Supplementary Information

Shear Induced Micromechanical Synthesis of Ti_3SiC_2 MAXene Nanosheets for Functional Applications

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Experimental Section

Synthesis of nanosheets of Ti_3SiC_2 (MAXenes) by micromechanical cleavage technique. Micronic Ti_3SiC_2 particles having an average size of 13 μm were procured from the 3-ONE-2 company, USA. Micromechanical milling was performed using Ultra-Fine mortar grinder (Retsch-RM 200, Germany) which works on pressure-friction principle. It consists of a mortar, pestle and a scraper. The role of the scraper is to feed the material in the area between the pestle and mortar. N,N Dimethyl formamide (DMF, Specctrochem, India), N-methyl pyrolidinone (NMP, Specctrochem, India) and distilled water were used as the solvent bath. Ti_3SiC_2 was first dispersed in 15 mL polar solvents. A highly polar chemical medium was employed based on a reason to prevent a reverse agglomeration of the exfoliated nanostructures which is critically important. In order to compensate for the natural evaporation of solvent, its level was maintained by the addition of small fractions of solvent during milling. A temperature was also monitored periodically to know the thermal-heating during milling. The dispersion after 24h milling was then ultrasonicated for 1h using ultrasonic processor (Sonics Vibra Cell, 20 kHz). The milled

dispersion was then centrifuged at 8000 rpm. Finally a separated nanodispersoid phase (hereafter denotes as exfoliated TSC) was obtained and characterized for the physical morphology, crystallinity, chemical stoichiometry and thickness.

Characterization. Powder X-ray diffraction (X'Pert Pro, Philips X-ray diffractometer) with a monochromator on the diffraction beam side (Cu Kα radiation, λ=0.154 nm) is used for analyzing the crystalline nature. Crystallite size was calculated by Scherrer's formula:

$D_{XRD}= 0.9\lambda/ \beta \cos\theta$

where λ is the X-ray wavelength,θ the Bragg angle and β the full-width at half-max.

The microstructure of bulk TSC before exfoliation was recorded using ZEISS EVO 18 Scanning Electron Microscopy (SEM). Transmission Electron Microscopy (TEM) and high resolution TEM (HRTEM) images were carried out using FEI Tecnai 30G2S-TWIN, operated at an accelerating voltage of 300 kV and coupled with EDAX facility. The zeta potential value was measured using Malvern Zetasizer 3000H. The stability of the MAXene dispersion was monitored with a Nephelometer (CL 52D, ELICO) as intensity of transmitted visible light through the fluid against time. The surface chemistry of the exfoliated MAXene nanosheets was examined by X-ray photoelectron spectroscopy (XPS) using SPECS GmbH spectrometer (Phoibos100MCD Energy Analyzer) with Mg Kα radiation (1253.6 eV) as the excitation source. The peak of C1s at 284.6 eV was taken as the reference energy position. UV/Vis spectrum of the TSC dispersion was recorded with spectrophotometer Shimadzu (UV 240 IPC). The resistivity of the green pellets was measure by standard spring loaded pressure contact four probe conductivity meter, Keithley 6221 DC current source and 2182 A nanovoltmeter at 30 °C.

Supplementary figures

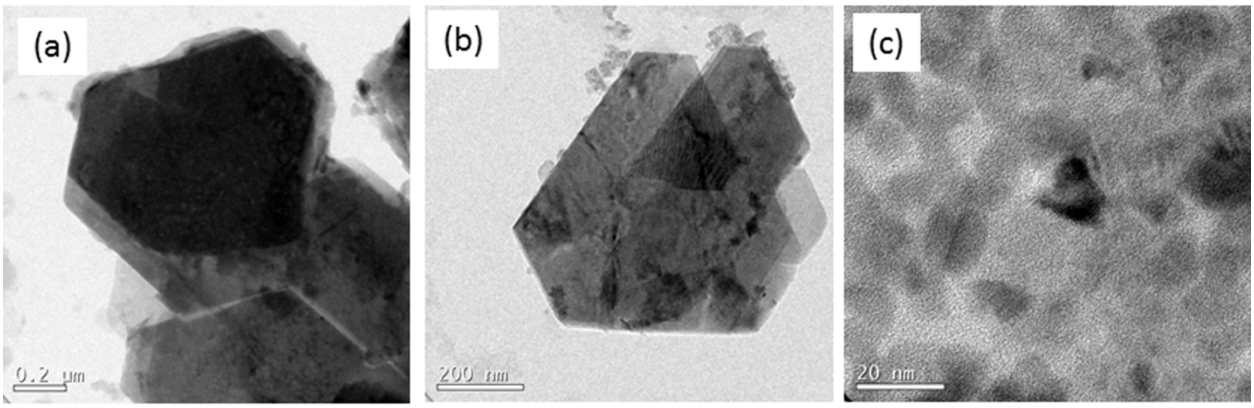

Fig. S1 TEM images of MAXene nanosheets obtained after micromechanical milling time of a) 12h, b) 24 h and c) 30h

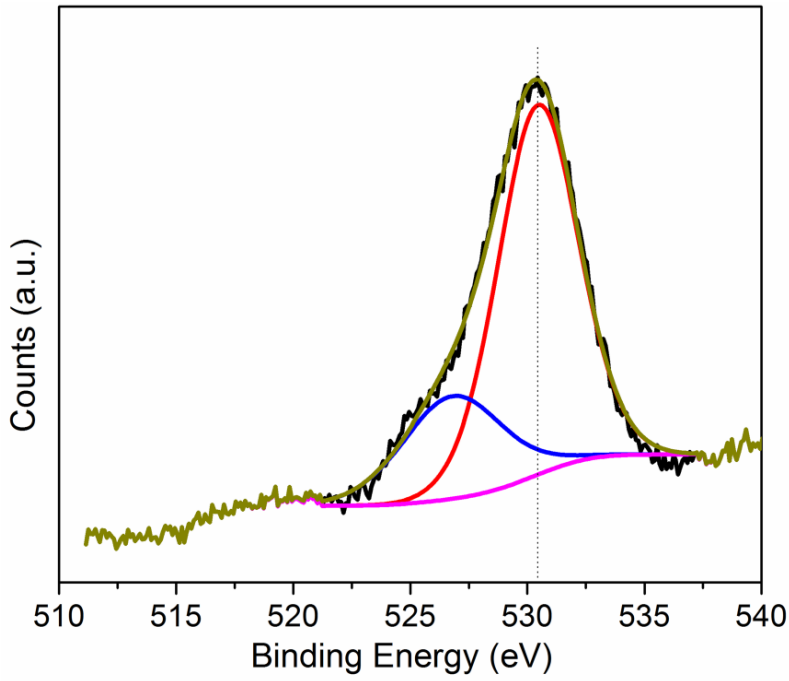

Fig. S2 The high resolution XPS of O1s showing the peaks corresponding to M-O bonds

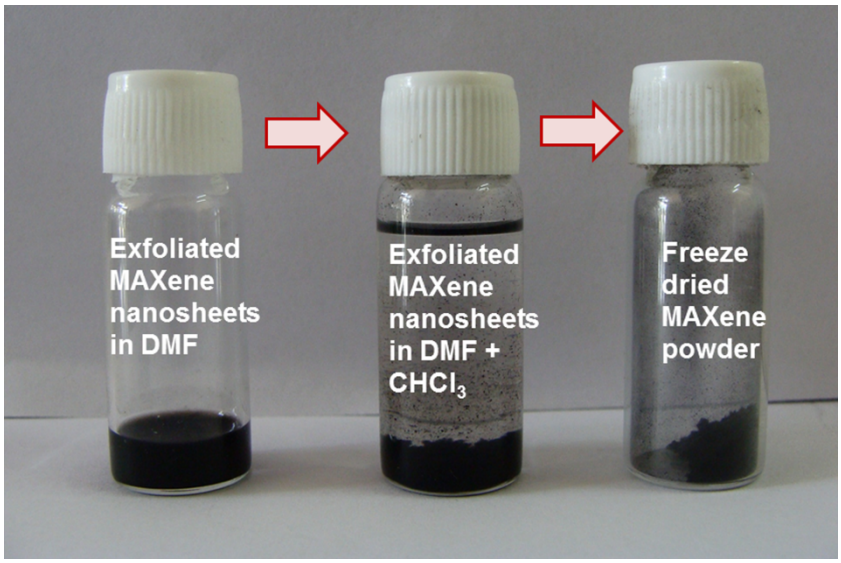

Fig. S3 The easy extraction of exfoliated MAXene nanosheets from dispersions using solvation technique. The naosheets get settled on dilution with less polar solvent and subsequent decantation and freeze drying gives free flowing nanosheets.

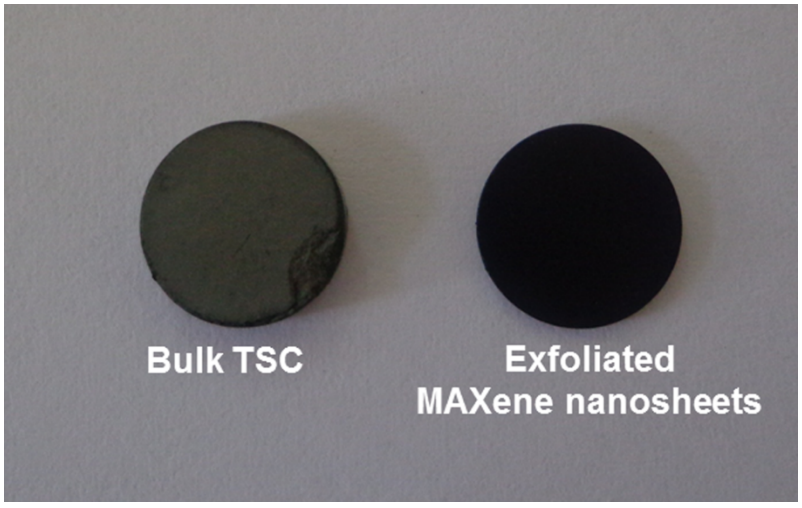

Fig. S4 The photographs of the green pelletes. It is clear that the exfoliated MAXene can be easily made into pellets with good shape retention, low density, green strength and easy for handling whereas the green strength of bulk TSC is very poor as evidenced by edge deformation seen in the image.