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Electronic Supplemental Information for

Controllable Synthesis and Lattice Vibration Anisotropy of 2D Sb_2O_3 Single Crystals

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Experimental Section

Synthesis of Sb_2O_3 monolayers

Two-dimensional Sb_2O_3 nanosheets were prepared using the microspacing sublimation method. First, Sb_2O_3 powder and NaCl particles were uniformly mixed and ground at a weight ratio of 2:1 to serve as the precursor for growth. Then, the growth hot stage was heated. When the temperature of the hot stage rose to ~500℃, a piece of glass was taken, and a small amount of the precursor was scattered in the center of the glass. The glass was then transferred to the center of the hot stage. Next, a freshly cleaved mica substrate was placed above the precursor powder, maintaining a spacing of either 1–3 mm or 300–1000 μm between the mica substrate and the precursor. The aforementioned setup was heated for 5–10 minutes. Subsequently, the mica substrate was removed from the hot stage and transferred under an optical microscope for observation, where the formed Sb_2O_3 nanosheets could be found on the mica substrate. It should be noted that the Sb_2O_3 nanosheets become thicker and larger with increasing growth temperature and extended growth time. Therefore, by adjusting the growth temperature and time, Sb_2O_3 nanosheets of different sizes and thicknesses can be obtained.

Transfer of materials

The synthesized 2D Sb_2O_3 flakes were transferred using a wet transfer method, with the commonly used Poly(methyl methacrylate） (PMMA) serving as the supporting layer. The Sb_2O_3 flakes were transferred from the mica substrate to either a SiO_2/Si substrate or a TEM copper grid for further characterization and testing. During the transfer process, a layer of PMMA was first spin-coated onto the prepared material at

a speed of 2000 rpm for 1 minute to form a uniform PMMA supporting layer. Next, the PMMA/α-Sb_2O_3/mica stack was placed on a heating stage and heated at 160°C for 10 minutes to solidify the PMMA and enhance its adhesion to the Sb_2O_3 layer. The stack was then immersed in deionized water at 70°C for 1 hour. Due to the hydrophobic nature of mica, the PMMA/Sb_2O_3 film tended to detach from the mica substrate in the deionized water. After immersion, the PMMA film was gently lifted with tweezers, separating the PMMA/Sb_2O_3 film from the mica substrate, leaving the PMMA/Sb_2O_3 membrane floating on the water surface. Subsequently, the target substrate (SiO_2/Si or TEM copper grid) was carefully dipped into the water to retrieve the PMMA/Sb_2O_3 film. The sample was then heated at 160°C for 10 minutes and further annealed in a vacuum oven at 60°C for 3 hours to strengthen the interaction between the sample and the target substrate. Finally, the sample was immersed in acetone and heated at 60°C for 5 minutes to remove the PMMA, completing the transfer of the sample to the target substrate.

Materials Characterization

The morphology, grain size, and thickness of 2D α-Sb2O3 flakes were characterized using a 3D laser confocal microscope (KEYENCE, VK-X1000) and an atomic force microscope (AFM) (Dimension, ICON-PT). Raman spectroscopy and angle-resolved polarized Raman spectroscopy measurements were performed on the samples using a Raman spectrometer (Horiba, iHR550+Smart Raman) with an excitation wavelength of 532 nm. The crystal phases and crystallinity of the crystals were analyzed by X-ray diffraction (XRD) (smartlab9), and the elemental composition of the Sb_2O_3 crystals was

analyzed by X-ray photoelectron spectroscopy (XPS) (ULVAC-PHI INC). The atomic structure of Sb2O3 was investigated by transmission electron microscopy (TEM) (Thermo Scientific Talos F200X). The bandgap of the samples was measured using ultraviolet-visible absorbance spectroscopy (UV-Vis).

Supporting Figures

Table S1 Quantitative analysis of EDS spectra for Sb_2O_3 crystals.

Element	Mass fraction (%)	Atomic fraction (%)
Sb	83.42	39.79
O	16.58	60.21

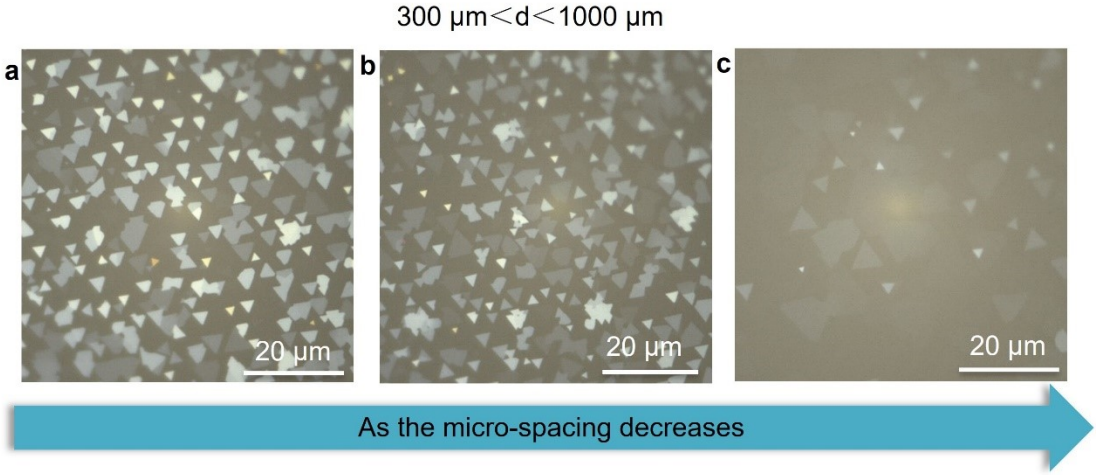

Fig. S1. (a-c) Optical images of Sb_2O_3 crystals prepared under different micro-spacing conditions.

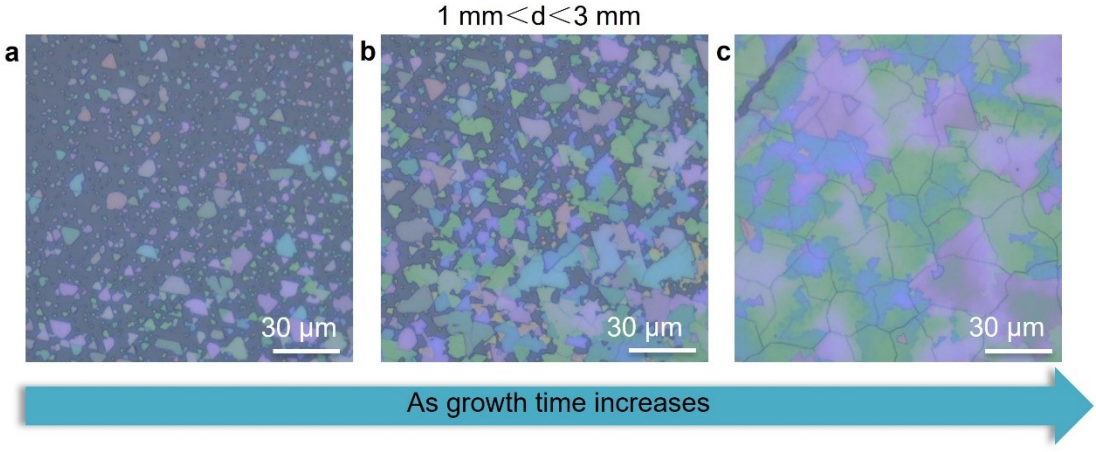

Fig. S2. (a-c) Optical images of Sb_2O_3 crystals prepared under different growth times.

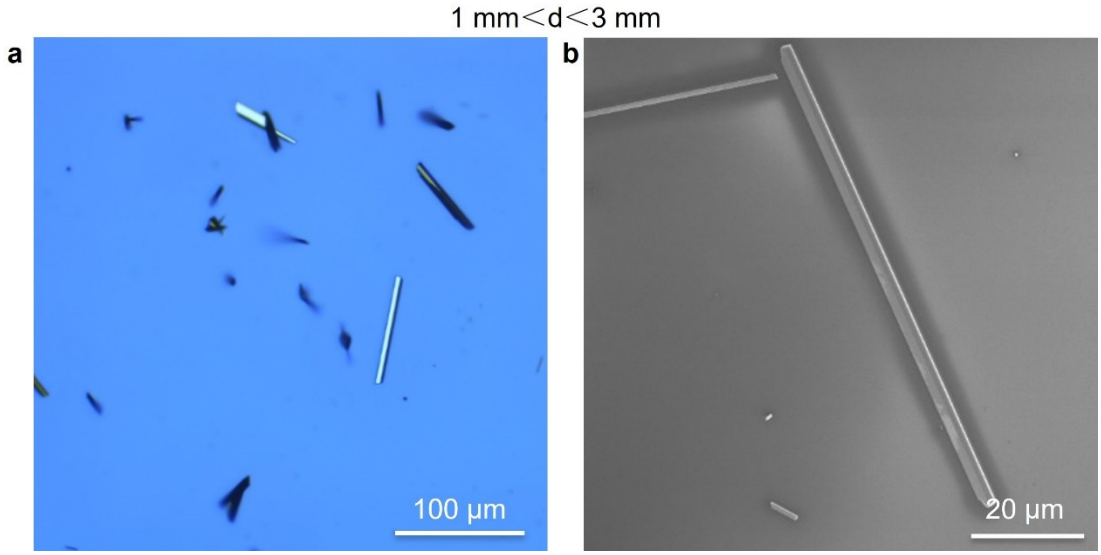

Fig. S3. (a) Optical image of the rod-shaped Sb_2O_3 crystal. (b) Scanning electron microscope image of the rod-shaped Sb_2O_3 crystal.

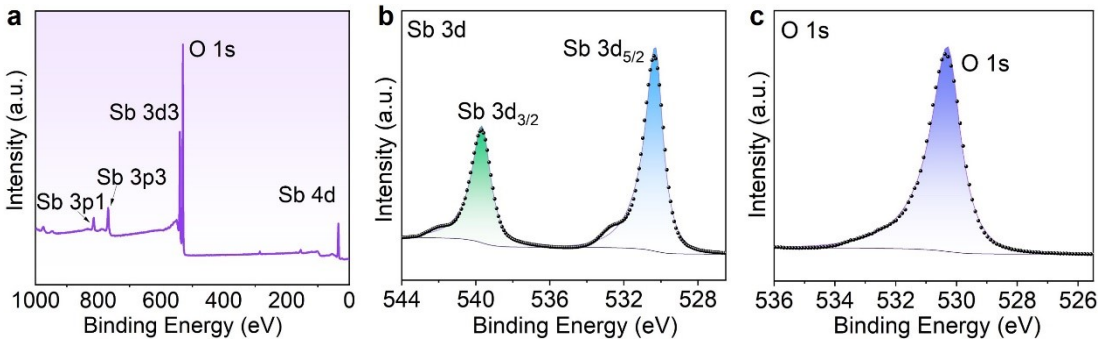

Fig. S4. XPS spectra of Sb_2O_3 powders. (a) Full XPS spectrum of Sb_2O_3 powders. (b) XPS spectra of Sb_2O_3 powders for Sb 3d. (c) XPS spectra of Sb_2O_3 powders for O 1s.

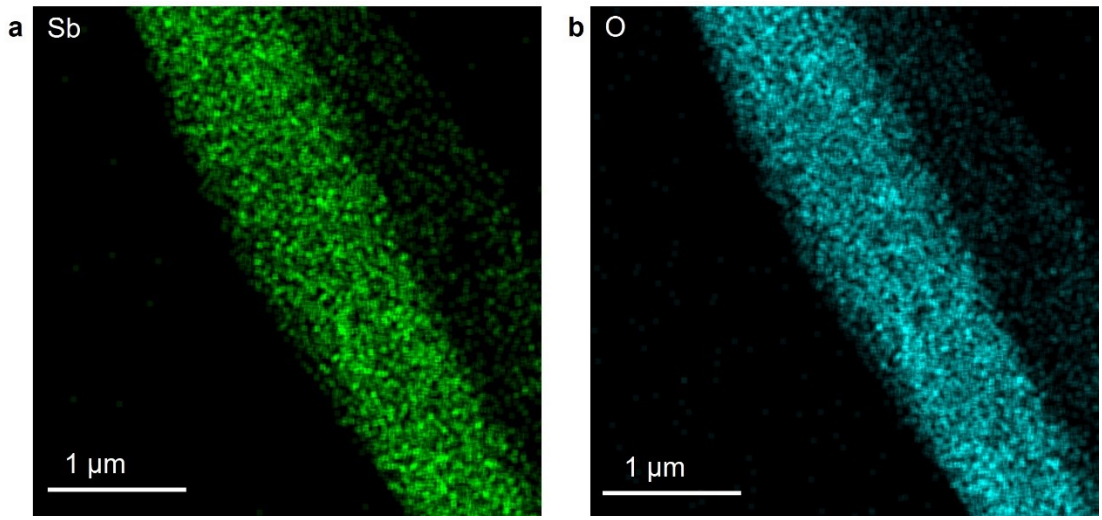

Fig. S5. (a-b) EDS images of rod-shaped Sb_2O_3 crystals, The uniform distribution of Sb and O elements is shown.

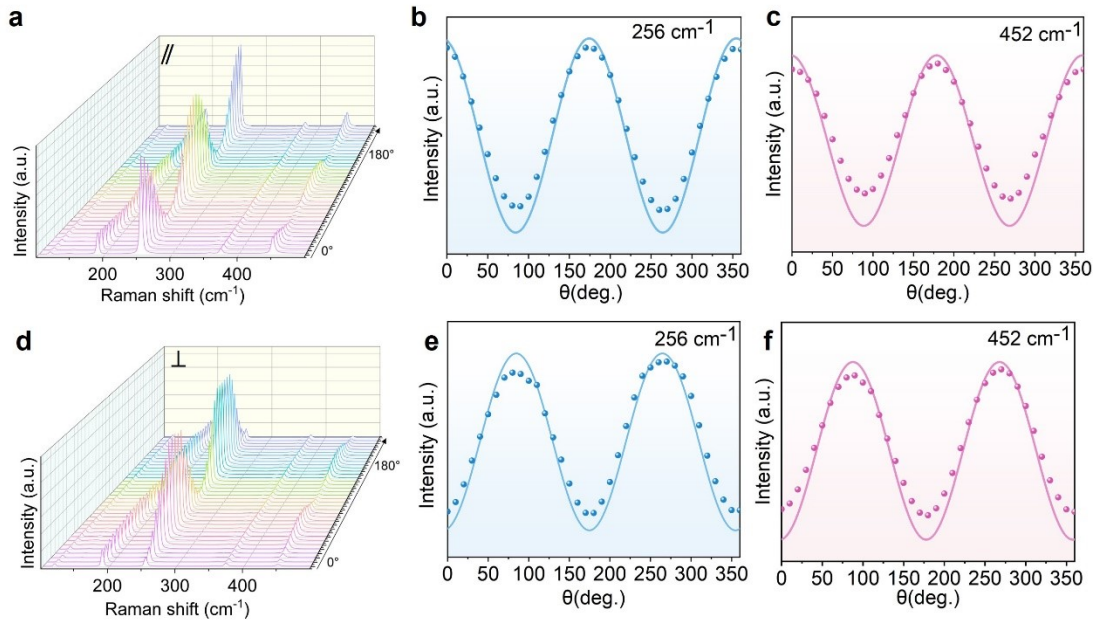

Fig. S6. (a, d) Polarizing Raman spectral waterfall diagram of Sb_2O_3 in parallel configurations and cross configuration. (b-c) Data plot of angular variation of Raman scattering intensity under parallel polarization configuration. (e-f) Data plot of the variation of Raman scattering intensity with angle in cross polarization configuration. Points are experimental data and curves are formula fitting curves.

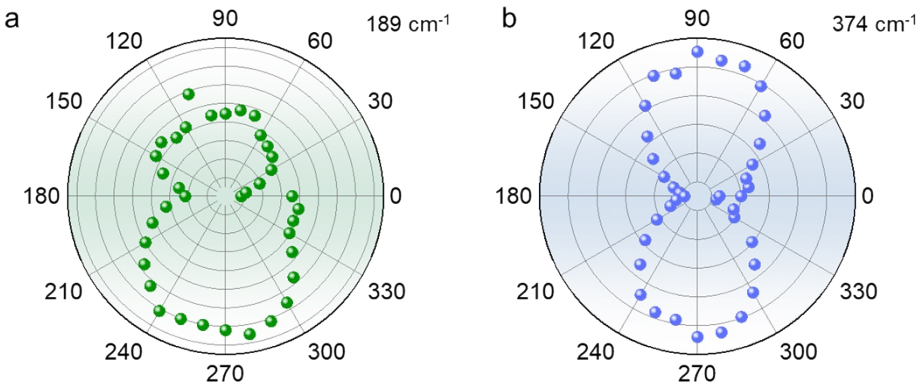

Fig. S7. Polar coordinates of Raman peak intensity at 189 cm^{-1} and 374 cm^{-1} in the cross-polarization configuration.