In Fig. 4, we show what results from subtracting the red line in Fig. 3 from the measured data, which as explained by the authors [2] was the procedure used to obtain the data in Fig. 1. According to Minkov et al. [1], the results shown in Fig. 4 reflect the behavior of the H_3S sample under applied magnetic field after background subtraction, and the deviation of the virgin curve from the linear behavior indicated by the dashed red line at magnetic field approximately 30mT (seen more clearly in Fig. 1, curve labeled rd) indicates that the magnetic field starts to penetrate the sample at that field, hence is labeled H_p. According to the authors, this reflects the lower critical field for the superconducting sample, after correcting for a demagnetization factor estimated to be 8.5 in Ref. [1].

Note however that the largest diamagnetic moment magnitude in the virgin curve shown in Fig. 4, at field approximately 90 mT, is approximately $0.3 \times 10^{-8} Am^2$, which is more than 6 times smaller than the negative moment for zero field that was measured shown in Fig. 3, approximately $-2 \times 10^{-8} Am^2$, which clearly does not reflect the diamagnetic properties of a hydride superconductor sample. Note also that the total diamagnetic signal measured at that field was approximately $-6 \times 10^{-8} Am^2$, i.e., more than 20 times larger in magnitude than the diamagnetic moment attributed to the sample.

Also note that the magnetic moment shown in Fig. 4 turns positive for applied magnetic field larger than 0.2T. If indeed it reflects the properties of the H_3S sample, as interpreted by Minkov et al. [1], it means that the sample is paramagnetic for applied fields larger than 0.2T. This is not consistent with the fact that this is supposed to be a type II superconductor, with an upper critical field estimated to be 97T [1].

Next, we would like to know the behavior of the magnetic moment for applied field larger than 0.3T, up to 1T. Unfortunately it is impossible to discern it from the published data, left panel of Fig. 2, due to the extremely low resolution of the figure. Fortunately, we know one point: for magnetic

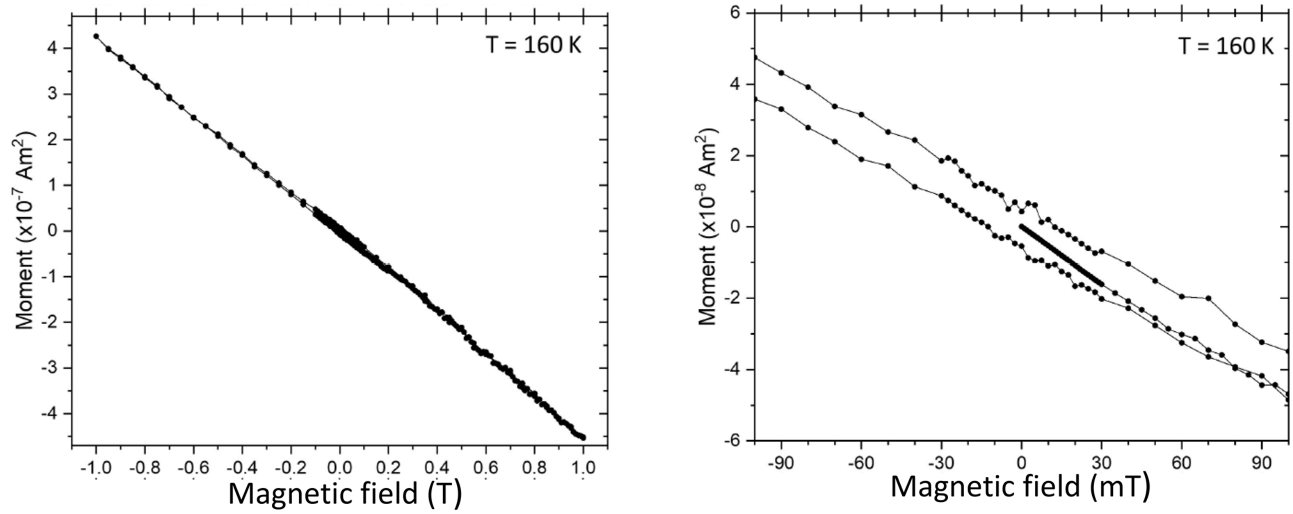

Fig. 2 From Supplementary Figure S10 of Ref. [1]. The figure caption read: *"M(H) magnetization data of the heated sample... The left panel shows the full range of hysteresis and the right panel shows the enlarged magnetic field range of −0.1 - 0.1 T"*

field 1T, the red line shown on the left panel of Fig. 3 goes through the measured data point at 1T. That is how the red line was constructed according to Ref. [2]. This implies that the moment extracted from subtraction of the red line, shown in Fig. 4, has to go to zero at magnetic field 1T. In Fig. 5, we show our educated guess of what the data may look like: we continued the curves smoothly, matching the first derivative at the largest field where we had information, and making it reach smoothly the blue known point of zero moment at H = 1T.

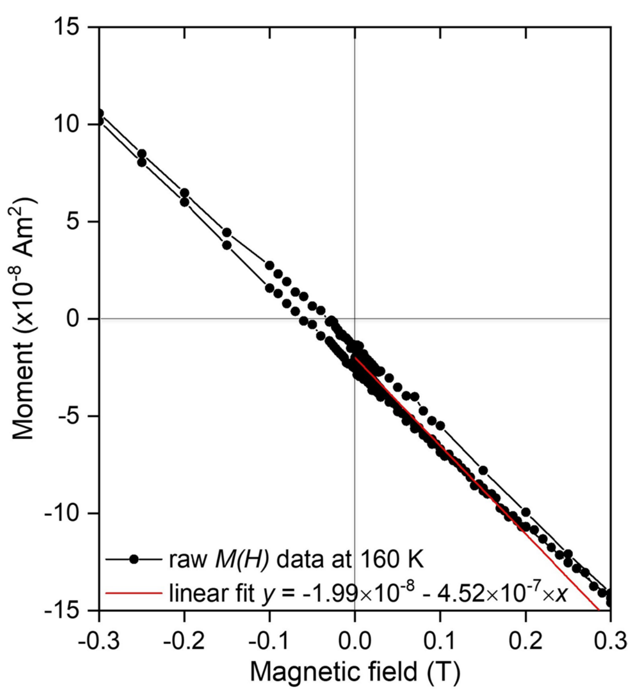

Fig. 3 Figure S12b of Ref. [1], that was added to the paper when the correction [2] was published. These are raw data in the range (-0.3T,0.3T), that differ from the ones shown in Fig. 2 by a uniform shift of the data: the magnetic moment is not zero for zero applied field, as shown by the thin horizontal and vertical lines added by us

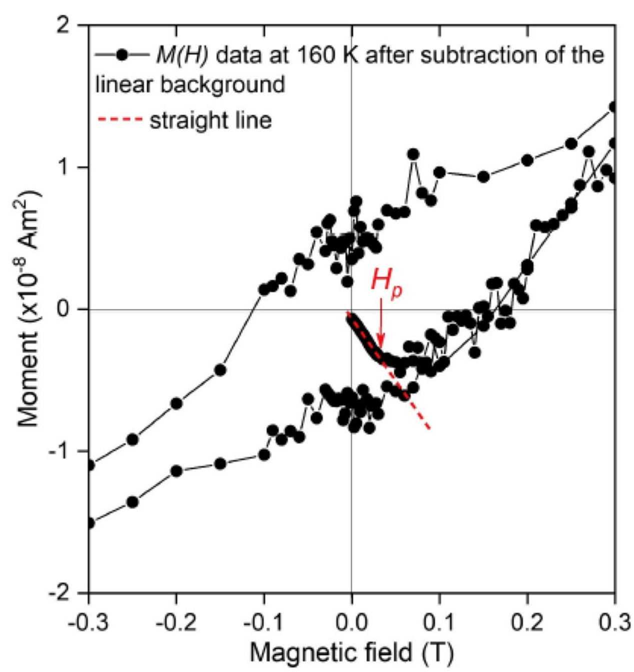

Fig. 4 Figure S12c of Ref. [1], added to the paper when the correction was published [2]. We have added the horizontal and vertical lines going through the origin. Note that the moment that resulted after subtraction of the red line in Fig. 2 is positive for applied magnetic field larger than 0.2 T. If this reflects the behavior of the sample, it implies the sample is paramagnetic for applied fields larger than 0.2T

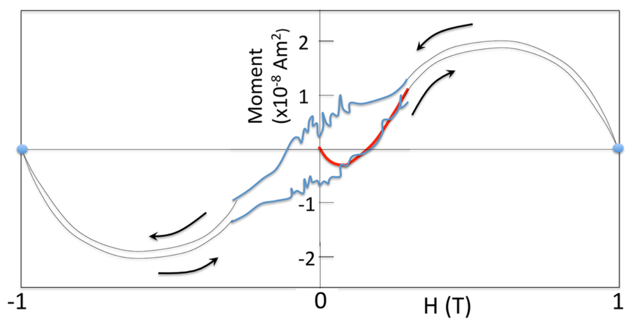

Fig. 5 Educated guess of what the moment inferred after subtraction of the straight red line from the measured data look like in the range of magnetic field up to 1T. Note that the curves go to zero at 1T

It would also be nice to know how the sample reacts to magnetic fields larger than 1T. Note, for example, that for the experiments on trapped flux reported by Minkov et al. in Ref. [3], magnetic fields up to 6T were used. In Fig. 6, we show an educated guess of what the magnetic moment could look like, where we have continued the curve beyond 1T with approximately the same slope. Note that it is likely that the magnetic moment would change sign again for fields larger than 1T, since it is unlikely that the curve would reach a minimum exactly at 1T, or discontinuously change the sign of its derivative at 1T. So if such behavior reflects the behavior of the sample, it has the remarkable property of being diamagnetic for fields smaller than 0.2T, paramagnetic for fields between 0.2T and 1T, and diamagnetic again for fields larger than 1T, which is still well below what is expected to be the upper critical field for this material.

If the behavior shown in Fig. 6 was what was measured, it allows us to make the following interesting suggestion. Suppose the authors had decided to draw the red line shown in Fig. 3 not according to the criterion they used, namely *"we have subtracted a linear background from the measured M(H) magnetization data. This linear background was determined as the straight line connecting two endpoints: the magnetic moment value at H = 0 T (the starting point of measurements) and the magnetic moment value at H= 1 T"*, but replacing $H = 1T$ by $H = 1.38T$. That is the value of H for the blue point in Fig. 6 where our hypothesized curve crosses the dotted line that goes through the origin. Note that the moment for small fields, indicated by the red curve, that is negative for field smaller than 0.2T according to the construction of the authors, lies right on top of the dotted line in Fig. 6. This means that if the authors had chosen to draw their red line through the $H = 1.38T$ data point rather than the one at 1T, upon subtraction of the background the inferred moment for the sample would have been always positive or zero, i.e., it would indicate that the sample is paramagnetic for all fields.

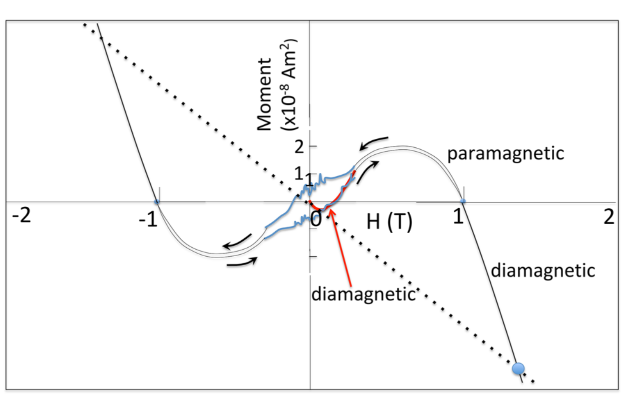

Fig. 6 Educated guess of what the magnetic moment may look like for applied field larger than 1T. We have also added the dotted line goes through the origin and through the value of the magnetic moment for $H = 1.38T$, and is on top of the moment curve (in red) for small fields. For its significance, see text

3 Measured Data for Other Temperatures

Figure 1, which is Fig. 3a of Ref. [1], shows curves for 9 different temperatures. Only for one temperature, T = 160K, were the raw data shown in the new Figure S12 published with the correction on September 1, 2023 [2]. For three other curves in Fig. 1, for temperatures 180K, 140K, and 100K, hysteresis loops were shown in Fig. S10 of [1]. However, it is hard to draw conclusions from them because they presumably contain undisclosed shifts of the origin, such as the one between Figs. 2 and 3 here, and in addition the red line used for subtraction similarly to the red line shown in Fig. 3 has not been disclosed for these temperatures. For the remaining 5 temperatures, labeled with question marks in Fig. 1, namely 120K, 80K, 60K, 40K, and 20K, neither the origin shifts nor the hysteresis loops measured nor the red line used in the subtraction process that was performed to arrive at the data shown in Fig. 1, have been disclosed.

Given this, the relation between the published curves shown in Fig. 1 and what was measured remains largely unknown. As a consequence, it is impossible to ascertain whether or not the curves shown in Fig. 1 contain any relevant information about the physical sample.

4 Other Transformations Performed on Measured Data

The subtraction of a linear function and the shift of the origin were not the only transformations that were performed in going from the measured data to the published data that originally had been reported as measured [8]. According to Ref. [2], *"the data were normalized to H = 15 mT data so that to have the same initial linear M(H) slope."* and *"we* *performed additional linear transformations so that the curves have the same initial linear M(H) slope. Importantly, these linear manipulations do not affect the onset of the deviation of the M(H) virgin curve from the linear dependence".*

Neither the paper nor the Author Correction give any information on the magnitude of these linear transformations that apparently changed the initial slope of the curves, nor do they give a reason for why doing such transformations is justified. If the diamagnetic signal measured is due to superconductivity, one would expect the initial slope to depend on sample size and geometry but be independent of temperature. Thus, under that assumption one could perhaps argue that changes in slope are not intrinsic and instead result from experimental artifacts and should be normalized away. Alternatively, without starting from the assumption that the samples are superconductors, one could infer from the fact that the slope of the signals changes with temperature the conclusion that the signals do not originate in superconductivity.

5 Unavailability of Underlying Data

The published paper Ref. [1] contains a data availability statement, that reads: *"The data that support the findings of this study are available from the corresponding authors upon reasonable request."* Clearly, the measured data before background subtraction for the 9 curves shown in Fig. 3a of Ref. [1] for H_3S, and for the 4 curves shown in Fig. 3b for LaH_{10}, are data that support the findings of that study. We now know that those data were processed by shifting the origin, subtracting a very large linear diamagnetic background, and performing additional normalizations, but do not know any of the details of these procedures.

Clearly, requesting the measured data to understand the relation between what was measured and what was published, and to what extent the transformations performed may affect the interpretation of the significance of the processed data to the understanding of the physical properties of the samples under study, is a reasonable request. Yet, the measured data are not available. We have requested these data repeatedly from the authors and from the journal starting on January 11, 2023, and not received any of them to date (September 5, 2023) [11, 12].

6 On the Nature of the "Author Correction"

The Author Correction [2] states that the original manuscript [8] contained errors, namely the omission of the information that the presented data had undergone various *"linear manipulations"*. If those errors had been inadvertent, these transformations and background subtraction should have been acknowledged immediately when the inconsistency in the published data was called to the attention of the authors in October 2022 [9], and a correction should have been published long ago. The fact that this did not happen suggests that the "errors" were not inadvertent but a deliberate attempt to hide information. This is supported by the fact that to this date the authors refuse to make their underlying data available for examination by readers [11, 12].

What could be the possible reasons for why this is done? One possibility is that the *"manipulations"* [2] of the data involved steps that are incompatible with generally accepted scientific norms, and this would be potentially revealed if the underlying data were available to readers.

7 Incompatibility of Published Raw Data with Published Processed Data

The caption of Fig. S12 of Ref. [1], that was added to the paper when the Author Correction [2] was published, reads: *"Subtraction of the linear background for the better illustration of the value of H_p, at which an applied magnetic field starts to penetrate into the sample. a, b Raw M(H) magnetization data measured at T = 160 K (black circles) and the linear background, which was determined as the straight line connecting two endpoints: the magnetic moment value at H = 0 T (the starting point of measurements) and the magnetic moment value at H = 1 T (the highest value of the applied magnetic field). c Corrected M(H) magnetization data after subtraction of the linear background (black circles). The value of H_p, at which an applied magnetic field starts to penetrate into the sample, was determined as the onset of the evident deviation of the M(H) data from the linear dependence (red dashed straight line)".*

This is supposed to explain how the data published in Fig. 3a of Ref. [1] (Fig. 1 here) for T = 160K were obtained. So we expected that the data points in Fig. S12c of the corrected Ref. [1] (Fig. 4 here), obtained after the background subtraction, would be the same as the data points in Fig. 3a. This is however not the case. Figure 7 shows the superposition of the two sets of data. The black dots show considerable more scatter than the red dots. Presumably, the black dots include both the data from the virgin curve and from the return curve in the hysteresis cycle, as seen in Fig. 4. However, there is clearly no correspondence between black and red points. Starting at field 30mT, there are 16 red points from Fig. 1 that lie on a smooth curve. There is no subset of black points that could correspond to those red points.

One could speculate that the black points and the red points originated in different runs. However there is much larger scatter in the black points than in the red points. In

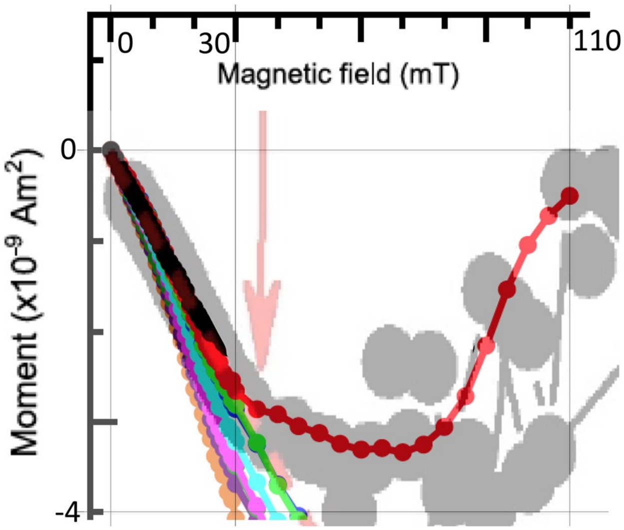

Fig. 7 Published data from Fig. 3a of Ref. [1] (Fig. 1 here) for 160 K (red dots), superposed to data from Fig. S12c of Ref. [1] (Fig. 4 here) (black dots). Thin vertical lines indicate field values 0mT, 30mT, and 110mT. Thin horizontal lines indicate moment $0 \times 10^{-9} Am^2$ and $-4 \times 10^{-9} Am^2$

addition, the purpose of Fig. S12 is to illustrate how the data in Fig. 3a of the paper were obtained, for which one would expect that a subset of the black points in Fig. 7 would match all the red points.

We conclude that the relation between measured data and the data published in Fig. 3a of Ref. [1] remains completely obscure even after the Author Correction, even for the single example addressed in the Correction, namely the inclusion of Fig. S12 to the manuscript.

It is also very peculiar that the authors chose to illustrate their data manipulation with the curve for 160K, instead of with one of the lower temperature curves in Fig. 1 where the features supposedly associated with superconductivity are much sharper, so that one would expect to see their signature in the raw data before background subtraction to be much more prominent than for the high temperature curve chosen.

8 Implications for Trapped Flux Experiments

In Ref. [3], results of experiments were reported where the same samples of the same materials discussed here were cooled to low temperature (10K), then a magnetic field of varying magnitude up to 6T was applied, then after 1 h it was removed, and then the remnant magnetic field was measured. It was argued that the remnant field was trapped flux, showing evidence that supercurrents circulate in these materials and that these materials are strong superconductors with very strong pinning centers, and the results were interpreted using the Bean model for hard superconductors. It was deduced that for H_3S the magnetic field H^* where the applied field reaches the center of the sample has magnitude $H^* \sim 0.8T$.

We do not know the raw magnetization data before background subtraction and other transformations that were measured for low temperature for Ref. [1], e.g., for the curve labeled 20K in Fig. 1. We can however guess that the inferred magnetization of the sample after background subtraction will look qualitatively similar to Figs. 4 and 5. However, one would expect for a superconductor with estimated upper critical field 97T [1] as H_3S is, that necessarily the diamagnetic response should persist for fields $H^* = 0.8T$ and much larger. Even for an ideal type II superconductor without pinning centers the diamagnetic response persists up to the upper critical field, and for superconductors with pinning centers the diamagnetic response should be even stronger since vortices that would weaken the diamagnetic response are prevented from penetrating due to pinning centers. Therefore the behavior shown in Figs. 4 and 5, and as pointed out in Ref. [10] even the behavior shown in Fig. 1 with the magnetization even at low temperatures being strongly reduced already for $H \sim 200\ mT$, is incompatible with superconducting behavior. Consequently, if the hysteresis loop shown in Fig. 4 is not reflecting the magnetization of a superconducting sample, there is no reason to believe that the magnetic moment that remains after the field is first applied and then removed, i.e., the value of the moment at the point where the upper curve of Fig. 4 crosses the *y*-axis, originates in superconducting currents creating trapped flux, as claimed in Ref. [3].

We have also pointed out [13] that the field dependence of the trapped flux reported by Minkov et al. under zero field cooling, which is linear for small fields [3], is incompatible with the conclusion that it originates in superconductivity, since it would be quadratic in that case, as seen for example in similar recent experiments on MgB_2 samples [14].

9 Discussion

From what we have now learned from Ref. [2], the experimental apparatus used to perform the measurements reported in Ref. [1] has a *"significant diamagnetic response"*, for reasons that have not been explained. The magnitude of that response is several times larger that the magnitude of the diamagnetic moments attributed to the sample in Ref. [1]. The field dependence inferred for the magnetic moment of the sample after background subtraction is totally incompatible with the behavior of a superconducting sample, as shown in Figs. 4, 5, and 6. All of this indicates that the observed diamagnetism is not due to superconductivity of the sample.

The authors of Ref. [1] seem to believe that the hysteresis shown in their magnetization cycles for the raw data is clear evidence for superconductivity. However, there could be a variety of reasons for this hysteresis due to the experimental apparatus that have nothing to do with superconductivity. One obvious check would be to perform these experiments with a sample before the laser treatment that is supposed to render it superconducting, and show that in that case no hysteresis is seen. The fact that the authors have not reported this simple check invalidates their claim that their presented results are evidence for superconductivity in their samples.

We have also argued in this paper that their background subtraction procedure that was used is arbitrary, and showed an example where choosing a different equally plausible straight line to subtract from hypothesized measured raw data would render a signal that is always paramagnetic. We have been unable to judge the validity of the authors' other additional normalization procedures performed because they have not disclosed the details of them nor have they presented arguments for why such procedures would be justified.

Finally, the fact that the underlying data are not made available [11, 12], and particularly not for low temperatures where signatures of superconductivity should be more apparent, is a big red flag that suggests that other features of the measured data may not be compatible with superconductivity. Moreover, the fact that underlying data are not made available [11, 12] leaves open the possiblity that some of the manipulations of the measured data that were performed to arrive at the published data may not be compatible with accepted scientific practice.

Eight years after the reported experimental discovery of high temperature superconductivity in a hydride under high pressure [15], there is no convincing magnetic evidence that these materials are superconductors [5]. The analysis in this paper eliminates the strongest claims in that regard. The resistance evidence has also been called into question [16–21]. Readers should draw their own conclusions.

Data Availability The data that support the findings of this study are available from the authors upon reasonable request.

Declarations

Conflict of Interest The authors declare no competing interests.

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11. We were informed by a Nature Comm. editor that the editors are in possession of these data, however they consider them "confidential" following the request of the authors of Ref. [1] and for that reason cannot be shared with us. The same editor told us that the journal will not publish an Editor Note informing readers that there are restrictions on data availability for Ref. [1] because the data are available to the editors
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