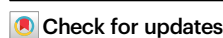


Long-term stability of superconducting metal superhydrides

Received: 3 April 2025

Accepted: 14 July 2026

Published online: 12 August 2026



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ARISING FROM Y. Zhou et al. *Nature Communications* <https://doi.org/10.1038/s41467-025-56033-3> (2025)

Zhou et al.¹ reported the synthesis of lanthanum superhydride, LaH_x ($x = 10.2\text{--}11.1$), by laser heating LaH₃ with NH₃BH₃ at a pressure of 170 GPa and investigated the temporal evolution of the NMR spectra of the reaction products. The authors¹ observed a gradual decrease in the ¹H-NMR signal intensity assigned to the synthesized metal hydride and a concomitant increase in molecular hydrogen over a period of 50 days, which they interpreted as a gradual decomposition of LaH₁₀ into LaH₃ and H₂. On the contrary, we demonstrate that, under their formation conditions, metal superhydrides are thermodynamically more stable than metal trihydrides, and we provide direct experimental evidence from X-ray diffraction and four-probe electrical resistance measurements showing that the *Fm-3m*-LaH₁₀ phase and its superconducting properties remain stable for more than 5 years. This long-term stability is consistent with predictions from quantum chemistry calculations.

Numerous theoretical studies predict that, under sufficiently high pressure, metal superhydrides MH_x (M = La, Y, Ce, Ca, etc., $x \geq 6$) become stable with respect to decomposition into hydrogen and lower hydrides. For example, theoretical predictions for the binary yttrium-hydrogen and lanthanum-hydrogen systems indicate that *Im-3m*-YH₆ is thermodynamically stable above 110 GPa^{2–4}, while *Fm-3m*-LaH₁₀ is stable above 150 GPa⁴. Subsequent experiments confirmed the formation of *Im-3m*-YH₆ at 160–180 GPa^{5–7} and *Fm-3m*-LaH₁₀ at 140–180 GPa^{8–14}, in reasonable agreement with the theoretical predictions.

Some metal superhydrides readily form in a hydrogen atmosphere at sufficiently high pressures at room temperature over the course of several weeks. However, laser heating accelerates the hydrogenation reaction at higher temperatures. When alternative hydrogen sources, such as NH₃BH₃, are used instead of H₂, laser

heating becomes essential to decompose ammonia borane and facilitate the release of free H₂.

The formation of superconducting *Fm-3m*-LaH₁₀, *P6₃/mmc*-YH₉, *Im-3m*-YH₆, and *Im-3m*-YD₆ phases by exposing corresponding metal trihydrides/trideuterides to hydrogen/deuterium at high pressures and room temperature has been previously reported^{6,11}. These metal superhydrides were characterized by X-ray powder diffraction and four-probe electrical resistance measurements, demonstrating high *T_c* values, as shown in Fig. 1.

Our experimental results contradict the findings of Zhou et al.¹, which suggest that lanthanum superhydride is unstable and decomposes into LaH₃ and H₂ at the pressures at which it was formed. On the contrary, our experiments show that the chemical reaction proceeds in the opposite direction—*forming* superhydrides rather than *decomposing* them. This provides direct experimental evidence that lanthanum and yttrium superhydrides are thermodynamically favored at high pressures. Consequently, these phases cannot spontaneously decompose into LaH₃ while releasing free H₂ at the conditions under which they were synthesized.

Next, we address the long-term stability of metal superhydrides, a key focus of our research since the discovery of near-room-temperature superconductivity in highly compressed hydrides. To this end, we have systematically monitored several synthesized superhydride phases over the years.

It is important to emphasize that the typical time interval between sample synthesis and subsequent resistance *R(T)* measurements, including those performed under external magnetic fields, ranges from several days to weeks^{6,10,11}, and can extend to several months for magnetic susceptibility measurements^{9,15}. Furthermore, synchrotron

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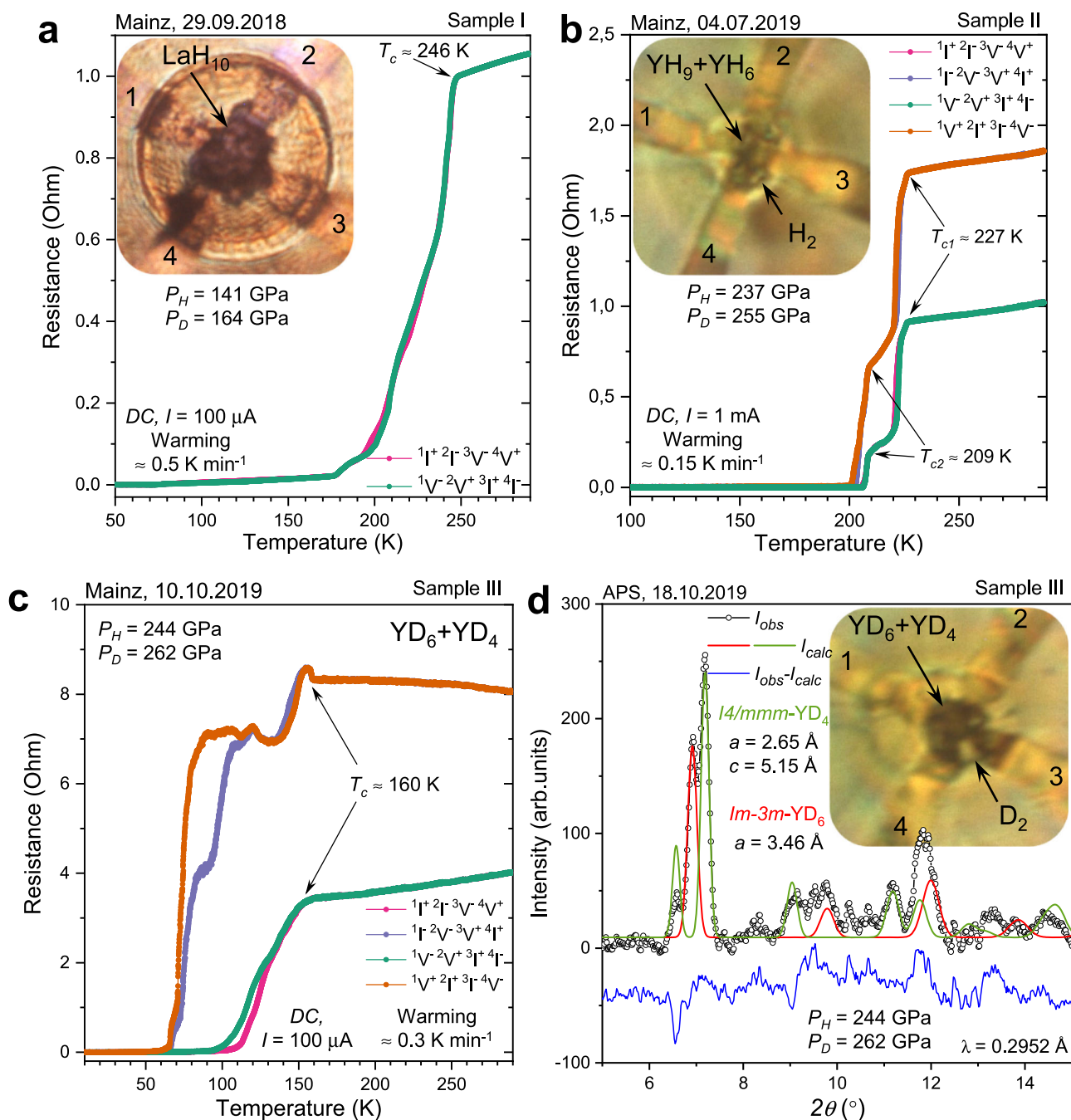


Fig. 1 | Formation of metal superhydrides with high-temperature superconductivity at high pressures and room temperature. a–c Temperature dependence of the electrical resistance $R(T)$ of metal superhydrides, demonstrating high-temperature superconductivity. **a** Sample I: LaH₃ and H₂ were pressurized to $P_H = 141$ GPa (estimated using H₂ (D₂) Raman vibron scale²⁵) or $P_D = 164$ GPa (estimated using diamond edge Raman scale²⁶) on 12.09.2018. After 17 days at room temperature, $R(T)$ measurements showed $T_c \approx 246$ K ($Fm-3m$ -LaH₁₀). **b** Sample II: YH₃ and H₂ were pressurized to $P_H = 237$ GPa ($P_D = 255$ GPa) on 10.06.2019. After 23 days at room temperature, $R(T)$ measurements showed $T_{c1} \approx 227$ K ($P6_3/mmc$ -

YH₉) with a second step at $T_{c2} \approx 209$ K ($Im-3m$ -YH₆). X-ray powder diffraction pattern and the Rietveld refinement of Sample II are shown in Supplementary Fig. 2 in ref. 6. **c** Sample III: YD₃ and D₂ were pressurized to $P_H = 244$ GPa ($P_D = 262$ GPa) on 23.09.2019. After 17 days at room temperature, $R(T)$ measurements showed $T_c \approx 160$ K ($Im-3m$ -YD₆). **d** X-ray powder diffraction pattern of Sample III and the Rietveld refinement confirms the presence of $Im-3m$ -YD₆ and $I4/mmm$ -YD₄ phases. The black circles are experimental data, the red and green profiles show the simulated contribution of the $Im-3m$ -YD₆ and $I4/mmm$ -YD₄ phases, and the blue profile represents the refinement residual.

X-ray diffraction experiments are often conducted weeks or even months after sample synthesis.

The authors of ref. 1 acknowledge that the superconducting $Im-3m$ -H₃S phase remains stable and retains its T_c of approximately 200 K over several years^{16–19}. Below, we present evidence demonstrating that the $Fm-3m$ -LaH₁₀ phase preserves its crystal lattice and high-temperature superconducting properties for over 5 years.

Figure 2 illustrates the stability of the $Fm-3m$ -LaH₁₀ crystal lattice over at least 55 months in sample IV, as confirmed by X-ray diffraction experiments. Sample IV was synthesized from a La piece and H₂ using pulsed laser heating at $P_H = 156$ GPa ($P_D = 172$ GPa) on February 17, 2020. Nine days later, an initial X-ray diffraction experiment at the Advanced Photon Source (Argonne) revealed that the sample primarily consisted of the $Fm-3m$ -LaH₁₀ phase, with a minor impurity of

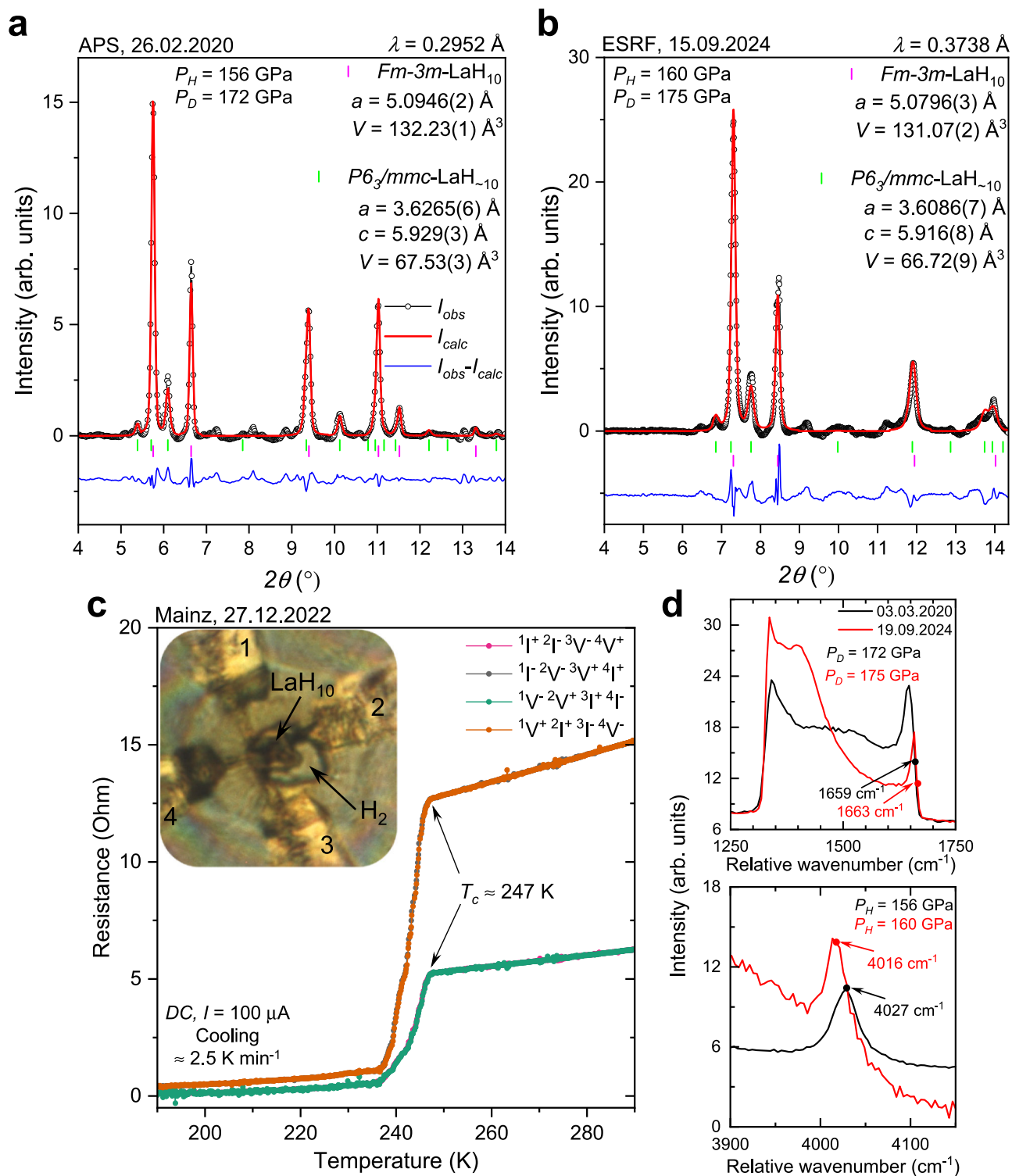


Fig. 2 | Long-term stability of the $Fm\text{-}3m\text{-LaH}_{10}$ crystal lattice. **a**, **b** X-ray powder diffraction patterns for sample IV were recorded 1 week and 55 months after the synthesis, respectively. The black circles represent the experimental data, while the red and blue profiles correspond to simulated patterns and the Le Bail refinement residuals, respectively. The magenta and green ticks indicate the calculated

positions of the diffraction peaks for the $Fm\text{-}3m\text{-LaH}_{10}$ and $P6_3/mmc\text{-LaH}_{10}$ phases. **c** $R(T)$ measurements performed 34 months after synthesis, showing a superconducting transition with a $T_c \approx 247 \text{ K}$. **d** Raman spectra, demonstrating the evolution of the estimated pressure in the diamond anvil cell over time, as manifested in the shift of the diamond Raman line edge (top) and the hydrogen vibron (bottom).

the $P6_3/mmc\text{-LaH}_{10}$ phase, commonly observed in samples containing $Fm\text{-}3m\text{-LaH}_{10}$ ^{10,11}. Four-probe electrical resistance measurements were conducted on December 27, 2022—34 months after synthesis—and revealed a superconducting transition at $T_c \approx 247 \text{ K}$. Fifty-five months after synthesis, a second X-ray diffraction experiment at the European

Synchrotron Radiation Facility (Grenoble) confirmed that the sample retained the same phase composition and, therefore, the same chemical composition. A minor decrease ($<0.5\%$) in the lattice parameters for both cubic and hexagonal phases was attributed to a slight pressure increase in the diamond anvil cell over time (see Fig. 2).

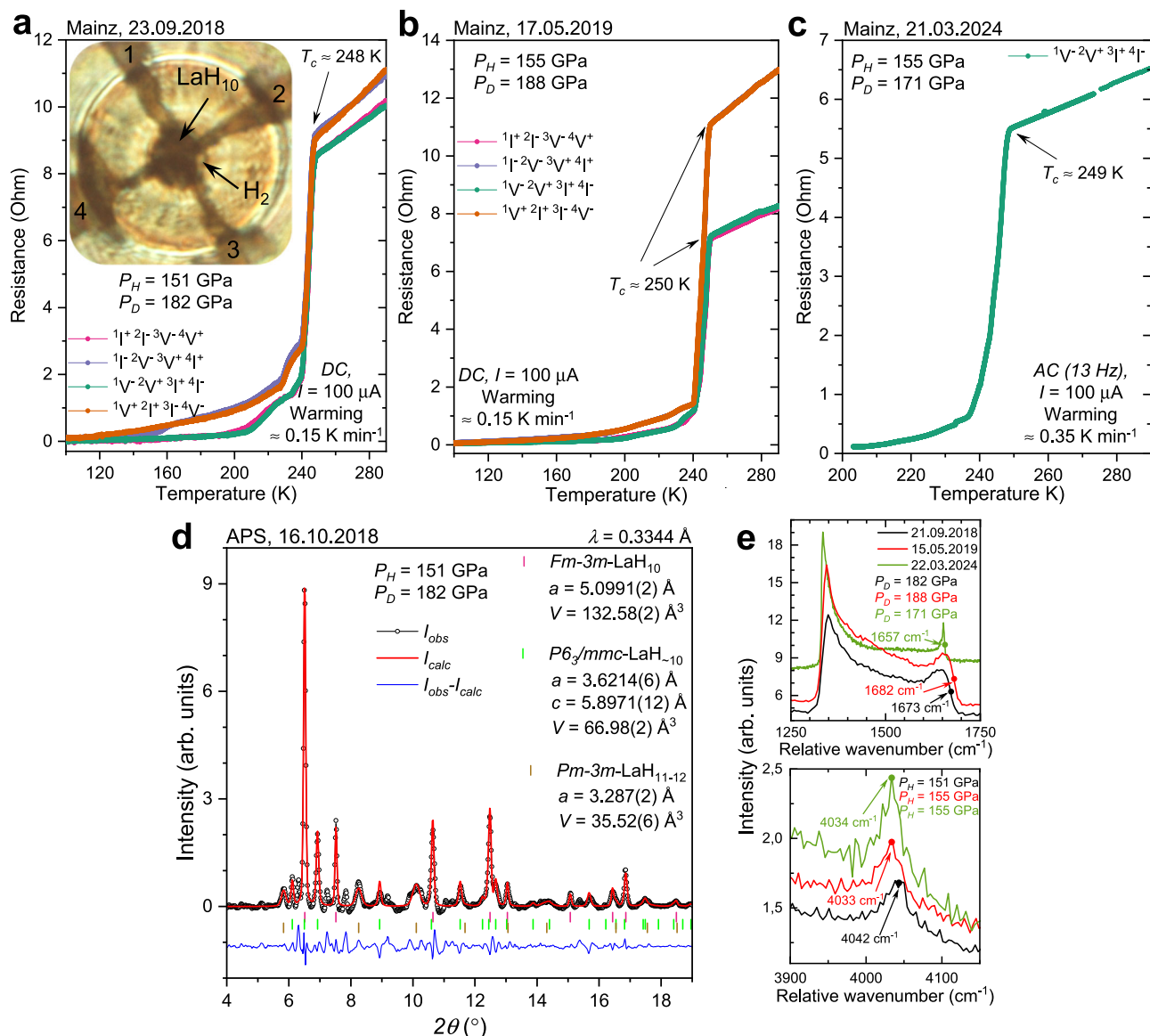


Fig. 3 | Long-term stability of the superconducting properties of the *Fm-3m-LaH₁₀* phase. **a–c** Electrical resistance $R(T)$ measurements for sample V taken respectively 2 days, 8 months, and 66 months after synthesis, showing a superconducting transition at approximately 250 K. **d** X-ray powder diffraction patterns for sample V collected about 1 month after the synthesis. The black circles represent the experimental data, while the red and blue profiles correspond to the

simulated pattern and the Le Bail refinement residuals, respectively. The magenta, green, and brown ticks indicate the calculated positions of the diffraction peaks for the *Fm-3m-LaH₁₀*, *P6₃/mmc-LaH₋₁₀*, and *Pm-3m-LaH₁₁₋₁₂* phases, respectively. **e** Raman spectra, demonstrating the evolution of the estimated pressure in the diamond anvil cell over time, as manifested in the shift of the diamond Raman line edge (top) and the hydrogen vibron (bottom).

Figure 3 summarizes the experimental data for sample V, synthesized from a La piece and H₂ using laser heating at $P_H = 151$ GPa ($P_D = 182$ GPa) on September 21, 2018. Subsequent $R(T)$ measurements revealed a superconducting transition at $T_c \approx 248$ K (see Fig. 3a), and X-ray diffraction confirmed that the sample contained a dominant *Fm-3m-LaH₁₀* phase with impurities of the *P6₃/mmc-LaH₋₁₀* and *Pm-3m-LaH₁₁₋₁₂* phases (see Fig. 3d).

A crucial experimental observation is the resistive $R(T)$ data in Fig. 3, showing that the superconducting transition was consistently reproduced 8 and 66 months after synthesis, with T_c values remaining around 250 K. Minor deviations in T_c are likely because of slight pressure variations in the diamond anvil cell over time.

Another significant experimental fact is that the LaH₁₀ phase remained intact even in the pressure range of 120–150 GPa^{10,14}, where the *Fm-3m-LaH₁₀* phase undergoes minor structural distortion into the *C2/m-LaH₁₀* phase¹⁰. This sample, synthesized at a P_H of 138 GPa via

laser heating on October 30, 2018, exhibited $T_c \approx 243$ K in $R(T)$ measurements conducted on the following day. X-ray diffraction experiments at APS confirmed the presence of the dominant *Fm-3m-LaH₁₀* phase on February 01, 2019. During transportation to the Los Alamos Magnet Laboratory, the sample pressure dropped to 120 GPa. Subsequent $R(T,H)$ measurements on April 19, 2019, and X-ray diffraction on May 31, 2019, revealed monoclinic distortions in the *Fm-3m-LaH₁₀* phase and a significantly lower $T_c \approx 191$ K. However, recompression to 136 GPa on June 19, 2019, restored $T_c \approx 241$ K, and the sample's superconducting transition was studied under high magnetic fields at Los Alamos until July 26, 2019. This study was published in ref. 10

These results demonstrate that even decompression down to 120 GPa does not lead to the decomposition of LaH₁₀ into phases with lower hydrogen content over several months. Thus, the contradictions with ref. 1 cannot be attributed solely to differences in the pressure scale used in their experiments.

Furthermore, it is important to note that no corroborating measurements, such as X-ray diffraction, were performed to verify the presence of the *Fm-3m*-LaH₁₀ phase in the samples analyzed via NMR in ref. 1. This is a critical issue, as NMR alone cannot unambiguously determine the number of phases and their compositions in a multiphase sample. In metal superhydride systems, structural characterization is essential, as different synthesis conditions can yield different phase compositions, particularly when hydrogen is not supplied in excess^{11,14,20–22}.

In addition to the NMR study, the authors¹ reported electrical resistance measurements for four samples from a separate set of experiments. However, all samples exhibited very broad resistive transitions with T_c values significantly below 250 K, indicating the absence of the *Fm-3m*-LaH₁₀ phase: two samples showed $T_{c,onset} < 160$ K, while other two had $T_{c,onset} < 80$ K, while $T_{c,zero} < 65$ K for all samples in ref. 1. Likewise, these samples were not structurally characterized, leaving their phase compositions uncertain.

Three different unannealed and annealed samples of the *Fm-3m*-LaH₁₀ phase (samples I, IV, and V) investigated in the present work exhibited T_c values in the range of approximately 246–250 K. We attribute these differences primarily to variations in pressure among the samples, namely $P_H = 140$ –160 GPa ($P_D = 164$ –188 GPa), according to the known pressure-dependence of T_c in the *Fm-3m*-LaH₁₀ phase¹¹. However, we cannot exclude the possibility that differences in the temperature-sweeping rate also contribute to variations of a few Kelvin in the measured transition temperature. A systematic study of the effect of temperature-sweeping rate on thermal hysteresis is presented in ref. 16.

Reference 1 also describes a sample in which T_c dropped from approximately 150 K to 80 K within a few days, attributing this to hydrogen desorption from the superconducting phase. However, this argument is flawed, as an increase in hydrogen content can also result in a decrease in T_c under certain conditions. For example, first-principles calculations²³ predict a systematic decrease of T_c with increasing hydrogen content in LaH_x hydrides with $6 \leq x \leq 8$: at $P = 180$ GPa, $T_c(R\text{-}3m\text{-LaH}_6) = 183$ –211 K, $T_c(C2/m\text{-LaH}_7) = 158$ –185 K, and $T_c(C2/m\text{-LaH}_8) = 106$ –127 K. Similarly, the predicted *P6/mmm*-LaH₁₆ phase exhibits a lower T_c of 156 K at $P = 200$ GPa than *Fm-3m*-LaH₁₀, despite its higher hydrogen content²³. Further examples include the *P4/nmm*-LaD₁₁ and *Pm-3m*-LaH_{11–12} phases, which were experimentally identified in a previous study¹¹. Although these phases have H(D)/La ratios exceeding 10, their superconducting properties are inferior to those of the *Fm-3m*-LaH₁₀ (LaD₁₀), most likely because they contain quasimolecular H₂ units rather than atomic hydrogen^{3,24}. Samples containing the *P4/nmm*-LaD₁₁ phase (see samples 8, 15, 16, and 17 in ref. 11) systematically demonstrated lower T_c s than samples containing the *Fm-3m*-LaD₁₀ phase (see samples 17 and 18 in ref. 11). The most illustrative example is multiphase sample 17 (see ref. 11): initially, it contained the *Fm-3m*-LaD₁₀ phase and exhibited superconductivity with $T_c \approx 180$ K at $P = 152$ GPa. However, after several subsequent laser-heating treatments, the *Fm-3m*-LaD₁₀ phase transformed to the *P4/nmm*-LaD₁₁ phase together with the same impurity phases, while T_c decreased to 155–160 K at $P = 147$ GPa. In contrast, the *Pm-3m*-LaH_{11–12} phase had an appearance of a semitransparent insulator (see sample 21 in ref. 11).

In summary, we have presented experimental evidence demonstrating that high-temperature superconducting metal superhydrides, namely *Im-3m*-YH₆ and *Fm-3m*-LaH₁₀, are more thermodynamically stable under their formation conditions than their corresponding metal trihydrides, in agreement with theoretical predictions. Furthermore, the hydrogen-rich *Fm-3m*-LaH₁₀ phase has been found to remain stable for at least 66 months after synthesis, maintaining both its crystal lattice and superconducting

properties (T_c), as confirmed by X-ray diffraction and four-probe electrical resistance measurements.

Data availability

The source data that support the findings of this study are provided in this paper. Data from the experiment at ID27 at ESRF is available at <https://doi.org/10.1515/ESRF-DC-2142507569>. Source data are provided with this paper.

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Acknowledgements

V.S.M., M.A.K., P.K., A.P.D., F.D., and J.Y. gratefully acknowledge Mikhail I. Eremets for his longstanding guidance and support, including assistance with sample preparation, electrical resistance and X-ray diffraction measurements, as well as insightful discussions of the results presented here. V.S.M., A.P.D., and F.D. express their gratitude to the Max Planck community for their support and to Prof. Dr. U. Pöschl for his continuous encouragement. The authors also thank Dr. Danila A. Barskiy for valuable discussions. The authors acknowledge the Advanced Photon Source (APS) at Argonne National Laboratory and the European Synchrotron Radiation Facility (ESRF) for providing synchrotron radiation facilities at beamline GeoSoilEnviro CARS, Sector 13 (proposals GUP-58419, GUP-65644, GUP-67864) and beamline ID-27 (proposal HC-5940).

Author contributions

V.S.M., M.A.K., P.K., and A.P.D. prepared the samples and conducted electrical resistance measurements. F.F.B. performed electrical resistance measurements under high magnetic fields in Los Alamos. V.S.M. and M.A.K. collected X-ray diffraction data with assistance from F.D., J.Y., J.K., G.A.S., V.B.P., S.C., M.M., and B.W. V.S.M. and M.A.K. processed the structural data. V.S.M., M.A.K., and E.F.T. wrote the manuscript with contributions from all co-authors.

Funding

M.A.K. acknowledges funding from the European Research Council under the European Union's Horizon 2020 Research and Innovation Program (Grant Agreement 948895, "MetElOne"). E.F.T. acknowledges support from the Ministry of Science and Higher Education of the Russian Federation within the framework of the state assignment for the IMP UB RAS, as well as research funding from the Ministry of Science and

Higher Education of the Russian Federation under the Ural Federal University Development Program (Priority-2030). The National High Magnetic Field Laboratory is supported by the National Science Foundation through NSF/DMR-2128556, the State of Florida, and the U.S. Department of Energy.

Competing interests

The authors declare no competing interests.

Additional information

Supplementary information The online version contains supplementary material available at <https://doi.org/10.1038/s41467-026-76475-7>.

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Peer review information *Nature Communications* thanks the anonymous reviewers for their contribution to the peer review of this work.

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