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Editors' Suggestion

Synthesis and superconductivity in (La, Ca)H_{10} under high pressure

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Tracking room-temperature superconductors represents a significant challenge and opportunity, while the discovery of binary lanthanum hydrides provides a crucial platform for exploring ternary hydrides with exceptional properties. The theoretically studied La-Ca-H system is also one of the attractive candidates for room-temperature superconductivity. In this work, we have successfully synthesized two La-Ca hydrides by doping Ca elements into the La-H system. The temperature dependence of the electrical resistance demonstrates the superconductivity of $Fm\bar{3}m$-(La, Ca)H_{10} and $P6_3/mmc$-(La, Ca)H_{10}, with superconducting critical temperature (T_c) of 247 K (173 GPa) and 230 K (167 GPa), respectively. Present results show that doping with 10% or 15% Ca has only a negligible effect on the T_c of LaH_{10}, which is consistent with Anderson's theory. Although the high T_c phase of La-Ca-H maintains a highly symmetric *fcc* structure, the doping of Ca may lead to premature H-cage distortion during the decompression process, thus affecting the structural stability of the system. Therefore, under the doping levels and synthesis conditions of this work, we think that Ca may not be an ideal dopant. Our study provides some hints on the key factors affecting superconductivity and informs the future search for room-temperature superconductors in more diverse hydride materials.

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I. INTRODUCTION

In the annals of superconductor history, the revelation of mercury's superconducting properties [1] heralded the dawn of humanity's quest for room-temperature superconductivity. In this field, high-pressure hydrides undoubtedly provide a broader scope for exploring high-temperature or even room-temperature superconductivity discoveries. At present, a large number of binary hydrides have been successfully predicted theoretically [2–5] followed by the successful synthesis of several of these hydrides under high-pressure conditions [6–10]. This has significantly enriched the family of high-temperature superconducting hydrides, propelling advancements in the field of superconductivity. Among these hydrides, cagelike hydrides have remarkable superconducting properties and are commonly found in the alkaline-earth and rare-earth metal superhydrides, such as CaH_6 [10,11], LaH_{10} [7,12–14], YH_6 [15], YH_9 [8,16], CeH_9 [9,17,18], and CeH_{10} [9]. Notably, the *fcc*-LaH_{10} with an H_{32} cage structure demonstrates exceptional superconducting properties [7], prompting the immediate desire to enhance its superconducting performance through doping/alloying in hopes of achieving room-temperature superconductivity.

Compared to binary hydrides, ternary hydrides offer a greater diversity in chemical composition, and are expected to be powerful candidates in the field of superconductors [19]. Recently, drawing from prototype structures such as LaH_{10} and CaH_6, multiple research teams have theoretically designed a series of cagelike hydrides, including ABH_8 [20–22] and (A, B)H_6 [23–25]. Theoretical calculations indicate that the superconducting temperatures (T_c) of $LaBeH_8$ [21] and YLu_3H_{24} [23] can reach as high as 185 and 288 K, respectively, at pressures of 20 and 110 GPa. Furthermore, through the substitution of nonmetallic elements B, C, and N for the hydrogen atoms located at the tetrahedral centers within LaH_{10}, a variety of M(B/C/N)$_2H_8$ structures that remain dynamically stable below 100 GPa have been designed [26–29]. In addition, Wang *et al.* discovered that doping 12.5% Ca into the La-H system can shift the Van Hove singularity (VHS) and increase the electronic density of states (DOS) at the Fermi level to increase the T_c of *fcc*-LaH_{10} by 15%, thus achieving room-temperature superconductivity [30]. And Yang *et al.* found that the $C2/m$-$CaLaH_{12}$ structure can be stabilized in the lower pressure range 30–55 GPa through calculations, with a T_c of 49 K at 30 GPa [31]. Unfortunately, despite the proposal of numerous exceptional ternary hydrides in theory, the complex nature of their precursors and phase diversity still presents significant challenges in synthesis and characterization. Therefore, experimental investigations of ternary hydrides are still in their infancy, and only a few ternary hydrides have been reported, such as (La, Y)H_{10} [32], (La, Nd)H_{10} [33], (La, Ce)H_{9-10} [34–36], (La, Al)H_{10} [37], and (La, Be)H_8 [38], etc. Nonetheless, the current body of theoretical and experimental research results suggests that ternary hydrides hold significant potential for enhancing the critical temperature and stability of superconducting systems.

Considering the theoretically proposed ternary La-Ca-H system with possible room-temperature superconductivity, in

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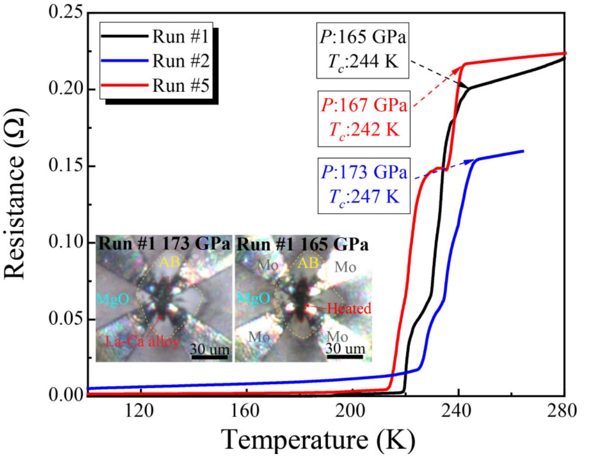

FIG. 1. The electrical resistance measurements of the La-Ca-H system for typical experimental runs. Temperature dependence of electrical resistance of La-Ca-H system under high pressures in runs 1, 2, and 5. Inset: Photographs of the sample before and after laser heating together with four electrodes in run 1.

this work, we aimed to synthesize La-Ca-H with similar proportions and explore its superconductivity. Two La-Ca hydrides were successfully synthesized by doping Ca into the La-H system. The temperature dependence of the electrical resistance demonstrates the superconductivity of $Fm\bar{3}m$-(La, Ca)H_{10} and $P6_3/mmc$-(La, Ca)H_{10}, with T_c of 247 K (173 GPa) and 230 K (167 GPa), respectively. Although the superconducting phase of La-Ca-H still maintains a highly symmetric *fcc* or *hcp* structure, the crystal structure and electronic properties of this system may be quite unique, leading to the failure to increasing its T_c by doping with Ca, at least within the range of doping levels and pressures investigated in this study.

II. RESULTS AND DISCUSSION

In this work, to investigate the formation and transport properties of La-Ca hydrides, we prepared two La-Ca alloys with Ca contents of approximately 10% and 15% by melting La and Ca in an inert atmosphere. The initial La-Ca alloy proportions and element distribution were analyzed using scanning electron microscopy (SEM) equipped with energy-dispersive x-ray spectroscopy (EDX) (Figs. S1 and S5 in the Supplemental Material [39]; see also Refs. [7,12,14,32,40–46] therein). The starting material was extracted from a uniform area on the surface of the alloy (the starting material was prepressed to approximately 1–2 μm) and then loaded into a diamond anvil cell (DAC) along with ammonia borane (NH_3BH_3, AB), which served as both a pressure transfer medium and a source of hydrogen. A total of five samples were compressed to 165–175 GPa at room temperature and then heated to about 1500 K for a few seconds. The laser heating caused a noticeable change in the color of the sample, as shown in the inset of Fig. 1. After quenching the synthesized sample to room temperature under high pressure, we performed subsequent superconductivity and structural characterization. To detect the superconductivity of the synthesized superhydrides, we carried out a series of electrical transport measurements. Representative electrical resistance data as a function of temperature at different pressures are shown in Fig. 1. In run 1, the synthesized sample had a T_c of 244 K at 165 GPa after heating two times (Fig. S2 [39]), and the resistance clearly drops to zero. Similar resistance drops are observed in run 2 and run 5, where the measured T_c reaches 247 K at 173 GPa and 242 K at 167 GPa, respectively, further confirming the superconducting state in the current experimental setup. However, the T_c values obtained in these runs are very close to *fcc*-LaH_{10}, indicating that Ca doping may have little effect on the T_c of LaH_{10}. In these runs, we also detected an additional resistance drop in the transition curve at 230±5 K, which may be caused by the presence of the mixed superconducting phases in the sample chamber.

To further confirm the superconductivity and study the upper critical field $\mu_0H_{c2}(0)$ of the synthesized superconducting phases, we applied an external magnetic field to different runs from 185 to 160 GPa [Figs. 2(a) and 2(b), and Figs. S4 and S6 of the Supplemental Material [39]]. Figures 2(a) and 2(b) show the measured resistance under different magnetic fields at 167 and 160 GPa in run 5. We observed that the decrease in resistance gradually shifts to lower temperatures as the applied magnetic field increases in the 0–8 T range, thus verifying the nature of the superconducting transition in La-Ca-H samples. To fit the $\mu_0H_{c2}(0)$, we have used the Ginzburg-Landau (GL) model [44], and the Werthamer-Helfand-Hohenberg (WHH) model [45], simplified by Baumgartner [46]. As shown in Fig. 2(c), the $\mu_0H_{c2}(0)$ produced by these two models at 167 GPa is 128 T (GL) and 176 T (WHH). With the pressure decreasing to 160 GPa, $\mu_0H_{c2}(0)$ increases slightly to 131 T (GL) and 180 T (WHH). For the WHH model, the coherence length $\xi_{\mathrm{WHH}}(0)$ is equal to 1.37 nm (167 GPa) and 1.35 nm (160 GPa), respectively. The magnitude of the short coherence lengths and high upper critical fields indicate that this phase is a typical type-II superconductor.

To reveal the crystal structure of the synthesized La-Ca polyhydrides, we performed *in situ* synchrotron x-ray diffraction (XRD) measurements on run 5. At 175 GPa, the peaks observed in run 5 can be labeled as $Fm\bar{3}m$ and $P6_3/mmc$ phases (Fig. 3). Detailed cell parameters are provided in Table S1 of the Supplemental Material [39]. Current XRD techniques are unable to locate hydrogen atoms due to the weak x-ray scattering cross section of light elements. It is often possible to compare the cell volumes of these two phases obtained in run 5 with previous experimental and theoretical results, leading to the hypothesis that the metal-to-hydrogen ratio in the $Fm\bar{3}m$ and $P6_3/mmc$ phases is approximately 1:10 (Fig. S9 [39]). The unit cell volume of the *fcc* phase obtained in the present work shows a slight increase compared to that of the *fcc*-LaH_{10} unit cell volume reported by Geballe *et al.* [12] at 175 GPa, which may be attributed to the doping of calcium in the lattice interstitials. Finally, combining the two phases obtained by XRD characterization and the superconductivity of the samples, we find that the behavior of the obtained ternary $Fm\bar{3}m$-(La, Ca)H_{10} is very similar to that of the binary $Fm\bar{3}m$-LaH_{10} [7]. Therefore, we conclude that the T_c around 242 K can be attributed to $Fm\bar{3}m$-(La, Ca)H_{10}, while the T_c around 230 K can be attributed to $P6_3/mmc$-(La, Ca)H_{10}.

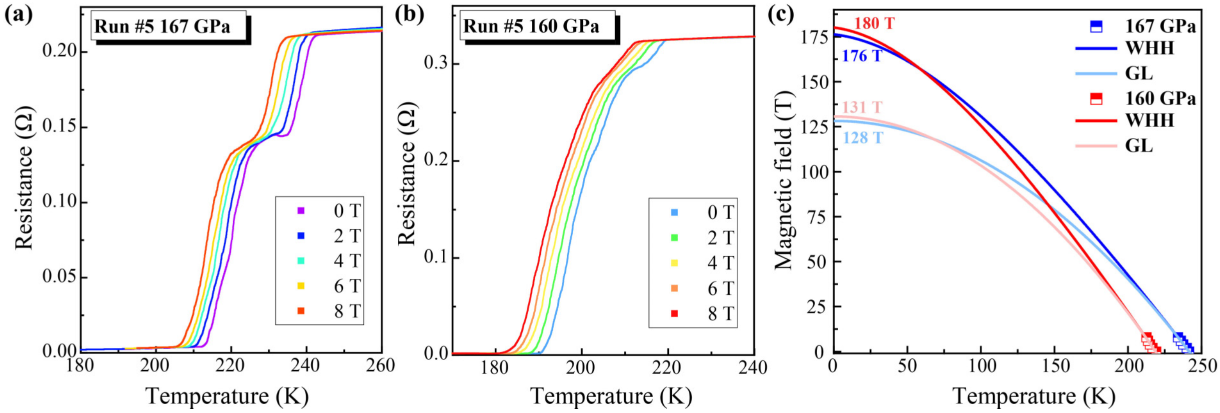

FIG. 2. Electrical resistance measurements of the superconducting transition in external magnetic fields. (a),(b) Temperature dependence of the resistance in an external magnetic field for run 5 at 167 and 160 GPa. (c) Extrapolated upper critical magnetic fields from run 5 for the La-Ca-H system, fitted with the GL [44] and simplified WHH [45,46] models. T_c values are determined by the criteria of the T_c^{onset} of normal state resistance, respectively.

In order to further compare the T_c-P trend of the ternary La-Ca-H system and binary La-H [7,14,47], Ca-H [10] system, we studied the T_c response to pressure of fcc-(La, Ca)H_{10}, as shown in Fig. 4. In different experimental runs, the T_c fluctuated slightly under pressures of 165–185 GPa. At 171 GPa, the highest T_c observed in the $La_{0.9}Ca_{0.1}H_{10}$ sample is 249 K, which is consistent with the experimental result of fcc-LaH_{10} at 250 K [7] under this pressure. However, the highest T_c of $La_{0.85}Ca_{0.15}H_{10}$ is 242 K (167 GPa), which is 3% lower than that of fcc-LaH_{10}. This also suggests that the slight difference in the content of Ca does not have a significant effect on the T_c of LaH_{10}. This finding contradicts the theoretical prediction that adding 12.5% Ca to LaH_{10} will increase its T_c by 15% [30]. Intriguingly, when the pressure was reduced to 165 GPa, T_c began to decline, and the trend of T_c's response to pressure became more similar to that of CaH_6 [10], which may be due to the premature occurrence of H-cage distortion induced by Ca doping.

Currently, our research indicates that in the synthesis of La-Ca hydrides, a combination of factors such as pressure, heating temperature, and heating time collectively influence the resulting products, leading to complex mixed-phase characteristics. However, during XRD analysis, we observed that the high-temperature superconducting phase with a T_c exceeding 200 K in La-Ca hydrides still maintains a highly symmetric fcc or hcp crystal structure. This observation aligns with the proposition from Matthias's rules that compounds with highly symmetric crystal structures are more likely to exhibit high-temperature superconductivity [48,49]. In addition to our work, previously discovered hydrides with high T_c have also primarily exhibited cubic or hexagonal phases [7–9,32–37]. Furthermore, although calculations by Wang *et al.* have shown that doping fcc-LaH_{10} with 12.5% Ca could increase its T_c by 15%, potentially achieving room

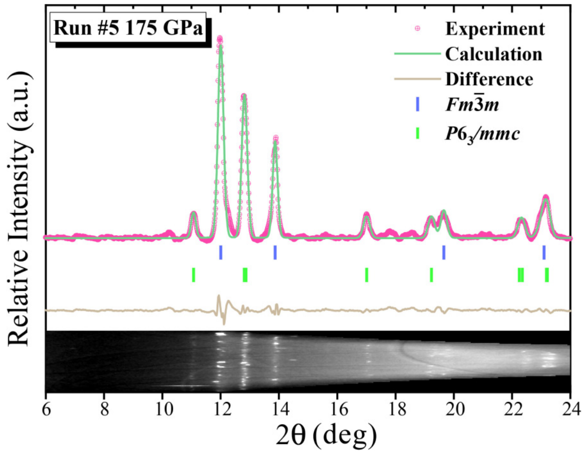

FIG. 3. The Synchrotron x-ray diffraction (XRD) patterns of the La-Ca-H system. (a) XRD pattern ($\lambda = 0.6199$ Å) and Le Bail refinement of the $Fm\bar{3}m$-(La, Ca)H_{10} and $P6_3/mmc$-(La, Ca)H_{10} at 175 GPa. The experimental XRD data, calculated data, and differences are represented by pink hollow circles, green lines, and brown lines, respectively. The bottom panel shows the cake view of the raw XRD patterns.

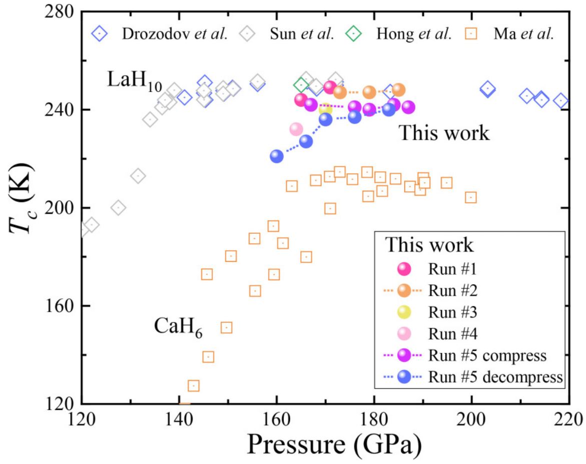

FIG. 4. T_c dependence on pressure for the La-Ca-H system. The pressure dependence of T_c for the $Fm\bar{3}m$-(La, Ca)H_{10} and binary La-H [7,14,47], Ca-H [10] system. The ratio of initial La-Ca alloy in runs 1 and 2 is $La_{0.9}$:$Ca_{0.1}$, while in runs 3–5 it is $La_{0.85}$:$Ca_{0.15}$. The solid circles represent the data from this work.

temperature superconductivity [30], current experiments have not yet found an accurate way to achieve such doping levels controllably. In this work, we attempted to control the doping level as close as possible to the theoretically optimal value. However, after doping with 10% or 15% Ca, we found that the addition of Ca had only a negligible effect on the T_c of LaH_{10}. Similarly, in the La-Y-H system, introducing different proportions of Y also had little effect on the T_c of LaH_{10} [32], consistent with Anderson's proposition that superconductors are generally insensitive to common physical and chemical impurities [50]. Although the doping of Ca did not lead to an increase in the T_c of LaH_{10}, it did have a certain impact on the structural stability. With decreasing pressure, in La-Ca hydrides with abundant defects and impurities, changes in the composition and symmetry of hydrogen sublattices occur earlier compared to fcc-LaH_{10}. This leads to a sharp decrease in T_c, thereby limiting the possibility of depressurizing superhydrides. Therefore, under the doping levels and synthesis conditions of this work, calcium is not a promising candidate as a doping agent.

III. CONCLUSIONS

In this work, we successfully synthesized La-Ca-H compounds in five independent runs through laser heating of La-Ca alloy with AB. The obtained temperature dependence of the resistance provides evidence for the superconductivity of $Fm\bar{3}m$-$(La, Ca)H_{10}$ whose T_c is about 247 K (173 GPa), respectively. By comparing with lanthanum hydrides, the present results show that doping with 10% or 15% Ca has only a negligible effect on the T_c of LaH_{10}, which is consistent with Anderson's theory [50]. Although the high T_c phase of La-Ca-H still maintains a highly symmetric *fcc* structure, the doping of Ca may lead to premature H-cage distortion during the decompression process, thus affecting the structural stability of the system. Therefore, under the doping levels and synthesis conditions of this work, we think that Ca may not be an ideal dopant. Our study provides some hints on the key factors affecting superconductivity and informs the future search for room-temperature superconductors in more diverse hydride materials.

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