

Laser-heated diamond anvil cell synthesis and recovery of metastable MnSb_2 and YbZn_2 for post-synthesis transport studies

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ABSTRACT

The creation and exploration of new materials under extreme pressure–temperature conditions has become increasingly reliant on laser-heated diamond anvil cell (LHDAC) techniques, which provide direct access to previously unexplored regions of multinary phase diagrams. Whereas numerous high-pressure phases have been identified *in situ*, systematic recovery and post-synthesis physical property characterization of these materials remain significant challenges. In this work, we describe the setup and implementation of an LHDAC-based synthesis and recovery workflow and demonstrate its application to metastable MnSb_2 and YbZn_2 phases. Synchrotron x-ray diffraction and spatial mapping confirm dominant formation of the targeted phases, whereas laboratory-based refinement quantifies phase fractions despite intrinsic microstrain and minor secondary phases. High-pressure transport measurements on recovered samples reveal pressure-tunable electronic instabilities in both systems. In MnSb_2 , pressure suppresses two high-temperature magnetic ordering anomalies, observed in transport, by ~ 5 GPa and, for higher pressures, induces a new low-temperature feature that increases with further pressure increase. In hexagonal high-pressure YbZn_2 , an electronic reconstruction emerges at ~ 11 GPa, characterized by semiconducting-like behavior from ~ 30 to 300 K and a broad low-temperature coherence crossover near 30 K. Our results establish LHDAC synthesis not only as a structural discovery tool but also as an experimental platform for investigating correlated quantum states stabilized far from equilibrium thermodynamic conditions.

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INTRODUCTION

The exploration of materials under extreme pressure and temperature conditions has entered a transformative era with the development of laser-heated diamond anvil cell (LHDAC) techniques.^{1–3} These methods provide access to a broad static pressure–temperature phase space while also allowing for *in situ* structural characterization using synchrotron x-ray diffraction, spectroscopy, as well as electrical transport and even thermodynamic measurements.^{4,5} The earliest demonstrations of laser

heating in DACs established the feasibility of reaching several thousand kelvin under multi-gigapascal pressures,^{6,7} laying the foundation for high-pressure materials synthesis and mineral physics investigations.

Subsequent advances in optical design, double-sided heating geometries, beam shaping, and synchrotron integration have significantly improved temperature stability and spatial uniformity.^{8,9} As a result, LHDAC has evolved into a powerful platform not only for probing deep-earth conditions³ but also for stabilizing novel condensed-matter phases inaccessible at ambient equilibrium.

In particular, LHDAC-enabled synthesis has led to the discovery of record-breaking high-temperature superconducting superhydrides, such as H_3S and LaH_{10} , with transition temperatures exceeding 200 K under megabar pressures.^{10–12} Beyond superconductivity, laser-heating techniques in DACs have been instrumental in establishing phase stability, melting behavior, and thermophysical properties of deep-mantle and core materials under multi-megabar conditions.^{13–15}

In parallel, LHDAC-based reactions have been widely used for high-pressure materials synthesis beyond geoscience and hydride systems. For example, transition-metal nitrides, carbides, and borides have been synthesized directly from the elements in laser-heated DACs, with several products recovered to ambient conditions and characterized *ex situ* by micro-XRD, Raman spectroscopy, transmission electron microscopy, energy-dispersive x-ray spectroscopy and related micro-beam techniques.^{16,17} The utility of LHDAC synthesis has also been demonstrated in the discovery of pressure-stabilized intermetallic compounds in otherwise inaccessible binary systems, including Fe–Bi, Cu–Bi, and Ni–Bi based phases.^{18–21} More recently, spatially resolved synchrotron diffraction combined with electrical-transport imaging has provided an important route for correlating local structural information with electronic behavior in high-pressure samples.²²

Together, these studies establish LHDAC synthesis, sample recovery, and spatially resolved characterization as important components of high-pressure materials discovery. Nevertheless, a critical bottleneck remains in extending these approaches toward reliable extraction, post-recovery phase evaluation, and systematic physical-property characterization of LHDAC-synthesized phases. The microscopic sample size, large thermal gradients during synthesis, accumulated microstrain, and mechanical constraints of the DAC environment often limit post-synthesis handling and *ex-situ* investigation.^{23,24} As a result, many high-pressure discoveries rely primarily on *in situ* structural signatures, while systematic physical property measurements on recovered samples remain relatively rare. Bridging this gap between extreme-condition synthesis and physical-property characterization is essential for advancing high-pressure materials research.

In this work, we describe the setup and implementation of an LHDAC-based synthesis and recovery workflow designed not only for high-pressure, high-temperature phase synthesis but also for practical sample recovery and subsequent characterization. To benchmark this approach, we selected two representative metastable intermetallic systems: MnSb_2 and YbZn_2 .

MnSb_2 crystallizes in the marcasite-type structure and can only be synthesized under high-pressure and high-temperature conditions.^{25,26} Recent studies have demonstrated that the high-pressure phase MnSb_2 (hp- MnSb_2) develops multiple magnetic phase transitions (200 and 118 K) upon cooling characterized by multi-sublattice, amplitude-modulated antiferromagnetic (AFM) order with temperature-dependent propagation vectors and inequivalent Mn sublattices.²⁶ First-principles calculations predict a sizable local moment on Mn, consistent with the experimentally observed ordered state. The coexistence of complex magnetic modulation and reduced symmetry makes MnSb_2 an appealing platform for examining the interplay between structural stabilization and electronic and magnetic ground states.

As for YbZn_2 , it can be stabilized in a high-pressure hexagonal MgZn_2 -type (C14) Laves phase (hp- YbZn_2) under high pressure. X-ray absorption spectroscopy and dynamical mean-field theory calculations indicate intermediate-valence behavior with a mean Yb valence of ~ 2.55 , and a density of states at the Fermi level dominated by Yb 4f contributions.²⁷ These features make hexagonal YbZn_2 a representative correlated intermetallic system in which pressure stabilization may significantly influence electronic properties.

Notably, both MnSb_2 and YbZn_2 are metastable at ambient pressure, allowing us to demonstrate not only successful LHDAC synthesis but also structural verification after decompression and enabling independent transport measurements under pressure. By integrating high-pressure synthesis, quantitative spatial structural analysis, and subsequent electrical measurements, we establish a reproducible experimental route for studying metastable intermetallic phases. Rather than emphasizing system optimization, we focus on demonstrating feasibility and recoverability, thereby providing a practical pathway for linking LHDAC synthesis to the discovery and characterization of new material properties in recovered metastable intermetallic phases.

METHOD

Development of the laser-heating DAC system

An in-house laser-heated diamond anvil cell (LHDAC) system was developed for high-pressure and high-temperature synthesis. The system accommodates multiple diamond anvil cell geometries and employs a continuous-wave fiber laser (Coherent, model: Compact SE 100, with $\lambda = 980$ nm and 100 W maximum output power) as the heating source. The laser beam is focused to a spot size of ~ 30 μm to one side of the DAC using a custom optical assembly equipped with a high resolution imaging system for real-time visualization. Temperature is monitored *in situ* by spectroradiometric measurements of the thermal emission collected from the laser-heated area through the optical path of the laser-heating system. The emitted radiation is collected by a fiber-coupled spectrometer (Ocean Optics HR-6VN400) for temperature estimation based on blackbody radiation fitting. Under typical operating conditions, temperatures up to ~ 3000 K can be achieved, while static pressures approaching the megabar regime are accessible depending on the anvil configuration (i.e., culet sizes ≤ 200 μm).

Precursor preparation

Polycrystalline precursor for MnSb_2 growth was prepared via solid-state reaction using elemental Mn and Sb in a molar ratio of 1:2. The mixture was sealed under vacuum in a silica tube in an Al_2O_3 crucible and heated to 785°C at 100°C/h for 24 h followed by air quenching of the still sealed tube. After cooling, the product was ground into powder for LHDAC use.

In a similar manner, YbZn_2 precursor was prepared from a Yb:Zn (1:2) mixture. The sealed ampoule was slowly heated to 850°C over 10 h, held at 850°C for 20 h, and then quenched in air. After cooling, the product was ground into powder for LHDAC use.

High-pressure synthesis in LHDAC

Two types of DACs were employed: a stainless-steel Mao–Bell-type cell and a Be–Cu cell compatible with a Quantum Design PPMS. Type Ia diamond anvils with 500 μm culets were used. Stainless-steel gaskets were pre-indented to $\sim 80\ \mu\text{m}$ thickness. A 500 μm hole was drilled and filled with a cubic BN–epoxy mixture, re-indented, and re-drilled to a final 200 μm diameter sample chamber. All pre-reacted precursors were ground into powder. A portion of the powders was pelletized, shaped into appropriate dimensions, and loaded into the sample chamber together with a few ruby spheres. The pressed NaCl (for MnSb_2 growth) and KCl (for YbZn_2 growth) thin layer pieces were inserted on both sides of the precursors and served as both pressure-transmitting media and thermal insulation. Pressure was determined by the ruby R_1 fluorescence shift.^{28,29} MnSb_2 was synthesized at 8.1 GPa and YbZn_2 at 9.6 GPa.

The DAC, loaded with the sample and pressure medium, was mounted on a three-axis (XYZ) micropositioning stage on the laser-heating optical line. Given that the laser has a focused beam spot size $\sim 30\ \mu\text{m}$ in diameter, to ensure a spatially uniform reaction across the sample chamber ($\sim 200\ \mu\text{m}$ in diameter), a raster heating strategy was adopted. The sample area was divided into a 4×4 grid, and each position was sequentially aligned with the laser focus by translating the DAC using the micropositioning stage. At each position, the sample was heated for ~ 60 s with a laser power of ~ 32 W for MnSb_2 and ~ 55 W for YbZn_2 .

The synthesis temperature was estimated from spectroradiometric measurements of the thermal emission collected from the laser-heated region. Under the heating conditions used in this work, the apparent temperatures were estimated to be ~ 1000 – 1400 K for MnSb_2 and YbZn_2 syntheses. These values should be regarded as approximate apparent temperatures rather than exact sample temperatures, due to uncertainties associated with emissivity, optical alignment, the finite laser-heated spot size, and strong axial and radial thermal gradients. The estimated overall uncertainty is on the order of ± 200 – 500 K. Because the present setup uses single-sided laser heating and single-sided optical collection, the temperature is expected to be highest near the laser-irradiated surface and to decrease along the axial direction through the sample and pressure-medium layers. This nonuniform temperature field likely contributes to the spatially inhomogeneous reaction products observed in the diffraction mapping.

We also need to note that, because the full rastering process took approximately one hour, different regions of the sample experienced sequential localized thermal cycles rather than one uniform heating event. Each grid position was directly heated for ~ 60 s, while neighboring regions could be partially reheated during subsequent laser exposures due to lateral heat diffusion. This rastering procedure was used to expose more regions of the sample chamber to laser-heated conditions and thereby promote more complete reaction over a larger sample area. At the same time, the repeated local heating, cooling, and partial reheating, together with the axial and radial thermal gradients associated with single-sided laser heating, may contribute to spatially nonuniform reaction progress, grain-size variation, accumulated microstrain, and peak broadening in the recovered samples. Further optimization of the growth protocol will be important for future studies. In particular, improved control of the rastering sequence, dwell time, heating overlap, and temperature

uniformity may help increase reaction homogeneity across the sample chamber while reducing local thermal strain, grain-size variation, and microstrain accumulation in recovered samples.

During the growth, the heating process was monitored in real time via optical microscopy, where the laser-heated regions exhibited a distinct increase in brightness, indicative of elevated temperature. The raster scan over the full grid was repeated once to improve reaction homogeneity and promote more complete phase formation. Currently, the rastering is accomplished by hand and the complete, two pass, process takes roughly one hour.

Due to the localized nature of single-sided laser heating, significant axial and radial temperature gradients are expected within the sample chamber. As a result, the reaction is anticipated to be most complete in the directly heated regions, whereas areas farther from the laser focus may remain partially reacted or retain precursor-related phases.

Synchrotron powder x-ray diffraction measurements

After laser heating, the loaded DACs were transferred to beamline 13-BM-C (GSECARS), Advanced Photon Source (APS), Argonne National Laboratory (ANL), for room-temperature high-pressure synchrotron powder x-ray diffraction (PXRD) measurements without decompression. This measurement sequence was used to verify phase formation under synthesis conditions before possible relaxation or back-transformation during recovery. Monochromatic x rays with a wavelength of $0.4283\ \text{\AA}$ were focused to a $10 \times 10\ \mu\text{m}^2$ spot. The LHDACs of as-grown MnSb_2 (SR006) and YbZn_2 (SR007) were directly mounted on the beamline setup for synchrotron PXRD measurements. Because synchrotron beam time was limited, a cross-shaped mapping strategy was used as a practical compromise between spatial coverage and measurement time. For the present sample-chamber size and $10\ \mu\text{m}$ step size, a full two-dimensional grid map would require ~ 225 diffraction patterns, whereas the cross-shaped scan required only about 30 points. This approach reduced the mapping time by nearly an order of magnitude while still probing spatial variations along two orthogonal directions across the laser-heated region. We, therefore, use the cross-scan as a semi-quantitative assessment of spatial inhomogeneity and phase formation, rather than as a complete two-dimensional reconstruction of the phase distribution.

The pressure of the sample was confirmed via the ruby fluorescence R_1 line before the measurement.^{28,29} Two-dimensional diffraction images were integrated using DIOPTAS,³⁰ and subsequent Rietveld refinements were carried out with GSAS-II.³¹

Le Bail refinements were employed to extract lattice parameters, unit cell volumes and estimate relative phase fractions at each spatial position based on the direct-derivation method.³² Phase fractions obtained by this method are intended to reflect the relative spatial distribution of the high-pressure phase, whereas the absolute value is not accurate due to the limited 2θ range accessible in the measurements. The weighted profile R -factor (R_{wp}) provides a measure of the agreement between observed and calculated diffraction profiles; however, in the context of multiphase Le Bail analysis of LHDAC samples, it should not be interpreted as a unique validation of the phase assignment. This is particularly relevant for micro-focused high-pressure diffraction data, where limited

powder averaging, preferred orientation, and overlapping reflections can allow different combinations of phases to yield comparable R_{wp} values. Therefore, phase identification in this work is based not only on the refinement quality but also on the consistency of peak positions, systematic absences, and agreement with known high-pressure phases reported in the literature.

Sample recovery procedure

After the synchrotron measurements, the DACs were decompressed to ambient pressure at room temperature for further study. In certain cases, decompression under low-temperature conditions can be employed to suppress kinetic relaxation processes and thereby improve retention of pressure-quenched metastable phases. Recovered samples were mechanically extracted from the gasket chamber at room temperature. For MnSb_2 , residual pressure medium (NaCl) was removed by rinsing in deionized water. The recovered samples were then separated into small pieces for laboratory PXRD and transport measurements. For YbZn_2 , because of its sensitivity to moisture, residual KCl was not fully removed immediately after recovery. Instead, before loading the YbZn_2 sample into the DAC for transport measurements, surface KCl was removed by repeated gentle rinsing with isopropyl alcohol (IPA).

In-house powder x-ray diffraction

The recovered samples were characterized using a Rigaku Synergy diffractometer equipped with a silver radiation source (K_α avg. ~ 0.56087 Å) in transmission mode, operating at 65 kV and 0.67 mA. The sample extracted from the DAC was shaped into ~ 50 μm size pieces and mounted on the nylon loop single crystal mount with N grease and subsequently placed on the goniometer for measurement. The beam spot size of the equipment is ~ 100 μm . Compared to synchrotron measurements, broader diffraction peaks were observed, attributed to a shorter sample-to-detector distance (~ 32 mm) and the microstrain and non-hydrostatic stress accumulated during decompression. The data were collected as two-dimensional CCD images and powder reduction was performed using CrysAlisPro (Rigaku OD, 2023), and subsequent Rietveld refinements were carried out with GSAS-II.³¹

Electrical transport measurements under high pressure

Following synthesis and extraction from the reaction LHDAC, the electrical resistance was measured up to 15 GPa using a Be–Cu DAC compatible with a Quantum Design Physical Property Measurement System (PPMS). The cell employed type-Ia diamond anvils with 500 μm culet size and a stainless-steel gasket. After pre-indenting the gasket to ~ 10 GPa, a concentric 500 μm hole was drilled by electric-discharge machining and back-filled with a cured mixture of cubic BN powder and epoxy (this cubic BN + epoxy insert is usually used to electrically isolate the sample and electrodes from the metallic gasket during the resistance measurements; however, in the work presented here, we did not actually put electrodes for *in situ* transport measurement). The insert was then compressed to 10 GPa again and re-drilled using a mechanical micro-drilling machine to a 200 μm diameter hole to form the sample chamber.

The bottom space of the sample chamber was first covered with a thin NaCl layer along with a small ruby sphere. MnSb_2 and

YbZn_2 samples extracted after synthesis were shaped into rectangles and placed above this layer, and the Pt foil electrodes were arranged in a van der Pauw configuration on the pressed sample. An additional thin NaCl layer was placed over the sample and electrodes to improve the hydrostaticity at high pressure. Pressure was determined from the ruby R1 fluorescence line.^{28,29}

RESULTS AND DISCUSSION

Synchrotron powder x-ray diffraction (PXRD) mapping was performed on the laser-heated diamond anvil cell (LHDAC) samples before decompression to evaluate phase formation under the synthesis conditions. As shown in Figs. 1 and 2, the spatial maps reveal clear formation of both hp- MnSb_2 and hp- YbZn_2 within the laser-heated regions, although the reaction products are spatially inhomogeneous. The relative target-phase fraction varies noticeably across the mapped regions, reflecting the combined effects of localized single-sided laser heating, rastered thermal cycling, and stress/thermal gradients within the DAC environment.

As shown in Figs. 1 and 2, the cross-shaped synchrotron PXRD maps provide a spatial assessment of phase formation in the laser-heated DAC samples. The relative target-phase fraction at each mapped position is shown by the color contrast in Figs. 1(a) and 2(a), while representative diffraction patterns along two orthogonal directions are shown in Figs. 1(d), 1(e), 2(d), and 2(e). The corresponding spatial evolution of the unit-cell volume and weighted profile R -factor (R_{wp}) is summarized in Figs. 1(b), 1(c), 2(b), and 2(c). These spatially resolved measurements reveal that the degree of reaction varies noticeably on a length scale of ~ 10 μm , reflecting the combined effects of axial thermal gradients inherent to single-sided laser heating and lateral inhomogeneity arising from the raster-based heating approach. Despite this spatial inhomogeneity, a key conclusion from the mapping is that a substantial fraction of the sample volume corresponds to the targeted phase. Averaging over the scanned positions indicates that $\sim 40\%$ or more of the probed regions are dominated by the high-pressure phase for both systems. Importantly, although the raster-based heating strategy is designed to approximate spatially uniform heating, the spatial maps reveal inhomogeneity in the phase distribution. This indicates that the local reaction conditions might be governed by a complex interplay between laser absorption, thermal conduction, and stress heterogeneity within the DAC environment.

Given the spatial variability revealed by the mapping, it is instructive to examine diffraction patterns from regions exhibiting the best phase purity. Figure 3 presents representative diffraction data collected from such “best-case” regions, corresponding to visually darker areas in the optical images in Figs. 1(a) and 2(a) that indicate stronger laser heating and more complete reaction.

For MnSb_2 (SR006), the selected diffraction pattern [Fig. 3(a)] is dominated by reflections that can be indexed to the high-pressure marcasite-type structure ($Pnmm$, SG No. 58), although diffraction peaks from likely unreacted Sb and MnSb remain detectable. This confirms that locally high phase purity can be achieved within the LHDAC-synthesized sample, even though the overall reaction remains spatially inhomogeneous. In addition, the relative phase fraction indicates that the targeted phase is significantly lower on

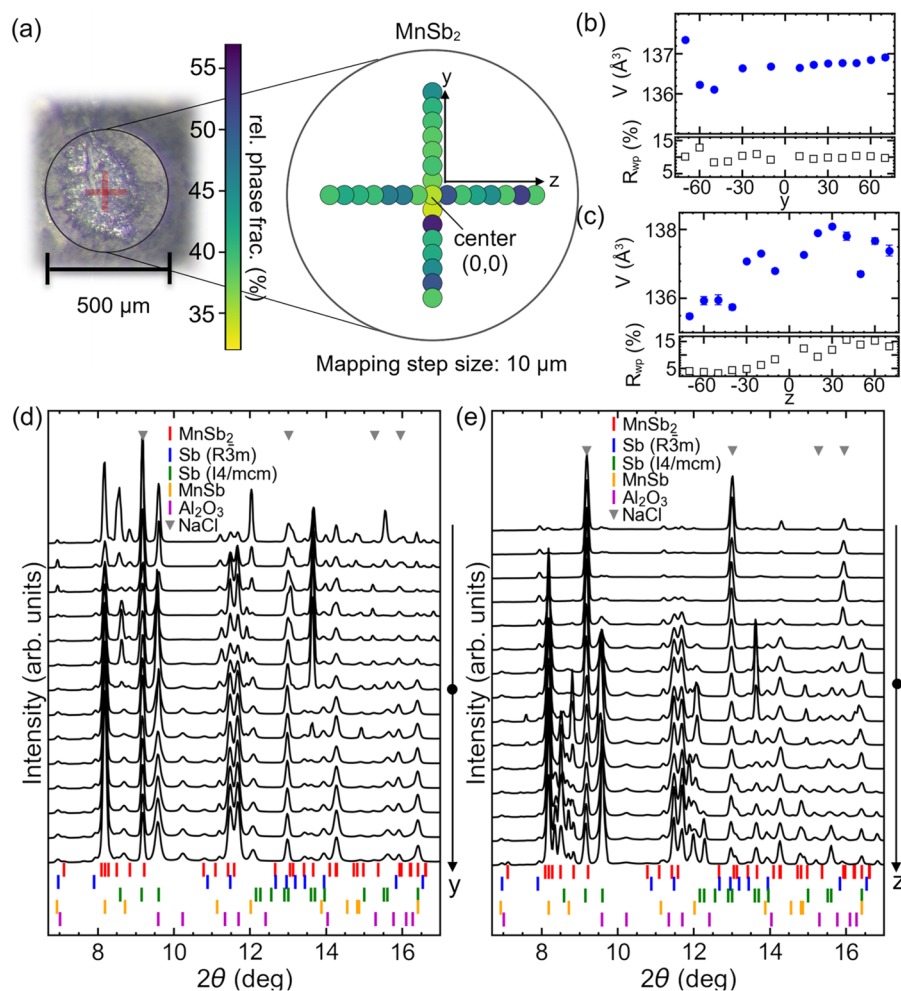


FIG. 1. Synchrotron powder x-ray diffraction spatial mapping using a cross-scanning strategy. (a) Schematic illustration of cross-shaped scanning geometry. The left panel shows a real-time optical image of the DAC sample chamber. The red cross stands for the mapping region. The color scale demonstrates the relative fraction of the main phase (hp-MnSb₂) at selected positions indicated within the central circular region (right panel). The relative phase fractions were roughly estimated from the sum Le Bail intensities weighted by the corresponding total scattering power per chemical formula weight, following the direct-derivation method. The refined unit-cell volume of hp-MnSb₂ and its weighted profile R -factor (R_{wp}) are plotted as a function of position along the (b) y - and (c) z -directions. The corresponding diffraction patterns collected along the y - and z -direction are shown in (d) and (e), respectively. The incident x-ray wavelength was 0.4283 Å.

the left side of the mapping region. This region corresponds to the area of outside sample's boundary where the signal is dominated in part by the NaCl medium.

Similarly, for YbZn₂ (SR007), the diffraction pattern shown in Fig. 3(b) corresponds to a region with the highest observed phase purity. The majority of reflections are consistent with the high-pressure hexagonal MgZn₂-type Laves phase ($P63/mmc$, SG No. 194), with contributions from low-pressure (lp-) YbZn₂ and Yb₃Zn₁₁ second phases. A quantitative estimate based on Le Bail refinement yields ~59.1 mol. % hp-YbZn₂, 29.0 mol. % lp-YbZn₂, and 11.9 mol. % Yb₃Zn₁₁.

All above results demonstrate that, although the reaction is not spatially uniform, the LHDAC synthesis produces the desired phase over a substantial portion of the sample chamber and that spatially resolved diffraction mapping provides a useful means to assess phase homogeneity and guide targeted secondary heating to improve phase purity.

It is also important to emphasize that these “best-case” patterns shown in Fig. 3 represent locally optimized regions rather than the average behavior across the sample chamber. Taken together with the spatial mapping results, this indicates that the LHDAC synthesis

produces a spatially inhomogeneous sample, in which well-reacted regions coexist with less-reacted areas.

For subsequent transport measurements, knowledge of the phase purity and distribution is essential due to spatial inhomogeneity. Therefore, identification of the spatial region where the target phase predominates is necessary for reliable downstream characterization. Following the synchrotron measurements, samples were recovered from the DACs, manually picked, shaped into small pieces (~50 μm), and characterized with PXRD mode using the in-house Rigaku Synergy diffractometer equipped with a silver radiation source, K_{α} avg. ~ 0.560 Å. Compared to the high-resolution synchrotron data collected at APS 13-BMC, the laboratory diffraction patterns exhibit noticeably broader peaks, as shown in Figs. 4 and 5. Such peak broadening can arise from the small sample to detector distance of the in-house system (~32 mm) compared with much larger sample-to-detector distance at the synchrotron (~194 mm) and the non-hydrostatic stress and lattice distortion accumulated during decompression, which may further contribute to strain-induced broadening.

Despite the broader diffraction features, the main structural phase can still be unambiguously identified, as shown in Figs. 4

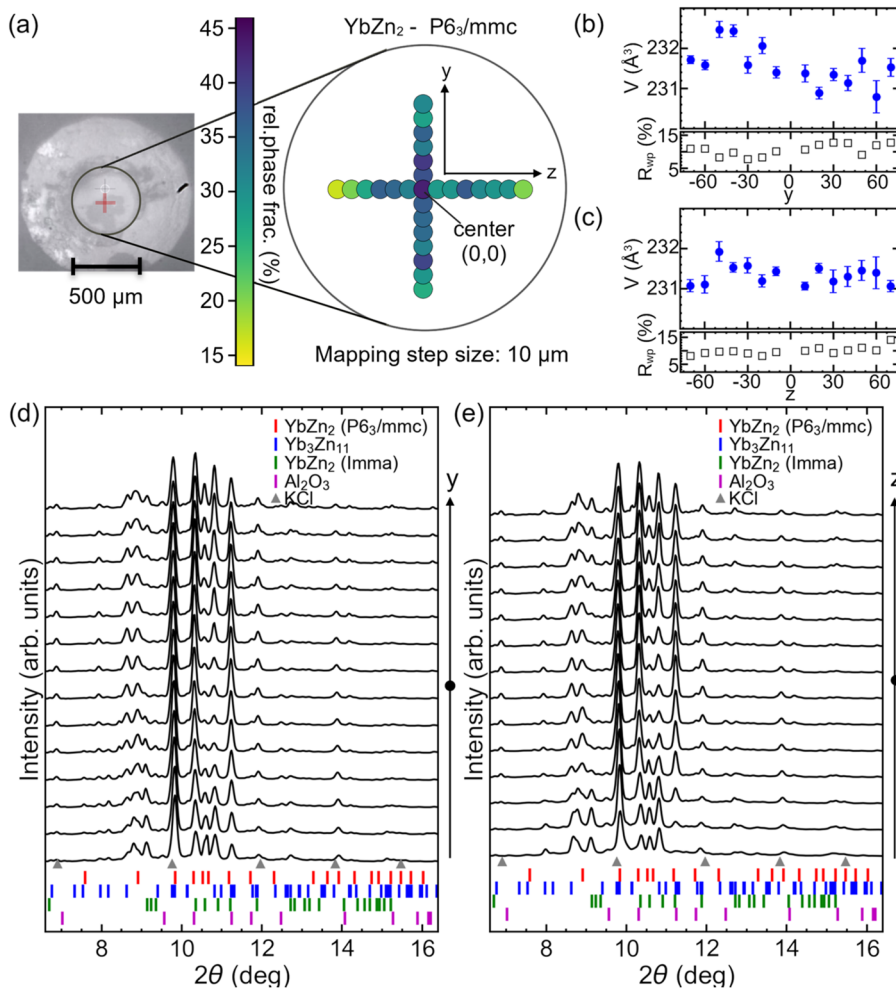


FIG. 2. Synchrotron powder x-ray diffraction spatial mapping using a cross-scanning strategy. (a) Schematic illustration of cross-shaped scanning geometry. The left panel shows a real-time optical image of the DAC sample chamber. The red cross stands for the mapping region. The color scale demonstrates the relative fraction of the main phase (hp-YbZn₂) at selected positions indicated within the central circular region (right panel). The relative phase fractions were roughly estimated from the sum Le Bail intensities weighted by the corresponding total scattering power per chemical formula weight, following the direct-derivation method. The refined unit-cell volume of hp-YbZn₂ and its weighted profile R_{wp} are plotted as a function of position along the (b) y - and (c) z -directions. The corresponding diffraction patterns collected along the y - and z -direction are shown in (d) and (e), respectively. The incident x-ray wavelength was 0.4283 Å.

and 5. To further illustrate the contributions of the individual phases to the laboratory PXRD patterns, independently calculated diffraction profiles for each refined phase are provided in Figs. S1 and S2 in the [supplementary material](#). The diffraction pattern contains reflections consistent with the targeted high-pressure phase, together with additional peaks arising from secondary or unreacted phases, indicating incomplete phase purity. During Rietveld refinement, microstrain effects were explicitly incorporated into the structural model, which significantly improved the fitting quality and allowed reliable extraction of lattice parameters and quantitative phase fractions. The refined phase percentages are summarized in the tables in [Figs. 4 and 5](#). This analysis enabled us to select samples with relatively higher phase purity for subsequent transport measurements.

This step is critical within the LHDAC workflow: by evaluating phase composition after recovery, samples with a relatively higher fraction of the target-phase can be selected for subsequent transport measurements. However, because the recovered samples remain multiphase, the measured resistance should be regarded as an effective transport response rather than the intrinsic

resistivity of a single phase. Secondary phases may affect the absolute value of resistance and residual resistance ratio and broaden the transition feature. In future studies, focused milling could provide a more precise approach for isolating selected phase-enriched regions from recovered LHDAC products, thereby improving the spatial correspondence between diffraction-based phase mapping and subsequent ex-situ physical-property measurements.

We performed high-pressure electrical resistance measurements on polycrystalline MnSb₂ recovered from LHDAC synthesis and reloaded into a DAC for measurement up to ~15 GPa. At ambient pressure, the temperature-dependent resistance shows metallic behavior with a convex shape, which qualitatively resembles the results of recent measurements on MnSb₂ single crystal at ambient pressure.²⁶ Although the residual resistance ratio (RRR ≈ 7) is significantly lower compared to the single crystals (RRR ≈ 60), the overall temperature dependence remains consistent, indicating that the electronic ground state is preserved despite enhanced scattering from grain boundaries and pressure-induced lattice strain. Remarkably, subtle resistive anomalies are discernible in the dR/dT plot, as shown in [Fig. 6\(b\)](#), and are consistent with the two magnetic

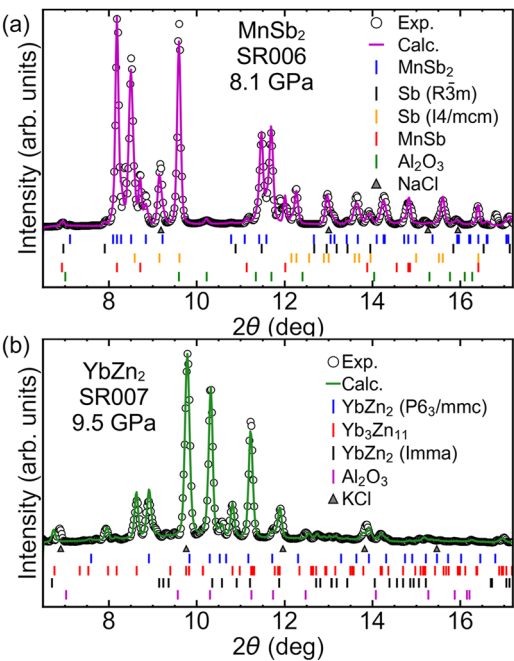


FIG. 3. Synchrotron PXRD patterns collected from the best phase-purity locations identified in the spatial mapping for post-growth (a) MnSb₂ (SR006, 8.1 GPa) and (b) YbZn₂ (SR007, 9.5 GPa). The incident x-ray wavelength was 0.4283 Å.

phase transitions (T_1 and T_2) that were found for MnSb₂ at ambient pressure.²⁶ Upon increasing pressure, T_1 and T_2 are rapidly suppressed and become indiscernible above ~1.3 GPa. This rapid disappearance implies that the associated instability is highly sensitive to lattice compression. As a result, the high-temperature linear resistance region extends to lower temperatures as pressure is initially increased. Interestingly, a pronounced low-temperature downturn in resistance, with the characteristic temperature T^* , emerges under pressure and becomes increasingly prominent with further

compression, as shown in Fig. 6(c). The character of this feature is manifested as a change in slope rather than a sharp discontinuity, indicating that the underlying transformation is either a broadened phase transition or some temperature driven crossover. The non-hydrostatic pressure condition due to the use of solid pressure transmitting medium could be part of the reason for the lack of sharp features of transition-like behavior. The systematic upward shift of T^* with pressure might suggest that compression modifies the low-temperature electronic or magnetic scattering environment in hp-MnSb₂, although transport data alone cannot determine whether this reflects magnetic ordering, a Fermi-surface reconstruction, or another crossover within the complex magnetic ground state. Additional measurements, such as high-pressure magnetic susceptibility, Hall effect, or neutron diffraction, would be required to determine the microscopic origin of this feature.

It is noteworthy that the MnSb₂ sample contains a substantial amount of residual elemental Sb, which may provide a parallel conduction path and influence the absolute resistance, residual resistance ratio, and apparent width of the resistive anomalies. Nevertheless, elemental Sb is not expected to produce magnetic-ordering-like features corresponding to T_1 and T_2 , whereas the observed anomalies are also consistent with the reported magnetic transitions of high-pressure MnSb₂. However, residual Sb can account for the low-temperature superconducting-like transition observed above 10.1 GPa, as shown in Fig. 6(a). The resistance drops sharply at low temperature and reaches zero with $T_c \sim 3.6$ K at 10.1 GPa; T_c slightly decreases to ~3.4 K at 14.8 GPa. Comparison with the known pressure-temperature phase diagram of elemental Sb³³ indicates that this superconducting transition is consistent with the high-pressure Sb-II phase, in $I4/mcm$ structure, within this pressure range,^{34,35} in agreement with our structural analysis showing coexistence of residual Sb with the MnSb₂ phase in Figs. 1 and 3.

Recent density functional theory calculations predict that marcasite-type MnSb₂ favors an AFM ground state with a sizable local moment (~2.8 μ_B per Mn), consistent with experimental observations of long-range magnetic order in the single crystal sample.²⁶ Neutron diffraction measurements reveal a complex multi-sublattice, amplitude-modulated AFM structure with temperature-dependent propagation vectors and inequivalent Mn

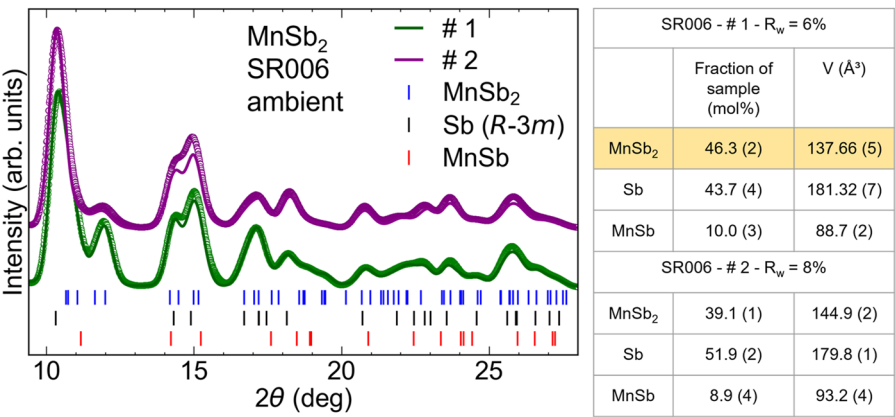


FIG. 4. Powder x-ray diffraction patterns from two selected recovered MnSb₂ pieces measured after decompression using a Rigaku Synergy diffractometer (integrated from two-dimensional diffraction images). The silver radiation source with K_α avg. ~ 0.560 Å is used. The additional reflections arise from Sb and MnSb phases. The table on the right summarizes the refined phase fractions and unit-cell volumes of each phase. Sample No. 1 was selected for subsequent electrical transport measurements.

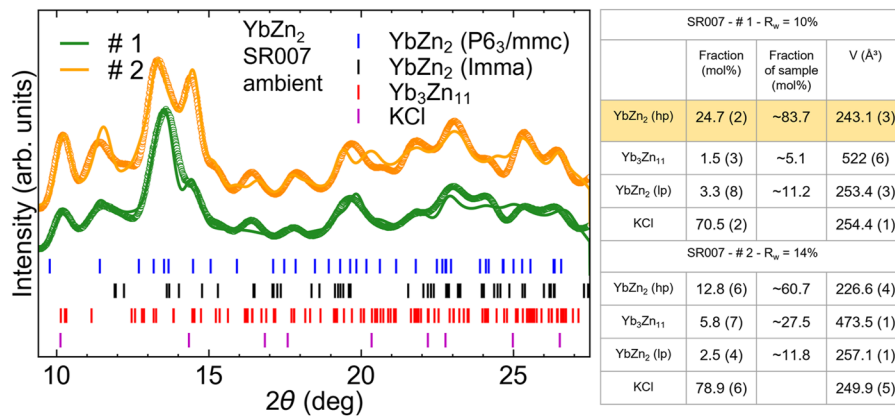


FIG. 5. Powder x-ray diffraction patterns from two selected recovered YbZn₂ samples measured after decompression using a Rigaku Synergy diffractometer with silver radiation source with K_α avg. ~ 0.560 Å. The patterns were obtained by integrating two-dimensional diffraction images. The additional reflections arise from the pressure-transmitting medium KCl, as well as lp-YbZn₂ and Yb₃Zn₁₁ phases. The table on the right summarizes the refined phase fractions and unit-cell volumes. Sample No. 1 was selected for subsequent electrical transport measurements, whereas sample No. 2 exhibits less reliable lattice-parameter refinement due to overlapping reflections and other-phases contributions.

sublattices. The coexistence of sizable local moments and metallic transport suggests that MnSb₂ lies in a delicate regime where magnetic exchange interactions, electronic bandwidth, and Mn–Sb hybridization are strongly intertwined. In such scenario, moderate lattice compression can significantly alter the magnetic exchange interactions and electronic bandwidth, thereby tuning the balance among competing magnetic configurations within the ordered state. The pressure-induced suppression of T_1 and T_2 , together with the emergence and monotonic enhancement of T^* , may, therefore, reflect a pressure-driven evolution of this complex magnetic ground state.

While the present transport data alone cannot unambiguously distinguish between magnetic ordering and a purely Fermi surface topology reconstruction in MnSb₂ system, the systematic pressure evolution and the monotonic increase of T^* strongly support the presence of a pressure-stabilized collective ground

state. Further probes, such as high-pressure magnetic susceptibility, neutron diffraction, or Hall measurements on a single crystalline sample, will be essential to determine the microscopic origin of this transition.

Electrical transport measurements were also carried out on the recovered hp-YbZn₂ sample, which was subsequently reloaded into a diamond anvil cell for compression up to 15 GPa. As described in the section entitled “Method,” surface KCl was removed before transport measurements. However, minor residual insulating KCl may still affect the effective current path and absolute resistance values. Therefore, we focus on the qualitative temperature dependence and pressure evolution of $R(T)$, rather than extracting intrinsic resistivity.

As reported previously, the hexagonal phase shows metallic behavior over the entire 2–300 K range without any visible phase transitions and follows a Fermi-liquid-like dependence,

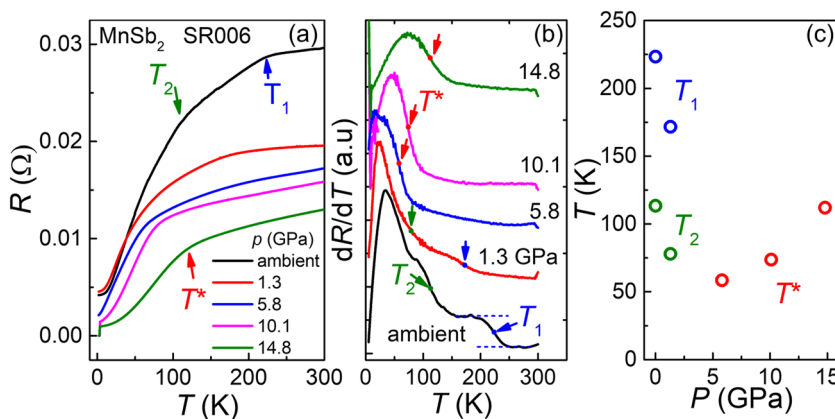


FIG. 6. (a) Temperature dependence of the resistance $R(T)$ for MnSb₂ (SR006, sample No. 1) at various pressures. The blue, green, and red arrows indicate the characteristic anomalies denoted as T_1 , T_2 , and T^* , respectively. (b) Temperature derivative dR/dT as a function of temperature. The criteria for determining T_1 , T_2 , and T^* are indicated in panel (b), defined by the midpoint of the shoulder features. (c) Pressure dependence of T_1 , T_2 , and T^* .

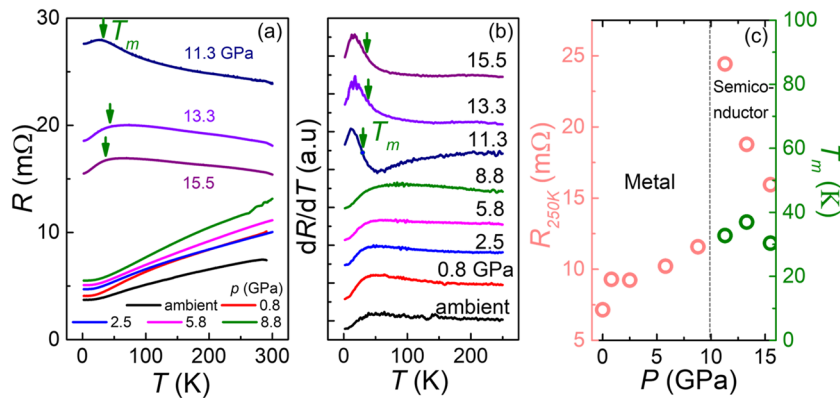


FIG. 7. (a) Temperature dependence of the resistance $R(T)$ for YbZn₂ (SR007, sample No. 1) at various pressures. The green arrows indicate the characteristic anomaly denoted as T_m . (b) Temperature derivative dR/dT as a function of temperature. The criterion for determining T_m is indicated in panel (b), defined as the midpoint of the shoulder-like feature. (c) Pressure dependence of the resistance at 250 K (left axis) and T_m (right axis).

$\rho = \rho_0 + AT^2$ at low temperatures.²⁷ In our measurement, as shown in Figs. 7(a) and 8, the metallic Fermi-liquid behavior (in our case, $R = R_0 + AT^2$) persists up to 8.8 GPa. The residual resistance R_0 increases monotonically with pressure, while the coefficient A remains nearly pressure independent. This evolution indicates that moderate lattice contraction enhances elastic scattering but does not immediately destabilize the coherent quasiparticle state. Although RRR at 1.8 K is ~ 1.7 under ambient condition, which is smaller than the reported value (~ 3), reflecting enhanced residual scattering in our polycrystalline specimen, the overall temperature dependence of the resistance is qualitatively identical.

Upon further increasing the pressure, a pronounced change emerges near 11.3 GPa [Fig. 7(a)]. At this pressure, the overall resistance exhibits an abrupt increase (increasing by roughly 2–5 times, depending on temperature), and the slope of resistance curve changes from positive to negative over a broad temperature interval, giving rise to semiconducting-like behavior at intermediate temperatures, as shown in Figs. 7(a) and 7(b). An Arrhenius-type analysis of this intermediate-temperature semiconducting-like regime is provided in Fig. S3 in the supplementary material. This evolution contrasts sharply with the robust metallic state observed at

the low-pressure range and strongly suggests an intrinsic pressure-driven modification of the electronic structure rather than a simple enhancement of scattering.

Notably, despite the semiconducting-like behavior at intermediate temperatures, a broad low-temperature downturn develops below ~ 30 K, labeled as T_m as shown in Figs. 7(a) and 7(b) where the resistance decreases upon further cooling. This crossover-like feature persists upon further compression to 15 GPa. While the overall resistance magnitude is gradually reduced at higher pressures, the characteristic temperature scale and the broad nature of the low-temperature downturn remain nearly unchanged. Such behavior suggests a pressure-induced two-regime transport scenario. Above ~ 30 K, the semiconducting-like temperature dependence indicates a strongly scattered electronic state, possibly associated with partial gap formation or incoherent 4f-conduction electron hybridization. In contrast, the low-temperature resistance downturn reflects the recovery of metallic-like transport or a loss of some, possibly magnetic, scattering below a well-defined temperature scale.

Two interpretations may account for this evolution. One scenario involves the gradual development of a correlated quasiparticle

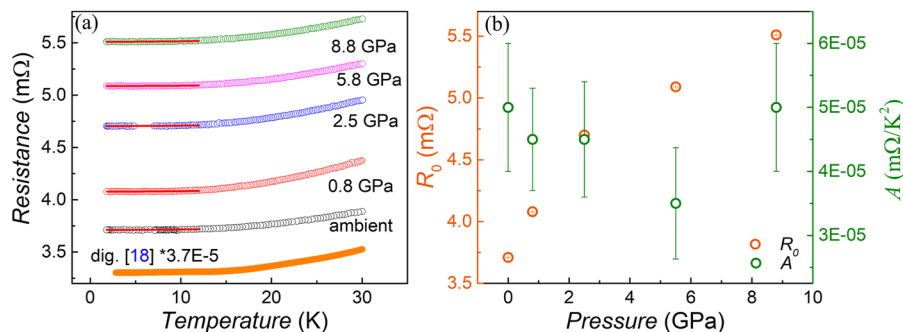


FIG. 8. (a) Power-law fits to the low-temperature $R(T)$ curves of YbZn₂ at various pressures. The data in the temperature range 1.8–12 K were fitted using the Fermi-liquid form, $R(T) = R_0 + AT^2$. The red curves represent the fitted results. The orange solid curve is digitized from Ref. 27, and its resistivity values were scaled by a factor of 3.7×10^{-5} for clarity and direct comparison with the present measurements. (b) Pressure dependence of the fitted Fermi-liquid parameters.

state under pressure. In this picture, the intermediate-temperature semiconducting-like response corresponds to an incoherent regime, while the low-temperature downturn marks the onset of coherent heavy-electron transport driven by enhanced f -conduction electron hybridization.

Alternatively, if the apparent broadness of the resistive anomaly near ~ 30 K arises from non-hydrostatic conditions associated with the use of a solid PTM as well as possibly inhomogeneous/locally stressed sample, the underlying transition may, in fact, be sharper and could correspond to the onset of magnetic ordering. Yb-based systems are often located near valence and magnetic instabilities, and pressure could stabilize a magnetically ordered phase. Such ordering would naturally lead to partial Fermi-surface reconstruction or superzone-gap formation, giving rise to semiconducting-like behavior above the ordering temperature.

The relatively broad nature of the feature and the absence of clear discontinuity prevent a definitive distinction between a continuous crossover and a second-order phase transition. Therefore, whereas the transport data strongly indicate a pressure-induced electronic reconstruction in hp-YbZn₂, complementary magnetic and thermodynamic measurements under improved hydrostatic conditions are required to clarify the microscopic origin of this anomaly. Taken together, these results demonstrate that the correlated electronic state of hp-YbZn₂ is highly tunable by pressure.

CONCLUSION

In conclusion, we have implemented and benchmarked an LHDAC-based synthesis and recovery workflow that enables the preparation, recovery, and post-synthesis characterization of metastable high-pressure intermetallic phases. By combining controlled laser heating, spatially resolved synchrotron diffraction mapping, quantitative laboratory diffraction, and pressure-dependent transport measurements, we establish a reproducible experimental workflow that bridges extreme-condition synthesis by LHDAC with correlated-electron investigations.

Using this workflow, we successfully synthesized and recovered metastable hp-MnSb₂ and hp-YbZn₂ phases. Synchrotron measurements confirm dominant formation of the targeted high-pressure phases. Crucially, laboratory-based PXRD characterization after recovery allows quantitative evaluation of phase fractions despite peak broadening arising from microstrain and non-hydrostatic stress. This step serves as a key bridge between *in situ* synthesis and ex-situ physical property measurements, enabling informed sample selection while also identifying secondary phases that must be considered when interpreting transport data.

Transport measurements reveal that both systems host electronically delicate states that are highly sensitive to lattice compression. In MnSb₂, pressure rapidly suppresses ambient-pressure magnetic ordering temperatures and stabilizes a distinct low-temperature correlated state, consistent with the proximity of competing magnetic and itinerant configurations predicted by theory. The monotonic enhancement of T^* under pressure indicates that modest lattice contraction is sufficient to shift the balance between nearly degenerate electronic ground states in the marcasite family. In hp-YbZn₂, a pronounced electronic reconstruction occurs near

~ 11 GPa, marked by an abrupt increase in resistance and a reversal of the temperature coefficient of resistance (metallic to semiconducting behavior). The emergence of semiconducting-like behavior, together with low-temperature metallic recovery, signals the formation of competing correlated regimes. Whether this anomaly reflects the onset of coherent heavy-electron behavior or pressure-stabilized magnetic ordering, the data demonstrate that hp-YbZn₂ resides near a critical electronic instability that can be accessed through moderate compression.

More broadly, this work demonstrates that LHDAC synthesis can evolve from a structural discovery technique into a practical platform for engineering and probing metastable quantum materials. By integrating spatial structural verification, quantitative post-recovery phase analysis, and downstream electrical characterization, our approach establishes a practical route for recovering LHDAC-synthesized materials and enabling comprehensive ex-situ investigations beyond *in situ* structural identification.

SUPPLEMENTARY MATERIAL

The [supplementary material](#) includes the following: independently calculated diffraction profiles of the individual refined phases for LHDAC-grown MnSb₂ (SR006, in Fig. S1) and YbZn₂ (SR007, in Fig. S2), based on the laboratory PXRD data from two recovered pieces of each sample. These profiles illustrate the separate phase contributions to the total diffraction patterns shown in Figs. 4 and 5 of the main manuscript. The [supplementary material](#) also includes an Arrhenius-type analysis of the intermediate-temperature semiconducting-like regime in YbZn₂ (Fig. S3).

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

S. Huyan: Conceptualization (lead); Data curation (lead); Formal analysis (lead); Investigation (lead); Methodology (lead); Project administration (lead); Supervision (lead); Validation (lead); Visualization (lead); Writing – original draft (lead); Writing – review & editing (lead). **R. F. S. Penacchio:** Data curation (lead); Formal analysis (lead); Validation (equal); Visualization (equal); Writing – original draft (equal); Writing – review & editing (equal). **D. Zhang:** Data curation (equal); Formal analysis (equal); Resources (equal); Writing – review & editing (equal). **Z. Li:** Data curation (supporting); Formal analysis (supporting); Writing – review & editing (supporting). **S. L. Morelhão:** Formal analysis (supporting); Supervision (supporting); Writing – review & editing (supporting). **R. A. Ribeiro:** Formal analysis (supporting); Methodology (supporting); Resources (supporting); Supervision (supporting); Writing – review & editing (supporting). **P. C. Canfield:** Conceptualization (lead); Funding acquisition (lead); Investigation (equal); Methodology (lead); Project administration (lead); Resources (lead); Supervision (lead); Writing – original draft (lead); Writing – review & editing (lead). **S. L. Bud'ko:** Conceptualization (lead); Funding acquisition (lead); Investigation (lead); Methodology (lead); Project administration (lead); Resources (lead); Supervision (lead); Writing – original draft (lead); Writing – review & editing (lead).

DATA AVAILABILITY

The data that support the findings of this study are openly available in Zenodo at <https://doi.org/10.5281/zenodo.20982166>, Ref. 36.

REFERENCES

- S. Anzellini and S. Boccato, “A practical review of the laser-heated diamond anvil cell for university laboratories and synchrotron applications,” *Crystals* **10**(6), 459 (2020).
- M. E. Alabdulkarim, W. D. Maxwell, V. Thapliyal, and J. L. Maxwell, “A comprehensive review of high-pressure laser-induced materials processing, part I: Laser-heated diamond anvil cells,” *J. Manuf. Mater. Process.* **6**(5), 111 (2022).
- R. Boehler, “Diamond cells and new materials,” *Mater. Today* **8**, 34–42 (2005).
- H.-K. Mao, X. J. Chen, Y. Ding, B. Li, and L. Wang, “Solids, liquids, and gases under high pressure,” *Rev. Mod. Phys.* **90**, 015007 (2018).
- B. Wei, L. Lin, J. Zhang, Z. Zhan, Z. Cheng, and J. Jiang, “In situ measurement techniques using diamond anvil cell at high pressure–temperature conditions: A review,” *Phys. Status Solidi RRL* **18**, 2300469 (2024).
- L. C. Ming and W. A. Bassett, “Laser heating in the diamond anvil press up to 2000 °C sustained and 3000 °C pulsed at pressures up to 260 kilobars,” *Rev. Sci. Instrum.* **45**, 1115–1118 (1974).
- L.-G. Liu, “High-pressure phase transformations and compressions of ilmenite and rutile. I. Experimental results,” *Earth Planet. Sci. Lett.* **10**, 167 (1975).
- V. B. Prakapenka, A. Kubo, A. Kuznetsov, A. Laskin, O. Shkurikhin, P. Dera, M. L. Rivers, and S. R. Sutton, “Advanced flat top laser heating system for high pressure research at GSECARS: Application to the melting behavior of germanium,” *High Pressure Res.* **28**, 225 (2008).
- Z. Konôpková, W. Morgenroth, R. Husband, N. Giordano, A. Pakhomova, O. Gutowski, M. Wendt, K. Glazyrin, A. Ehnes, J. T. Delitz, A. F. Goncharov, V. B. Prakapenka, and H.-P. Liermann, “Laser heating system at the extreme conditions beamline, P02.2, PETRA III,” *J. Synchrotron Radiat.* **28**, 1747 (2021).
- A. P. Drozdov, M. I. Eremets, I. A. Troyan, V. Ksenofontov, and S. I. Shylin, “Conventional superconductivity at 203 kelvin at high pressures in the sulfur hydride system,” *Nature* **525**, 73 (2015).
- A. P. Drozdov, P. P. Kong, V. S. Minkov, S. P. Besedin, M. A. Kuzovnikov, S. Mozaffari, L. Balicas, F. F. Balakirev, D. E. Graf, V. B. Prakapenka, E. Greenberg, D. A. Knyazev, M. Tkacz, and M. I. Eremets, “Superconductivity at 250 K in lanthanum hydride under high pressures,” *Nature* **569**, 528 (2019).
- M. Somayazulu, M. Ahart, A. K. Mishra, Z. M. Geballe, M. Baldini, Y. Meng, V. V. Struzhkin, and R. J. Hemley, “Evidence for superconductivity above 260 K in lanthanum superhydride at megabar pressures,” *Phys. Rev. Lett.* **122**, 027001 (2019).
- M. Murakami, K. Hirose, K. Kawamura, N. Sata, and Y. Ohishi, “Post-perovskite phase transition in MgSiO₃,” *Science* **304**, 855 (2004).
- S. Tatenò, K. Hirose, Y. Ohishi, and Y. Tatsumi, “The structure of iron in Earth’s inner core,” *Science* **330**, 359 (2010).
- S. Anzellini, A. Dewaele, M. Mezouar, P. Loubeyre, and G. Morard, “Melting of iron at Earth’s inner core boundary based on fast X-ray diffraction,” *Science* **340**, 464 (2013).
- A. Friedrich, B. Winkler, E. A. Juárez-Arellano, and L. Bayarjargal, “Synthesis of binary transition metal nitrides, carbides and borides from the elements in the laser-heated diamond anvil cell and their structure-property relations,” *Materials* **4**, 1648–1692 (2011).
- M. E. Alabdulkarim, W. D. Maxwell, V. Thapliyal, and J. L. Maxwell, “A comprehensive review of high-pressure laser-induced materials processing, part III: Laser reactive synthesis within diamond anvil cells,” *J. Manuf. Mater. Process.* **7**, 57 (2023).
- J. P. S. Walsh, S. M. Clarke, Y. Meng, S. D. Jacobsen, and D. E. Freedman, “Discovery of FeB₂,” *ACS Cent. Sci.* **2**(11), 867–871 (2016).
- S. M. Clarke, M. Amsler, J. P. S. Walsh, T. Yu, Y. Wang, Y. Meng, S. D. Jacobsen, C. Wolverton, and D. E. Freedman, “Creating binary Cu-Bi compounds via high-pressure synthesis: A combined experimental and theoretical study,” *Chem. Mater.* **29**(12), 5276–5285 (2017).
- K. M. Powderly, S. M. Clarke, M. Amsler, C. Wolverton, C. D. Malliakas, Y. Meng, S. D. Jacobsen, and D. E. Freedman, “High-pressure discovery of β -NiBi,” *Chem. Commun.* **53**, 11241–11244 (2017).
- J. P. S. Walsh and D. E. Freedman, “High-pressure synthesis: A new frontier in the search for next-generation intermetallic compounds,” *Acc. Chem. Res.* **51**(6), 1315–1323 (2018).
- A. H. Manayil Marathamkottail, K. Wang, N. P. Salke, M. Ahart, A. C. Mark, R. Hrubciak, S. Chariton, D. Smith, V. B. Prakapenka, M. Somayazulu, N. Velisavljevic, and R. J. Hemley, “X-ray-diffraction and electrical-transport imaging of superconducting superhydride (La,Y)H₁₀,” *Nat. Commun.* **16**, 11222 (2025).
- R. Giampaoli, I. Kantor, M. Mezouar, S. Boccato, A. D. Rosa, R. Torchio, G. Garbarino, O. Mathon, and S. Pascarelli, “Measurement of temperature in the laser-heated diamond anvil cell: Comparison between reflective and refractive optics,” *High Pressure Res.* **38**, 250–269 (2018).
- C. E. Yen, Q. Williams, and M. Kunz, “Thermal pressure in the laser-heated diamond anvil cell: A quantitative study and implications for the density versus mineralogy correlation of the mantle,” *J. Geophys. Res.: Solid Earth* **125**, e2020JB020006, <https://doi.org/10.1029/2020jb020006> (2020).
- H. Takizawa, M. Shimada, Y. Sato, and T. Endo, “High-pressure synthesis of MnSb₂ with the marcasite-type structure,” *Mater. Lett.* **18**, 11 (1993).
- M. Xu, M. Boswell, Q. Ding, P. Cheng, A. Sapkota, Q. Zhang, D. Yahne, S. L. Bud’ko, Y. Furukawa, P. C. Canfield, R. A. Ribeiro, and W. Xie, “Pressure-stabilized MnSb₂ with complex incommensurate magnetic order,” *arXiv:2603.09635* (2026).
- D. A. Salamatina, K. V. Klementiev, V. N. Krasnorussky, M. V. Magnitskaya, N. M. Chitchelekachev, V. A. Sidorov, A. V. Semeno, A. V. Bokov, M. G. Kozin, A. V. Nikolaev, A. V. Salamatina, A. Velichkov, M. V. Mikhin, M. Budzynski, and A. V. Tsvyashchenko, *J. Alloys Compd.* **946**, 169275 (2023).
- A. Dewaele, M. Torrent, P. Loubeyre, and M. Mezouar, “Compression curves of transition metals in the Mbar range: Experiments and projector augmented-wave calculations,” *Phys. Rev. B* **78**(10), 104102 (2008).

- ²⁹G. Shen, Y. Wang, A. Dewaele *et al.*, "Toward an international practical pressure scale: A proposal for an IPPS ruby gauge (IPPS-Ruby2020)," *High Pressure Res.* **40**, 299–314 (2020).
- ³⁰C. Prescher and V. B. Prakapenka, "DIOPTAS: A program for reduction of two-dimensional x-ray diffraction data and data exploration," *High Pressure Res.* **35**, 223–230 (2015).
- ³¹B. H. Toby and R. B. Von Dreele, "GSAS-II: The genesis of a modern open-source all-purpose crystallography software package," *J. Appl. Crystallogr.* **46**, 544–549 (2013).
- ³²H. Toraya, "Review of the direct derivation method: Quantitative phase analysis with observed intensities and chemical composition data," *Powder Diffr.* **36**(3), 159–168 (2021).
- ³³J. Wittig, "A study of the superconductivity of antimony under pressure and a search for superconductivity in arsenic," *J. Phys. Chem. Solids* **30**, 1407–1410 (1969).
- ³⁴D. Schiferl, D. T. Cromer, and J. C. Jamieson, *Acta Crystallogr., Sect. B* **37**, 807 (1981).
- ³⁵O. Degtyareva, M. I. McMahon, and R. J. Nelmes, *High Pressure Res.* **24**, 319 (2004).
- ³⁶S. Huyan, R. Penacchio, D. Zhang, Z. Li, S. L. Morelhaio, R. A. Ribeiro, P. C. Canfield, and S. L. Bud'ko (2026), "A practical laser-heated diamond anvil cell synthesis technique and recovery workflow for metastable MnSb₂ and YbZn₂ phases [Dataset]," Zenodo. <https://doi.org/10.5281/zenodo.20982166>