

Silicon nanoparticles preparation and the formation of carbon-silicon hybrid nanoparticles for sensor applications

Mussabek G.^{1,2}, Zhylybayeva N.^{1,2}, Zaderko A.N.³, Baktygery S.^{1,2},
Yermukhamed D.^{1,2}, Taurbayev Ye.^{1,2}, Lisnyak V.V.^{1,3}

¹*Institute of Information and Computational Technologies, Pushkina Str., 125, 050010 Almaty, Kazakhstan.* ²*Al-Farabi Kazakh National University, al-Farabi Ave., 71, 050040 Almaty, Kazakhstan;*

³*Taras Shevchenko National University of Kyiv, Volodymyrska Str., 64/13, 01601 Kyiv, Ukraine,*
E-mail: vladyslav_lisnyak@yahoo.com



Preparing silicon nanoparticles (Si NPs) is a problem that needs a nontrivial solution. For this aim, we performed by preparing Si NPs using different routes, including the reduction of silica NPs and silicon-organic compounds and mechanical grinding using porous silicon materials of various origins. Si NPs have developed inner surface and moderate chemical reactivity. The sensitive layer of Si NPs was modified with carbon adatoms by using chemical routes and by carbonization of organic compounds, including sugars, on the surface of Si NPs. We carried these modifications to regulate adsorptive properties for preparing advanced vapor sensors. Prepared by different processes, silicon nanomaterials, in the form of hybrids and composites, were briefly characterized by TEM and SEM methods. We carried out the modification of Si NPs and Si nanoscale filaments with nanocarbon and nanostructured carbon. The formation of hybrids of various types, including the desired ones, in which nanocarbon modifies the nanosilicon surface, occurs because of high-temperature structuring processes. They are passed during the carbonization of carbon-containing precursors. We have developed and successfully applied synthetic techniques for the experimental production of hybrids bound by a thin interface and surface-modified Si NPs. The sensor characteristics of some surface-modified Si NPs were examined with ammonia and alcohol vapors. The obtained nanohybrids with the carbon decorated surface at the interfacial boundaries and the edge defects/oxidized surface of Si NPs showed sensor response towards hydrogen and ammonia in the gas phase being indifferent to low molecular weight alcohols.

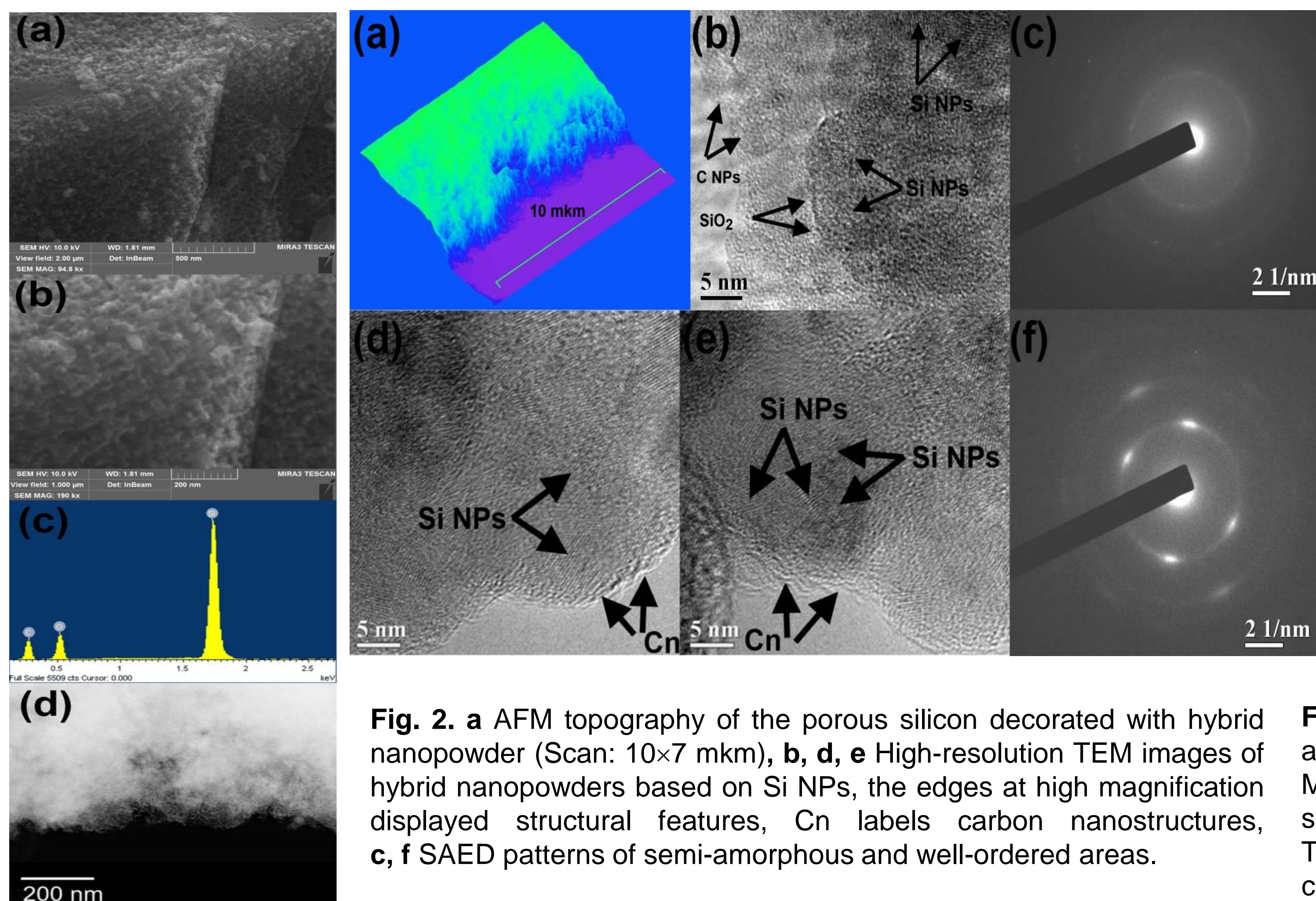


Fig. 2. **a** AFM topography of the porous silicon decorated with hybrid nanopowder (Scan: 10×7 mkm), **b, d, e** High-resolution TEM images of hybrid nanopowders based on Si NPs, the edges at high magnification displayed structural features, Cn labels carbon nanostructures, **c, f** SAED patterns of semi-amorphous and well-ordered areas.

Fig. 1. **a, b** SEM images at low and high magnifications of hybrid aggregates (integrated in the sandwich structure), **c** EDX analysis, **d** TEM image of the edges at high magnification displayed a well-developed porous structure.

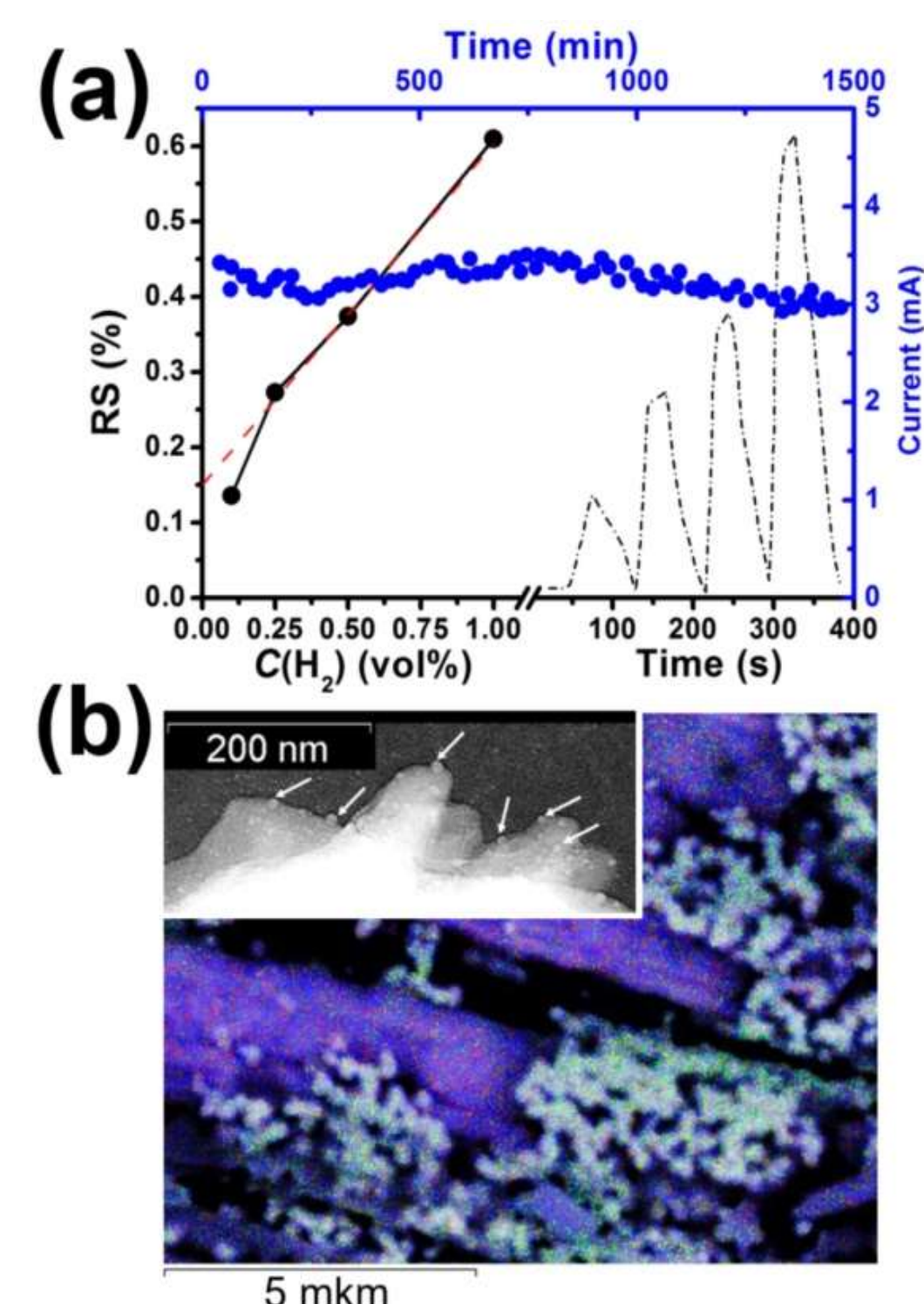


Fig. 3 **a** RS against $C(H_2)$ and transient RS at different $C(H_2)$ in N_2 at 100 °C. Measurements were done with the sandwich structure at 1.0 V (black scales). The response stability under stated conditions (blue scale); **b** TEM image (upper insert) and SEM-EDX elemental mapping of the cross-sectional image of the sandwich structure: blue – Si, red – Pd; white – C, green – O.

Figure 3a shows that the value of RS is influenced by the presence of H_2 in the inert N_2 carrier gas at 100 °C. The maximal RS registered at the maximal volume concentration ($C(H_2)$) of 1 vol% H_2 in N_2 is about 61% at 1.0 V. The RS change has a linear character within the studied $C(H_2)$, $C(H_2) = 0.25\text{--}1.0$ vol%, $RS = 0.15 \pm 0.02 + (0.45 \pm 0.01) \times C(H_2)$. The recovery time for all studied $C(H_2)$ is in the range of 48 ± 4 s. The response time at the $C(H_2) = 0.1, 0.25, 0.5$, and 1 vol% of H_2 is 24, 17, 15, and 14 s, correspondingly. However, the recovery process over the sandwich structure takes a longer time (48 ± 4 s) for the studied $C(H_2)$.

In this contribution, we show the growth of CNS carbon nanostructures on the surface of partially oxidized Si NPs. Low pyrolysis temperature of 400 °C causes the decoration of Si NPs by 2D carbon structures and carbon NPs. Oxidized Si NPs with an oxide shell of below 1 nm can serve for the growth of different carbon nanostructures. So, our results are important for developing Si NPs/carbon interfaces. Based on TEM analysis, we showed the formation of hierarchical nanostructures when carbon NPs nucleate and growth on the partially oxidized Si NPs. The obtained hybrid based on Si NPs, considering the surface modification and decoration with Pd NPs, presumably, both at the interfacial boundaries and at the edge defects or on the oxidized surface of Si NPs, showed the possibility of being sensory materials for H_2 detection in the gas phase. The proposed hybrid NPs decorated with Pd NPs are of considerable interest (as sensor material) to be incorporated into semiconductor sensor devices.

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