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ABSTRACT

The recent report of superconductivity in nitrogen-doped lutetium hydride (Lu-H-N) at 294 K and 1 GPa brought hope for long-sought-after ambient-condition superconductors. However, the failure of scientists worldwide to independently reproduce these results has cast intense skepticism on this exciting claim. In this work, using a reliable experimental protocol, we synthesized Lu-H-N while minimizing extrinsic influences and reproduced the sudden change in resistance near room temperature. With quantitative comparison of the temperature-dependent resistance between Lu-H-N and the pure lutetium before reaction, we were able to clarify that the drastic resistance change is most likely caused by a metal-to-poor-conductor transition rather than by superconductivity. Herein, we also briefly discuss other issues recently raised in relation to the Lu-H-N system.

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Since the discovery of superconductivity in mercury at ~4 K in 1911, searching for materials with superconductivity at higher temperatures closer to practical conditions has been a primary and enduring goal. The recent report of room-temperature superconductivity at near-ambient pressure in nitrogen-doped lutetium hydride (Lu-H-N) by Dasenbrock-Gammon *et al.*¹ (hereafter referred to as D-G) seems a great step approaching this ultimate goal. Specifically, they claimed “evidence of superconductivity on a nitrogen-doped lutetium hydride with a maximum T_c of 294 K at 10 kbar.” However, the failure to observe the drastic temperature-dependent resistance change above 200 K—a prerequisite for superconductivity—in high-pressure synthesized Lu-H-N compounds by researchers worldwide in all independent follow-up studies^{2–8} casts a heavy shadow on the authenticity of the claims. There are three sober questions relating to this work, which are as follows. What is the sample that produces the sharp resistance jump near room temperature? What are the reasons for the non-reproducibility of D-G’s results by others who have

followed their method of synthesis and for the inscrutable low success rate (35%) in synthesizing the right sample even for the authors of Ref. 1? What causes the observed sharp resistance jump?

In our work, using a well-controlled experimental protocol, we repeatedly reproduced the near-room-temperature sudden change of electrical resistance in the Lu-H-N sample, and we quantitatively compared this behavior with the initial pure Lu in a normal metallic state. These results enabled us to scrutinize the origin of the sharp resistance change, which we attribute to a metal-to-poor-conductor transition rather than to superconductivity.

We developed a reliable protocol to minimize the extensive range of possible extrinsic influences on the sample^{1,9} by starting the reaction from a piece of pure Lu foil loaded with an H₂/N₂ gas mixture, and we conducted *in situ* Van der Pauw four-probe resistance measurements to monitor the entire reaction process in a diamond anvil cell (DAC) under different pressures and temperatures using a Physical Property Measurement System (Quantum Design). In this way, the temperature-dependent resistance of the sample (including

the initial pure Lu metal and subsequently the reacted Lu-H-N) at target pressures with different reaction times can be obtained without extra sample changes, manipulation, or exposure to air (more experimental details can be found in the supplementary material). Micrographs of the Lu foil with the H_2/N_2 gas mixture and a Van der Pauw four-probe circuit at ~ 10 GPa and room temperature (295 K) are shown in the insets of Fig. 1.

The initial resistance–temperature curve (blue squares in Fig. 1) shows a positive temperature coefficient of resistance and residual resistance below ~ 15 K, demonstrating a typical normal metallic state of Lu. However, after 5 days holding the sample at ~ 10 GPa and 295 K, an apparent reaction between Lu and the H_2/N_2 gas mixture is evidenced by the sample color change (from silver to dark blue), the slightly increased sample size, and the shrunken sample chamber (a considerable amount of H_2/N_2 gas was consumed). Indeed, the temperature-dependent resistance curve of the reacted sample (red circles in Fig. 1) becomes quite different from the initial curve obtained from the pure Lu metal: a sharp upsurge in resistance emerges at ~ 250 K during warming.

Although this confirms the sharp resistance transition reported by D-G,¹ it should be emphasized that with the measurement of the initial pure Lu metal as the baseline, we can conclude that the entirety of the resistance curve—both above and below the sharp change at ~ 250 K in the reacted sample—is higher than that obtained from the initial pure Lu metal. In other words, the sharp change is

caused by the sample forming a much worse metal above 250 K. Its resistance at temperatures below 250 K is still higher than that of pure Lu, contradicting the claim of superconductivity. This experiment has been repeated and reproduced without failure. The sharp resistance change has also been observed when lowering the pressure to 5 and 2 GPa.

The temperature dependence of the resistance of the reacted sample below ~ 230 K is linear and basically parallel to that of the initial Lu metal. Therefore, the treatment of the low-temperature linear part of the curve as an extra meaningless system background by the authors of Ref. 1 to derive zero resistance is not justified. The hallmark—and indeed the most desirable property of a superconductor—is its zero electrical resistance, which enables an electric current to flow without any energy dissipation. In this respect, the absence of zero resistance and the presence of an upsurge in resistance rules out the possibility of a superconducting transition in the Lu-H-N system and makes this Lu-H-N sample interesting only as a poor metal or semiconductor, not a superconductor.

In addition, the sample color change from blue to pink was claimed in Ref. 1 to be a critical feature associated with the sudden resistance change. Therefore, many groups worldwide have devoted tremendous efforts to searching for this pink crystal with superconductivity.^{4,5,8,10,11} Although researchers have synthesized blue Lu-H-N samples using various methods and observed the blue-to-pink transition during compression, they all failed to reproduce the near-room-temperature sharp resistance transition.^{2,4–8,12} The authors of Ref. 1 did not show sample micrographs with clear details of how the four platinum (Pt) leads were connected to the sample or how the temperature-dependent resistance gradually evolves with sample color changes. Our results indicate that it is unnecessary to link the blue-to-pink color change with a sharp resistance transition, as is consistent with previous conclusions relating to non-superconducting pink Lu-H-N samples.² The micrographs in a very recent paper (Figs. 1 and S1 in Ref. 13) using the same sample provided by the authors of Ref. 1 reveal that the D-G sample showing the sharp resistance change is not a pure substance but actually an uneven composite mixture; it has an undefined texture and ratio of metallic and insulating phases showing shining metallic, gray, black, and pink colors under reflective lighting, rather than a pure pink phase as shown in the inset of Fig. 2(a) of Ref. 1. The low success rate of D-G *et al.*¹ and lack of success by others^{2,4–8,12} are consistent with the expected resistance of a random multiple metal–insulator phase mixture. Clearly, zero resistance cannot be defined in such a mixture, and a controlled baseline, such as the resistance of the initial pure Lu before the reaction as measured in the present work, is essential for consideration of superconductivity.

In summary, by taking the approach of *in situ* resistance measurements of pure Lu foil during its reaction with the H_2/N_2 gas mixture within a broader pressure and temperature space, we confirmed that the abrupt resistance transition near room temperature can be reproduced in the dark-blue Lu-H-N sample but not under the experimental conditions provided in Ref. 1. The increase of the sample resistance after the reaction when compared with that of the initial pure Lu metal rules out attributing the observed sudden resistance change to any superconducting transition. With the concrete and reproducible results in this work, we hope that the scientific community will be able to invest its precious time and resources in the right direction and work toward addressing the many

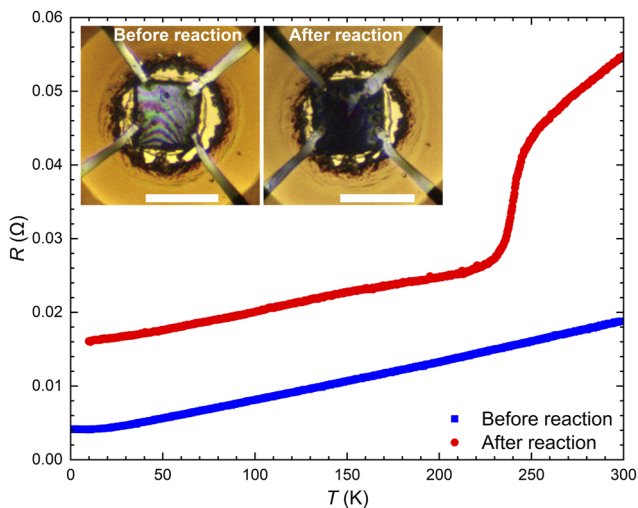


FIG. 1. *In situ* electric resistance measurements of the Lu foil sample loaded with H_2/N_2 gas mixture in a DAC at 10 GPa and 295 K. The insets present optical micrographs of the Lu foil sample as loaded (left) and after a 5-day reaction (right), showing the four Pt electrodes. The metallic luster of the surface of the Lu foil sample changes to dark blue after 5 days of reaction. The colorful fringes are due to light interference between the gap from the sample to the diamond-anvil surface, which becomes invisible after reaction mainly due to much less light being reflected by the dark sample surface. The obvious consumption of H_2/N_2 gas mixture was reflected by sample-chamber shrinkage after 5 days of reaction. The sample size also slightly increased after the reaction. The blue and red curves show the temperature-dependent raw resistance values during warming from 2 K (10 K) to 300 K for the initial pure Lu metal before reaction and the same sample after reacting for 5 days at 10 GPa and 295 K, respectively.

questions raised by the Lu-H-N system other than its ostensible near-room-temperature, ambient-pressure superconductivity.

See the supplementary material for experimental details.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

Di Peng: Data curation (equal); Formal analysis (equal); Investigation (equal); Methodology (equal); Writing – original draft (equal). **Qiaoshi Zeng:** Conceptualization (equal); Formal analysis (equal); Investigation (equal); Methodology (equal); Supervision (equal); Writing – original draft (equal); Writing – review & editing (equal). **Fujun Lan:** Data curation (equal); Formal analysis (equal); Investigation (equal); Methodology (equal). **Zhenfang Xing:** Data curation (equal); Formal analysis (equal); Investigation (equal). **Yang Ding:** Formal analysis (equal); Validation (equal); Writing – review & editing (equal). **Ho-kwang Mao:** Conceptualization (equal); Formal analysis (equal); Supervision (equal); Writing – review & editing (equal).

DATA AVAILABILITY

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

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