Electronic Supplementary Information

Evolution of amorphous selenium nanoballs in silicone oil and their solvent induced morphological transformation

Arun Kumar Sinha, Anup Kumar Sasmal, Shyamal Kumar Mehetor, Mukul Pradhan and Tarasankar Pal*

Department of Chemistry,

Indian Institute of Technology, Kharagpur-721302, India

E-mail: tpal@chem.iitkgp.ernet.in

Synthesis of Se

Synthesis of Se nanoballs

In the typical preparation, 0.01-0.06 g of Se powder was mixed with 40 mL pure silicone oil in a round bottom flask equipped with a condenser at room temperature, and then the reaction mixture was elevated to 230-240 °C in argon atmosphere on sand bath and the temperature was maintained until all Se metal dissolved. In this case, a transparent deep yellow solution was formed and it is considered as liquid Se. Then, this hot solution was allowed to stand overnight to cool down, and a brick red colored colloidal dispersion was obtained. The as formed red color Se material was separated from silicone oil by centrifugation at 8000-10000 rpm. Then the separated red Se was redispersed in tetrahydrofuran (THF) and immediately separated from THF by centrifugation. The washing of silicone oil by THF was done at least 5 times and dried at room temperature in the dark. Now the separated and dried Se nanoballs were stored in dark for characterization.

When more than 0.06 g Se in 40 mL silicone oil was used for the typical synthesis, one extra observation is that red Se was deposited as thin film on the upper surface of the round bottom flask due to sublimation.

Molecularization of Se in silicone oil:

In a 250 mL round bottom, 40 mL silicone oil was taken and black commercial Se powder (0.01-0.06 g) was mixed to it. The bulk selenium was dispersed in silicone oil by gentle shaking. The mixture was heated under argon atmosphere on a sand bath at about

230-240 °C. Selenium, having a lower melting point than the boiling point of silicone oil, melts and was homogeneously dispersed in silicone oil as a yellow solution. The open end of the round bottom flask was tightly closed with a standard joint glass stopper under hot condition, taken out, wrapped with a heat resistant cloth (hot cloth) and shaken vigorously. This is molecularization of selenium in silicone oil similar to the molecularization of sodium metal in boiling xylene. After the vigorous jerk the hot cloth was removed and the round bottom flask having yellow colloidal selenium dispersion was allowed to cool to room temperature for further use. Room temperature cooling made the dispersion brick red. This caused the transformation of Se into tiny selenium nanoballs (SNBs).

Synthesis of Se Nanoflowers

After removing silicone oil by THF, the as formed Se material was store in THF at room temperature for 7 days; the red colored Se material was gradually changed to grey-black Se. After the color change, the material was dried and stored for characterization.

Synthesis of Se nanowires

After removing silicone oil by acetone, the as formed Se material was stored in acetone at room temperature for 7 days; the red colored Se material was gradually changes to deep red Se. After the change of color, the material was dried and stored for characterization.

Fig. S1 UV-Vis spectrum of colloidal Se nanoballs in silicone oil

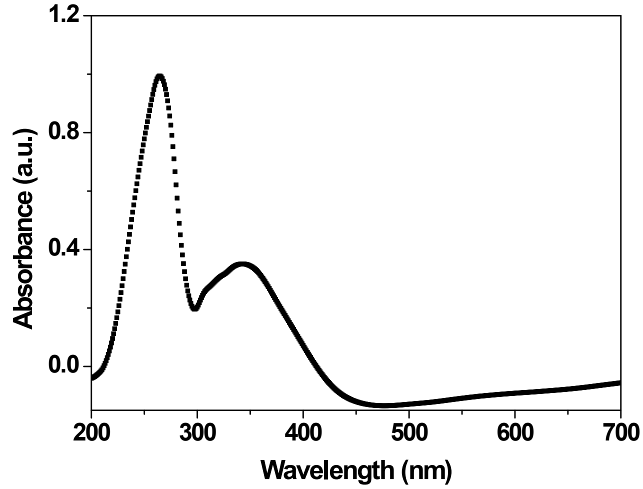
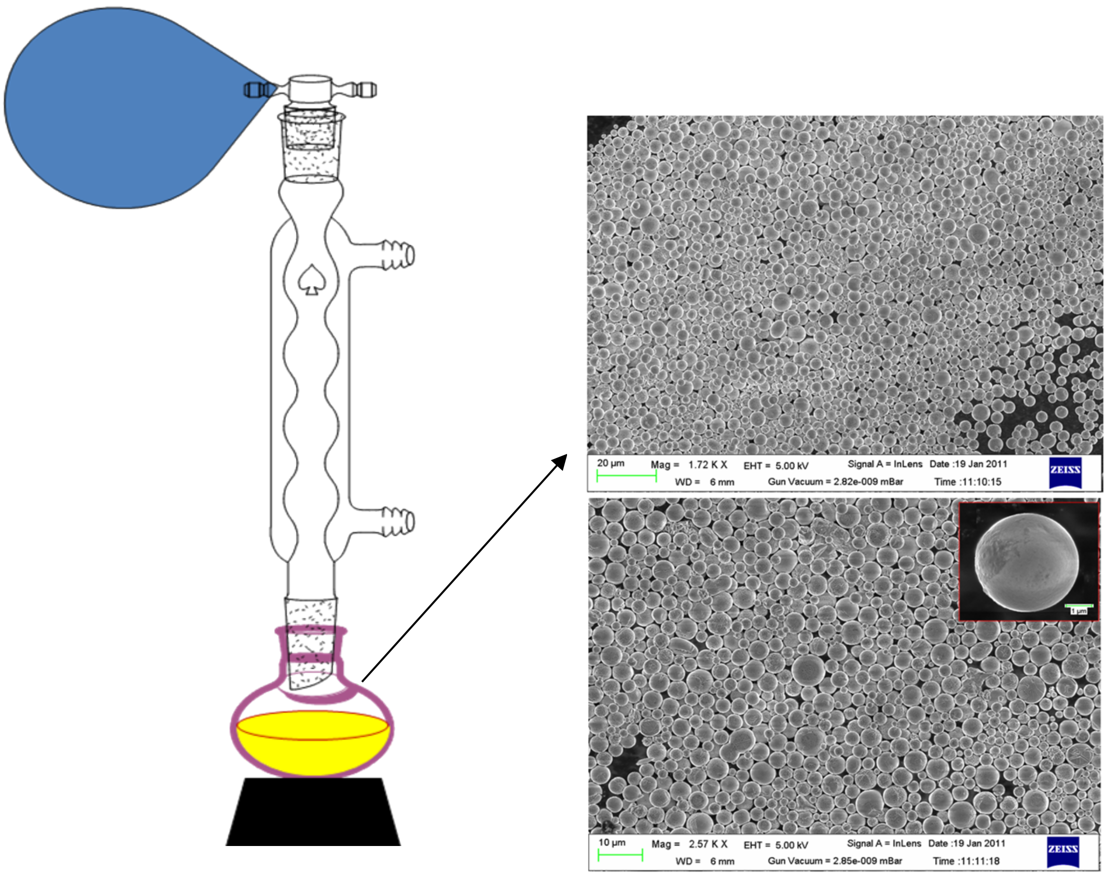


Fig. S2 Formation of Se microcrystal on upper wall of the round bottom flask during melting of the material in silicone oil by evaporation.



During this heating process, liquid Se in silicone oil was slowly evaporated and deposited on the upper surface of round bottom flask. It is observed that a perfect spherical micro crystal of Se is formed on the upper glass surface.

Fig. S3 EDS pattern of Se nanoflower (a) and nanowire (b)

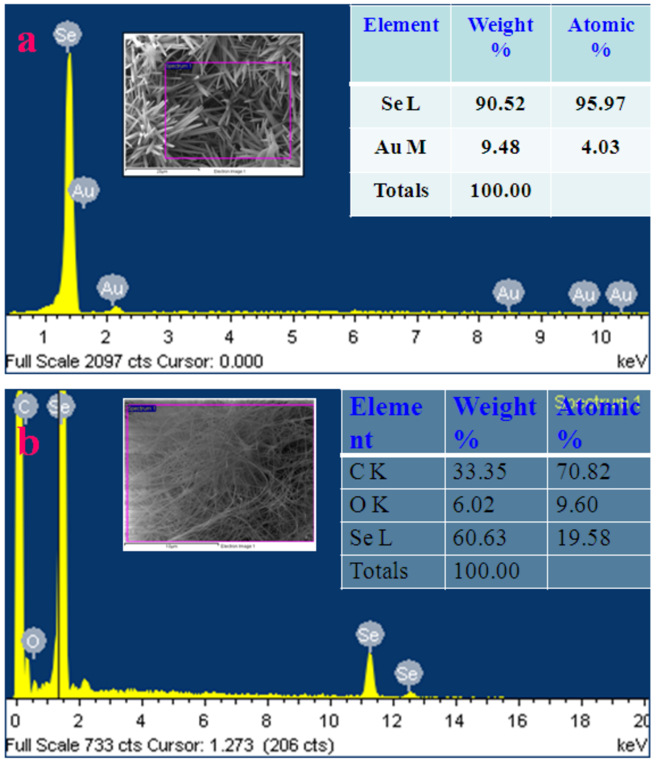


Fig. S4   DTA analysis of different Se material

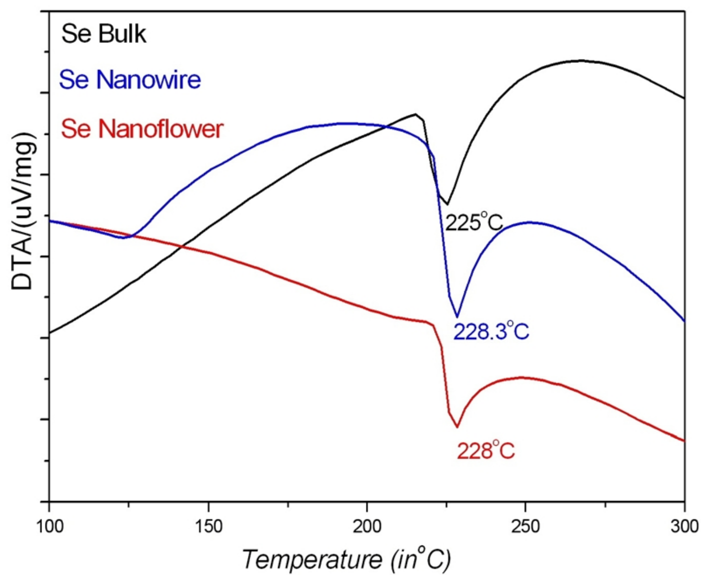


Fig. S5 DRS-spectra of powder bulk Se, Se nanowires and Se nanoflowers

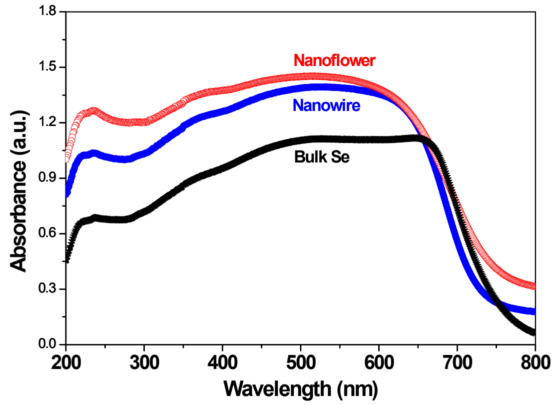


Fig. S6 Se-PMMA composite material