In the last few years, the superconducting transition temperature, $T_c$, of hydrogen-rich compounds has increased dramatically, and is now approaching room temperature. However, the pressures at which these materials are stable exceed one million atmospheres and limit the number of available experimental studies. Superconductivity in hydrides has been primarily explored by electrical transport measurements, whereas magnetic properties, one of the most important characteristic of a superconductor, have not been satisfactorily defined. Here, we develop SQUID magnetometry under extreme high-pressure conditions and report characteristic superconducting parameters for $Im-3m$-$H_3S$ and $Fm-3m$-$LaH_{10}$—the representative members of two families of high-temperature superconducting hydrides. We determine a lower critical field $H_{c1}$ of $\approx 0.82$ T and $\approx 0.55$ T, and a London penetration depth $\lambda_L$ of $\approx 20$ nm and $\approx 30$ nm in $H_3S$ and $LaH_{10}$, respectively. The small values of $\lambda_L$ indicate a high superfluid density in both hydrides. These compounds have the values of the Ginzburg-Landau parameter $\kappa \approx 12-20$ and belong to the group of “moderate” type II superconductors, rather than being hard superconductors as would be intuitively expected from their high $T_c$s.
The Bardeen–Cooper–Schrieffer\textsuperscript{1} and Migdal–Eliashberg\textsuperscript{2,3} theories of conventional phonon-mediated superconductivity imply that high frequency phonons and strong electron-phonon interactions are favorable for high-temperature superconductivity. Hydrogen, which has the highest naturally-occurring phonon frequencies due to its low mass, could be the best candidate material for high-temperature superconductivity\textsuperscript{4,5}. Although the realization of superconductivity in pure hydrogen has been hindered by the extreme pressures required to reach the superconducting state (∼500 GPa), the idea of "chemical precompression" of hydrogen by heavier chemical elements in hydrogen-rich compounds has brought great success. Following the discovery of $T_c \approx 203$ K in $\text{H}_2\text{S}$ at ∼150 GPa\textsuperscript{6,7}, higher $T_c$ were subsequently reported in so-called metal superhydrides including $T_c \sim 220$ K in CaH$_2$\textsuperscript{8,9,10}, $T_c \sim 243$ K in YH$_3$\textsuperscript{11}, and $T_c \sim 250$ K in LaH$_{10}$\textsuperscript{12–14}. These major leaps toward room temperature superconductivity are the result of fruitful synergy between theory, computation, and experiment.

Superconductivity in hydrogen-rich compounds has since been demonstrated in numerous experiments\textsuperscript{15,16}, however, it was identified based mostly on electrical transport measurements. Magnetic measurements, which are, inter alia, a crucial and independent test of superconductivity are scarce. They have not provided reliable experimental values of a lower critical field $H_c$ at and the London penetration depth $\lambda_L$ in hydrogen-rich superconductors. Magnetic field screening in the superconducting state of the 1m-3m-H$_2$S phase below 203 K was demonstrated using a superconducting quantum interference device (SQUID) and was in good agreement with the sharp drop of resistance in corroborating electrical resistance measurements of the same sample\textsuperscript{7}. However, $H_c$ was only roughly estimated from the hysteric loops of $M(H)$ data instead of the initial virgin portion of magnetization curves of zero-field-cooled (ZFC) sample. More recently, the diamagnetic response in H$_2$S\textsuperscript{17} and LaH$_{10}$\textsuperscript{18} were qualitatively demonstrated by alternating current magnetic susceptibility measurements adapted for diamond anvil cells (DACs)\textsuperscript{19}.

In the present work, we created an effective approach for accurate magnetometry measurements of samples under megabar pressures by measuring the reference magnetic signal of the whole DAC assembly before the synthesis of a superconducting compound. This technique allows us to accurately determine the values of $H_c$, $\lambda_L$, the Ginzburg–Landau parameter $\kappa$, and the critical current density $j_c$ in 1m-3m-H$_2$S and 1m-3m-LaH$_{10}$ high-temperature superconductors.

Results
Synthesis and characterization of superconducting samples. Samples of H$_2$S and LaH$_{10}$ were synthesized via a chemical reaction between sulfur or lanthanum trihydride and hydrogen at high pressures, in the stability field of the final products. The samples were prepared by sandwiching thin plates of S or LaH$_3$ between two thicker layers of NH$_3$BH$_3$ and pressurized in miniature nonmagnetic DACs to ∼170 GPa. The reference background magnetization signal was collected from the whole assembly of DAC including the pressurized precursor compounds in a SQUID magnetometer. The samples were subsequently heated using a pulsed laser to synthesize the desired superconducting products. Several photos of samples are shown in Fig. 1. Ammonia borane was chosen as an alternative source of hydrogen\textsuperscript{20}, as it readily decomposes at high temperature and releases free H$_2$. This approach was successfully implemented for synthesis of hydrides earlier\textsuperscript{11,13}. In contrast to synthesis in an atmosphere of pure H$_2$, the use of NH$_3$BH$_3$ simplified the experimental procedure and significantly enlarged a size of the final products. The latter is crucial for SQUID measurements because the measured magnetic moment is proportional to square of a sample radius. In addition, the use of NH$_3$BH$_3$ allowed for correct reference magnetization measurements in contrast to pure hydrogen, which can cause uncontrollled spontaneous hydrogenation and formation of the superconducting phases at high pressure prior to the laser heating.

Figure 1g, h shows X-ray diffraction patterns from the dominant 1m-3m-H$_2$S and 1m-3m-LaH$_{10}$ phases in the heated samples. Although LaH$_{10}$ sample contained $P_S = 155 \pm 5$ GPa for 1m-3m-H$_2$S and $P_S = 130 \pm 8$ GPa for 1m-3m-LaH$_{10}$ was determined more precisely based on the variation of the refined lattice parameters across the sample (see Supplementary Information). The 1m-3m crystal lattice of LaH$_{10}$ sample is likely slightly distorted as (111), (220) and (311) diffraction peaks are broader than (200) peak. These peaks were shown to be most sensitive to the monoclinic structural distortions in LaH$_{10}$ at pressures below ∼138 GPa\textsuperscript{21}.

$M(T)$ magnetization measurements. The ZFC samples with the 1m-3m-H$_2$S and 1m-3m-LaH$_{10}$ phases exhibit clear diamagnetic signal below their respective $T_S$, indicated that they had become superconducting after laser heating (see Fig. 2 and Supplementary Figs. S1 and S2). The pronounced changes were detected in raw voltage curves of direct current (DC) scans measured before and after laser heating (see Fig. 2a, b). The pressurized unheated precursors have two minima in DC scans at ∼0 and −1.5 cm, which correspond to the centered position of the sample in DAC and the center of the massive part of DAC body including a piston and a cap, respectively (see Supplementary Fig. S3). After the synthesis of 1m-3m-H$_2$S and 1m-3m-LaH$_{10}$ phases, an additional diamagnetic signal originating from the superconductor below its $T_c$ appeared at 0 cm. The distinctive step on the resulting $M(T)$ dependence associated with superconductivity was observed in both heated samples at 2, 4 and 10 mT (see Fig. 2c–f). The LaH$_{10}$ sample has a broader superconducting transition, which is most likely caused by a larger pressure gradient across the sample and a strong $T(P)$ dependence on the verge of structural instability in this pressure range\textsuperscript{21}. The observed values of $T_c$ ∼231 K in 1m-3m-LaH$_{10}$ and ∼196 K in 1m-3m-H$_2$S are in excellent agreement with the previously-reported values from four-probe electrical transport measurements of samples at the same pressures\textsuperscript{21–24}.

It is worth noting whereas the superconducting transition is pronounced in ZFC measurements, its signature is subtle or almost undetectable in field-cooled measurements (see Fig. 2g, h and Supplementary Figs. S1 and S2). The weak flux expulsion or its absence is well-known for type II superconductors with strong pinning of vortices\textsuperscript{25}. Strong pinning prevents vortices inside the sample from leaving the sample below the $H_{c2}(T)$ value. The very low fields are favorable for the detection of the Meissner state, because in this case the $H_{c2}(T)$ line is crossed in the vicinity of $T_c$ where critical currents are smaller and the pinning is weaker\textsuperscript{26}. We also observed strong suppression of the Meissner effect in the test measurements performed on a powder sample of MgB$_2$ (see Supplementary Information). No flux expulsion at all was also reported in some publications on Fe-based superconductors\textsuperscript{27,28}. 

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When measuring a superconducting sample with a large magnetization value, it is important to consider demagnetization effects. The total magnetic field $H_t$ inside a sample is given by:

$$H_t = H - H_d,$$

where $H$ is the external applied field and $H_d$ is the demagnetization field. The demagnetization field is given by $H_d = -NM$, where $N$ is a shape-dependent demagnetization factor and $M$ is the magnetization of a sample. For a long and thin sample in a parallel field $N \approx 0$, while for a short and flat sample in a perpendicular magnetic field the demagnetization correction $NM$ can be enormous.

We estimated an effective demagnetization factor $N$ in the studied samples using the measured values of magnetization, assuming an ideal diamagnetic signal in low fields ZFC measurements. Then the demagnetization correction for a perfect diamagnet (magnetic susceptibility $\chi = -1$) can be written as follows:

$$\Delta M = -\frac{1}{1 - N} V,$$

where $V$ is the volume of a sample. The absolute value of $\Delta M$, the difference in $M$ between a normal metal state (above $T_c$) and a superconducting state (below $T_c$), was extracted from the measurements by subtraction of the reference data of the compressed sandwiched samples (see Fig. 2). A thickness of 2.8 and 1.9 $\mu$m, respectively, by considering: (1) the visual expansion of samples during pressurizing, (2) the pressure-induced compressibility of $S$ and $LaH_3$, and (3) the increase of product volumes after the hydrogenation reaction (see details in Supplementary Information). If we put the values of $\Delta M$ and $V$ in Eq. (2) the demagnetization correction is $-8.5$ for the sample of $Im-3m-H_3S$ and $Fm-3m-LaH_{10}$ phases as shown inset. $P_b$, $P_i$, and $P_s$ are pressure values estimated from the position of diamond edge and hydrogen vibron in Raman spectra and refined lattice parameters of the final products, respectively (see details in “Methods”).

An alternative way of the evaluation of the diamagnetic factor would be approximating of the shape of samples by thin solid disks and using the equation for effective demagnetization factor from ref. 29 (see detailed discussion in “Methods”). We consider the $N$ values from $M(T)$ data more reasonable and reliable and will use them in the rest of the paper.

**$M(H)$ magnetization measurements.** Measurements of the magnetic field dependence of magnetization allow us to estimate the characteristic superconducting parameters $H_{c1}$, $\lambda_L$, $\kappa$, and $J_c$. 

**Fig. 1 Synthesis of the $Im-3m-H_3S$ and $Fm-3m-LaH_{10}$ phases.** a Illustration of the miniature DAC used for magnetic measurements in SQUID. b Scheme showing the typical arrangement of the sandwiched precursors in the DAC. c, d Photos of the sandwiched samples, $S + NH_3BH_3$ and $LaH_3 + NH_3BH_3$, after loading at $P_b \sim 1$ and $\sim 3$ GPa, respectively. e, f Photos of the $H_3S$ and $LaH_{10}$ samples after compression and subsequent pulsed laser heating. g, h X-ray powder diffraction patterns collected from the synthesized $Im-3m-H_3S$ and $Fm-3m-LaH_{10}$ samples. The black circles and red and blue curves correspond to the experimental data, Rietveld refinement fits and residues, respectively. The green ticks indicate the calculated peak positions. The (101) reflection stemming from the $P6_3/mmc-LaH_{10}$ impurity phase is marked by asterisk. The fragments of the crystal structure with the characteristic SH$_6$ and LaH$_{32}$ coordination polyhedra are shown as insets. The large yellow and cyan and small black spheres represent the $S$, La and H atoms in the crystallographic unit cells, respectively. The spatial distribution across the heated samples and the estimated diameter of the superconducting phases are shown as inset.

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The value of $H_m$, 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). The extrapolation of $H_m(T)$ to lower temperatures yields $H_m(0 K) \sim 96 \text{ mT}$ for $\text{Im-3m-H}_3\text{S}$ and $\sim 41 \text{ mT}$ for $\text{Im-3m-LaH}_{10}$. Applying the demagnetization correction we obtain values of $H_m(0 K) \sim 820 \text{ mT}$ for $\text{H}_3\text{S}$ and $\sim 550 \text{ mT}$ for $\text{LaH}_{10}$. The Ginzburg–Landau parameter $\kappa$ can be evaluated from the equation:

$$\frac{H_{c2}}{H_m} = \frac{2\kappa^2}{\ln 2}$$

(3)

where $H_{c2}$ is an upper critical field. Inserting the experimental estimations of $H_{c2}(0 K) \sim 97 \text{ T}$ for $\text{Im-3m-H}_3\text{S}$ and $\sim 143.5 \text{ T}$ for $\text{Fm-3m-LaH}_{10}$, gives $\kappa \sim 12$ and $\sim 20$ for $\text{H}_3\text{S}$ and $\text{LaH}_{10}$, respectively. A coherence length $\xi(0 K) \sim 1.8 \text{ nm}$ for $\text{H}_3\text{S}$ and $\sim 1.5 \text{ nm}$ for $\text{LaH}_{10}$ were evaluated using the available experimental data, which gives a London penetration depth of $\lambda_L(0 K) \sim 22 \text{ nm}$ in $\text{Im-3m-H}_3\text{S}$ and $\sim 30 \text{ nm}$ in $\text{Fm-3m-LaH}_{10}$. The temperature dependence of $\lambda_L$ is shown in Supplementary Fig. S4. At low temperatures, the s-wave model of conventional superconductivity well reproduces the data for both compounds. The thermodynamic critical field value is given by:

$$H_c(0 K) = \frac{\sqrt{H_{c1}(0 K)H_{c2}(0 K)}}{\sqrt{\ln 2}}$$

(4)

$H_c(0 K) \sim 5.6 \text{ T}$ for $\text{H}_3\text{S}$ and $\sim 5.1 \text{ T}$ for $\text{LaH}_{10}$. The robustness of the dissipation-free vortex solid phase in hydrides can be evaluated via Ginzburg–Levanyuk number:

$$G_i = \frac{1}{2} \left( \frac{2\mu_0 \kappa^2 T_c \lambda^2}{\phi_0^2} \right)^2$$

(5)

where $\kappa$ is the Boltzmann constant, $\mu_0$ is the vacuum permeability and $\phi_0$ is the magnetic flux quantum. $G_i$ quantifies the scale of fluctuations responsible for vortex melting and vortex creep in a superconductor. $G_i(0 K) \sim 9 \times 10^{-7}$ for $\text{H}_3\text{S}$ and $\sim 6 \times 10^{-6}$ for $\text{LaH}_{10}$, which is substantially smaller than what is reported for cuprate and pnictide high-temperature superconductors and comparable to that of $\text{Nb}_3\text{Sn}$. Despite high $T_c$ both hydrides display a moderate $\kappa$ which results in weaker vortex fluctuations.
and explains why the reported vortex liquid region remains narrow even at high magnetic fields\textsuperscript{21,23}.

It is important to estimate the range of values of the superconducting parameters of Im-3m-H\textsubscript{3}S and Fm-3m-LaH\textsubscript{10}, which depends on a sample thickness. We evaluated the minimum and maximum values of a sample thickness as 2.1–3.1 μm for H\textsubscript{3}S and 0.6–2.5 μm for LaH\textsubscript{10} (see details in Supplementary Information). Thus, the lower and upper limits are:

\[ \sim 0.74–1.09 \text{T for } H_{c1}(0 \text{ K}), \sim 18–23 \text{ nm for } \lambda_L(0 \text{ K}), \text{ and } \sim 10–13 \text{ for } \kappa \text{ in Im-3m-H}_3\text{S}; \text{ and } \sim 0.42–1.75 \text{ T for } H_{c1}(0 \text{ K}), \sim 14–35 \text{ nm for } \lambda_L(0 \text{ K}), \text{ and } \sim 10–23 \text{ for } \kappa \text{ in Fm-3m-LaH}_10. \]

For LaH\textsubscript{10}, the larger dispersion is a result of a substantial increase of N in case of the lower limit of a sample thickness. The estimated superconducting parameters for both hydrides are summarized in Table 1.

A general behavior of M(H) (see Fig. 3c, f) is typical for type II superconductors, in particular, the difference in a magnetic moment between the forward and reverse sweep of an applied magnetic field increases with a decrease of temperature. However, it was not possible to observe the extent of the superconducting magnetization hysteresis and the characteristic field H*, above which magnetization becomes reversible, because the magnetic signal of the DAC becomes much higher in comparison with the magnetic response of H\textsubscript{3}S and LaH\textsubscript{10} samples for the reliable subtraction of the background.

We also estimated the critical currents using the Bean critical state model\textsuperscript{33,34}. This model assumes that when the sweeping magnetic field fully penetrates the sample the density of the screening current equals the critical current value j\textsubscript{c}. The difference in magnetic moment Δm between forward and reverse field is due to the reversal of the direction of the screening moment, and can be used to evaluate the magnitude of the bulk critical current. A concentric screening current pattern with density j\textsubscript{c} creates a magnetic moment

\[ m = \frac{1}{2} j \text{c} h r^2, \]

where h is the thickness and r is the radius of the disk-shaped sample, often approximated by j\textsubscript{c} = 30 Ψ, where m, V, and r are in CGS units.

Fig. 3 M(H) magnetization data for Im-3m-H\textsubscript{3}S and Fm-3m-LaH\textsubscript{10} at high pressure. a, b Magnetic moment associated with the penetration of the applied magnetic field into the Im-3m-H\textsubscript{3}S phase at P\textsubscript{S} = 155 ± 5 GPa and the Fm-3m-LaH\textsubscript{10} phase at P\textsubscript{S} = 130 ± 8 GPa based on virgin curves of the M(H) magnetization data at selected temperatures. The curves were superimposed by performing linear transformations for a better representation. A linear background, defined as a straight line connecting M(H = 0 T) and M(H = 1 T) at corresponding temperature, was subtracted. After that the data were normalized to H = 15 mT data so that to have the same initial linear M(H) slope. c. Temperature dependence of a penetration field H\textsubscript{p} for Im-3m-H\textsubscript{3}S and Fm-3m-LaH\textsubscript{10} derived from the virgin curves of M(H) magnetization data. Black circles and red curves correspond to the experimental data and fits, respectively. e, f Hysteretic part of M(H) measurements of H\textsubscript{3}S and LaH\textsubscript{10} in the normal metallic state above T\textsubscript{c} and the superconducting state below T\textsubscript{c}, correspondingly.
and \( j_c \) is in A cm\(^{-2}\). We found that \( j_c \) reaches values of \( \sim 7 \times 10^6 \) A cm\(^{-2}\) for both LaH\(_{10}\) and H\(_3\)S at 100 K (see Supplementary Fig. S5). This high value of the critical current indicates strong vortex pinning, which corroborates negligible magnetization signal observed during field cooling, as well as very high irreversibility field \( H^* \), or vortex melting/de-pining field, reported in magnetotransport measurements\(^{6,12,21,33,35}\). The critical currents in LaH\(_{10}\) estimated from the magnetization measurements are of the same order of magnitude but somewhat higher than the value of \( 1.2-2.8 \times 10^6 \) A cm\(^{-2}\) at 4.2 K measured in presumably yttrium-doped lanthanum superhydride using electrical transport technique\(^{35}\). This discrepancy can be attributed to the fact that in the transport measurements \( j_c \) is constrained by those parts of the electrical current path where the superconductivity is least robust, while the magnetization signal is dominated by the parts of the sample with most robust superconductivity.

**Discussion**

It is informative to compare our findings with the available magnetic data from previous studies on H\(_3\)S\(^{7,36}\). Although \( H_{c1} \sim 30 \) mT was reported in an earlier study of Im-3m-H\(_3\)S\(^7\), this value was strongly underestimated. Firstly, \( H_{c1} \) was determined from the hysteretic loops of \( M(H) \) data instead of the initial virgin magnetization curves, which were not measured. Secondly, the real shape of the superconducting phase was not determined and the corresponding demagnetization correction were not applied. In addition, the magnetic signal was superposed by a much stronger paramagnetic signal, presumably stemming from the body of DAC.

In another work, authors applied forward nuclear resonant scattering technique using the \(^{119}\)Sn Mössbauer isotope as a sensor and reported a value of \( H \sim 68 \) T, which was expelled by the sample at \( \sim 120 \) K\(^{36}\). The sample was synthesized by pressure-induced disproportionation of H\(_3\)S at \( \sim 150 \) GPa as in ref. \(^7\), however the superconducting phase was not characterized—neither crystal structure nor \( T_c \) were determined. Since the geometry and arrangement of a tin foil and superconducting sample, which are required for calculations of demagnetization and end effects, are unknown, it is not possible to quantify the value of \( H_{c1} \) from this experiment.

In summary, we have performed magnetization measurements using a specially-designed miniature DAC for representative members of two families of hydrogen-rich superconductors—H\(_3\)S, which contains covalent H-S bonds, and LaH\(_{10}\), which has ionic bonding between La and H. The present data demonstrate that the diamagnetic signal is absent in the pressurized S and LaH\(_3\) precursor compounds and only appears after laser heating of the samples and the resultant chemical synthesis of the respective superconducting phases. In contrast to high-\( T_c \) superconductors of the cuprate family, the Im-3m-H\(_3\)S and Fm-3m-LaH\(_{10}\) phases have significantly lower values of \( \lambda_c \). The low values of \( \kappa \) indicate that both compounds belong to “moderate” type II superconductors not far from the clean limit. Both H\(_3\)S and LaH\(_{10}\) hydrides possess good superconducting characteristics; in addition to high values of \( T_c \) they exhibit high critical current densities and have high values of lower and upper critical fields. These make hydrogen-rich compounds promising materials for technological use, provided that they can be stabilized at ambient or accessible pressure conditions.

**Methods**

**Diamond anvil cell.** The samples were synthesized in miniature DACs, which were specially designed for a standard commercial SQUID magnetometer (either Quantum Design MPMS or Cryogenic Limited S700X) with the sample space diameter of 9 mm by reworking and modifying the prototype piston-locking nut design. The design of the DAC was briefly described in ref. \(^{7}\). To minimize the magnetic signal over a wide temperature range simultaneously providing a high mechanical strength, the body of the DAC was made of a high-purity Cu-Ti alloy with 3 wt% Ti\(^{35}\). This material has the lowest magnetic susceptibility among the known hard metallic alloys: \( \chi = 8 \times 10^{-8} \) mT \(^{-1} \) g \(^{-1} \) at 1.8 K and at the same time is hard enough to build parts of the DAC (its tensile strength is \( \sim 1800 \) MPa\(^{36}\). As parts such as piston and diamond seats are subjected to the highest load, they were made of harder Cu-Be alloy with 1.8-2.0 wt% Be. Such combination of materials allows us to construct the miniature DAC with an outer diameter of 8.8 mm, which is capable to reach pressures as high as 220 GPa retaining the low overall magnetic response.

The diamonds were beveled at 9° to a diameter of \( \sim 250 \) µm with a culet size of \( \sim 75 \) and \( \sim 90 \) µm. In total, 200-µm-thick rhenum gasket was pre-indented to a thickness of 20 and 30 µm, and a hole with a diameter of about the culet size was drilled using a laser. All elements of the high-pressure cell assembly were thoroughly etched in acids in order to remove a possible contamination with magnetic pieces, which could stem from the manufacturing of the DAC parts, polishing of the diamonds and cutting of the gaskets. All parts of the DACs and prepared gaskets were etched in 3 M hydrochloric acid for 30 min, and diamonds were etched in a mixture of concentrated nitric and hydrochloric acids in 1:3 molar ratio for 90 min in an ultrasonic cleaner.

**Preparation of samples.** Sulfur (99.999%, Alfa), NH\(_4\)BH\(_3\) (97%, Sigma-Aldrich), and LaH\(_{10}\), which was synthesized from La (99.9%, Alfa Aesar) and H\(_2\) (99.999%, Spectra Gases), were used as initial reactants. In contrast to metallic La, LaH\(_3\) was beneficial because it required less hydrogen for the full hydrogenation. The loading of samples in DACs were handled in an inert Ar atmosphere with the O\(_2\) and H\(_2\)O residual contents of <0.1 ppm. NH\(_4\)BH\(_3\) act both as a source of H\(_2\) and a thermal isolator from the diamonds during laser heating. The thin plates of S, LaH\(_3\), and NH\(_4\)BH\(_3\) for the sandwiched samples were molded out the corresponding powder samples by squeezing them between two large 1-mm-diameter diamond anvils. The thickness of the plates was monitored by the interference of the visible light.

The sandwiched samples, in which 8-µm-thick S or 6-µm-thick LaH\(_3\) plates were interposed between two \( \sim 15-15 \) µm-thick layers of NH\(_4\)BH\(_3\) were put in the hole of pre-indented metallic gaskets. Then samples were pressurized to \( P_0 \) of \( \sim 167 \) GPa with a pressure gradient across the culet of about \( \pm 7 \) GPa. The decomposition of NH\(_4\)BH\(_3\) and synthesis of the superconducting Im-3m-H\(_3\)S and Fm-3m-LaH\(_{10}\) phases were performed using the one-side heating with Nd:YAG laser pulse (a wavelength \( \lambda = 1.064 \) µm, the duration of pulses of 3 µs, and frequency of 10\(^4\) Hz). We heated S + NH\(_4\)BH\(_3\) sample at \( \sim 700 \) K and LaH\(_3\) + NH\(_4\)BH\(_3\) sample at \( \sim 2000 \) K by traversing the \( \sim 5 \) µm-diameter laser spot horizontally and vertically across the diamond culets. Several photos of the initial, pressurized and heated samples are summarized in Supplementary Figs. S6 and S7.

Importantly, the integrity of a superconducting phase in a final sample is crucial for detecting of the diamagnetic signal by a SQUID. For example, LaH\(_3\) completely transformed to the Fm-3m-LaH\(_{10}\) phase already after the first laser heating at \( \sim 1000 \) K according to the X-ray diffraction data, nevertheless the superconducting transition was not observed in the magnetic measurements (see Supplementary Fig. S8). We guessed that this was because the sample was not uniform and consisted of separate parts, from which the sum magnetic signal is smaller than that from the one uniform disk of the same integral area. Our rough estimations gave a factor of \( \sim 5 \) difference in the signal between one 60-µm-diameter disk and 20 12-µm-diameter disks with a thickness of 2 µm just because of different demagnetization factors. Additionally, the smaller disks might have a smaller total volume resulting in an increase of a factor to \( \sim 10 \), so the sum magnetic signal becomes less than the sensitivity of a SQUID. To improve the integrity of the

### Table 1

| Sample     | \( P_0 \), GPa | \( T_c \), K | Size, µm | \( \lambda_c \), mT | \( H_{c1}(0 \) K), mT | \( H_{c2}(0 \) K), T | \( \kappa \) | \( \gamma \) |
|------------|---------------|-------------|----------|-------------------|-------------------|-------------------|---------|--------|
| Im-3m-H\(_3\)S | 15.5 ± 5      | ~196        | 85       | 2.8 (2.1-3.1)     | 8.5 (7.7-11.4)    | 96 ± 2            | 0.82 (0.74-1.09) | 22 (18-23) |
| Fm-3m-LaH\(_{10}\) | 130 ± 8   | ~231        | 70       | 1.9 (0.6-2.5)     | 13.5 (10.2-42.6)  | 41 ± 2            | 0.55 (0.42-1.75) | 30 (14-35) |

The upper and lower limits of corresponding parameters are parenthesized.
superconducting phase by sintering, we again heated LaH$_3$ but at significantly higher temperatures of ~2000 K. As a result, the pronounced superconducting transition appeared in the subsequent magnetic measurements.

**Estimation of pressure.** The pressure values in samples were estimated using three different techniques. Initially we determined the pressure in the compressed sandwiched samples using the diamond anvil cell (DAC) technique. We found the pressure in the DAC to be close to that of a similar calculation using a different technique. We then used the pressure values derived from the DAC experiments for calibration of the distance between sample and detector.

For the micrometer-size superconducting samples in DACs we were able to determine the pressure in the compressed samples using three different techniques. Initially we determined the pressure in the compressed sandwiched samples using the diamond anvil cell (DAC) technique. We found the pressure in the DAC to be close to that of a similar calculation using a different technique. We then used the pressure values derived from the DAC experiments for calibration of the distance between sample and detector.

**X-ray diffraction measurements.** X-ray diffraction measurements were done in the same sample DACs at the beamlines 13-IDD at GSECARS, Advanced Photon Source (λ = 0.2952 Å, a beam spot size of ~2 × 3.5 µm$^2$), Pilatus 1 M CdTe detector) and PO22 at PETRA III, DESY (λ = 0.2885 Å, a beam spot size of ~2 × 2 µm$^2$). The reference samples of LaB$_6$ and CeO$_2$ were used for calibration of the distance between sample and detector. The average value of the refined lattice parameter $a$ across the sample is 3.057(8) Å for 1m-3m-H$_3$S phase and 5.175(3) Å for Fm-3m-LaH$_{10}$ phase. Taking into account the available structural data of H$_3$S and LaH$_{10}$, we deduced the 140-mm-long straw made of kapton polyimide, which was specially designed for the experiments.

**Magnetization measurements.** Magnetization measurements were done in the S700X SQUID magnetometer by Cryogenic Limited, a miniature DAC was attached to the 140-mm-long straw made of kapton polyimide, which was specially designed for the experiments.

To minimize the errors associated with the sample positioning at different temperatures, the temperature-induced expansion of the rod, which holds the sample, was additionally calibrated within the wide temperature range using the ferromagnetic signal from the same steel piece.

For the micrometer-size superconducting samples in DACs we were able to extract a small magnetic signal of a superconductor from the measured overall magnetic moment including that of the bulky body of DAC, diamonds and rhenium gasket. We first measured the magnetic signal of the DACs with the starting pressurized precursor compounds before laser heating, in which S and LaH$_{10}$ were normal metals. Then we subtracted these reference data from the response of the miniature high-pressure cell, which increases with the applied magnetic moment collected from the same DACs after laser heating and chemical transition appearing in the subsequent magnetic measurements.

**Rhenium gasket.** We used a rhenium gasket. We performed additional linear background subtraction for the data collected from the laser-heated and chemically reduced matrix. The response of the miniature high-pressure cell, which increases with the applied magnetic moment, was subtracted from this data.

**Curves of the temperature dependence of magnetic moment.** The curves of the temperature dependence of magnetic moment for both materials were determined from the magnetization measurements. The London penetration depth was determined using the equation $\Delta T_c(0) = \frac{\Delta T_c}{2}\sqrt{\frac{3}{2}\pi}$, where $m_F$ is the effective mass, $c$—speed of light, $n_F$—density of superconducting electrons, $e$—electron charge. Using $n_F$ for $n_F$, from the measurements of the Hall effect at room temperature and assuming no mass enhancement, the $\Delta T_c(0)$ was ~18 nm for H$_3$S and ~6 nm for LaH$_{10}$. These values are reasonably consistent with the more accurate estimations using the values of the upper and lower critical fields.

**Demagnetization correction.** We estimated the effective demagnetization correction as ~8.5 for the 0.85 × 2.8 µm$^2$ sample of 1m-3m-H$_3$S and ~13.3 for the ~0.70 × 1.9 µm$^2$ sample of Fm-3m-LaH$_{10}$ using the measured value of magnetization.

The demagnetization factor $N$ can be also calculated for the given geometry of a superconductor according to ref. 29. This gives demagnetization correction ~20 and ~24 for the 1m-3m-H$_3$S and Fm-3m-LaH$_{10}$ samples, respectively. We note that the difference in values of $N$ for a thin disk- and thin ellipsoid-shaped (due to the cupping effect in diamond anvils at high pressures) samples is negligible if a diameter is much larger than a thickness. For the sake of simplicity, we consider the samples as thin disks in our estimates. The larger values of the computed demagnetization correction stem from the ignoring of variation of thickness in a sample and imperfections of sample integrity, which are, conversely, already included in the experimental value of $\Delta M$. A magnetic field penetrates into a superconductor at these imperfections decreasing an effective demagnetizing factor $N$. Thus, using the measured value of $\Delta M$ we obtain a lower estimate of $N$, since a decrease of a sample volume in a case of variation of a sample thickness will lead to an increase of $N$. For a good single-crystal, where imperfections of a sample are minimal, the demagnetization correction should be of the same value if calculated from a sample geometry or its value of magnetization. For instance, we have almost the same values of demagnetization correction derived from the prior ZFC $M(T)$ measurements of the test single-crystal of Bi$_{2}$Sr$_{2}$Ca$_{2}$Cu$_{3}$O$_{8}$ with a size of a size of 100 × 80 × 10 µm$^3$ (see details in Supplementary Information). In particular, $\Delta T_c/8$ from the value of $\Delta M$ and $\Delta T_c/7$ from the geometry of the sample.

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

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