Electronic Supplementary Information (ESI†)

Synthesis of very small diameter silica nanofibers using sound waves

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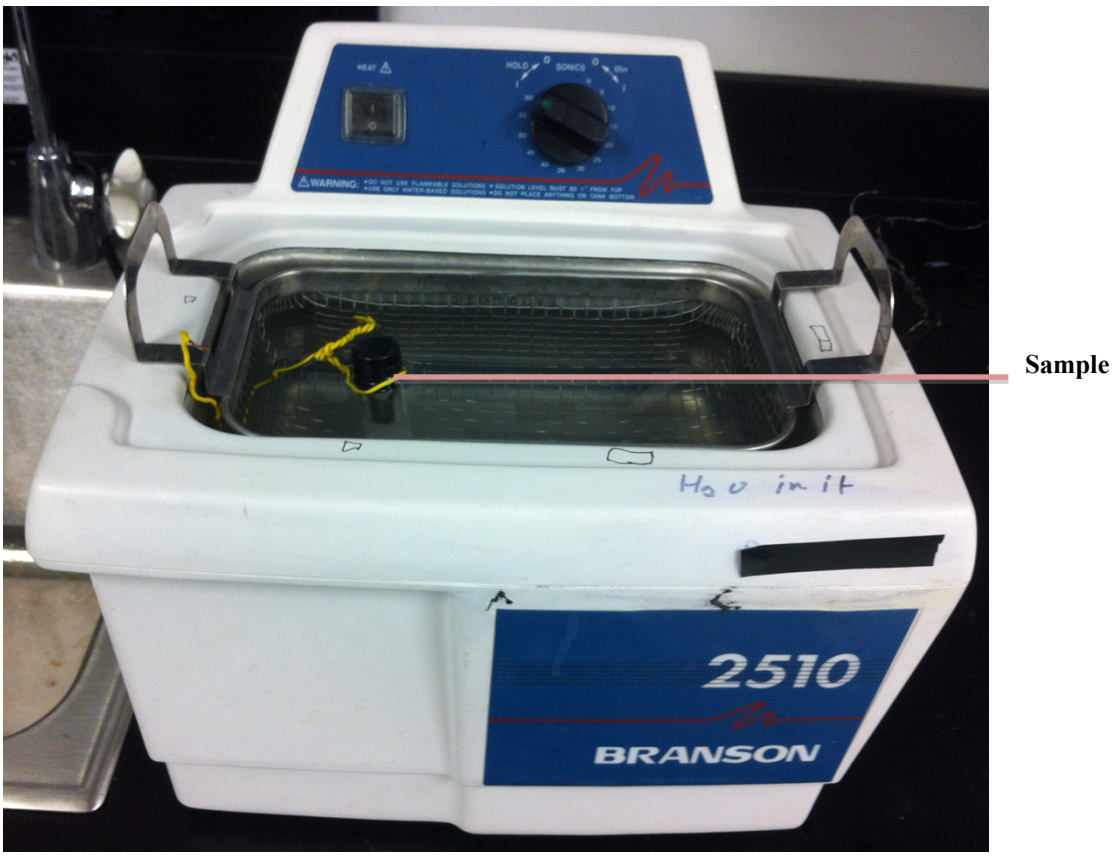
S1.

Chemicals: Sodium citrate ($Na_3C_6H_5O_7$), absolute ethanol (C_2H_5OH, 200 proof), Tetraethylorthosilicate (TEOS), pentanol ($C_5H_{11}OH$), and ammonium hydroxide (NH_4OH, 28–30 %) were purchased from Fisher Scientific. Polyvinylpyrrolidone (PVP: MW 40,000) was purchased from Sigma Aldrich. The required solutions were made by mixing the above chemicals.

Synthesis of silica nanofibers and general observations: PVP (0.5 g) was dissolved in pentanol (5 mL) in a glass vial by sonication. Water (140 µL), sodium citrate (50 µL; 0.18 M), absolute ethanol (475 µL), and ammonium hydroxide (100 µL) were added to the glass vial and sonicated for a few minutes. Finally, 50 µL of TEOS was added to the above reaction mixture and sonicated [40 kHz; 4 hr.; bath sonicator: Branson 2510 ultrasonicator; power setting: 100 watt (set by the manufacturer)]. The 4 hr. time is the minimum time, longer sonication also gives similar nanofibers. After sonication the sample was kept at room temperature to stop the sonication process. Placing the sonicated sample for sometime (overnight is recommended) at room temperature. It helps in settling down the large rods, and thus their maximum separation from the nanofibers. The position of the sample in the sonicator affects the diameter and reproducibility of the fibers. For example, thinner fibers (ranging from 20 nm to 60 nm, with main population ≈ 30 nm) were obtained at the positions as shown in the picture below. If sample was placed in the center, the thicker fibers with an average diameter ≈ 60 nm were obtained. It appeared that the actual acoustic force experienced by the sample affects the fiber diameter. In all cases short or broken fibers were also observed. We assume either these fibers got broken during the processing or start growing in the later phase of the reaction and couldn't get sufficient TEOS for long growth.



Picture showing the experimental set up.

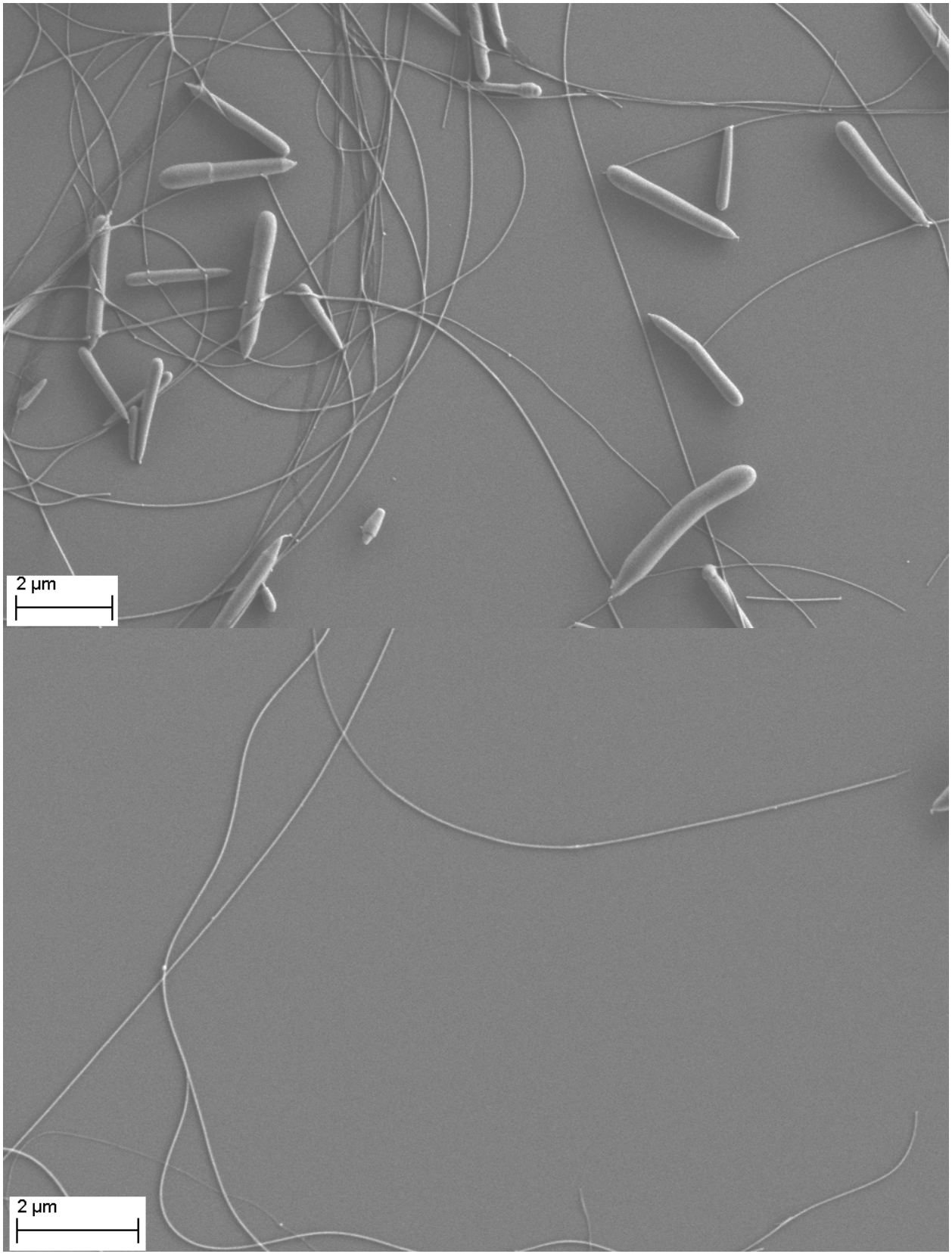
Purification of nanofibers: Centrifuged the nanofibers at 4000 rpm for 10 minutes, and discarded the supernatant. Washed the nanofibers with ethanol by centrifuging at 4000 rpm three times and discarded the supernatant every time. Finally centrifuged the nanofibers at 500 rpm for 10 minutes to remove the large rods and made the samples from supernatant for SEM studies.

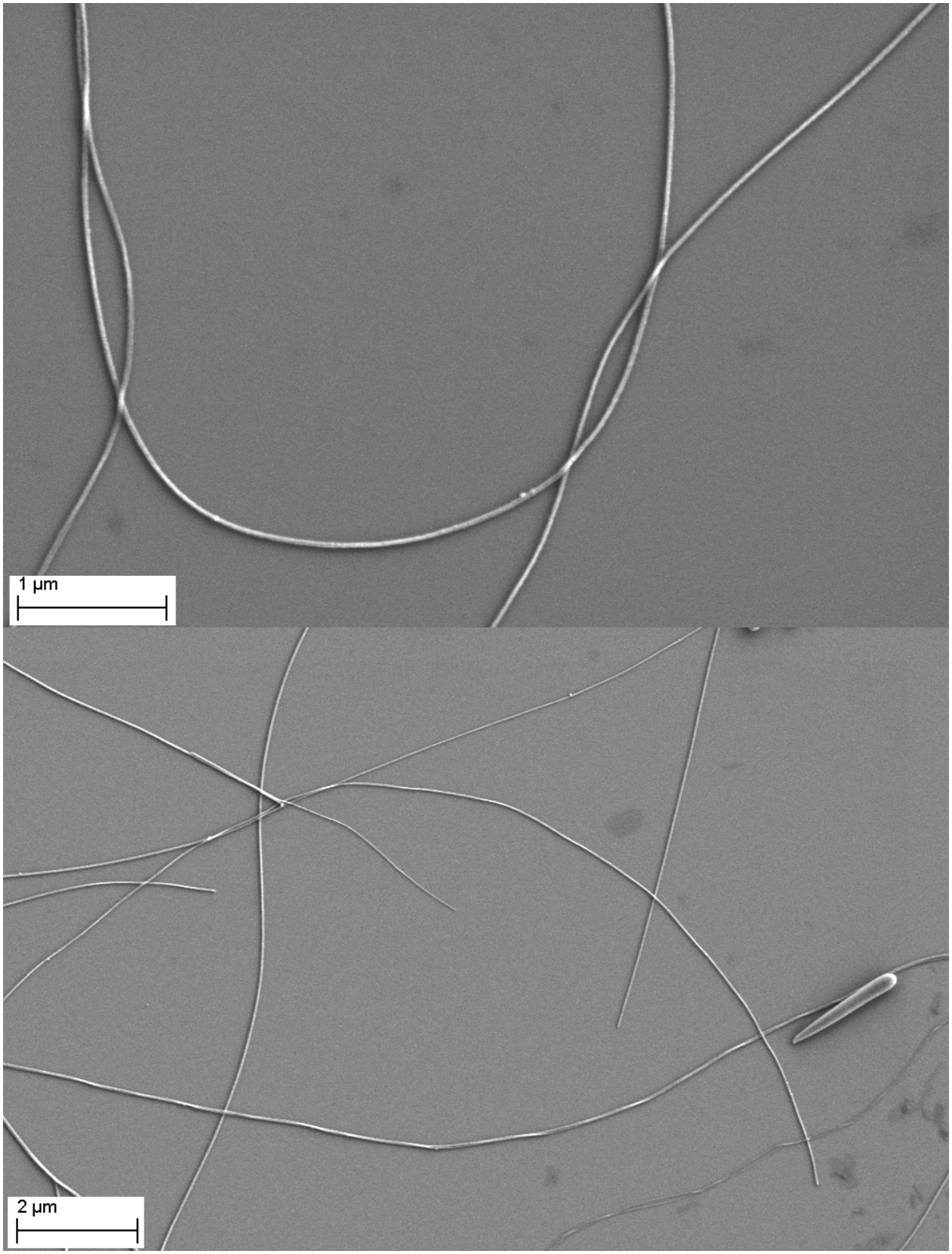
SEM imaging: SEM imaging were done using Merlin 200 instrument. SEM samples were prepared by depositing the purified sample on a silicon wafer. Silicon wafers were employed to minimize the charging of the silica samples (no gold sputtering was employed).

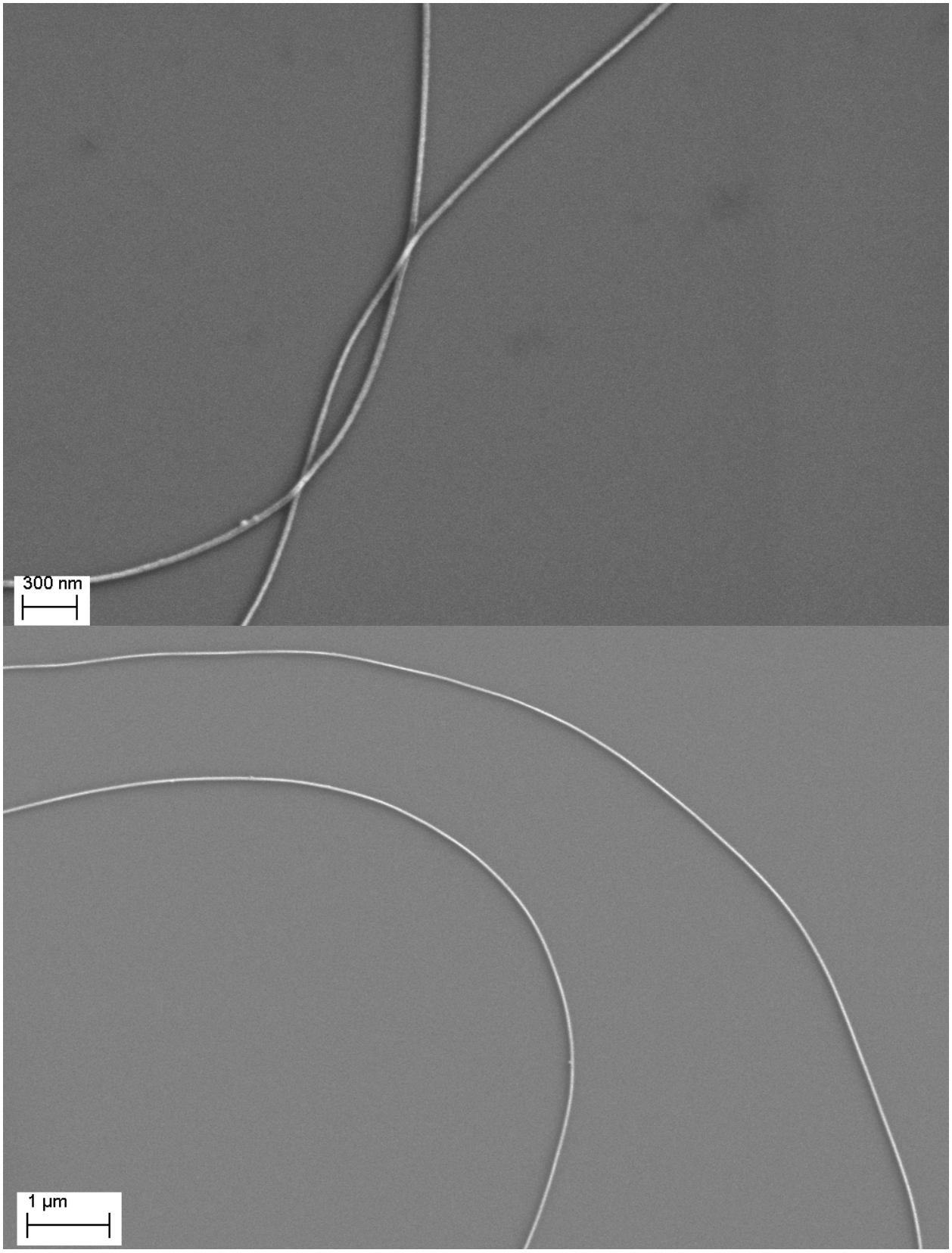
TEM studies: The above-purified nanofibers were washed with water to remove any adhering PVP. Final silica nanofibers were suspended in absolute ethanol to avoid any degradation in water.

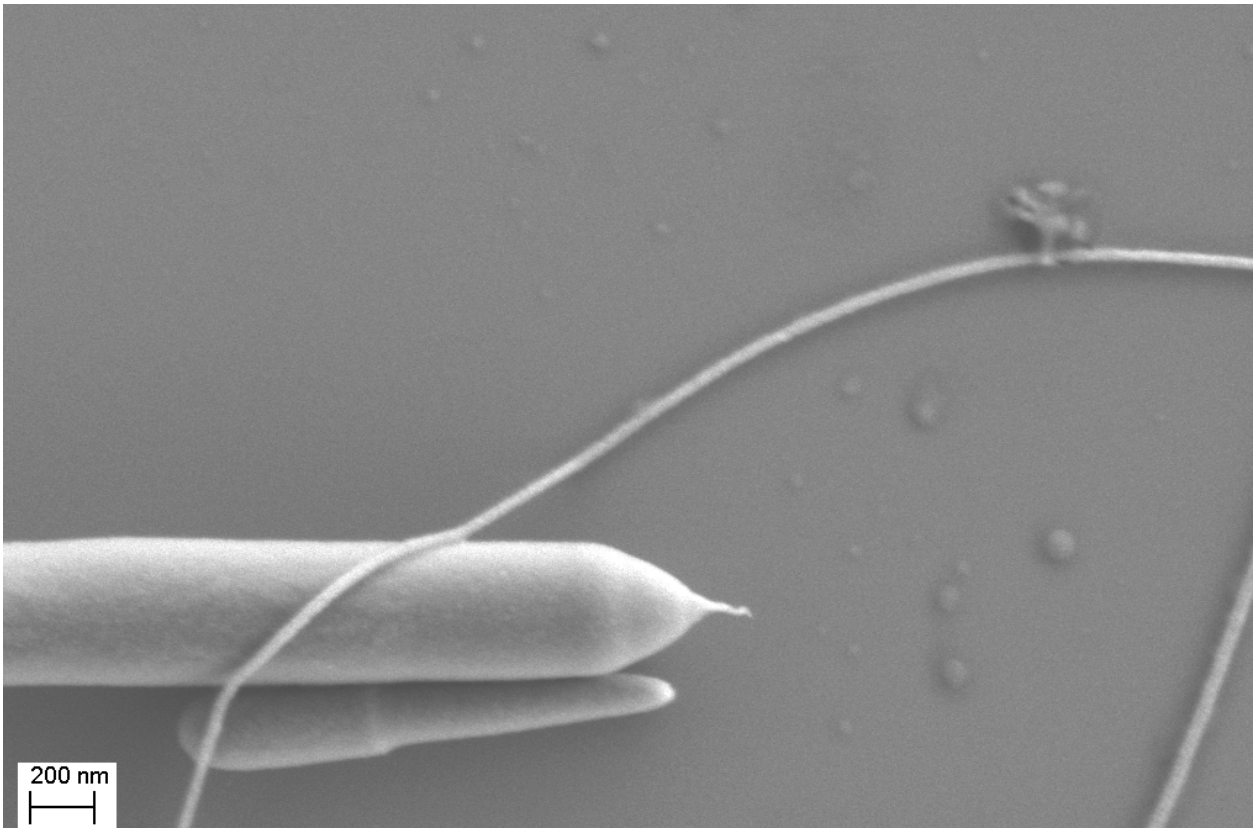
Selected area electron diffraction (SAED) and TEM data were collected using a Zeiss Libra 120 with an Omega energy filter. SAED and TEM experiments were performed at 120 kV with an emission current of 4 μA in order to minimize electron-beam-caused microstructural change or sample damage. The chosen SAED aperture has a 1 μm diameter with a typical camera length of 576 mm. The SAED patterns are calibrated against aluminum standards. TEM samples were prepared by depositing ≈ 2 μL of prewashed sample on carbon coated copper grids and wiping out the excess sample with filter paper.

S2: More SEM images of the silica nanofibers.

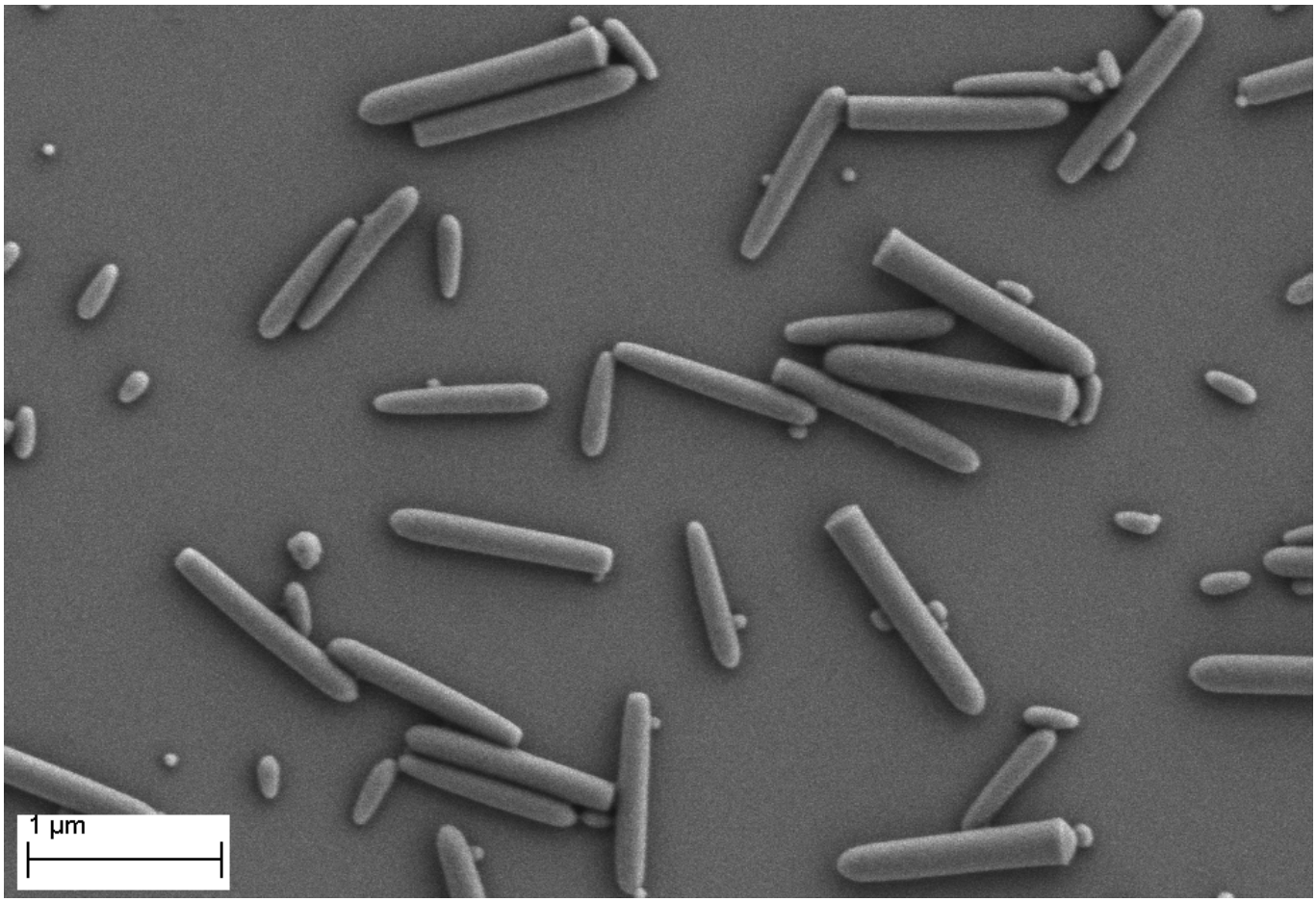



1 µm
2 µm

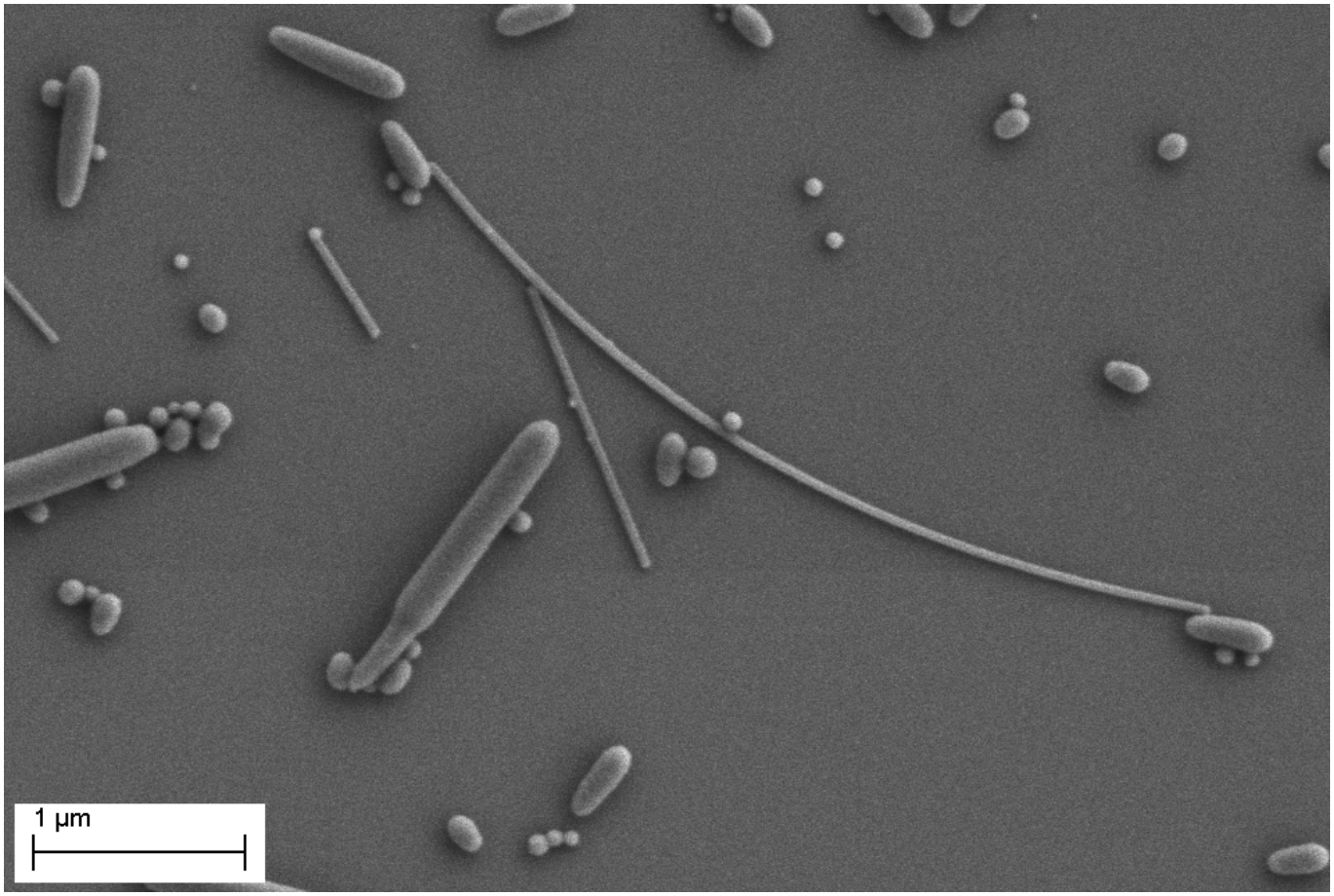

300 nm
1 µm


200 nm

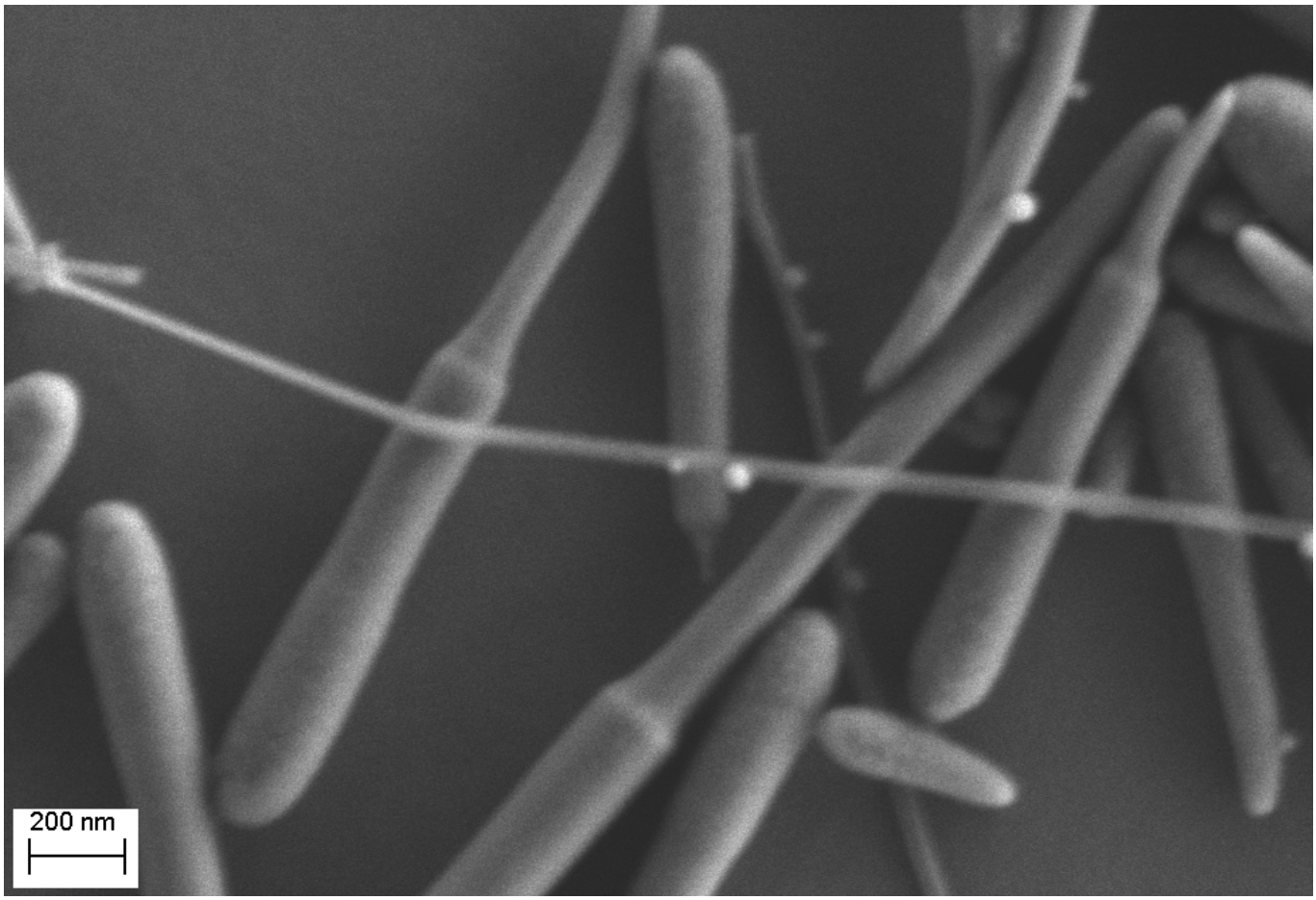
S3.



SEM image showing no tapering and nanofiber formation when preformed rods sonicated after 24 hr. of initial rod growth.

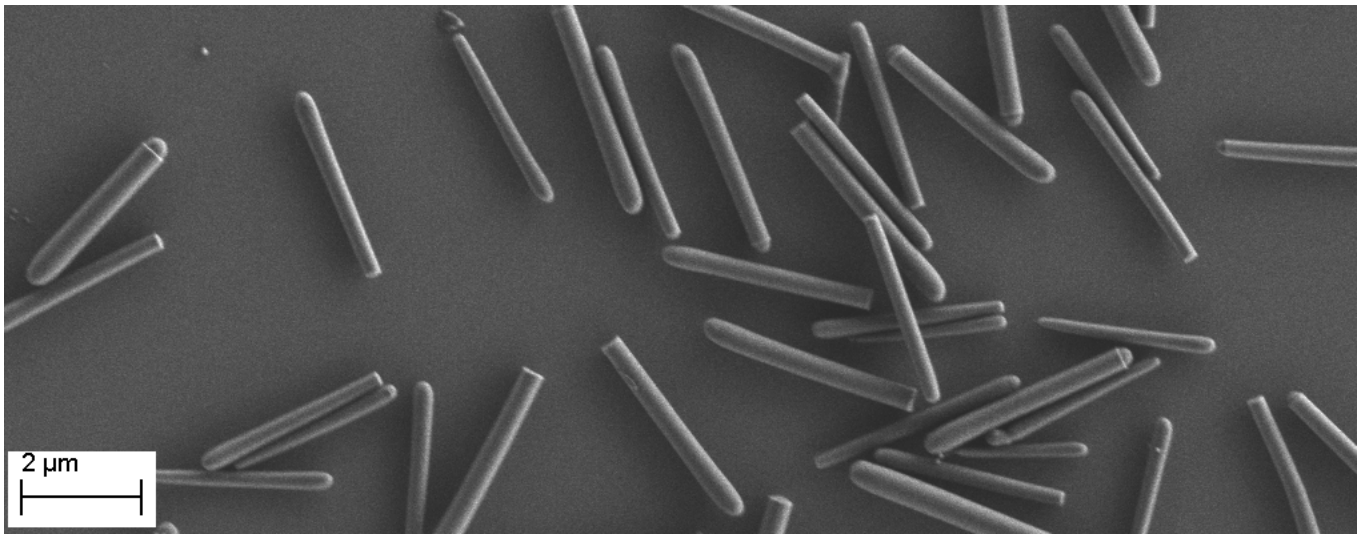


SEM image showing rod tapering and nanofiber formation when preformed rods sonicated after 24 hr. with another equivalent addition of TEOS.

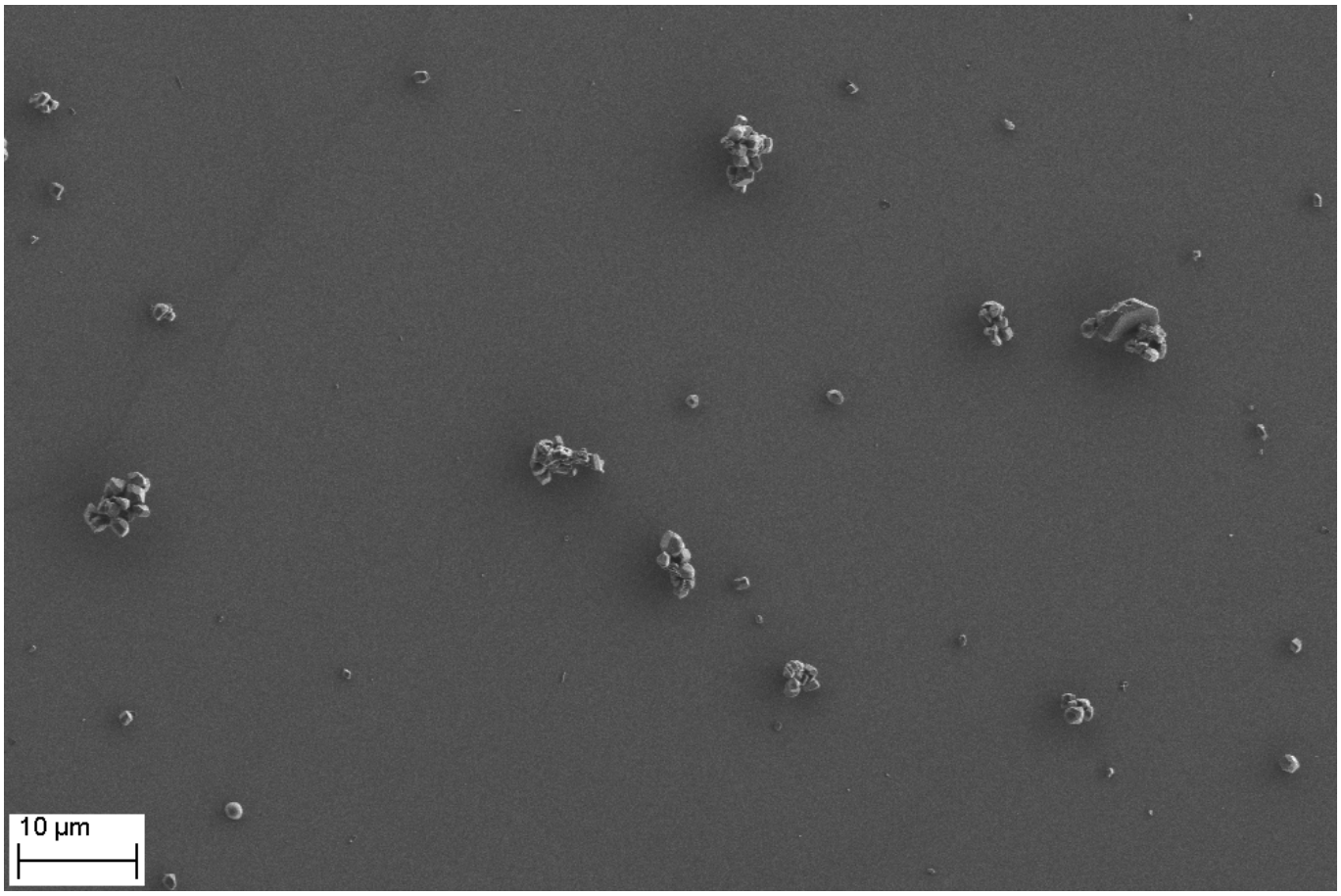


SEM image showing rod tapering and nanofiber formation when preformed rods sonicated after 3 hr. of initial growth.

S5



SEM image when TEOS is added after sonicating the solution for 6 hr.



SEM image of a sample sonicated without any TEOS.