Electronic Supplementary Information for the manuscript submitted to Journal of Materials Chemistry C

Colloidal solutions of niobium trisulfide and niobium triselenide

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1. Experimental details

The syntheses of niobium trichalcogenides were carried out using high temperature ampoule method starting from high purity elements. Before synthesis the silica ampoule V=16 mL was thoroughly cleaned, dried, heated in vacuum at 800°C to remove traces of absorbed H_2O and O_2 and cooled under vacuum.

Synthesis of NbS_3

The stoichiometric mixture of niobium powder (2.500 g, 0.0269 mol) and sulphur (2.590 g, 0.0809 mol) was loaded into ampoule which was evacuated to 10^{-5} bar and sealed under dynamic vacuum. The ampoule was heated in electrical furnace as follows: heating to 600°C during 12 h, keeping at 600°C during 120 h, and cooling to room temperature for 5 h. Then the ampoule was cut and the resulting powder was taken out and put under Ar. Yield of NbS_3 is quantitative. Powder pattern of NbS_3 fits well that calculated for triclinic NbS_3 (PDF Number: 71-468, triclinic, Sp. Gr. P-1).

Synthesis of $NbSe_3$

The stoichiometric mixture of niobium powder (3.000 g, 0.0323 mol) and selenium powder (7.650 g, 0.0969 mol) were placed in a quartz vacuum-degassed ampoule and sealed. The ampoule with reaction mixture was heated in a furnace during 6 h to the temperature of 600°C and then kept at this temperature for 74 h. After that the furnace was cooled down to room temperature during 14 h and opened. Yield of $NbSe_3$ is quantitative. XRD powder pattern of the synthesized $NbSe_3$ corresponds to the pattern calculated for monoclinic $NbSe_3$.

2. Uv-*vis* characterization of dispersions

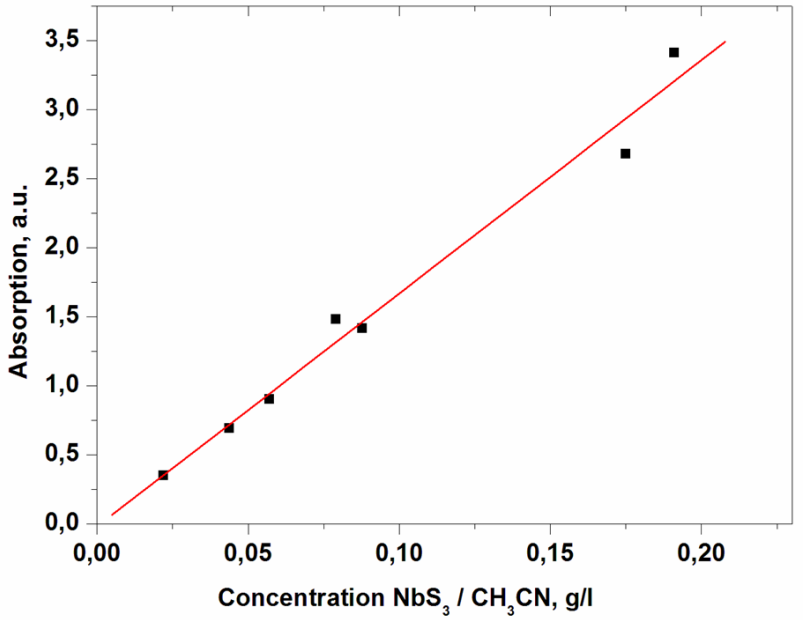


Figure S1. Correlation dependence between the concentration of NbS_3 / CH_3CN dispersion and the value of absorption band at 589 nm.

3. DLS characterization of dispersions

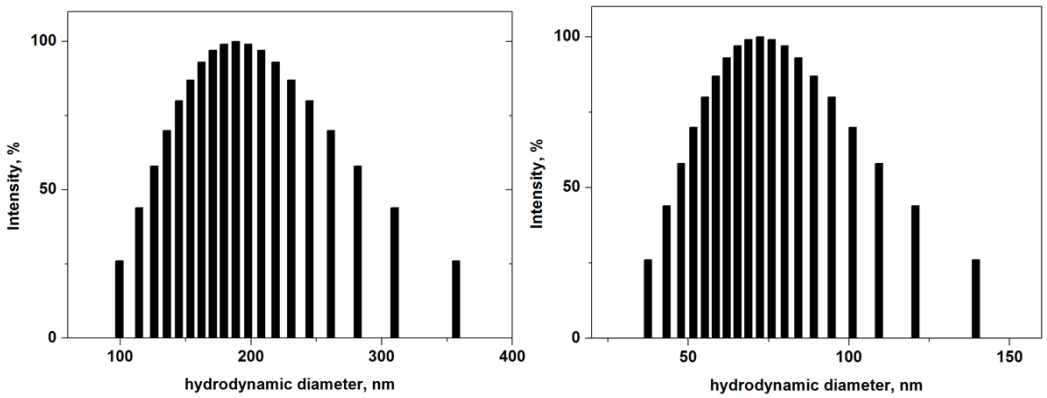


Figure S2. Monomodal DLS analysis of the hydrodynamic diameters distributions for NbS_3 colloidal dispersions in 45% EtOH water solution (left); in isopropanol (right).

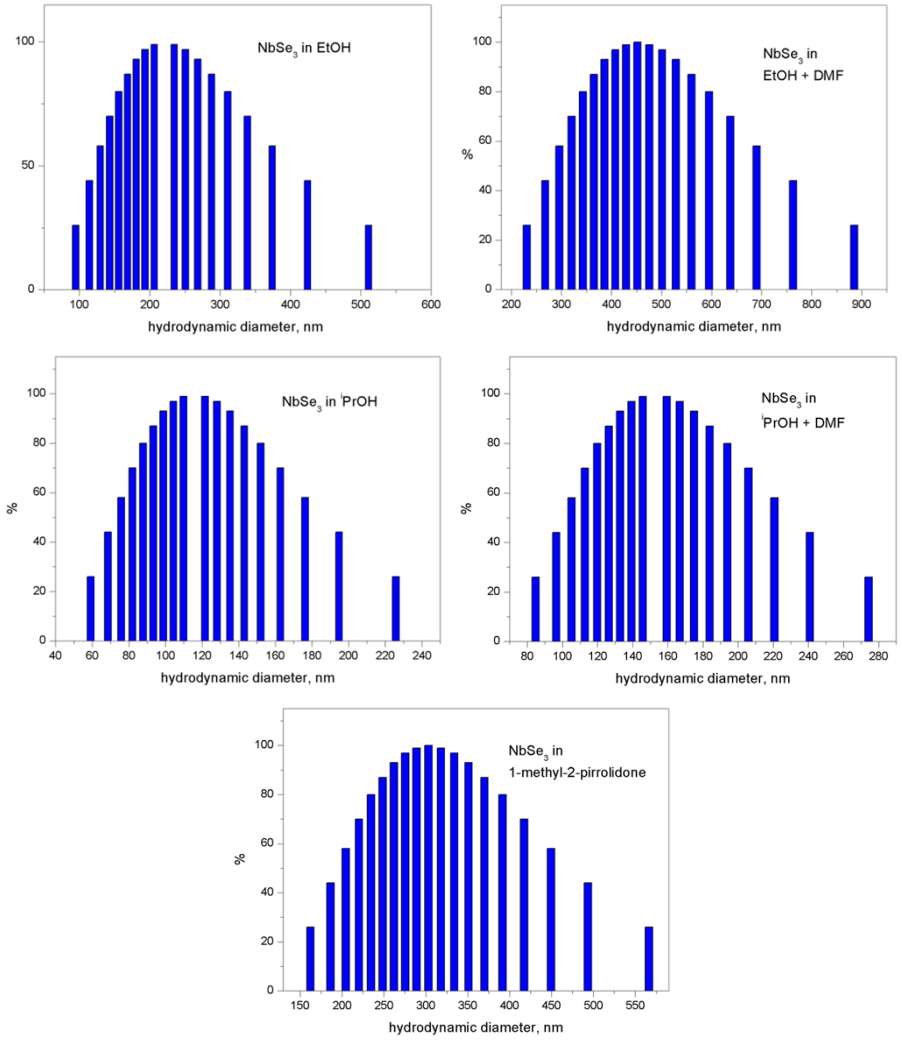


Figure S3. Monomodal DLS analysis of the hydrodynamic diameters distributions for $NbSe_3$ colloidal dispersion in various solvents

4. SEM characterization of NbS_3 samples filtered through Anodisc membrane from its colloids

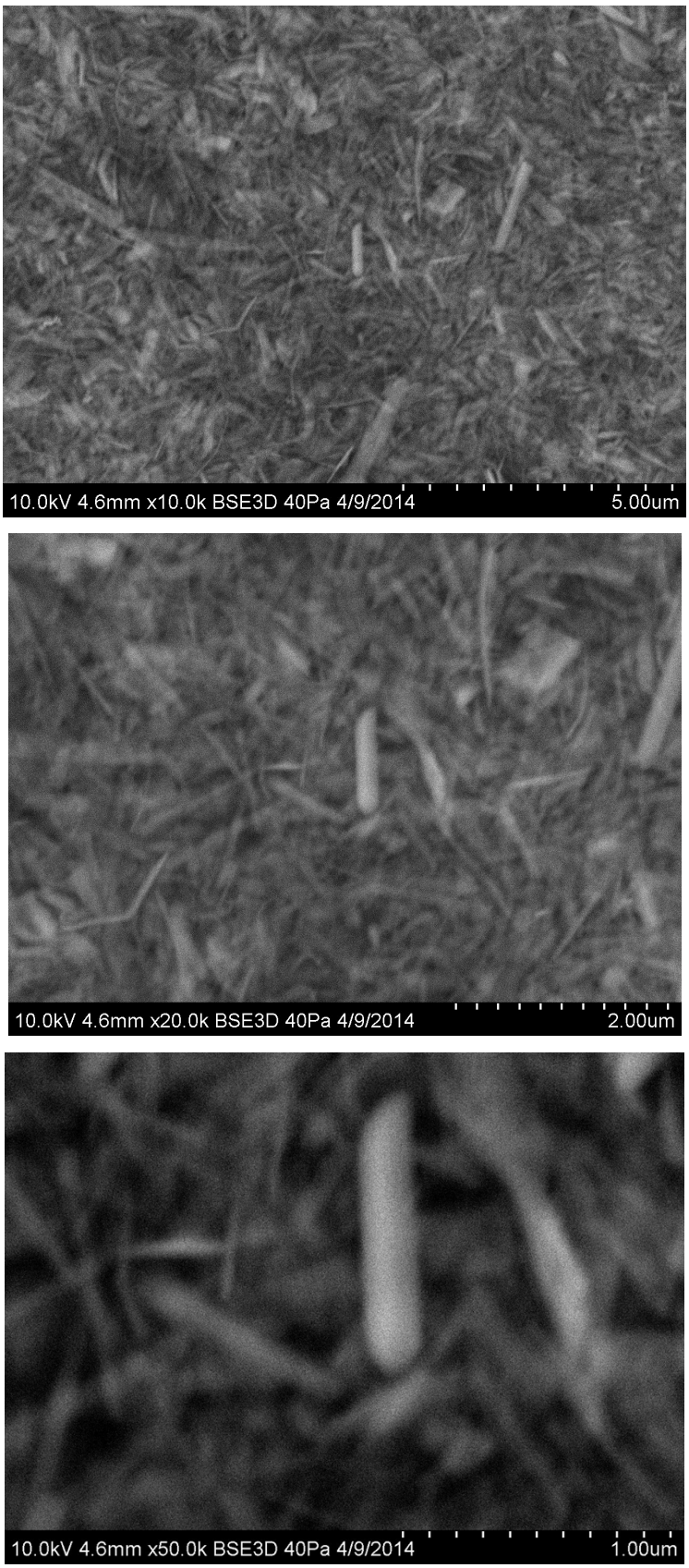


Figure S4. SEM images of NbS_3 nanoparticles filtered from its colloid through Anodisc membrane.

5. AFM characterization of NbS_3 and $NbSe_3$ samples deposited from acetonitrile dispersion

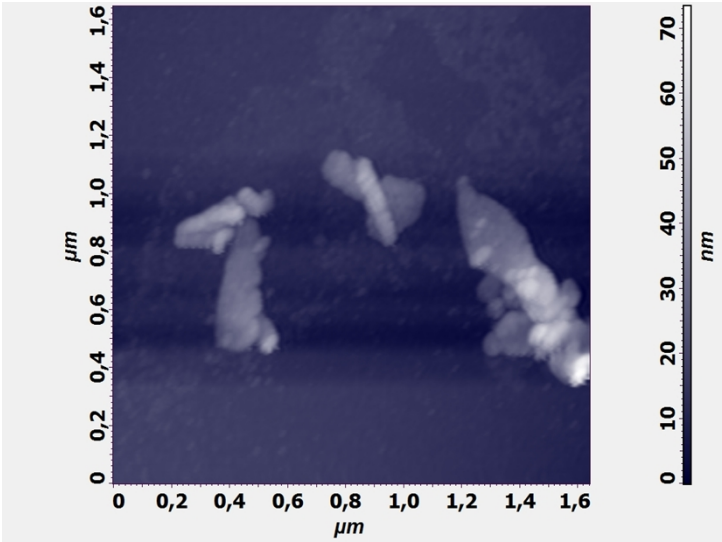
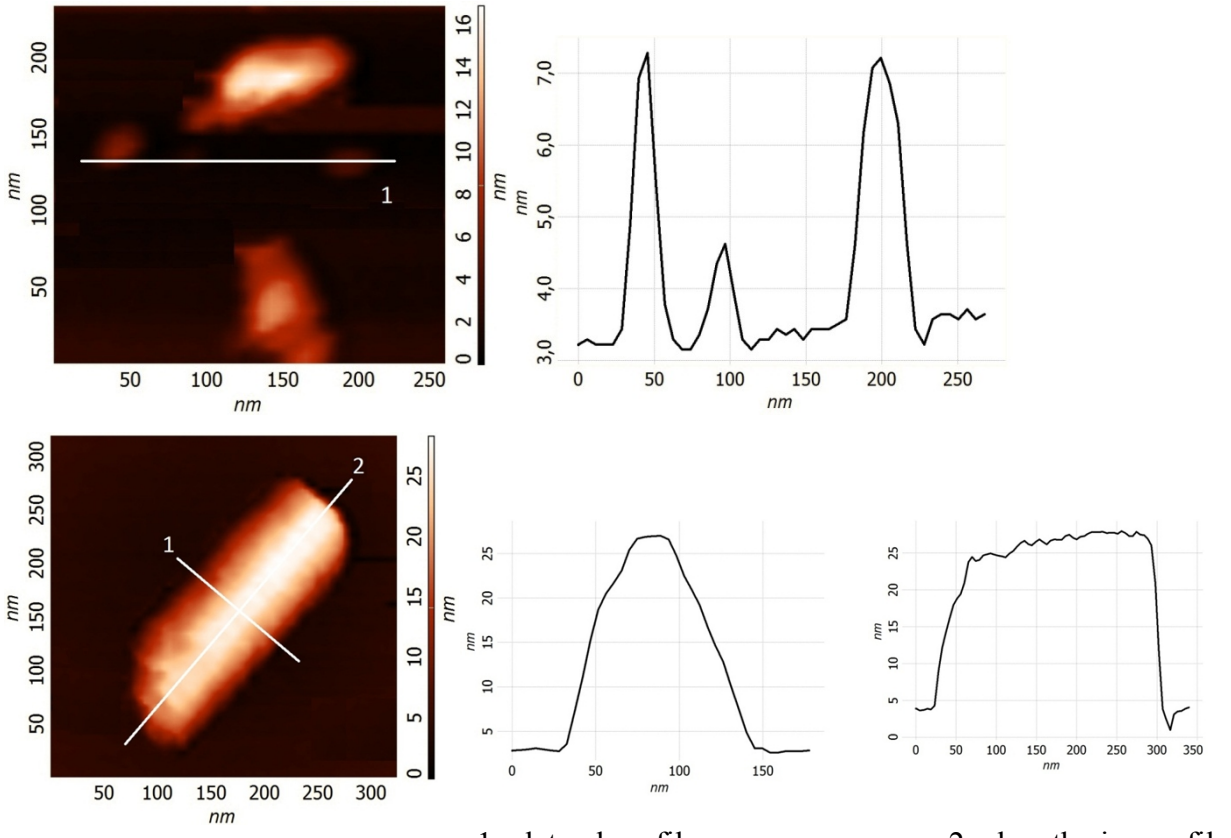


Figure S5. AFM image of $NbSe_3$ deposited from acetonitrile colloid solution.



1 – lateral profile 2 – lengthwise profile

Figure S6. AFM images of small particles of NbS_3 in the sample and its profiles.

6. TEM characterization of NbS_3 samples deposited from acetonitrile dispersion

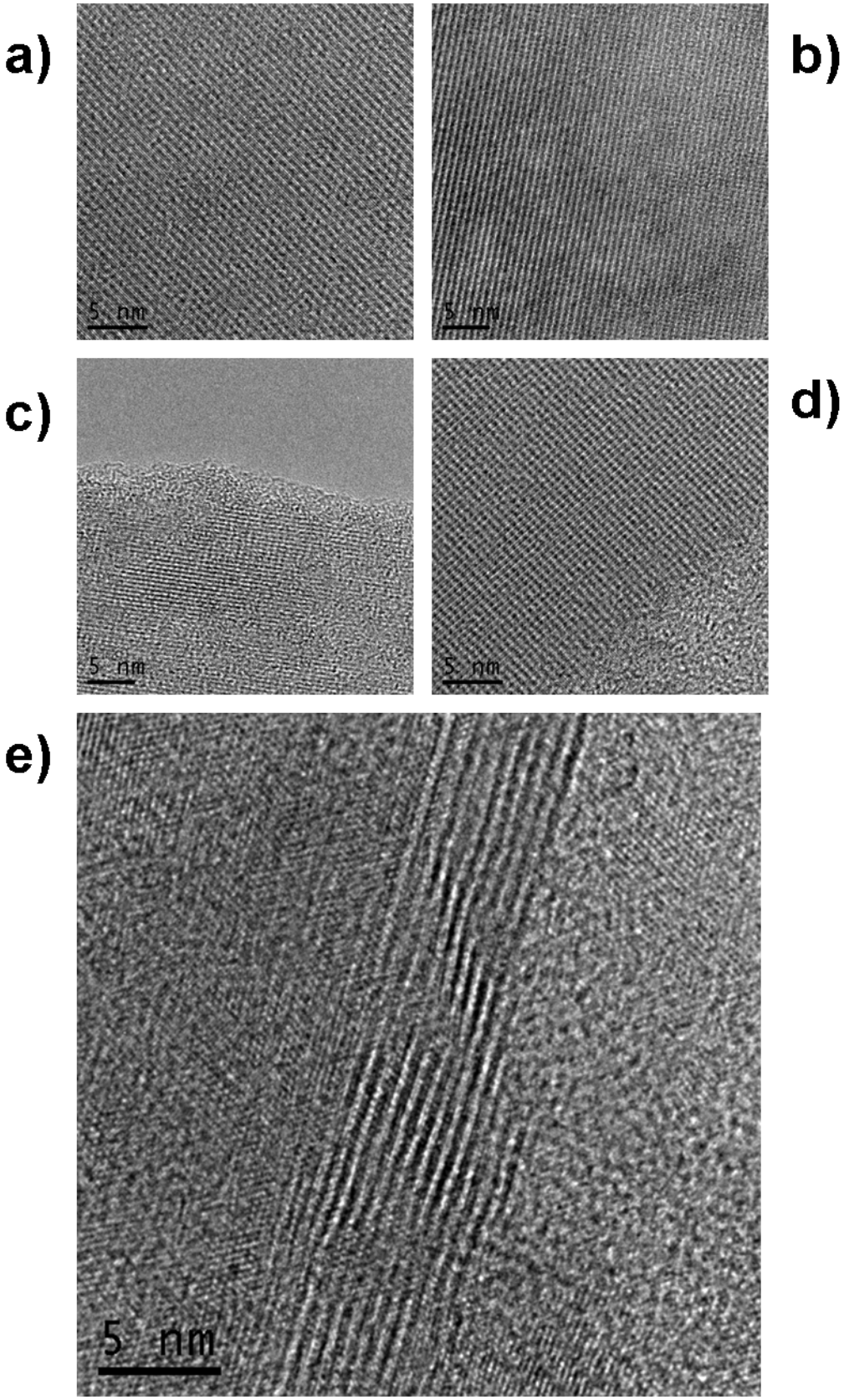


Figure S7. TEM images of NbS_3 high-crystalline NbS_3 particle (a, b); edges of NbS_3 particles (c, d); Figure c shows thin amorphous shell on the surface; NbS_3 layer packing (e).

7. Optical images of thin films of NbS_3 prepared by filtration and spray method

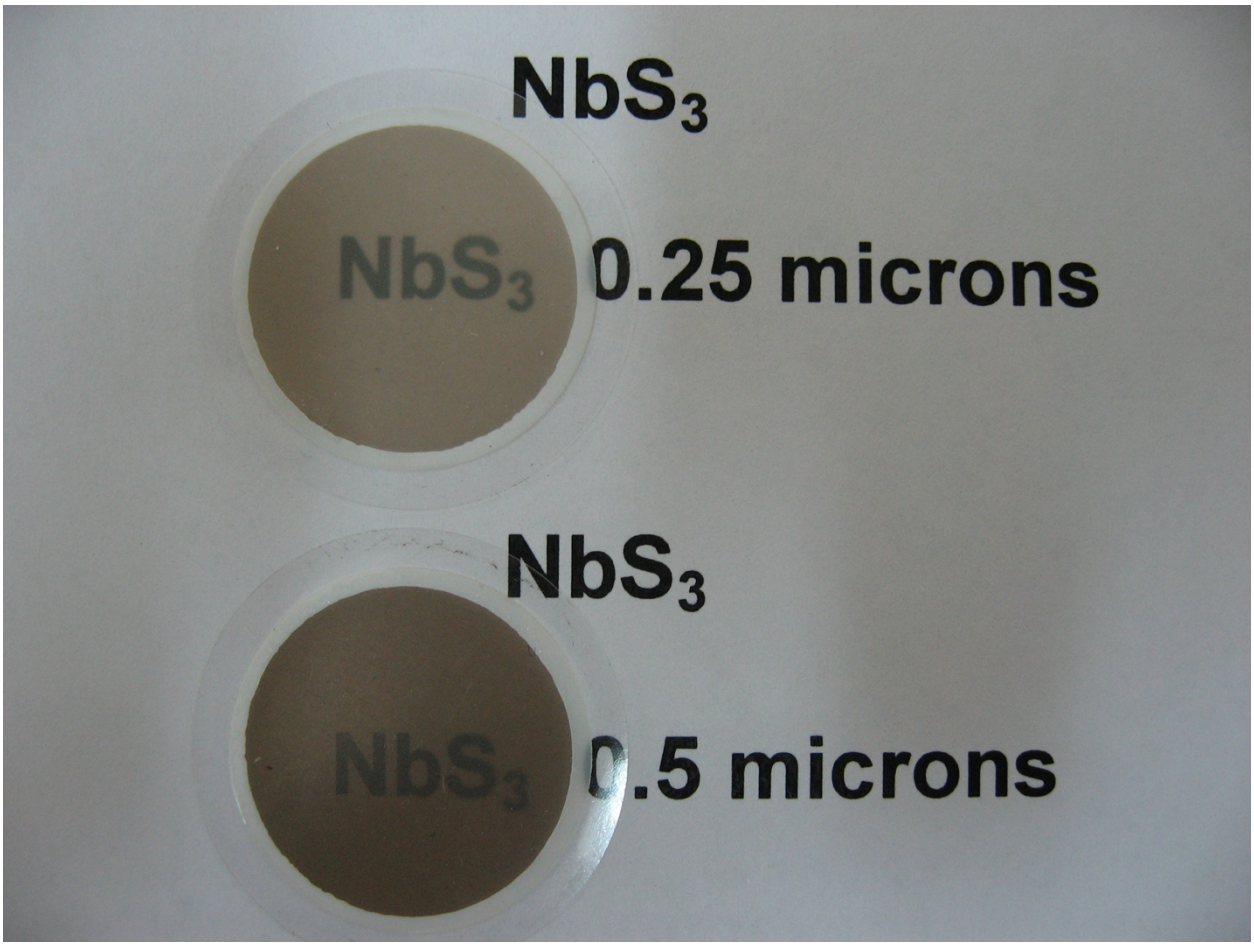


Figure S8. Photograph of NbS_3 films filtered on Whatman anodisc (diameter is 25 mm, pores are 0.02 μm)

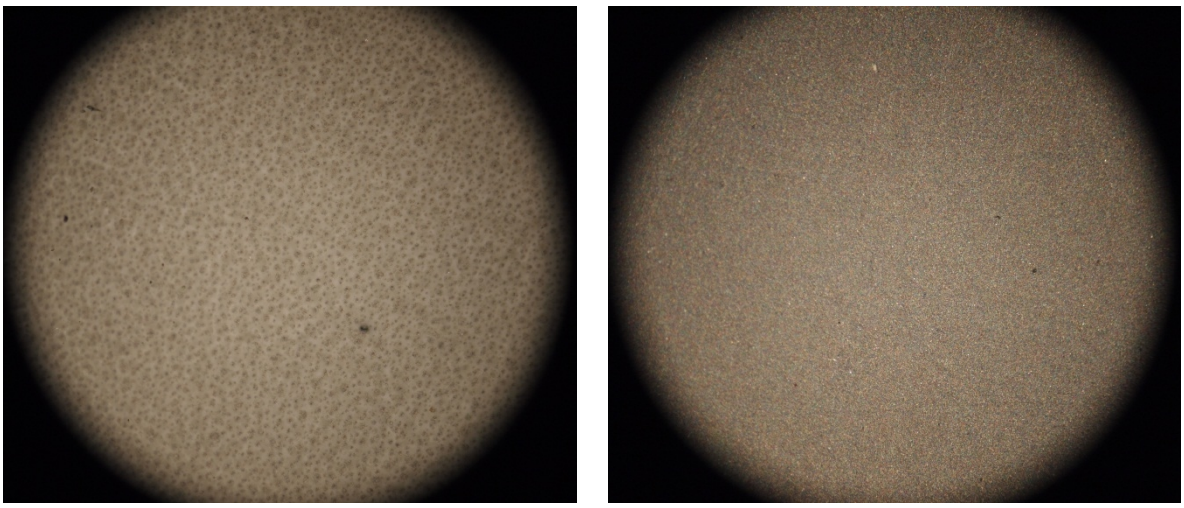


Figure S9. NbS_3 film preparation by spray-method: in process of spraying (left); the resulting NbS_3 film (right).

8. XRD characterization of NbS_3 films

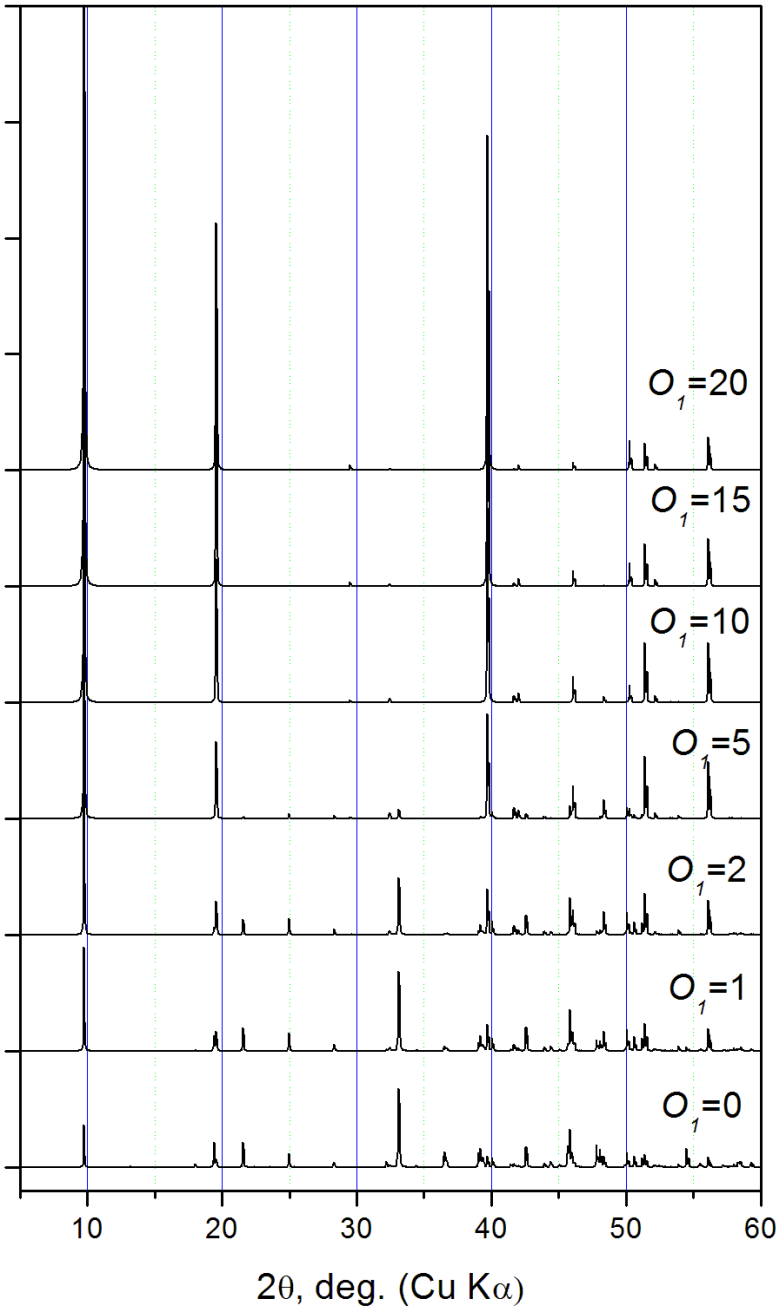


Figure S10. Simulation of textured X-Ray diffraction patterns for NbS_3 films with variation of empirical coefficient O_1.

9. IR spectroscopy data

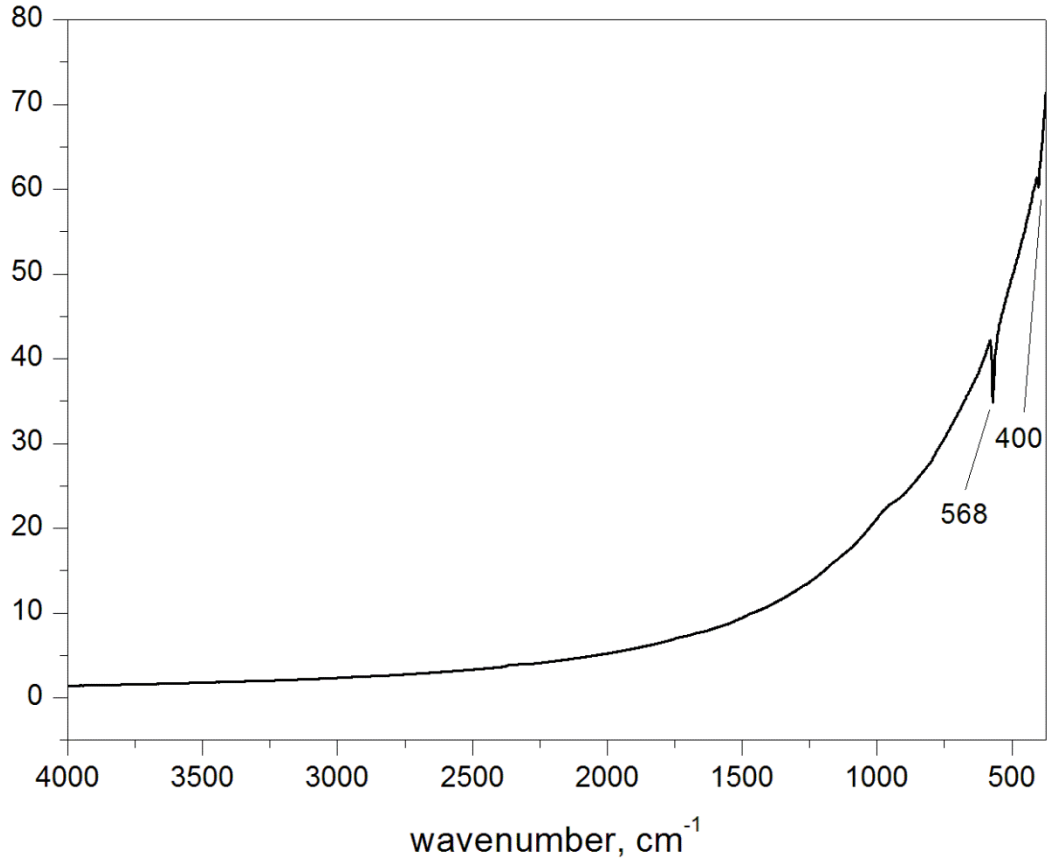


Figure S11. IR-spectrum of dispersed NbS_3 solid. The band at 568 cm^{-1} corresponds to S–S bonds, and the band at 400 cm^{-1} to Nb–S bonds.