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Characterization of Silicon-Germanium Oxide Nanocomposites Fabricated by the Marine Diatom Nitzschia Frustulum

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Diatoms are nature's priceless manufacturers of 3-D intricate nanocomposites [1]. We report here the incorporation of germanium into the marine diatom *Nitzschia frustulum*. Diatom cell mass bearing both silicon and germanium was produced by a two-stage photobioreactor cultivation process. Organic materials in the diatom cell mass were removed by hydrogen peroxide treatment to expose the silica-rich diatom frustule (exoskeleton). Specifically, 100 mg of filtered fresh cell mass from the cultivation process was mixed with 20 mL of 30 wt% hydrogen peroxide solution at 60 ºC for 2 hours under continuous stirring. The suspension was cooled, neutralized, and then centrifuged. The resulting pellet was washed with distilled water and then air dried at 80 ^{o}C. Prior to SEM analysis, the diatom frustules were dispersed onto carbon tape affixed to an aluminum stub. For TEM, the powdered diatom sample was placed on a holey carbon TEM copper grid and then shaken for several seconds to allow the frustules to attach to the grid. An FEI Sirion field emission SEM and a Tecnai F-20 field emission TEM/STEM equipped with an EDS were used to characterize the samples. An overview and topology of the sample was obtained from SEM analysis and a TEM was used to determine the internal structure of the frustules. The physical locations of the nanoparticles containing germanium were obtained from STEM/EDS analysis.

Fig. 1-2 show *N. frustulum* frustules in a low magnification SEM and a high magnification TEM image, respectively. In Fig. 1 (a) the ribbed and honeycomb structure of the frustules is clearly evident. Fig. 1 (b) is a high magnification image of a diatom frustule showing pores with an average diameter of about 160 nm. The EDX spectrum in Fig. 3 (b) shows that germanium (Ge) was incorporated into the diatom frustule. The Ge signal [identified at 2.1 KeV ($L\beta_1$ peak), 9.8 KeV ($k\alpha$)] was obtained from the location indicated by the white dot on the material with darker contrast in the STEM image in Fig. 3 (a). Other characteristic energy peaks were identified as 0.52 KeV for O, 1.21 KeV for Mg; 1.74 KeV for Si; 2.0 keV for P; 2.3 keV for S; 2.62 keV for Cl; 2.67 KeV and 3.67 KeV for Ca; and 6.4 for Fe. Trace elements in the analysis (Ca, Fe, Mg, P, S) were the result of nutrient uptake by the living diatom. Although well-preserved diatom frustules were observed in the samples, some were damaged. Work is currently in progress to minimize this type of damage and to control the amount of Ge incorporated into the diatoms. We are also working to form crystalline structures derived from these diatom frustules [2].

References

[1] K. Sandhage et al, Adv. Matter, 429-433 (2002).
[2] This research was supported by the National Science Foundation (NSF) Nanoscale Science and Engineering Initiative, Nanoscale Interdisciplinary Research Team Award, BES-0400648 and DMR-0353738 (REU Site).

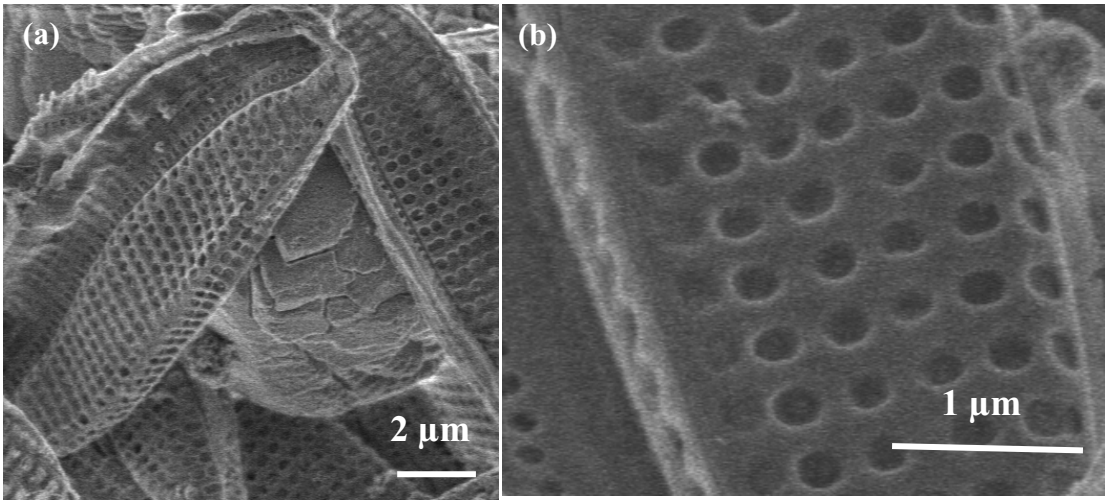

Fig. 1 (a) shows a *Nitzschia frustulum* at low magnification in an SEM. The ribbed and honeycomb structure is clearly evident. (b) is a high magnification image of the diatom frustule showing pores an average of about 160 nm in diameter.

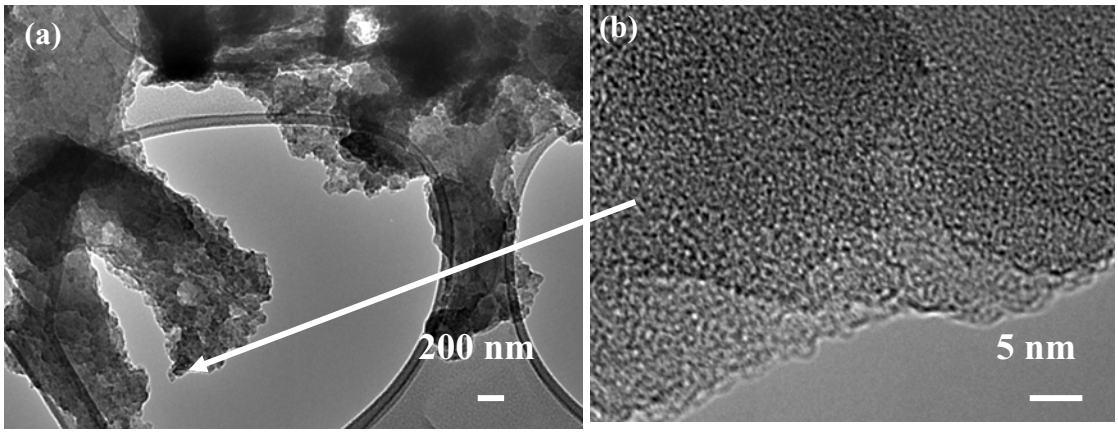

Fig. 2 (a) TEM overview of nanoclustered fragments *Nitzschia frustulum* cell mass. (b) HRTEM of the nanoclustered fragments of diatom cell mass from the area indicated by arrow.

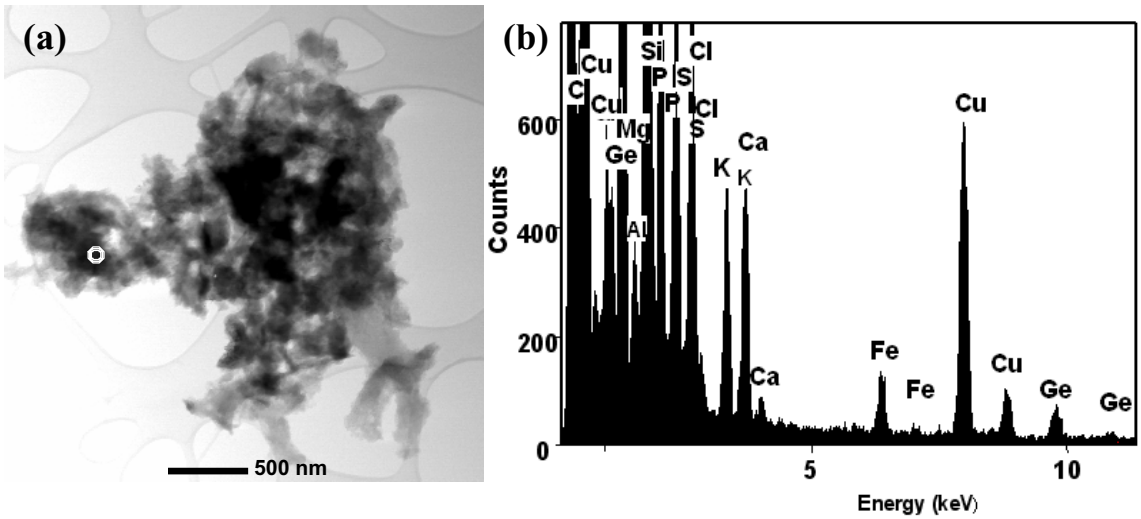

Fig. 3 (a) An STEM image of diatom frustules. (b) EDX quantitative and qualitative analysis showing the presence of germanium in the diatom frustule fragments.

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