Room-temperature skyrmion phase in bulk $\text{Cu}_2\text{OSeO}_3$ under high pressures

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A skyrmion state in a noncentrosymmetric helimagnet displays topologically protected spin textures with profound technological implications for high-density information storage, ultrafast spintronics, and effective microwave devices. Usually, its equilibrium state in a bulk helimagnet occurs only over a very restricted magnetic field–temperature phase space and often in the low-temperature region near the magnetic transition temperature $T_c$. We have expanded and enhanced the skyrmion phase region from the small range of 55 to 58.5 K to 5 to 300 K in single-crystalline $\text{Cu}_2\text{OSeO}_3$ by measuring the isothermal ac magnetic susceptibility $\chi'(H)$ at $T=0$ with an alternating current (ac)-modulation field of 3 Oe at 10 Hz and direct current (dc) magnetization $M(H)$ at different temperatures $T$ up to 300 K and pressures $P$ up to 42.1 GPa, respectively. All magnetization measurements have been carried out in the zero-field-cooled (ZFC) mode to eliminate the possible $H$- and/or $T$-history dependence of the skyrmion state. Representative $\chi'(H)|_{T=0}$ results of $\text{Cu}_2\text{OSeO}_3$ at ambient pressure and at different temperatures are shown in Fig. 1. The magnetic fields ($H_{A1}$, $H_{A2}$, $H_{A3}$, and $H_{A4}$) that define the various phase boundaries at ambient are indicated by the arrows in the same figure. As exhibited in Fig. 1, $\chi'(H)|_{T=0}$ displays anomalous behavior within a narrow field region ($H_{A1}$, $H_{A2}$) or a $\Delta H \equiv H_{A1} - H_{A2}$.

In a noncentrosymmetric helimagnetic compound, the complex competitions among the various magnetic interactions in decreasing strengths, that is, the exchange interaction, the Dzyaloshinskii–Moriya (DM) spin-orbit interaction, and the crystalline anisotropy, result in a generic but complex magnetic field ($H$)–temperature ($T$) phase diagram. For instance, on cooling below the magnetic transition temperature $T_c$, $\text{Cu}_2\text{OSeO}_3$ undergoes a paramagnetic-to-helical magnetic transition in a low $H$ less than ~0.5 kOe but a paramagnetic-to-conical magnetic transition in an intermediate $H$ below ~2 kOe and a paramagnetic-to-ferrimagnetic transition in a large $H$ above ~2 kOe (1–4). In this generic $H$–$T$ phase diagram, the skyrmion phase occurs in a very restricted region near $T_c$ ~58 K, as depicted schematically in Fig. 1. Magnetic skyrmions on the scale of approximately tens of nanometers emerge with vortex-like spin textures and form the skyrmion lattice state, which has been detected by means of small-angle neutron scattering (SANS) (2), resonant elastic X-ray scattering (6, 7), Lorentz force transmission electron microscopy (LTEM) (1, 8, 9), magnetic force microscopy (10, 11), electron holography (12), optical polarization rotation measurements (13), and magnetization measurements (1, 3, 4, 14, 15). As a result, great potential has been envisioned for skyrmions for high-density information storage, ultrafast spintronics, and efficient microwave devices (16, 17). To facilitate such a vision, we have decided to enhance the equilibrium skyrmion phase space in bulk $\text{Cu}_2\text{OSeO}_3$ to a broader and higher-temperature region by the application of pressure. We have successfully increased the skyrmion temperature space of $\text{Cu}_2\text{OSeO}_3$ from 55 to 58.5 K to 5 to 300 K, up to room temperature, under pressures up to 42.1 GPa, following a series of structural phase transitions, in agreement with predictions based on the Ginzburg–Landau free energy consideration. The observations will allow for easier device operations and show that high-temperature skyrmion lattices may be found in more helimagnets with different structures.

Results and Discussion

Magnetic Studies at Ambient and under High Pressure. We have adopted the magnetic technique to identify the skyrmion phase in single-crystal $\text{Cu}_2\text{OSeO}_3$ by measuring the isothermal ac magnetic susceptibility $\chi'(H)|_{T=0}$ with an alternating current (ac)-modulation field of 3 Oe at 10 Hz and direct current (dc) magnetization $M(H)|_{T=0}$ as a function of magnetic field $0 < H \leq 1 \text{kOe}$ at different temperatures $T$ up to 300 K and pressures $P$ up to 42.1 GPa, respectively. All magnetization measurements have been carried out in the zero-field-cooled (ZFC) mode to eliminate the possible $H$- and/or $T$-history dependence of the skyrmion state. Representative $\chi'(H)|_{T=0}$ results of $\text{Cu}_2\text{OSeO}_3$ at ambient pressure and at different temperatures are shown in Fig. 1. The magnetic fields ($H_{A1}$, $H_{A2}$, $H_{A3}$, and $H_{A4}$) that define the various phase boundaries at ambient are indicated by the arrows in the same figure. As exhibited in Fig. 1, $\chi'(H)|_{T=0}$ displays anomalous behavior within a narrow field region ($H_{A1}$, $H_{A2}$) or a $\Delta H \equiv H_{A1} - H_{A2}$

Significance

Skyrmion materials hold great promise for information technology due to the extremely low current needed to modify the spin configurations and the small size of magnetic domains. To facilitate their application, one great challenge is to break the magnetic field–temperature phase space restriction for the skyrmion state. We found that the temperature region for the skyrmion phase in bulk $\text{Cu}_2\text{OSeO}_3$ can be greatly enhanced under physical pressure, making applications more practical by the use of strained heterostructures, for example. The observation of additional structures suggests that the skyrmion state may be insensitive to the underlying crystal structure. This work will stimulate research on finding skyrmion materials with different crystal structures and retaining the room-temperature skyrmion state at ambient condition.
region $\sim 200$ Oe, over a small temperature range $(T_{A1}, T_{A2})$ or a $\Delta T = T_{A2} - T_{A1} = 58.5$ to $55$ K range $\sim 3.5$ K, where the skyrmion state has been shown to exist by LTEM (1) and SANS (2). These results are summarized in Fig. 1B, which shows the skyrmion state embedded in the magnetic phase diagram of Cu$_2$OSeO$_3$ together with the known helical, conical, and ferromagnetic phases, in agreement with previous reports (1–4). The schematics for the conical phase, the skyrmion phase, and the skyrmion core based on our model calculation are displayed in Fig. 1C. The narrow field-temperature window for the skyrmion state is also evident from the isothermal $M(H)_{57 \text{ K}}$, which displays the rapid deepening in the slopes of $M(H)_{57 \text{ K}}$ and $\chi'_{ac}$ as a function of magnetic field up to $1,000$ Oe at $57$ K. arb., arbitrary.

Our recently developed ultrasensitive high-pressure magnetization technique (19) was used to investigate the skyrmion phase evolution under high pressure. Under pressure the magnetic phase transition temperature $T_c$, which defines the upper bound of the skyrmion phase, is shifted toward higher temperature as shown in Fig. 2. Above $7.9$ GPa, it becomes increasingly difficult to distinguish the magnetic transition from the background signal based on dc magnetization measurements. We have measured the $\chi'_{ac}(H)_{T,P}$ as a function of $H$ at different $P$ up to $42.1$ GPa and $T$ up to $300$ K and the accompanying $H$–$T$ regions where the skyrmion state occurs at selected pressures, for example 2.5, 7.9, 26.2, and 42.1 GPa, as shown in Fig. 3A–D, respectively. It is clear that the temperature region $(T_{A1}, T_{A2})$ for skyrmions, or $\Delta T = (T_{A2} - T_{A1})$, has been expanded from $(55$ K, $58.5$ K), that is, $\sim 3.5$ K, at ambient to $(5$ to $10$ K, $>300$ K), that is, $>290$ K, at $42.1$ GPa by lowering $T_{A1}$ and raising $T_{A2}$ to above $300$ K via pressures.

Fig. 1. Magnetic measurements of Cu$_2$OSeO$_3$ at ambient pressure. (A) ac susceptibility as a function of dc bias field up to $900$ Oe at different temperatures for a single-crystal Cu$_2$OSeO$_3$ sample at ambient pressure. The curves are shifted vertically within the same scale. (B) Magnetic phase diagram for a single-crystal Cu$_2$OSeO$_3$ sample at ambient pressure. (C) Schematics for conical and skyrmion phases. The direction of the arrows denotes the direction of magnetization and the color denotes the $z$-component of magnetization. The conical phase is plotted from the conical ansatz in SI Appendix, section S2, while the skyrmion phase is schematic. (D) $M$, (E) $dM/dH$, and (F) $\chi'_{ac}$ as a function of magnetic field up to $1,000$ Oe at $57$ K. arb., arbitrary.

Fig. 2. dc magnetization measurements of Cu$_2$OSeO$_3$. $M$ vs. $T$ measured on a single-crystal sample under magnetic field of $250$ Oe and at different pressures up to $7.9$ GPa in ZFC mode. The arrows indicate the ferromagnetic transition temperature at different pressures.
in agreement with the results shown in Fig. 2 for $T_c$ and makes the skyrmion state accessible without the aid of liquid cryogen for device applications. At the same time, the field region ($H_{TA1}$, $H_{TA2}$) or $\Delta H \equiv H_{TA2} - H_{TA1}$ remains almost unchanged, keeping the accessible field low, despite the great expansion in $\Delta T$. The observation is in qualitative agreement with previous reports at low pressures up to 1.4 GPa by Wu et al. (4) and up to 5.7 GPa by Sidorov et al. (20), who raised $T_{A2}$ from ~56 K to 60.5 K and $T_c$ to 75 K, respectively. It is worth mentioning that while $T_{A2}$ and $\Delta T$ are observed to increase smoothly with pressure, they suffer a drastic increase at 7.9 GPa, suggesting a possible pressure-induced structure transition to be explored below.

**Synchrotron X-Ray Measurements under High Pressure.** The sudden expansion of $\Delta T$ resulting from the rapid increase of $T_{A2}$ around 7.9 GPa and the precipitous drop of $T_{A1}$ around 26.2 GPa for the skyrmion state strongly suggest possible pressure-induced structural transitions in Cu$_2$OSeO$_3$. Because of the small volume (~0.003 mm$^3$) of our sample in a high-pressure diamond anvil cell (DAC), we decided to carry out the structural study using synchrotron X-ray diffraction (XRD). Room-temperature synchrotron XRD with a wavelength of 0.6889 Å (18 keV) was performed and the patterns were analyzed. As shown in Fig. 4, they display the cubic $P2_12_12_1$ phase with the lattice constant $a$ of 8.9193 Å, consistent with a previous report (3). The same crystal structure persists as the pressure increases to 3.96 GPa. However, at 5.28 GPa, new Bragg reflection peaks emerge, indicating the breaking of crystal symmetry. This pattern can be indexed within the orthorhombic phase with the $P2_12_12_1$ space group (losing the threefold rotational symmetry) with lattice parameters $a = 8.7988$ Å, $b = 8.7790$ Å, and $c = 8.7409$ Å. At ~7.01 GPa, Cu$_2$OSeO$_3$ undergoes a second structural transition to the monoclinic phase with the $P2_1$ space group (losing the $I_3$ screw axis symmetry). Consequently, the schematic diagram of relevant pressure-induced structural phases was established and is shown in Fig. 4B, where the cubic $P2_12_12_1$ phase, the orthorhombic $P2_12_12_1$, and the monoclinic $P2_1$ are marked in black, blue, and red, respectively. The results add two additional structure phases below 11 GPa, the limit of our synchrotron XRD experiment, that can host the skyrmions.

**Raman Measurements under High Pressure.** Raman spectra taken with increasing pressure are presented in Fig. 5A and provide further experimental evidence for the existence of several crystallographic

![Fig. 3. ac susceptibility of Cu$_2$OSeO$_3$ as a function of magnetic field at different critical pressures: (A) 2.5 GPa, (B) 7.9 GPa, (C) 26.2 GPa, and (D) 42.1 GPa. The evolution of the “dip figure” indicates that the temperature region for the possible skyrmion state expands under pressure. At 7.9 GPa, the upper limit of the temperature range, $T_{A2}$, increases to 300 K, the highest temperature measured in this experiment. At 26.2 GPa, the lower limit of the temperature range, $T_{A1}$, extends to 5 K. With increasing pressure up to 42.1 GPa, the “dip feature” becomes more pronounced while the temperature range remains at between 5 to 10 K and 300 K.](image-url)

![Fig. 4. Pressure dependence of XRD patterns. (A) Evolution of room-temperature synchrotron XRD patterns for a polycrystalline Cu$_2$OSeO$_3$ sample under high quasi-hydrostatic pressure up to 10.47 GPa, indicating multiple structural phase transitions. (B) Schematic diagram representing the pressure-induced structural phase transitions in Cu$_2$OSeO$_3$. It should be noted that 1) the initial cubic $P2_12_12_1$ phase transforms into the orthorhombic $P2_12_12_1$ phase at 5.28 GPa and 2) the second structural transition from the orthorhombic $P2_12_12_1$ to the monoclinic $P2_1$ occurs at 7.01 GPa.](image-url)
TA2 increases as the pressure increases. At the transition to phase IV, TA2 extends to a lower temperature, that is, 5 to 10 K, while TA1 remains at room temperature.

For a noncentrosymmetric helimagnet, the Ginzburg–Landau free energy can have the form \( f = f_0 + J(VM)^2 + DM \cdot (\nabla \times M) \) (22–24), where \( M \) is the magnetization, \( (VM)^2 \equiv \sum_{ij} a_{ij} M_i M_j \), and \( J \) and \( D \) denote Heisenberg and DM interactions among magnetic moments, respectively. The uniform part of the free energy is \( f_0 = a(T - T_m)M^2 + bM^4 \) at temperature \( T \), with phenomenological parameters \( a \), \( b \), and \( T_m \) determined by microscopic details of the magnet. From the free energy described above, another temperature scale \( T_0 = D^2/Ja \) can be obtained by dimensional analysis. As demonstrated in SI Appendix, section S2, the temperature region for skyrmions \( \Delta T \equiv (T_{A2} - T_{A1}) \) should be proportional to the temperature scale \( T_0 \). It should be noted that \( D \) measures the breaking of centrosymmetric symmetry, so as we increase the pressure and reduce the symmetry, \( D \) should increase and show sudden jumps near structural transitions. As a result, the structural transitions induced by high pressure lead to \( \Delta T_I < \Delta T_{II} < \Delta T_{III} < \Delta T_{IV} \), where \( \Delta T \) represents the temperature region for skyrmions in phase \( i \) (\( i = I \) to IV). These results are consistent with experimental results presented in Figs. 3–5. In general, the critical temperatures can be computed by numerical methods such as Monte Carlo simulations (25). It is worth mentioning that since the surface and/or interface can affect DM interaction (1), perhaps associated with the strain relaxation due to the sample thickness, the change of sample thickness under pressure may also play an important role in the enhanced skyrmion region we observed.

**Summary**

In summary, we have carried out a systematic study of the high-pressure effect on the skyrmion phase in Cu2OSeO3 via magnetization, synchrotron X-ray, and Raman spectra measurements. We also performed Ginzburg–Landau free energy analysis on the crystal symmetry and DM interaction terms, which affect the temperature region for the skyrmion phase. We found that pressure favors the skyrmion phase in this system and that the structural transitions induced by higher pressure help to extend the temperature region of the skyrmion phase immensely. SANS measurements under high pressure are currently being taken to obtain direct evidence for the pressure-induced room-temperature skyrmion phase. The results demonstrate the potential role of stress/strain in the stabilization of the skyrmion phase at room temperature. They further suggest that chemical pressure may be deployed to retain the phase at ambient condition, similar to stabilizing high-temperature superconductivity by replacing ions in the compound with smaller isovalent ions (26, 27). The appearance of the skyrmion phase in different crystal structures of Cu2OSeO3 implies that the skyrmion phase is not sensitive to the symmetry of the underlying host, suggesting that more host materials can be found. New synthesis routes, for example chemical doping/catalyzing or pressure quenching, may be helpful in retaining the high-pressure and room-temperature skyrmion phase in this and other materials at ambient pressure for applications.

**Materials and Methods**

**Crystal Growth.** The single-crystalline samples of Cu2OSeO3 were grown from the polycrystalline compound by the chemical vapor deposition method. Pure polycrystalline Cu2OSeO3 was synthesized using the solid-state-reaction technique by heating pressed pellets of stoichiometric mixture of high-purity CuO and SeO2 in an evacuated quartz tube at 510 to 600 °C for 72 h followed by slowly cooling to room temperature. To improve the sample quality, intermediate breaking, grinding, and pressing of the pellet was performed several times. The XRD patterns of the samples at ambient determined by a Rigaku D-MAX-BII diffractometer show the high quality of the samples investigated.
Magnetization Measurements under High Pressure. The skyrmion state has been shown to be easily identified by the ac susceptibility ($\chi''$) as a function of dc magnetic field (H) at different temperature (T). To determine the pressure effect on the skyrmion state we have deployed our ultrasensitive high-pressure magnetization technique using the DAC incorporated within a Quantum Design Magnetic Property Measurement System. The technique allows us to measure the dc and ac magnetization of Cu2OSeO3 single crystals with diagonal ~100 μm and thickness of a few micrometers at a T between 5 and 300 K for an H up to 1 kOe under pressure up to 42.1 GPa. Since the skyrmion state has been shown to be thermal- and field-history-dependent, we chose to measure all isothermal and isobaric magnetizations, $\chi''(H)$, following the ZFC mode. The pressure experienced by the sample inside the DAC is determined by the fluorescence line of ruby powders and the Raman spectrum from the culet of the top diamond anvil. A pair of 300-μm-diameter culet-sized diamond anvils was used. The anvils were made from nonmagnetic Ni–Cr–Al alloy. Each gasket was preindentated to about 80- to 100-μm culet size. The aperture of the DAC is up to 85° for obtaining high-Q diffraction data. Stainless steel gaskets were preindentated to about 80- to 100-μm thickness, drilled in the middle of the indentation to obtain a ~200-μm hole, and placed between two diamonds to form a pressure sample chamber. The pressure chamber with the sample and a small amount of ruby powder was loaded and sealed with silicone oil as the pressure medium (Alfa Aesar, Polydimethylsiloxane, trimethylsiloxy terminated, M.W. 410 CAS:43669). Pressure was determined using the ruby R1 fluorescence line as a pressure marker by Raman spectrometer iHR550 (Horiba Jobin Yvon).

Raman Spectroscopy under High Pressure. The pressure cell and sample were prepared using a method similar to that described in Magnetization Measurements under High Pressure. All light scattering measurements were performed in the back-scattering geometry at room temperature using the triple Raman spectrometer T64000 (Horiba Jobin Yvon) equipped with a microscope, a liquid-nitrogen-cooled charge-coupled-device detector, and an Ar+ laser ($\lambda_{\text{exc}} = 488$ nm) as the excitation source. Laser excitation power was kept below 1 mW in order to minimize heating of the sample. The spectral resolution is 1.5 cm$^{-1}$.

Data Availability. All data, materials, and experimental procedures that support the findings of this study are shown in Materials and Methods and SI Appendix.

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