

Percolation-induced resistivity drop in lutetium dihydride with controllable electrical conductivity over six orders of magnitude

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The recent report of near-ambient superconductivity in the nitrogen-doped lutetium hydride has attracted considerable attention. Subsequent follow-up studies confirmed the pressure-induced color changes in both N-free and N-doped LuH₂ but failed to reproduce superconductivity. It remains a puzzle why the samples in the original report exhibited pronounced resistance anomaly reminiscent of the superconducting transition. Here, we show that percolation of metallic grains with high conductivity through the insulating surfaces in cold-pressed LuH₂ samples can occasionally produce sharp resistance drops, which even display magnetic field and/or current dependences but stay far from zero resistance. The insulating surface of LuH₂ grain should be attributed to the modification of hydrogen stoichiometry or the contamination by oxygen/nitrogen, resulting in an increase of resistance by over six orders of magnitude. Such an effect is more significant than that discovered recently in LaH_{3±x}, which may indicate that LuH₂ can be a potential superionic conductor. Our results call for caution in asserting the resistivity drops as superconductivity and invalidate the background subtraction in analyzing the corresponding resistance data.

LuH₂, percolation, resistance drop, superconductivity, superionic conductor

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1 Introduction

Room-temperature superconductivity has been a long-sought

goal ever since the discovery of superconductivity in 1911. The recent report of near-ambient superconductivity in nitrogen (N)-doped lutetium (Lu) hydride [1] has thus attracted considerable attention [2–18] since it, if proven to be true, would represent a milestone towards the ultimate goal. According to this report, the samples recovered from the reaction of a thin Lu foil with H₂/N₂ (99:1) mixture at 2 GPa and

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65°C consist of a major cubic $\text{LuH}_{3-\delta}\text{N}_\epsilon$ with the minor phases of $\text{LuN}_{1-\delta}\text{H}_\epsilon$ and Lu_2O_3 . In addition to a high superconducting critical temperature (T_c) of ~ 294 K at 1 GPa, the sample was observed to exhibit visible color changes from blue through pink to bright red upon compression. The reported high- T_c superconductivity was found to exist only in the pink-colored phase in the pressure range of 0.3-3 GPa and seems to be well supported by the electrical transport, magnetic, and thermodynamic measurements in ref. [1].

However, subsequent follow-up studies immediately cast doubts on the results of this report [2-12]. For example, a density-functional-theory study on structural stability and optical absorption suggested that the parent structure of the N-doped Lu hydride should be LuH_2 with the cubic fluorite structure rather than the originally proposed cubic LuH_3 [2]. Pressure-induced color changes from blue through pink to bright red were indeed verified in both N-free [9] and N-doped LuH_2 [3,5], and the underlying mechanism was revealed through the optical reflectivity measurements under high pressures [16]. However, no superconductivity was observed down to 1.5 K under pressures up to 50 GPa [3,5,6,9,10], in striking contrast to the original report [1]. For the $\text{LuH}_{2\pm x}\text{N}_y$ samples prepared with the large-volume press, some kink- or hump-like features were observed in the temperature-dependent resistivity around room temperatures at high pressures [3,5]. But these features are distinct and far weaker than those reported in ref. [1] showing an abrupt drop in resistivity. Since the essential feature of resistivity drop in ref. [1] has not been reproduced, the debate on the mystery of near-ambient superconductivity remains unsettled [3,5].

Since the available experimental and theoretical investigations consistently ascertained the actual chemical composition of the samples in ref. [1] as N-doped LuH_2 [2-5,13,14], we decided to carry out comprehensive investigations on the parent compound LuH_2 . During the course of our study, we noticed that the resistivity of cold-pressed (CP) LuH_2 pellets made of commercially purchased powders can vary sensitively upon modifying the grain size or surface conditions via the grinding process. Surprisingly, we can occasionally yet repeatedly observe abrupt resistivity drops at high temperatures, which also show dependences of magnetic fields and/or electrical current, reminiscent of the observations in ref. [1]. However, our detailed investigations exclude the possible causes of structural, magnetic, or superconducting transition for the observed resistivity drop. Instead, we attribute it to the percolation of the metallic grains through the insulating layers on the grain surface, which are likely produced by the modification of hydrogen stoichiometry or the pollution by oxygen/nitrogen in the air. The present work calls for caution in asserting the resistivity drops as superconductivity, especially for those samples involving multiple components. Meanwhile, our results show that LuH_2 is an ideal platform to study the percolation phe-

nomenon because its surface contribution to resistance can be controlled by the grinding process. We found that its electronic conductivity can be reduced by over six orders of magnitude via the simple grinding process, which is more pronounced than that discovered recently in the nanosized $\text{LaH}_{3\pm x}$ [19], making LuH_2 a potential superionic conductor.

2 Results

It has been reported that the stoichiometric bulk LuH_2 is a paramagnetic metal with high electrical conductivity comparable to simple metals. To evaluate the intrinsic transport properties of LuH_2 , we picked up a large grain of ~ 200 μm in length from the as-received powder samples (See the [Supporting Information](#) (SI) for details about the samples) and measured its temperature-dependent resistivity, $\rho(T)$, by using the standard four-probe method. The inset of Figure 1(a) shows the photograph of the measured grain attached with four gold leads by silver paste. As shown in Figure 1(a), the LuH_2 grain displays a typical metallic behavior with a small room-temperature resistivity of $\sim 9.3 \times 10^{-6}$ Ωcm , which is on the same order as high-purity copper. The resistivity decreases upon cooling down and saturates below ~ 20 K to a constant value of $\sim 0.15 \times 10^{-6}$ Ωcm , giving rise to a relatively large residual resistivity ratio $\text{RRR} \equiv \rho(300\text{K})/\rho(2\text{K}) = 62.7$. The $\rho(T)$ below 40 K can be described by the power law, viz. $\rho(T) = \rho_0 + AT^n$ with $n = 3.5$, as shown by the dotted line in Figure 1(a). The obtained larger n than the typical Fermi-liquid behavior ($n = 2$) indicates that the electron-phonon interactions might play an important role in addition to the electron-electron interactions in LuH_2 at low temperatures. These results are comparable to those reported previously [20,21] and thus confirmed an intrinsic metallic behavior with high conductivity for the blue-colored LuH_2 .

When the as-received LuH_2 powder with different grain sizes ranging from ~ 200 μm to submicron was directly cold pressed at 4 GPa into dense pellets by using a large-volume press, we find that its resistivity is enhanced by almost four orders of magnitude in comparison with that of the above LuH_2 grain. The samples made in this way are labeled as “As-received+CP” hereafter. We measured $\rho(T)$ of over ten samples and the $\rho(T)$ curves for four representative samples shown in Figure 1(b). Although the $\rho(T)$ still shows metallic behavior, the room-temperature resistivity has been increased significantly to the level of $2\text{-}5 \times 10^{-2}$ Ωcm , and the RRR value is reduced significantly to 1.1-1.3. As mentioned above, the as-received powder contains LuH_2 grains of different sizes, such enhanced resistivity should be attributed to the presence of submicron-sized grains, which contributed significantly to resistivity due to grain boundary scatterings. However, the enhancement of $\rho(T)$ by nearly four orders of magnitude is surprisingly large, which indicates that the

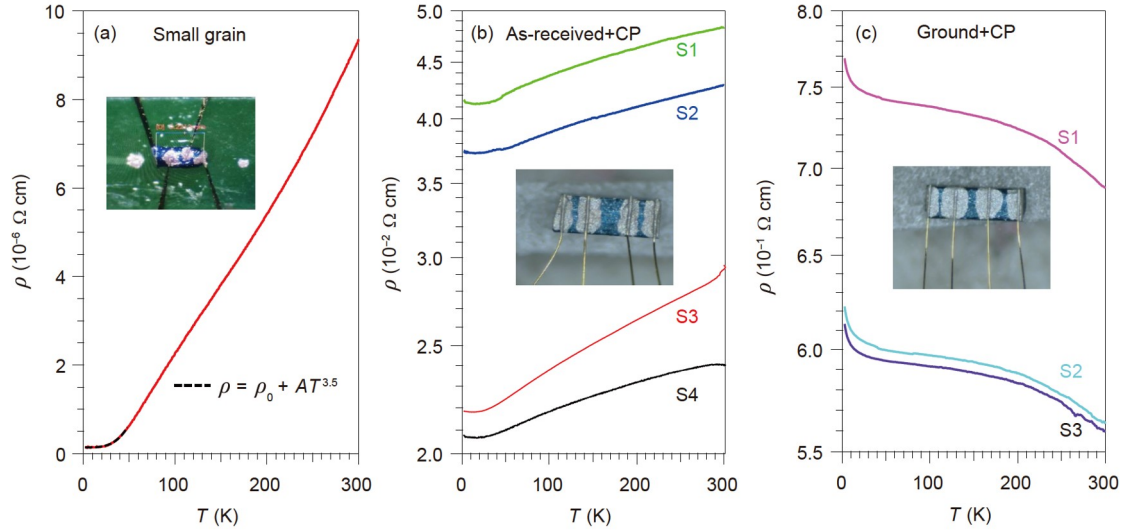


Figure 1 (Color online) Temperature-dependent resistivity $\rho(T)$ of LuH_2 samples: (a) the small grain picked up from the as-received powder; (b) the cold-pressed samples made of the as-received powder; (c) the cold-pressed samples from the ground LuH_2 powder. The inset shows the samples' photographs for measuring resistivity.

surface of the LuH_2 grains scatters electrons strongly.

During the $\rho(T)$ measurements of “As-received+CP” LuH_2 samples, we occasionally observed in one sample a pronounced resistivity drop at about 200–250 K as shown in Figure 2(a). In comparison with the typical samples shown in Figure 1(b), this sample is featured by a larger resistivity of $\sim 1.6 \Omega \text{ cm}$ at room temperature. For the first measurement upon cooling down under 0 T, its $\rho(T)$ starts to decrease quickly from ~ 250 K and then levels off below ~ 212 K, showing about a 10% drop in resistivity. This anomaly shows up repeatedly during the thermal cycling between 300 and 2 K, and it can even be shifted down by magnetic fields. At first glance, this anomaly seems to signal the occurrence of superconductivity, which, however, is not supported by our following studies. For this specific sample, this anomaly is rather robust and always shows up for many runs at different currents, Figure 2(b). It is noticeable that the anomaly moves to higher temperature at larger current and the magnitude of resistivity also varies for different runs.

To verify if the observed resistivity drops correspond to a superconducting transition, we measured the dc magnetic susceptibility of the same sample used for the above resistivity measurements. Before the susceptibility measurements, the electrical leads were detached, and the sample surface was slightly polished to remove the silver paste. Unfortunately, we did not observe any feature due to the Meissner effect within the limit of our high-resolution SQUID. Then, we reattached electrical leads on the same sample to measure its resistivity again. To our surprise, the anomalous drop in resistivity disappears and cannot be reproduced on the identical sample. This puzzled us and motivated us to investigate the underlying mechanism to

reproduce the resistance drops in the cold-pressed LuH_2 .

As mentioned above, we noticed that the “As-received+CP” LuH_2 samples show a ten-thousand times larger resistivity and the specific sample showing resistivity drop possesses an even larger room-temperature resistivity in comparison with that of the bulk grain. We then realized that it could be an effective way to further enhance the resistivity of LuH_2 by reducing the grain size or increasing the surface area. To test this hypothesis, we manually ground the as-received powder for 5 min in the air with a mortar and pestle, and then cold-pressed it into dense pellets at 4 GPa as done above. This sample is labeled as “Ground+CP” to distinguish it from the above “As-received+CP” sample. As shown in Figure 1(c), the $\rho(T)$ of most “Ground+CP” samples increase slightly upon cooling down, displaying a weakly localized non-metallic behavior. In addition, the room-temperature resistivity also increases further by one order of magnitude to $\sim 7 \times 10^{-1} \Omega \text{ cm}$. This result demonstrated that the additional grinding process is effective in modifying the transport properties of LuH_2 samples.

Interestingly, for some “Ground+CP” LuH_2 samples, we can reproducibly observe the sharp resistivity drop as shown in Figure 3 for a representative example. The panels in Figure 3 are displayed in the same order as the measurement sequences. As seen in Figure 3(a), the $\rho(T)$ at 0 T exhibits an abrupt drop at 50 K during the first cooling-down process while it jumps back at ~ 200 K during the warming-up process, resulting in a significant thermal hysteresis. Upon increasing magnetic field to 7 T at room temperature and measuring $\rho(T)$ again, Figure 3(b), the resistivity anomaly takes place at ~ 160 and 210 K for the cooling-down and warming-up processes, respectively. The positive field effect

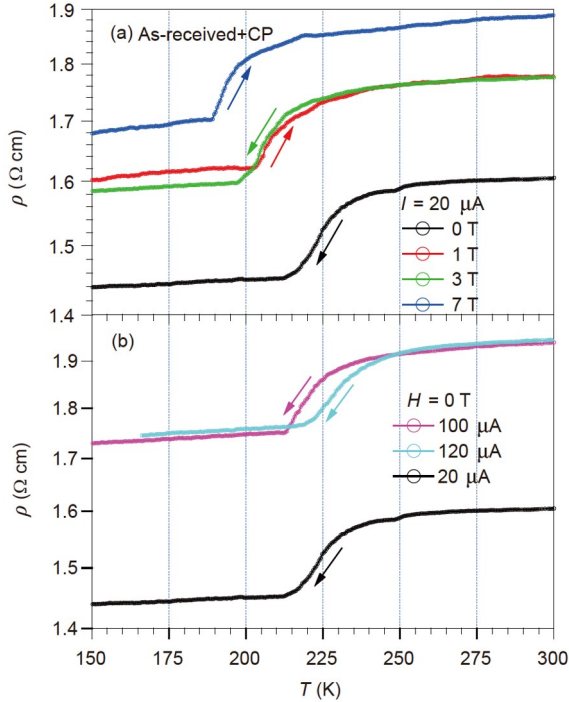


Figure 2 (Color online) Temperature-dependent resistivity $\rho(T)$ of one “As-received+CP” LuH_2 sample that shows resistivity drop: (a) measured at different magnetic fields with the same electrical current, and (b) measured at zero field with different electrical currents.

thus excludes the possibility of superconductivity. When the field is reduced to zero and the current is increased from 20 to 100 μA , Figure 3(c), the anomalous drop in resistivity still stays at high temperatures of 150 and 230 K with a slightly wider hysteresis. Finally, when the current is reduced to the original 20 μA , Figure 3(d), however, the hysteresis becomes much reduced while keeping the resistivity drop at 150–170 K. Similar behaviors can be reproduced on another sample, Figure S2, but the temperatures of the resistivity anomalies are different. In comparison with the “As-received+CP” samples, the “Ground+CP” samples with larger resistance show a high probability to present resistivity drops. We have measured a total of six “Ground+CP” samples, and two of them exhibited a pronounced drop in resistivity. However, such an abrupt resistivity drop does not follow any regular dependences of the magnetic field or electrical current according to our results. This indicated that it is not an intrinsic effect caused by the magnetic field but takes place randomly due to a combined effect of thermal and magnetic field variations. In addition, all resistivity measurements in this work were carried out by using the commercial PPMS in the standard way, with a continuous sweep of temperature at a rate of 2 K/min. The difference between the instrument thermometer and the sample’s real temperature should be negligible or much smaller than the hysteresis observed here.

To clarify the origin of such a resistivity-drop anomaly, we first checked the powder XRD at room temperature for these

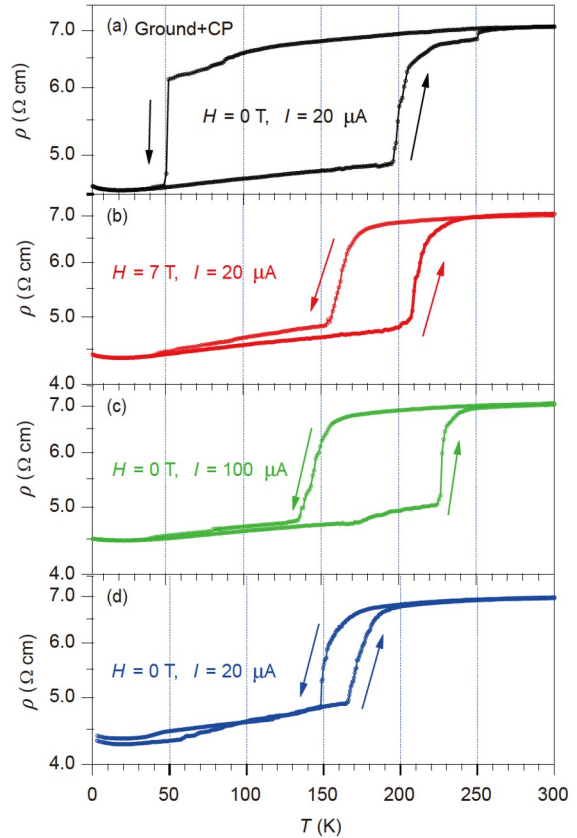


Figure 3 (Color online) Temperature-dependent resistivity $\rho(T)$ of one “Ground+CP” LuH_2 sample with 5-min grinding. Panels (a) to (d) are organized in the same order as the measurement sequences. The arrows indicate the cooling-down and warming-up processes.

three samples. As shown in Figure S3, both the XRD patterns and the calculated lattice parameters for these samples are almost identical, while the peaks of the CP samples are slightly broadened up due to the presence of stress/strain introduced by the grinding and cold-pressing processes. Then, we further measured variable-temperature XRD of the ground powder from 300 down to 100 K, Figure 4(a). The absence of any peak splitting and smooth evolution of lattice parameters as a function of temperature rule out the structural phase transition for LuH_2 in the investigated pressure range. Linear fitting to the temperature dependence of unit-cell volume $V(T)$ yields a thermal expansion coefficient (TEC) of $\alpha_V = 11.98 \times 10^{-6} \text{ K}^{-1}$, or a linear TEC of $\alpha_L = 3.98 \times 10^{-6} \text{ K}^{-1}$, Figure 4(b). We also measured the temperature dependence of magnetic susceptibility for these samples, Figure S4, which show similar paramagnetic behaviors without discernable anomalies in the temperature range 2–350 K, thus excluding the possibility of magnetic transition as the cause of the sudden resistivity drop.

Then, we probe the intrinsic electronic properties of the CP samples made from as-received and ground powders via specific-heat measurements. As seen in Figure 5(a), except for a dip anomaly around 220 K associated with the Apiezon

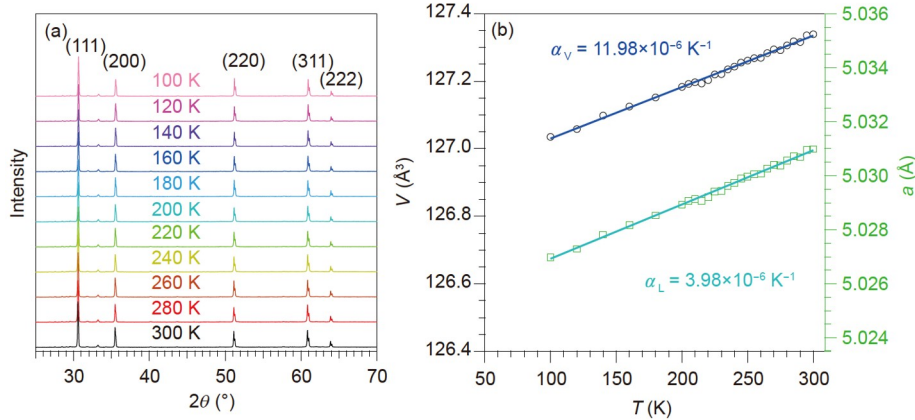


Figure 4 (Color online) (a) Variable-temperature XRD of the ground powder from 300 down to 100 K. (b) Temperature dependences of unit-cell volume and lattice parameter with linear fitting curves shown by solid lines.

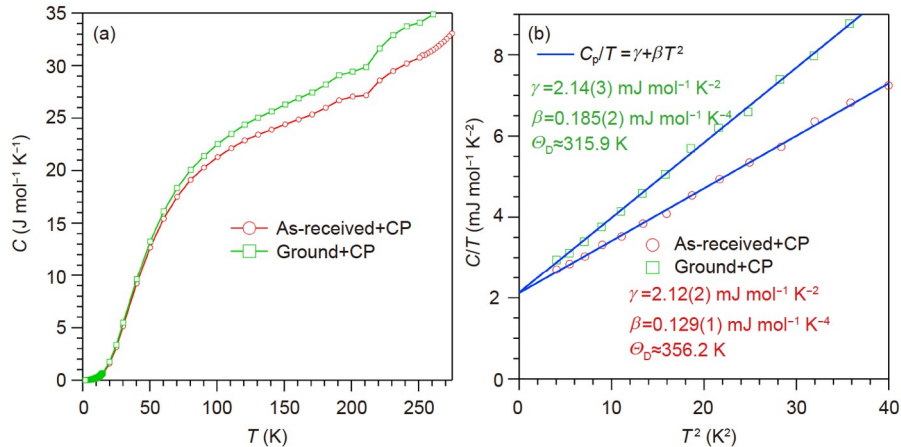


Figure 5 (Color online) (a) Specific heat $C(T)$ for two CP LuH_2 samples without and with additional grinding in the wide temperature range from 2 to 270 K under zero field. (b) The plot of C/T vs. T^2 in the low-temperature range. A linear fitting was applied to extract the Sommerfeld coefficient γ and the Debye temperature θ_D .

N grease, no obvious anomalies can be discerned on the $C(T)$ curves in the whole temperature range, further confirming the absence of structural, magnetic, or superconducting transitions. It is noted that the $C(T)$ of the “Ground+CP” sample is slightly larger than that of the “As-received+CP” sample, especially at high temperatures. The $C(T)$ data below 7 K were replotted in Figure 5(b) in the form of C/T vs. T^2 , which follows nicely a linear behavior due to the lattice and electronic contributions. A linear fitting to the C/T vs. T^2 data yields almost identical Sommerfeld coefficient $\gamma = 2.1 \text{ mJ mol}^{-1} \text{ K}^{-2}$ for these two samples, even though their $\rho(T)$ data in Figure 1(b) and (c) show distinct behaviors. Such a small γ is consistent with the intrinsic metallic ground state for the LuH_2 samples, while the observed weak localization of $\rho(T)$ for the “Ground+CP” sample should be mainly attributed to the strong scattering of electrons by the insulating layers on the grain surfaces.

Although the LuH_2 is stable at ambient conditions, the surfaces of LuH_2 can be easily damaged or contaminated by

oxygen/nitrogen, resulting in an insulating layer on the grain surfaces according to a previous study [21]. Thus, operations of Lu and LuH_2 were usually carried out under mineral oil [22]. In the present study, however, all operations were performed in the air, which can inevitably produce insulating layers due to the modification of hydrogen stoichiometry and/or pollution by oxygen/nitrogen on the grain surfaces. This effect is expected to be magnified and expedited through the additional grinding process, in consistent with the observations of much enhanced resistivity in the “Ground+CP” sample shown in Figure 1(c). To elaborate it, we elongated the grinding time to 30 min and indeed observed further enhancement of resistivity in comparison with that ground for 5 min, as shown in Figure S5. The incorporation of heavier oxygen/nitrogen on the surfaces can partially account for the smaller Debye temperature for the “Ground+CP” sample with respect to that of the “As-received+CP” sample, shown in Figure 5(b). Further controlling experiments, e.g., grinding the LuH_2 samples in an argon-filled

glove box, are needed to clarify the origin or mechanism of the surface degradation.

Therefore, the present work demonstrates that the CP LuH₂ samples consist of metallic grains with high conductivity and insulating surfaces, and their relative ratio can be modified through the grinding process in the air. For the “As-received+CP” sample, the metallic grains still dominate the transport properties and retain the metallic behavior in $\rho(T)$ as observed in Figure 1(b). When the contributions from the insulating surfaces are enhanced by grinding as-received powder, the “Ground+CP” samples usually display a weakly localized $\rho(T)$ behavior as seen in Figure 1(c). Under the right conditions, percolations of the metallic grains through the insulating surfaces can give rise to sudden drops of resistivity upon varying temperatures. As a probability issue, the percolation takes place randomly and it can thus rationalize the observed irregularities of the resistivity drop in the studied “Ground+CP” samples. It seems that the CP LuH₂ sample made of as-received powder after grinding for 5 min gives a high rate to reproduce the resistivity drop for our studied samples. For those CP samples made of well-ground powders, the contributions from the insulating layers become dominant, preventing the occurrence of percolation.

As seen in Figures 2 and 3, the drop of resistivity in the “As-received+CP” sample is about 10%, while the drop in the “Ground+CP” samples can reach as high as 30%. Therefore, the magnitude of the resistivity drop associated with the percolation process seems to be tunable, and the relative volume ratio between the conducting and insulating regions should be an important controlling parameter. As mentioned above, the purpose of the grinding process is to increase the insulating surface so that the resistance drop is enlarged. Given the fact that the resistivity drop disappears once we reattach the electrical leads, it seems that the percolation occurs within a relatively thin layer of the sample’s surface. In this regard, it is highly possible that the resistance drop/jump can be significantly enhanced if the thickness of the sample is reduced, and this effect might be magnified in the thin-film samples. In addition, the observation of a large hysteresis indicates that the percolation occurs through a thermal driven process associated with the contraction/expansion of conducting grains. More studies are needed to address these intriguing issues in the future.

To substantiate the above results, we further evaluated theoretically the electronic and superconducting properties of LuH₂ and related compounds LuH₃ and LuN by performing first-principles calculations under 0 and 10 GPa via density functional theory at the GGA-PBE level (See the SI for details). The calculation results are given in Figure S6 and summarized in Table S1. Here, we set the empirical parameter $\mu^* = 0.1$ for the effective screened Coulomb repulsion. According to our calculations, LuH₂ is metallic and becomes superconducting only at very low temperatures

($T_c = 0.026$ K), in good agreement with ref. [10]. The hydrostatic pressure of 10 GPa cannot help to enhance T_c . The LuH₃ ($Fm\bar{3}m$) is thermodynamically unstable based on our evaluation as the energy above the hull is as high as 91 meV/atom. LuN is also not a superconductor under 0 and 10 GPa.

Based on the above studies, we can conclude that the observed resistance drops in the CP LuH₂ samples have nothing to do with superconductivity, magnetic, or structural phase transitions, but are mostly likely attributed to the percolation of metallic grains through the insulating surfaces. We speculate that the insulating surfaces of LuH₂ originate from the modification of hydrogen stoichiometry or the contamination by oxygen/nitrogen during the grinding process in the air. The reduced Debye temperature on the “Ground+CP” sample is consistent with this scenario. More studies are needed to clarify the mechanism of surface degradation in LuH₂. If this scenario is correct, intermixing of highly conducting LuH₂ with other insulating phases such as LuN can also produce sudden drops in resistivity. The present study thus demonstrates that an abrupt drop in resistivity does not mean the occurrence of superconductivity, which calls for caution in interpreting the anomalies in resistivity. In this case, it is definitely invalid to perform background subtraction in treating the resistivity data.

In addition to the Lu-H-N system, possible high- T_c superconductivity has been claimed in the inhomogeneous systems such as the La-based multiple hydrides with T_c above 550 K [23], the Ag-Au mesoscopic thin film with $T_c \sim 260$ K [24], the Ir-substituted perovskite-type manganese oxides with T_c above 100 K [25], and palladium hydride and deuteride with $T_c \sim 52$ -61 K [26]. These unidentified superconductors unusually exhibit narrow resistance drops or kink-like transitions in the temperature-dependent resistivity $\rho(T)$ with large thermal hysteresis. The lack of the Meissner effect has sparked further debate. In light of the present work, these reports on high- T_c superconductivity in inhomogeneous systems with multiple components deserve further reexaminations.

Furthermore, a recent study has shown that the creation of nanosized grains and defects through high-energy ball milling can suppress the electronic conductivity of LaH_{3±x} by more than five orders of magnitude [19], which is consistent with our results of LuH₂. Such a significant reduction of electronic conductivity transforms LaH_{3±x} to a superionic conductor. It is noteworthy that by simply varying the grinding time in the air, the electronic conductivity of LuH₂ can be suppressed by more than six orders of magnitude as shown in Figure S7, exhibiting a more pronounced effect than that discovered in LaH_{3±x} [19]. Considering that the grain sizes produced by hand grinding the powder in the mortar are still relatively large, the electronic conductivity of LuH₂ is expected to be further reduced if nanosized grains are created. Our results indicate that LuH₂ has the potential to

be a superionic conductor and deserves further investigation.

3 Conclusion

In summary, we demonstrate that the resistivity of polycrystalline LuH_2 is very sensitive to grain conditions and can be modified from metallic to weakly localized behavior with controllable electrical conductivity over six orders of magnitude through a simple grinding process in the air. In light of the recent results on $\text{LaH}_{3\pm x}$, our results indicate that LuH_2 has the potential to be a superionic conductor. By controlling the grinding time, we can repeatedly observe the abrupt resistivity drop at high temperatures in the “Ground+CP” LuH_2 samples. We proposed that it is the percolation of the metallic grains through the insulating layers on the grain surfaces that produces the resistivity drops. Our present results call for caution in asserting the resistivity drop as superconductivity and invalidate the background subtraction in analyzing the resistivity data.

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Conflict of interest The authors declare that they have no conflict of interest.

Supporting Information

The supporting information is available online at <http://phys.scichina.com> and <https://link.springer.com>. The supporting materials are published as submitted, without typesetting or editing. The responsibility for scientific accuracy and content remains entirely with the authors.

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