Evidence against superconductivity in flux trapping experiments on hydrides under high pressure & On magnetic field screening and expulsion in hydride superconductors

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It has recently been reported that hydrogen-rich materials under high pressure trap magnetic flux, a tell-tale signature of superconductivity [1]. Here we point out that under the protocol used in these experiments the measured results indicate that the materials don’t trap magnetic flux. Instead, the measured results are either experimental artifacts or originate in magnetic properties of the sample or its environment unrelated to superconductivity. Together with other experimental evidence analyzed earlier, this clearly indicates that these materials are not superconductors.

In a second part, we discuss magnetic field screening and expulsion.

PACS numbers:

\section{I. INTRODUCTION}

Following the paper “Conventional superconductivity at 203 kelvin at high pressures in the sulfur hydride system”, published in 2015 [2], several other hydrogen rich materials under high pressure have been reported in recent years to be high-temperature superconductors based on observed drops in resistance versus temperature [3–19]. Many more such materials have been determined to be conventional high-temperature superconductors based on theoretical evidence [20–25]. However, little magnetic evidence has so far been provided in support of the claims of superconductivity [2, 26–29], and what evidence does exist has been strongly called into question [30–33].

In particular, these materials show no trace of magnetic flux expulsion, i.e. the Meissner effect, when cooled in the presence of a magnetic field [2, 26, 27]. They also apparently are able to screen very large applied magnetic fields [28]. This has been interpreted as indicating that the materials are “hard superconductors” with very strong pinning centers that prevent both flux penetration and flux expulsion [26–28]. We have argued that if that is the case the materials should also trap large magnetic fields [34], and that observation of flux trapping would provide definitive evidence that the materials can sustain persistent currents, hence are indeed superconductors [34].

Experiments aimed at detecting flux trapping were recently performed by Minkov et al. and the results analyzed and reported in Ref. [1]. Ref. [1] interprets the measured data as clearly indicating that the materials are superconductors. Instead, we analyze here the information presented in Ref. [1] and conclude that it proves the absence of superconductivity in these materials.

\section{II. EXPERIMENTAL PROTOCOLS}

The flux trapping experiments on sulfur hydride ($H_3S$) [1] were performed under zero-field-cooling (ZFC) conditions for 13 values of applied field ranging from 0 to 6T, and under field cooling (FC) conditions for one field value only, 4T. The results for both protocols for field 4T were reported to agree [1]. In the ZFC protocol, the sample was cooled to low temperatures in zero magnetic field, a magnetic field was then applied and gradually increased to reach value $H_M$, then after 1 hour the external field was gradually decreased to zero, then the resulting magnetic moment was measured with a SQUID magnetometer.

Fig. 1 shows the experimental data and a theoretical fit to the data given in Ref. [1]. Note in particular that the measured magnetic moment rises linearly from zero when the field exceeds the threshold value $H_p$, both for the experimental data and for the theoretical fit.

The experimental results were reportedly analyzed in Ref. [1] assuming the Bean model [36] controls the behavior of fields and currents in the material. From the...
III. OUR ANALYSIS

Just as Ref. [1], we assume the validity of the Bean model. However we disagree that Eqs. (2a), (2b) used by the authors of [1] is the proper way to calculate the trapped magnetic moment under ZFC conditions. Instead, we argue that Eq. (2a) is the proper way to calculate the trapped moment under FC conditions, provided Eq. (2b) is replaced by

$$r = r(H_M) = \frac{d}{2}(1 - \frac{H_M - H_p}{H^*})$$

(3)

for $H_M < H^* + H_p$, $r = 0$ for $H_M > H^* + H_p$, with $H_p = 0$. This is illustrated in the left panels of Fig. 2. For ZFC conditions instead, the diagrams shown in the right panels of Fig. 2 apply. For that case, the magnetic moment is given by

$$m = m_s[1 - 2\left(\frac{r_1}{d/2}\right)^3 + \left(\frac{r_2}{d/2}\right)^3]$$

(4)

where, for $H_M < H^* + H_p$

$$r_1 = \frac{d}{2}(1 - \frac{H_M - H_p}{2H^*})$$

(5a)

$$r_2 = \frac{d}{2}(1 - \frac{H_M - H_p}{H^*})$$

(5b)

For $H^* + H_p < H_M < 2H^* + H_p$, $r_1$ is given by Eq. (5a) and $r_2 = 0$, and for $H_M > 2H^* + H_p$, $r_1 = r_2 = 0$.

Fig. 3 shows what these expressions predict for the trapped magnetic moment versus magnetization field $H_M$ for the parameters assumed in Ref. [1]. Most importantly, the moment rises from zero linearly under

FIG. 2: Magnetic fields and currents predicted by the Bean model under field-cooled (FC) and zero-field-cooled (ZFC) protocols. Here we assume $H_p = 0$ for simplicity.

experimental results, Ref. [1] inferred the parameters:

$H_p = 0.042T$—threshold value of the applied field where it begins to penetrate the sample at low temperatures. Assuming demagnetization $1/(1 - N) = 8.5$, this implies a lower critical field value $H_{c1} = 0.36T$.

$H^* = 0.835T$—minimum applied field that reaches the center of the sample (called “full penetration field”), with assumed sample diameter and height $d = 85\mu m$, $h = 2.5\mu m$.

The measured moment was found to increase with magnetic field $H_M$ up to a maximum value of approximately $m_s = 15.9 \times 10^{-9} Am^2$ for $T = 30K$ when the applied magnetic field was $\sim 1.7T \equiv H_M^{Crit}$ or larger. Following the Bean model, Ref. [1] concluded that

$$H_M^{Crit} = 2H^* + H_p$$

(1)

from which the value of $H^*$ was extracted.

The theoretical fit performed in Ref. [1] assumed the magnetic moment is given by (with $j_c$ the critical current)

$$m = \int r^{d/2} \pi r^2 j_c hr' = m_s[1 - \left(\frac{r}{d/2}\right)^3]$$

(2a)

$$r = r(H_M) = \frac{d}{2}(1 - \frac{H_M - H_p}{2H^*})$$

(2b)

so that $r(H_p) = d/2$, $r(2H^* + H_p) = 0$.

FIG. 3: Expected trapped magnetic moment versus magnetization field $H_M$ for the parameters assumed in Ref. [1] under FC and ZFC protocols. For small $H_M$ the dependence is linear (quadratic) for FC (ZFC) protocols. The experimental points are also shown.
FC conditions and \textit{quadratically} for ZFC conditions. As seen in the inset, for small fields the ZFC moment is very much smaller than the FC moment and in stark disagreement with the experimental observations.

The experimental results of Ref. \cite{1} are actually well fit by our FC calculation for all values of the magnetization field \( H_M \) if we take the value of \( H^* \) to be twice as large as inferred in Ref. \cite{1}, \( H^* = 1.67T \). This is shown in Fig. 4. We conclude that this agreement is accidental, since the experimental protocol was ZFC for all but one experimental point \cite{1}.

In order to try to fit the low field ZFC experimental data to the ZFC calculation we would have to take a much smaller value of \( H^* \). Fig. 5 shows the results for \( H^* = 0.2T \), chosen to fit as well as possible the low field data. In addition to not fitting the low field data very well, the higher field data deviate strongly from the theoretical ZFC curve. For this assumed value of \( H^* \) the trapped moment saturates for \( H_M^{\text{sat}} = 0.44T \) (Eq. (1)), in clear contradiction with the experimental data that show no saturation till \( H_M > 1T \).

\section*{IV. DISCUSSION}

Is it possible that under the ZFC protocol of the experiment with the field \( H_M \) applied for 1 hour, the field could penetrate sufficiently so as to mimic the FC protocol? It is not possible, because Ref. \cite{1} also measured the rate of flux creep and there was negligible flux creep over a 1 hour period even at temperatures as high as 165K. Also, according to the NRS experiment \cite{28} the flux didn’t penetrate over times substantially larger than 1 hour.

Therefore, the experimental results of Ref. \cite{1} shown in Fig. 1 of this paper are incompatible with the interpretation that the magnetic moment observed originates in flux trapping. If the magnetic moment had originated in flux trapping, it would rise quadratically from zero as function of the magnetization field \( H_M \) under the ZFC conditions of the experiment, not linearly as observed. Therefore, the experiment indicates that there is no flux trapping in this material, \( H_S \). As argued in Refs. \cite{32, 34}, if the material doesn’t trap flux, and in addition it does not expel flux, then the material is not a superconductor.

The question then arises, what is the origin of the magnetic moments measured in Ref. \cite{1} shown in Fig. 1? We suggest they are either experimental artifacts associated with the experimental apparatus used (SQUID magnetometer) or magnetic moments of localized spins originating either in the sample or in the diamond anvil cell environment (gasket, etc). It is also possible that the measurements could signal unexpected collective magnetic behavior of hydrogen-rich materials under high pressure, as suggested in Ref. \cite{37}.

To confirm the results of our analysis we suggest that it would be of interest to repeat the measurements of Ref. \cite{1} under FC conditions. We expect that the results will be similar to the results under ZFC conditions, in contradiction with what is expected from trapped flux shown in Figs. 3 and 4, namely a marked difference between FC and ZFC behavior, and consistent with the hypothesis that the origin of the magnetic moments measured is localized spins rather than delocalized supercurrents. We suggest that it would also be informative to perform these experiments using FC and ZFC protocols for a known hard superconductor and verify the expected qualitatively different behavior shown in Figs. 3 and 4.
Finally, we would like to point out that the interpretation of the measurements of magnetic moment of Ref. [1] as originating in flux trapping with $H^*/4 = 0.27$T appear to be in contradiction with the magnetic moment measurements presented in Ref. [26]. For example, according to the former (see our Fig. 2 top right panel) for an applied field $H \sim H^*/4 = 0.27$T the magnetic field should still be excluded from more than 75% of the sample even at temperature $T \sim 100K$ (see Fig. 1c of [1]). Instead, the magnetic moment measurements shown in Fig. 3a of Ref. [26] indicate that the diamagnetism has essentially disappeared at that point.

Acknowledgments

FM was supported in part by the Natural Sciences and Engineering Research Council of Canada (NSERC) and by an MIF from the Province of Alberta. We are grateful to the authors of Ref. [1] and particularly V. Minkov for clarifying information.

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V. SECOND PART

The above manuscript was posted on arxiv on 14 Jul 2022 and published in J Supercond Nov Magn 35, 3141–3145 (2022). What follows is a new manuscript, under consideration for publication in Nature Communications as “Matters Arising”, submitted to arxiv on 11/14/2022, that arxiv decided should be combined with the above manuscript to allow posting.

On magnetic field screening and expulsion in hydride superconductors

Ref. [1] presents evidence for magnetic field screening and “subtle” evidence for magnetic field expulsion in hydrides under high pressure, which is argued to support the claim that these materials are high temperature superconductors. We point out here that data presented in Ref. [1] appear to be inconsistent (i) with one another, (ii) with other measurements reported by the same authors on the same samples [2, 3], and (iii) with expected behavior of standard superconductors. This suggests that these magnetic phenomena reported for these materials are not associated with superconductivity, undermining the claim that these materials are high temperature superconductors.

In 2015, Eremets and coworkers reported high temperature superconductivity in sulfur hydride (hereafter $\text{H}_3\text{S}$) under pressure [2], starting the hydride superconductivity epoch. Since then to the present, considerable evidence for superconductivity in various pressurized hydrides has been presented based on resistance measurements [4], however little magnetic evidence for superconductivity has been reported so far. In their original paper [2] Eremets and coworkers presented some magnetic evidence based on SQUID measurements. After a long hiatus, new evidence was presented this year in Nat. Comm. 13, 3194 (2022) [1]. That evidence is the subject of this comment. We focus here on the magnetic measurements reported for sulfur hydride ($\text{H}_3\text{S}$), but exactly the same considerations apply to the same measurements reported for lanthanum hydride ($\text{LaH}_{10}$) in Ref. [1], the only other hydride material for which magnetic measurements have been reported to date.

Figure 6 left and center panels reproduce Fig. 3a and Fig. 3e of ref. [1]. To the best of our understanding from carefully reading the paper, both panels show in their light-blue and blue curves respectively the same quantity: magnetic moment versus magnetic field, for the same sample at the same temperature (100K) and same pressure (155 GPa). The middle blue curve in the center panel is the virgin curve, which starts (when properly shifted vertically, as shown in Fig. S10 of [1]) with zero magnetic moment for zero applied field. It should be the same as the light blue curve labeled 100K on the left panel. Yet the curves look very different. The left panel curve shows an upturn for magnetic field beyond 95mT while the center panel curve show no upturn. When plotting both curves on the same scale in the right panel in Fig. 6 it is apparent that they are very different in magnitude and shape.

It should also be noted that the rapid decrease in the magnitude of the magnetic moments beyond the minimum points of the curves shown in Fig. 6 left panel is inconsistent with what is expected for a type II superconductor with very large upper critical field [5], estimated in Ref. [1] to be $H_{c2}(T = 0) \sim 97T$. For example, at $T = 100K$ $H_{c2}(T)$ should be above 60T. When corrected for demagnetization factor estimated as $1/(1 - N) \sim 8.5$ in Ref. [1], it implies that the curve labeled $T = 100K$ should evolve smoothly from its value attained at $H \sim 95mT$ approaching zero at or beyond $H_{c2}(T)(1 - N) \sim 7T$. This is qualitatively inconsistent with the behavior seen in Fig. 6 left panel that shows that the magnetic moment magnitude has already decreased to less than 15% of its maximum value for a field as small as $H \sim 0.2T \sim H_{c2}(T)/35$. Furthermore, in the presence of strong pinning, which Ref. [1] claims has to exist in order to explain the absence of flux expulsion in their samples, the decay of the induced diamagnetic moment should be even slower than for an ideal type II superconductor [7, 36], hence very much slower than what is shown in Fig. 6 left panel.

We also point out that the magnitude of diamagnetic moment versus temperature under zero field cooling reported in Ref. [1] Figs. 2e and S1 left middle panel differs by a factor of 4 or more from the same quantity reported in 2015 in Ref. [2] Figs. 4a and extended data Fig. 6c for samples estimated to be of similar size, with the earlier result showing the larger moment. While in field cooling experiments one may expect substantial variations in magnetic moment depending on sample quality, this is not expected to be the case for zero field cooling experiments.

Figure 7 shows as a blue curve the magnetic moment versus magnetic field at temperature 100K from the left panel of Fig. 6, i.e. Fig. 3a of Ref. [1], compared with the magnetic moment versus magnetic field for a hysteresis cycle at the same temperature for the same sample at the same pressure reported in Fig. 4a of Ref. [3], that was used to obtain the critical current data shown in Fig. S5 of Ref. [1]. The blue curve on the left panel of Fig. 7 should be the virgin curve for this hysteresis cycle, joining smoothly the green curve, as is universally seen in such measurements for superconductors. One such typical example is shown on the right panel of Fig. 7, from Ref. [7]. It can be seen that the blue curve on the
FIG. 6: Left panel: magnetic moment versus applied field for $H_s S$ under pressure, from Fig. 3a of Ref. [1]. Center panel: Magnetic moment versus applied field in a hysteresis cycle, from Fig. 3e of Ref. [1]. The middle blue curve in the center panel is presumably the virgin curve, which should be identical to the light blue curve on the left panel labeled 100K. Right panel: quantitative comparison of the virgin curves for 100K from the left panel (3a) and the center panel (3e).

FIG. 7: Green curve, left panel: hysteresis cycle for magnetic moment of $H_s S$ at 100K, from Fig. 4a of Ref. [3]. Those data were used to obtain the critical current data shown in Fig. S5 of Ref. [1]. The blue curve on the left panel shows the magnetic moment versus magnetic field for 100K from the light blue curve on the left panel of Fig. 6, which is Fig. 3a of Ref. [1]. Right panel: a typical hysteresis cycle for a type II hard superconductor, from Ref. [7]. The virgin curve starting at the origin smoothly joins the hysteresis loop curve.

The signature property of superconductors, that cannot be mimicked by localized magnetic moments, is the Meissner effect, the ability to expel magnetic fields when cooled in a field (FC). In Ref. [1], the authors claim to find “subtle Meissner effect in FC measurements at 2 mT” indicated by the light blue curve shown in their Fig. S1 middle left panel. However, when the same data are plotted in Fig. SI1 middle left panel of Ref. [3] without the light blue curve, no evidence for a Meissner effect is seen. While for some standard superconductors with strong pinning the percentage of flux expulsion (Meissner fraction) can be very small for larger fields, it rapidly increases for small fields, as shown e.g. in Refs. [8–11]. The Meissner fraction is expected to depend on the ratio $H/H_{c1}$ [12], and for $H_s S$ $H_{c1}$ is estimated to be 0.82T [1], which is more than an order of magnitude larger than lower critical fields for standard superconductors with high $T_c$ such as cuprates and pnictides. So the field 2mT of Fig. S1 of Ref. [1] is equivalent to a field of less than 2 Oe for those other materials, for which a sizable Meissner fraction is found [8–11]. It should also be noted that in Ref. [2] Extended Data Fig. 6 (c) the authors plotted magnetic moment under FC for magnetic fields down to 0.2mT showing no evidence for a Meissner effect. Additionally, the Meissner fraction is expected to increase as the thickness of the sample decreases [10, 13], and the samples used in these high pressure experiments are rather thin.

Elsewhere we have also called attention to the facts that (i) Fig. SI1 of Ref. [1] lower left panel shows that the ZFC and FC magnetic moment curves for the precursor sample, not expected to be superconducting, also show an unexplained divergence around 200K [14], (ii) the behavior of magnetic moment versus temperature shown in Fig. 6 is incompatible with the claim of Ref. [15], referenced in Ref. [1] in support of superconductivity of sulfur hydride, that a magnetic field as large as 0.68T...
is excluded from the sample [16], and (iii) ac magnetic susceptibility measurements for sulfur hydride [17] referenced in Ref. [1] as evidence for superconductivity were shown to result from an experimental artifact [18].

Recently, the authors of Ref. [1] also reported measurement of trapped magnetic flux in their samples as evidence for superconductivity [19]. We pointed out [20] that the reported linear behavior of trapped moment versus field in zero field cooling experiments [19] is inconsistent with the expected behavior of hard superconductors [6]. In addition, the magnetic moment measurements reported in Ref. [1] indicate that at low temperatures magnetic fields of up to 95mT are excluded from the sample (see Fig. 6 left panel here), which is inconsistent with the reported finding in Ref. [19] that applied fields as small as 50mT penetrate and are trapped by the same samples.

In summary, we argue that the matters pointed out here cast doubt on the interpretation of Ref. [1] that the reported measurements originate in superconductivity.

Acknowledgments

We acknowledge some helpful correspondence with the authors of Ref. [1]. JEH is grateful to R. Prozorov for illuminating discussions. FM was supported in part by the Natural Sciences and Engineering Research Council of Canada (NSERC) and by an MIF from the Province of Alberta.

Author contributions: JEH initiated the study. JEH and FM analyzed the data and prepared the manuscript.

Competing interests: the authors declare no competing interests.

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

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