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# Pulsed ionized mesh-like assembly of hybrid silica nanostructures with controlled resistivity



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#### ABSTRACT

The adaption and inclusion of electrical technologies with various applications have motivated researchers to develop the most efficient and effective sensors, batteries and capacitors for safe, reliable and prolonged operations. However, miniaturization of sensors and practically associated with batteries and capacitors have driven them to employ nanofibers and nanotubes made of various material for reliable operations. Considering wide acceptance and available research for silicon material, nanofibers and nanoparticles made using silicon can be a great candidate for manufacturing of such sensors and electrical components. To employ nanofibers for those applications, understanding of its resistive characteristics can be crucial. In our study, we have manufactured hybrid silicon nanofibrous structure using novel approach of pico-second laser ablation and performed 4-point resistivity test to comprehend the change in its electrical behavior with variations in different laser parameters like frequency, power and scanning parameter of number of loops. Significant change in resistivity and topological structure was observed for surface ablated with various laser and scanning parameters.

#### 1. Introduction

Manufacturing of high capacity rechargeable Li-Ion batteries for automobile, cellular and medical devices along with fabrication of various effective, optimized and miniaturized sensors for different applications such as bio-sensing and health monitoring are a great interest of 21st century [1–3]. Nanofibers made of various materials and carbon nanotubes have been increasingly accepted in applications like sensors, capacitors, batteries and implants as it provides exceptional surface area and performance suitable for the application [4–9]. Along with the topological property of nano and micro structures, control of an electrical property such as resistivity is vital for suitability of the approach in various applications [10-14]. Carbon nanotubes are proven to be promising for such applications as increase in surface area causes enhancement in current with decreased impedance which can be desirable depending on the application [15,16]. Along with the promising performance, it may offer some disadvantages such as lack of solubility in aqueous media, low purity, alignment issues and non uniform resistive property based on the fabrication method [17]. Contrary, silicon being abundant and considering the wide acceptance in semiconductor industry, noticeable surge has been seen in attempt to use silicon nanofibers for such applications [18-20]. Due to importance of the electrical characteristics in micro and nano manufacturing, silicon hybrid

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Received 23 April 2018; Received in revised form 8 May 2018; Accepted 15 May 2018 Available online 18 May 2018 0169-4332/ © 2018 Elsevier B.V. All rights reserved. nanofibrous structure with controlled resistivity can be greatly beneficial. For fabrication of nano patterned silicon surface with desired characteristics, modern methods like photo-lithography, etching, chemical-physical vapor deposition and atomic layer deposition offers great suitability combined with significant limitations, complexity and huge capital investments [21–24]. In this study, we propose a inexpensive, relatively less complex and novel approach of picosecond laser ablation to form silicon hybrid nanofibrous structure with controlled resistivity. Various laser parameters like frequency, power and scanning pitch was varied to achieve nano fibrous structure with diverse resistance characteristics.

#### 2. Materials and methods

Silicon n-type  $\langle 100 \rangle$  wafers (source: University Wafer Inc., manufactured using Czochralski process - one side polished) were processed using a Ytterbium pulsed fiber laser (IPG Laser Model: YLPP-1-150V-30) with a wavelength of 1064 nm. The laser diameter of 7.6 mm was reduced to 6 mm using iris diaphragm which was then focused towards XY galvanometer scanner (JD2208 by Sino-Galvo). Employing F-theta lens with focal length of 63.5 mm, input aperture of 14 mm, and beam displacement of 18.7 mm, this scanner provided theoretical laser spot diameter of 20  $\mu$ m. Laser patterning parameters like scanning speed,

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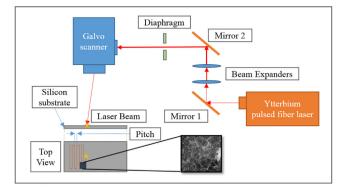


Fig. 1. Experimental setup for silicon nanofiber fabrication.

pitch and number of loops (NL) were controlled using MarkingMate 2.7 software. Fig. 1 depicts general experimental setup for silicon processing. Here, pitch refers to the center distance between two consecutive line pulsed by laser beam and number of loops is the count of laser passed through same ablated surface. For example, 3 NL would mean laser beam has passed 3 times over the same surface. Sample preparation was done with constant pulse width of 150 ps, pitch of 0.025 mm and scanning speed of 100 mm/s. Sets of specimen was prepared with combination of frequency of 1200 kHz, 800 kHz and 600 kHz, power of 10 W, 15 W and 20 W along with variation in number of loops from 1 to 3.

#### 3. Resistance and surface characterization

#### 3.1. 4-Point resistivity measurements

4-Point probe (NAPSON Corporation TC - 150u - 100 g) was used to measure direct resistivity of prepared samples. It was measured on several different locations on each sample to compensate any deviations in homogeneity of ablated surface.

#### 3.2. Surface characterization: Scanning Electron Microscope (SEM), energy-dispersive X-ray spectroscopy (EDX) and Raman spectroscopy

Various means for characterization of surface was employed for the study. FEI Quanta 3D 200/600 scanning electron microscope (SEM) was used to collect desired images for topological study of processed surface. In addition, the study of combinations of formed material and it's degree of presence was done using collected data and images from EDX (Equipment used: EDAX Genesis 4000 energy dispersive X-ray system) and Raman spectroscopy tests (Equipment used: Renishaw Raman Imaging Microscope System 2000).

#### 3.3. Image analysis: ImageJ

By analyzing the images produced by SEM, the estimation of topological features related to ablated surface was done using the software ImageJ 1.501 by Wayne Rasband at the National Institutes of Health, USA.

#### 3.4. Light spectroscopy

Light spectroscopy was done using Ocean Optics USB 2000+. All the samples was analyzed using visible spectrum range. Data was extracted using Spectrasuit software. In addition, Fiber-Lite MI-150 was used as source of light with full possible intensity for all of the analysis.

#### 3.5. Statistical analysis

Statistical analysis was done using Microsoft Excel to ensure authenticity of the results. 5% of error is assumed for individual diameter measurement of nano fiber and re-solidified silicon nano particles (R-SiNp). Also, average values and dimensions are shown with error bar marked on the graph which represents standard deviation of 1 for ImageJ analysis and 4-point probe measurements. In addition, One Way Anova analysis with one factor was carried out to check the difference between means with 95% of confidence level. The comments were provided where the means are not significantly different with the predetermined confidence value.

#### 4. Results and discussion

When the laser is aimed on the material surface, it transfers electromagnetic energy to the present electron on top most surface. Those electrons are excited and ejected because of the sudden gain in energy. It also causes the temperature rise on the same surface enabling ionization and formation of plasma. Accumulation of energy on the same spot for longer period of time causes the expansion of plasma which behaves like a shock-wave resulting in a surface particle removal. The expanded plasma contains electrons, ions and nano particles. Plum core being relatively hot and as moving away from center it gets comparatively cold which helps the nano particles to move away from the center of plum and to solidify in relatively colder region. Such rapid accumulation of silicon nano particles causes the formation of nano fibrous structure [25-28]. Such accumulation can be identified through TEM (Transmission electron microscopy) results of nanofibers as shown in Fig. 2(a). As the ablation process being conducted in air, the re-solidified particles and nanofibers may get oxidized [26,29]. In case of ablation with pico-second laser, individual pulse duration being shorter than time required for conventional melt down of crystals, with enough

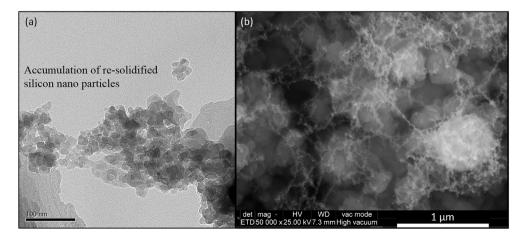


Fig. 2. (a) Formation of nano fibers caused by accumulation of R-SiNp. (b) SEM result of laser ablated silicon surface - mixture of R-SiNp and nano fibers.

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