Growth and Helicity of Noncentrosymmetric Cu$_2$OSeO$_3$ Crystals

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1. Introduction

Investigation of complex magnetic systems is generally limited by an inability to obtain sufficiently large, pure high-quality crystals. This is especially true for the noncentrosymmetric magnets with chirality. In this class of materials, the interactions that may lead to symmetry breaking magnetic order do not cancel each other when evaluated over the unit cell. The most well-studied chiral systems are MnSi, Mn1–xFexGe, and semiconducting Fe1–xCo3Si. In these chiral magnets, the principal magnetic phases are helical phases, a single-domain conical phase, and a skyrmion state (known as A-phase). They appear in a small magnetic field–temperature (B–T) pocket close to transition temperature Tc. In the chiral atomic framework of this crystal family, the orbital motions of localized electrons also take helical paths. The neighboring spins of localized electrons are coupled by the relativistic spin–orbit interaction called Dzyaloshinskii–Moriya (DM) interaction. As the sign of the DM interaction is determined by the chemical composition, it emphasizes that the magnetic chirality is intrinsically dependent on the lattice handedness. It has been shown experimentally in Mn1–xFexGe crystals that skyrmion helicity is directly determined by the crystal helicity.

Cu2OSeO3 is one of the most important members of the chiral group with the P213 chiral cubic crystal structure. It is the first insulator in which the skyrmion lattice has been observed with a very similar B–T phase diagram as the other related members of this chiral group. Recently, some new magnetic phases like tilted conical spiral, low-temperature skyrmion lattice phase, and elongated skyrmions have been observed in Cu2OSeO3. The insulating behavior of this magnetic material makes the study of the decisive role of crystal helicity especially more interesting by excluding other contributions due to conduction electrons. To understand the unique magnetic structure of Cu2OSeO3, several different techniques have been used including muon spin rotation/relaxation (μSR), Lorentz transmission electron microscope, ac-susceptibility measurements, terahertz electron spin resonance, and time-resolved magneto-optics. Recently, generation of spin currents has been studied in Cu2OSeO3 by spin-pumping experiments.

Considering the large interest in the magnetic properties of Cu2OSeO3, it is important to look for new, efficient, and fast single-crystal growth techniques. Conventionally, Cu2OSeO3 crystals are grown by the vapor transport method with HCl gas as transport agent (TA). With this growth method only one helicity has been reported. The other helicity has not been reported to the best of our knowledge. It is known that the structural and magnetic chiralities for Cu2OSeO3 crystals are directly
related with each other.\textsuperscript{19} Therefore to use both magnetic chiral-
ities, it is needed to improve the growth techniques not only to
speed up the growth rate but also to get crystals with both chir-
als. Here, we report a new and fast way for the growth of
Cu2OSeO3 single crystals with SeCl4 as TA. We observed
very-high-quality crystal growth yielding both chiralities with this
new growth technique. The crystal structure of Cu2OSeO3
crystals has been studied before\textsuperscript{20–22} with different diffraction
techniques. Here, we used the simplest single-crystal X-ray dif-
fraction (XRD) to establish the absolute structures for both hand-
eedness, which also emphasizes good quality of the crystals.

2. Experimental Section

Single crystals of Cu2OSeO3 were grown by the standard chemi-
cal vapor transport method. However, the novelty of this growth
was the use of selenium tetrachloride (SeCl4) as a TA. Previously,
SeCl4 was mainly used to grow molybdenum and tungsten dis-
elenides. In literature, Cu2OSeO3 is usually grown by HCl gas.\textsuperscript{23}
Here, we report the growth of chiral magnets with SeCl4 as TA,
which is new and different from literature.\textsuperscript{24} For growth, trans-
parent quartz ampoules (30 mm inside diameter, 30 cm length)
were used. They were first carefully cleaned with ethanol, ace-
tone, 10% HF, and demineralized water overnight at 200 °C
before the charge was introduced. SeCl4 is very hygroscopic;
therefore, it was weighed and introduced into the transport tubes
in a glove box in nitrogen atmosphere. Mixtures of high-purity
CuO (Alfa-Aldrich, 99.995%) and SeO2 (Alfa-Aldrich, 99.999%)
powders in a molar ratio of 2:1 were sealed in an evacuated
quartz ampoule with 0.54 g of SeCl4 (Alfa-Aldrich, 99.5%).
After a few minutes of degassing, the part of the ampoule con-
taining chemicals was immersed in liquid nitrogen and sub-
sequently evacuated and sealed after the chemicals cooled below
evaporation temperatures. The ampoule was then placed hori-
zontally into a tubular three-zone furnace having 18 cm-long
zones separated by a distance of 3 cm. The temperature of the
furnace was raised gradually by 50 °C h\textsuperscript{−1} to 600 °C. To get
rid of unwanted nucleation centers, a reverse temperature grad-
ient was applied by adjusting the temperature of the source zone
\(T\textsubscript{hot}\) to 610 °C and the deposition zone \(T\textsubscript{cold}\) to 660 °C for 24 h.
Afterward, \(T\textsubscript{hot}\) and \(T\textsubscript{cold}\) were adjusted to 610 °C and 570 °C,
respectively, for growth. These furnaces were regulated by a
PID electronic regulator (SHINKO) with ±0.5 °C temperature
stability at 500–650 °C. After 2 weeks, shiny crystals were seen
at the deposition zone. After 4 weeks, the ampoules were
quenched at the source zone so that all gas vapors condensed at
the source zone. The extreme hygroscopic nature of SeCl4
resulted in the presence of water in the ampoules, in spite of
all precautions taken. The presence of water created the vapor
phase of hydrogen chloride (HCl) and chalcogen oxichloride
(SeOCl\textsubscript{2}), thus making the analysis of the transport mechanism
more complex. However, we observed that the presence of mois-
ture slowed down the transport process. This transport method
with SeCl4 resulted in reasonably big and thick crystals. To com-
pare the efficiency of the growth method, we also synthesized the
crystals with HCl gas as TA, as reported in literature.\textsuperscript{23}
The growth conditions are shown in Table 1. The crystal structure
of Cu2OSeO3 crystals was investigated by single-crystal XRD

| TA     | \(T\textsubscript{hot} [°C]\) | \(T\textsubscript{cold} [°C]\) | Duration of growth [d] | Maximum size of crystals [mm\textsuperscript{3}] |
|--------|-----------------|-----------------|----------------|-----------------|
| HCl\textsuperscript{24} | 620             | 580             | 49              | 130–150          |
| SeCl4  | 610             | 570             | 23              | 210–224          |

on a D8 Venture diffractometer. The crystal quality was checked
by collection of a full sphere of XRD data using high-precision
scans. The morphology and elemental analysis were examined
using Philips XL 30 scanning electron microscopy (SEM),
equipped with an energy-dispersive spectrometer (EDS) system,
which was operated at an accelerating voltage of 20 kV. The mag-
netization measurements were carried out using a Quantum
Design magnetic measurement system (MPMS-XL) 7 SQUID
magnetometer.

To further study the phase magnetic phase diagram of
Cu2OSeO3, we carried out ferromagnetic resonance (FMR)
measurements using a broadband spin-wave spectroscopy tech-
nique\textsuperscript{21,25} on both HCl and SeCl4 grown crystals. For this pur-
pose, we used two polished crystals with similar dimensions:
HCl-grown sample with size 1.5 \(\times\) 2.5 \(\times\) 0.5 mm\textsuperscript{3} and
SeCl4-grown crystals with size 2.9 \(\times\) 2.7 \(\times\) 1 mm\textsuperscript{3}. The crystals
were polished using the technique reported in the study by
Aqeel et al.\textsuperscript{26} The coplanar waveguides (CPWs) with a signal line of
50 μm width and gap of 25 μm width were patterned. The
CPWs were directly patterned onto the oriented polished crystals
with (110) and (111) surfaces, respectively. They were patterned
by e-beam lithography followed by e-beam evaporation of Ti
(10 nm)/Au (150 nm). The excitation field distribution of the
CPWs is shown in Figure 4b. The samples with CPWs were
mounted on a continuous-flow cryostat. A vector network ana-
lyzer (VNA) was used to measure the resonance signals. The tem-
perature reading of the cryostat was different from the MPMS
system used to measure the magnetization data due to the place-
ment of the temperature sensor. The temperature difference
(7–10 K) between both setups was adjusted in the experiment.
A rotatable electromagnet was used to provide the static magnetic
field up to 500 mT.

3. Results

A tiny single crystal grown with SeCl4 was selected for
morphology and element analysis, as shown in Figure 1a. The
as-synthesized crystal has a rough surface with many tiny nano-
particles attached on it. The molar ratios of Se, Cu, and O are
determined to be very close to the stoichiometric Cu2OSeO3 sam-
ple. Furthermore, the EDS elemental mapping (Figure 1b–d)
demonstrates that the Cu, O, and Se atoms are uniformly distrib-
uted, which unambiguously reveals the uniformity of the single
crystal. That is, a homogeneous and high-quality sample was suc-
scessfully synthesized with such a simple method. Table 2 shows
the parameters used to establish the absolute structure of
Cu2OSeO3 single crystals. Cu2OSeO3 crystals display the
P2\textsubscript{1}3 space group and the ions occupy the Wyckoff positions (WFs)
The chirality of the crystals has been characterized by the Flack parameter analysis. The Flack parameter is defined for a racemic twin of noncentrosymmetric crystals as the ratio between the scattering power of the two opposite-handed domains, giving rise to a resonant contribution in the X-ray-scattering amplitudes. A Flack parameter equal to zero corresponds to a single domain of the chiral structure (enantipure) and a Flack parameter equal to 1 represents a single domain structure but with opposite chiralities. The Flack parameter \( x \) is determined simultaneously with the atomic coordinates and atomic displacement parameters during the least-squares refinement of the crystal structure, using the twin model and thus enabling to determine the absolute structure of the crystal of a pure enantiomeric sample. The Flack parameter \( x \) for intensities of \( hkl \) reflections is as follows.

\[
I_{calc} = (1 - x)|F_{hkl}|^2 + x|F_{-h-k-l}|^2
\]

Here, \( |F_{hkl}| \) and \( |F_{-h-k-l}| \) represent the structure factors. The full sphere of Bragg reflections was used for refinement. Results of least-square refinement gives a Flack \( x \) of 0.013 (17), indicating two absolute structures having opposite chirality. The deviation factor is defined as

\[
R_1 = \frac{\sum |F_{obs} - |F_{calc}||/ \sum |F_{obs}|}{2}
\]

The reliability factors \( R_1 \) and \( wR_2 \), between the model and the data set, were found to be 0.0217 and 0.1473, respectively. We measured eight crystals to resolve the absolute structure, in which we found five right-handed and three left-handed enantiomers. The atomic coordinates for absolute structures for left-handed and right-handed enantiomers of \( \text{Cu}_2\text{OSeO}_3 \) (Figure 2) are shown in Table 3.

Figure 3a shows the temperature dependence of field-cooled magnetization measurements under an applied field \( H \) varying from 100 to 1500 Oe with a \( T_c \approx 60 \, \text{K} \).

The FMR spectra were measured as reported earlier[13] and the background-free transmission signal was defined as

\[
\Delta S_{21}^2 = (|S_{21}(H)| - |S_{21}(H_0)|)^2.
\]

\( S_{21}(H) \) and \( S_{21}(H_0) \) are the complex transmissions measured with a VNA at fixed magnetic fields \( H \) and \( H_0 \), respectively. An example of such spectra measured for HCl- and SeCl4-grown samples is shown in Figure 4d,e, respectively. These spectra are used to construct the high-temperature magnetic phase diagram of \( \text{Cu}_2\text{OSeO}_3 \) shown in Figure 4f.

\[\text{Temperature} \quad 100 \, \text{K} \]

| Crystal system         | Cubic         |
|------------------------|---------------|
| Space group            | \( P2_1 \_3 \) |
| Wave length            | 0.7107 Å      |
| Unit cell dimension    | 8.9446 Å      |
| \( \theta \) range for data collection | 3.147°−32.25° |
| Limiting indices       | \(-13 \leq h \leq 13\) |
|                       | \(-11 \leq k \leq 11\) |
|                       | \(-13 \leq l \leq 13\) |
| Reflected collected/unique | 0.0367       |
| Final \( R \) indices | 0.0312        |
| Absolute structure parameter | −0.01(2)     |

that are shown in Table 3. The precision scans of XRD for full-sphere approximation show the high quality of \( \text{Cu}_2\text{OSeO}_3 \) single crystals without any twinning.

Table 3. Atomic coordinates and WFs for \( \text{Cu}_2\text{OSeO}_3 \) for both handedness.

| WP | \( x \)   | \( y \)   | \( z \)   | \( x \)   | \( y \)   | \( z \)   |
|----|----------|----------|----------|----------|----------|----------|
| Cu (1) | 4a | 0.88589(3) | 0.88589(3) | 0.88589(3) | 0.11404(4) | 0.11404(4) | 0.11404(4) |
| Cu (2) | 12b | 0.13439(3) | 0.12108(3) | 0.87247(3) | 0.86549(4) | 0.87895(4) | 0.12754(4) |
| Se (1) | 4a | 0.45963(3) | 0.45963(3) | 0.45963(3) | 0.54031(4) | 0.54031(4) | 0.54031(4) |
| Se (2) | 4a | 0.88589(3) | 0.88589(3) | 0.88589(3) | 0.85649(4) | 0.87895(4) | 0.12754(4) |
| O (1) | 4a | 0.01031(3) | 0.01031(3) | 0.01031(3) | 0.78802(4) | 0.78802(4) | 0.78802(4) |
| O (2) | 12b | 0.76232(2) | 0.76232(2) | 0.76232(2) | 0.98974(3) | 0.98974(3) | 0.98974(3) |
| O (3) | 4a | 0.27029(2) | 0.48318(2) | 0.46954(2) | 0.23730(3) | 0.23730(3) | 0.23730(3) |
| O (4) | 12b | 0.27257(2) | 0.18681(2) | 0.03276(2) | 0.72786(3) | 0.81329(4) | 0.96738(3) |
4. Discussion

The vapor transport technique\[23\] commonly used for the growth of Cu$_2$OSeO$_3$ single crystals is relatively slow and complex due to use of the HCl gas as TA. However, the method reported in this article is very simple and easy due to use of solid TA SeCl$_4$ and is also found to be relatively fast. A disadvantage of SeCl$_4$ TA could be the strong silica attack and its strong hygroscopic nature, which can be easily settled using the TA in an inert and dry atmosphere. SeCl$_4$ is frequently used in the past as an efficient TA for the growth of diselenides WSe$_2$ and MoSe$_2$. Like SeCl$_4$, TeCl$_4$ can also be an efficient TA. TeCl$_4$ is more stable and less hygroscopic compared with SeCl$_4$ which makes it a more suitable TA compared with SeCl$_4$ for vapor transport growth. However, TeCl$_4$ can dope the crystals and therefore, SeCl$_4$ is more suitable for growth of undoped Cu$_2$OSeO$_3$ crystals. The decomposition of SeCl$_4$ will give a mixture of selenium and dichlorine that can result in possible gaseous oxygen compounds during transport of SeCl$_4$, which can be SeO$_2$, SeOCl$_2$, and SeO. Chlorine resulting from the decomposition of SeCl$_4$ probably plays an efficient role in transport but the role of the selenium is not very clear in transport. In the case of the presence of water, the transport would be more complicated by also involving HCl vapors. We observed a clear decrease in the deposition rate by exposing the SeCl$_4$ TA to the air.

We observed a clear decrease in the deposition rate by exposing the SeCl$_4$ TA to the air. The absolute structures were solved for six different crystals, grown with SeCl$_4$ as TA. During refinement, the Goodness of Fit (GooF) was found to be 0.9–1.03 and the scale factor $K$ is 0.95–1.0, which confirms the high quality of these crystals. Four out of six analyzed crystals showed the same helicity and the other two crystals showed the opposite. The helicity can be defined from the WF of magnetic ions. In the case of Cu$_2$OSeO$_3$, Cu(1) and Cu(2) ions are located at 4a and 12b WF, as shown in Table 3. The 4a WF of Cu(1) in Cu$_2$OSeO$_3$ is $(x, x, 0.5+x, 0.5-x, -x-x, 0.5+x, 0.5-x, 0.5-x, -x, 0.5+x, 0.5+x, 0.5-x)$, where $x \approx 0.136$ or $x \approx 1-0.136 = 0.863$, corresponding to two enantiomers. The crystals having Cu(1) at $x = 0.863$ are

![Figure 2](image)

**Figure 2.** The two chiral crystal structures of Cu$_2$OSeO$_3$ where green and blue spheres represent Cu(1) and Cu(2) atoms. The top views are along the body diagonal of the cube (along [111] axis). a) Right-handed and b) left-handed crystals.

![Figure 3](image)

**Figure 3.** a) Temperature dependences of the field-cooled magnetic susceptibilities in different applied magnetic fields along [001] direction. b) Magnetic field dependence of the magnetization at different temperatures, with magnetic field applied along [111] direction. The inset shows the slope-change behavior at the fields $\approx 350$ and 1100 Oe at 5 K, as indicated by arrows. The $\frac{dM}{dH}$ versus $H$ at different temperatures with magnetic field applied along [111] and [100] crystallographic directions. The shaded region represents the skyrmion lattice phase.
defined as right-handed enantiomer and others with $x = 0.136$ as left-handed enantiomer, as shown in Table 3. The structure of Cu$_2$OSeO$_3$ with the same set of coordinates for the right-handed crystals shown in Table 3 is also defined as right-handed in the study by Dyadkin et al.\[19\]. There, the crystals are defined as right-handed on the basis of similarity of $4a$ WF of Cu(1) ion in Cu$_2$OSeO$_3$ and Mn in MnSi (right handed).

The crystal helicity can also be defined by considering the closeness of the structural symmetry of the $P2_13$ space group with the absolute structure of $P4_132$, as proposed in the study by Chizhikov et al.\[29\] $P4_132$ space group contains only right-handed screw axes 4; therefore, the right-handed crystals of $P2_13$ space group can be easily distinguished by comparison. The same approach is also mentioned for B20 structures.\[10\] The set of coordinates determined with this definition for right-handed crystals is found to be consistent with the obtained absolute structure for the right-handed crystals, as shown in Table 3.

The magnetization and resonance data shown in Figure 3, 4 show the presence of the skyrmion lattice phase along with other magnetic phases of Cu$_2$OSeO$_3$. To determine the linewidths and peak positions from the transmission signal, Lorentzian peak fitting was used. An example of such peak fitting is shown in Figure 4c. The resonance spectra measured for both samples grown with HCl and SeCl$_4$ qualitatively show no clear difference (see Figure 4d,e). For both samples, resonance signal with similar linewidth is observed, confirming that the crystals grown with the new method have the same damping as those grown with HCl.\[31\] The phase boundaries of the skyrmion lattice for both samples grown with HCl and SeCl$_4$ coincide with each other (see Figure 4f).

5. Conclusion

We have demonstrated a simple route that allows the growth of Cu$_2$OSeO$_3$ single crystals in a relatively short duration. The XRD analysis shows high quality of single crystals. We observed both right-handed and left-handed enantiomers of Cu$_2$OSeO$_3$ and the absolute structure was fully determined by the Flack parameter analysis of the refined XRD pattern. The growth of crystals with both left- and right-handed structural chiralities can be useful to understanding the coupling between structural and magnetic chiralities. The understanding of coupling is important to control the magnetic textures such as skyrmions for spintronics applications.

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Conflict of Interest
The authors declare no conflict of interest.

Data Availability Statement
Research data are not shared.

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