

Enhanced Thermal Stability of Promising Nano-Porous Silicon Powder

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Abstract

A direct synthesis method is introduced to prepare nano-porous silicon-nickel nanocomposite (nPS/Ni) powder for thermal isolation applications. In this paper, we study the thermal stability of nanocomposites consisting of nanoparticles metal incorporated into the pores of a porous silicon by a very simple method. The nickel element is chemically deposited whereas the nanoparticles are precipitated on the pore surfaces. The (nPS) and (nPS/Ni) nano-materials are thermally measured under nitrogen at temperatures of 40°C - 1000°C, noticeable, demonstrating better thermal stability of (nPS/Ni) until 900°C than in the case of (nPS) at 600°C. Then, the improving of the thermal stability of the nPS powder is facilitated using it in many applications of the thermal insulation process.

Keywords

Chemical Synthesis, Thermal Analysis, Porous Materials, Silicon-Nickel Nanocomposite

1. Introduction

Well-known for its interesting properties, porous silicon (PS) has received considerable attention and many applications in various fields were pointed out. Because of easy integrability with the highly advanced modern silicon technology, as-prepared and oxidized PS has been effectively used as material for the fabrication of micro-hotplates in low power thermal micro-sensors and thermal effect microsystems. Nano-porous-silicon (nPS) is known to have a thermal conductivity that is orders of magnitude smaller than the bulk crystalline silicon from which it is formed [1]. Nanoporous materials are especially promising for thermal insulation and isolation, because they provide the

unique capability to use the microscopic structure to control or inhibit the flow of heat. nPS is of particular technological significance because it can be directly integrated a top conventional silicon microfabricated device as a thermal, structural, or optical material [1] [2]. It allows, for example, decreasing heat exchange between hot and cold junctions of a thermopile based microsensor and consequently, to increase its measurement sensitivity [3].

Till now, two main methods for ensuring the thermal isolation of Microdevices or microsystems were used. First, thin Si structures such as membranes or cantilever beams with high thermal resistance can be realized by micromachining of bulk Si. However, such structures have poor mechanical stability. Secondly, different materials (quartz, polymer films, etc.) having low thermal conductivity values can be applied, but the use of such materials is not compatible with standard semiconductor technologies. The main advantages of this alternative approach are the following: 1) thick nPS layers can be obtained easily and rapidly by simple and chip technological means, 2) nPS-Si structures are more mechanically more stable than thin Si microstructures; 3) nPS is compatible with usual semiconductor technologies [4]. nPS has not been characterized extensively in terms of thermal properties. In particular, no thermal data have been reported for as-prepared and oxidized nPS.

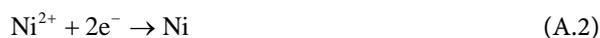
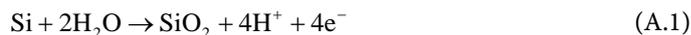
Nevertheless, this large surface area makes nPS chemically unstable, and its oxidation is a common natural process. In fact, the storage of nPS samples in ambient air makes important changes in its electrical and optical properties. Porous materials can be thermally oxidized through different methods. Specifically, the thermal oxidation of nPS under controlled temperature and oxygen atmosphere shows an increase in the surface roughness at 200°C - 400°C and a decrease of 600°C - 800°C [5].

In this work, we present a study on the metallization of nPS by immersion in Ni solution. The presence of Ni on the porous silicon surface improves the thermal stability of the nPS which can be used in fabrication of heaters and thermal sensors.

2. Methods

The nPS powders were prepared by a stain-etching method, depending on our previous study [6] [7]. The produces nPS powder was filtered out from the etching solution and dried in room air for 24 h. The porous layer was formed on the surface of the Si powder owing to the oxidation of the Si surface and simultaneous dissolution of the oxidized Si.

Ni layers were deposited on the nPS powder by a simple chemical method. The as-prepared nPS powder was immersed in an aqueous NiCl₂ solution at 80°C [8]. With this treatment, Ni was deposited on the nPS surface via redox reactions, *i.e.*, Ni ions in the solution were reduced and Si layers were simultaneously oxidized. The corresponding chemical reactions can be expressed as



Finally, the nanocomposite (nPS/Ni) powder was filtered out again from the nickel

salt solution and dried at 60°C overnight for two days. The structure and morphology of producing powder are characterized using XRD (X-ray 7000 Shimadzu diffractometer). The formation of chemical bonds for the formation process of (nPS/Ni) was determined by Raman Spectroscopy (Senteral-Bruker Raman micro-spectroscopy), and TGA (Thermal Gravimetric Analyzer-Schimadzu-TGA50-Japan) which is used to evaluate the thermal stability of the produced powders. In a desired temperature range, if a species is thermally stable, there will be observed the mass change.

3. Results and Discussion

XRD was used to inspect the purity and crystallographic structure of the intrinsic etched silicon nanoparticles. As shown in **Figure 1**, diffraction features of nPS as a production of alkali etching process. **Figure 1(a)**, detects XRD patterns of nPS powder

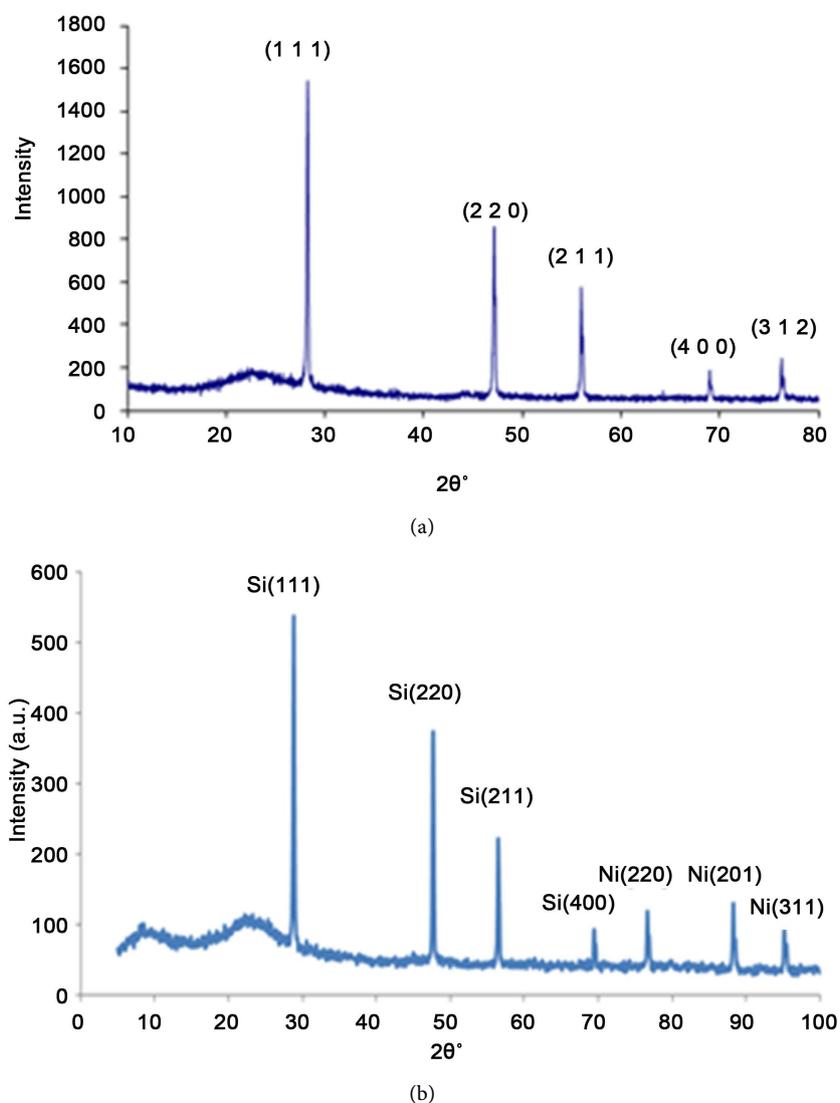


Figure 1. XRD patterns of (a) nPS powder and (b) (nPS/Ni) nano-composite powder.

which is prepared using wet-alkali etching process (6wt% KOH + 30 vol% n-propanol), a wide peak between $20^\circ - 30^\circ$ due to an amorphous structure of Si—O layer on nPS particles surface [9], (111) plane appears at $2\theta = 28.2302^\circ$ of Si structure, accompanied with the peaks at $2\theta = 47.1286^\circ$, 55.9607° , 68.9825° and 76.2396° corresponds to planes (220), (211), (400) and (312), respectively, of Si structure (JCPDS Card No. 01-079-0613 and 00-027-1402). **Figure 1(b)**, shows the XRD peaks in the $2\theta = 0^\circ - 100^\circ$ range for the as-prepared nPS/Ni sample, it describes the polycrystalline shape of nPS powder which is coated with nanocrystalline Ni particles for producing nano-composite (nPS/Ni) powder. Also, it shows a wide peak between $10^\circ - 20^\circ$ due to an amorphous structure of Ni—O which form the chemical bond with nPS particles [10], in addition, small diffraction peaks at $2\theta = 76.68^\circ$, 88.34° and 95.24° , arising from the reflection on (220), (201) and (311) planes, respectively, of Ni structure (JCPDS Card No. 01-089-7129).

The Raman spectra analysis indicated that the Ni content deposited on the nPS surface {the nPS powder has a larger surface area as compared to the Si powder} increased by increasing the deposition time and Ni salt until arriving to the saturation case, which appeared at (4Ni:1PS) percentage. After Ni was deposited, the nPS powders changed from hydrophobic to hydrophilic because of the oxidation of the nPS surface. **Figure 2** shows the Raman-spectra peak at 516 cm^{-1} for nPS powder and the slight shift of peak to 514.5 accompanied by a broad peak in the range $50 - 212\text{ cm}^{-1}$, as a result of, n-Ni deposition within the pores for the formation of nano-composite (nPS/Ni). The optical phonon band in the Raman spectrum shifts to lower frequencies with decreasing crystallinity in nPS material than in case of bulk Si-material [11]. Contains red-shift has been reported for decreasing the size of the crystallites as a result of forming the (nPS/Ni) nanocomposite.

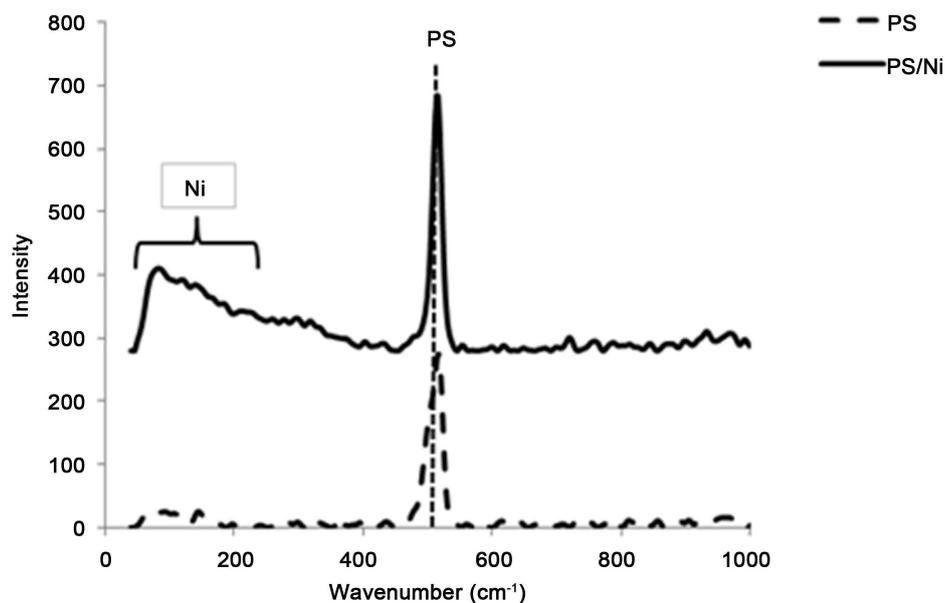
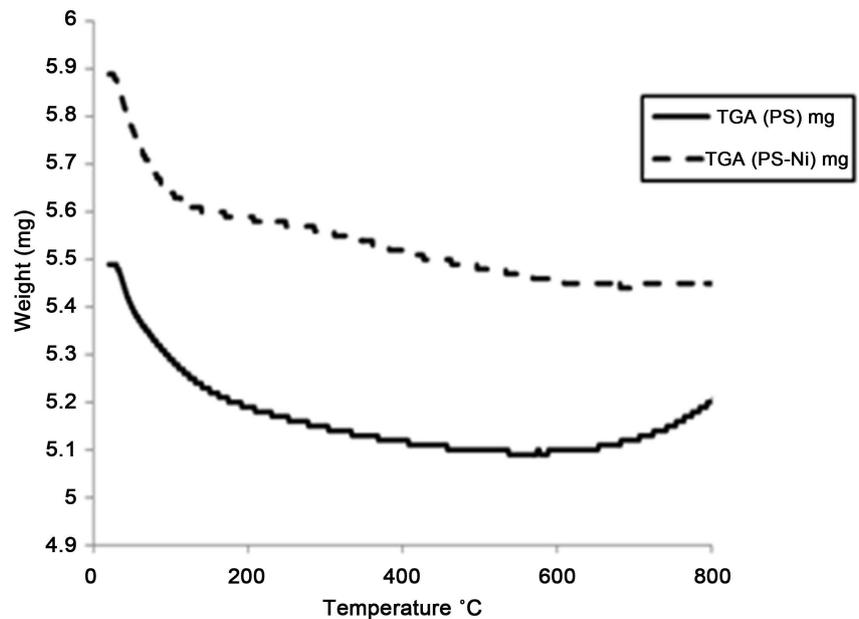
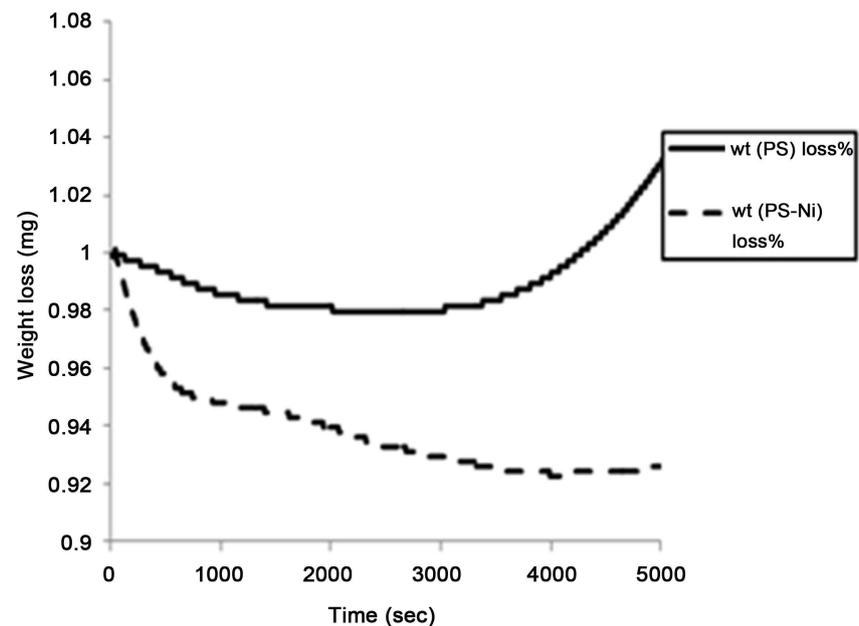


Figure 2. Raman spectra of nPS and (nPS/Ni) nano-composite powders.

Thermal stability of nPS and (nPS/Ni) are the stability of its molecule of each of them at high temperatures; *i.e.* a molecule with more stability has more resistance to decomposition at high temperatures. Once the structural characteristics of the samples were determined. **Figure 3** presents the results of the thermal gravimetric analysis (TGA) of the two samples (nPS and nPS/Ni) under an air atmosphere to a maximum temperature of 1000°C. **Figure 3(a)** and **Figure 3(b)**, show that, the major weight loss is attributed



(a)



(b)

Figure 3. TGA curves of; nPS powder and (nPS/Ni) nanocomposites.

to surfactant decomposition and is observed between (40°C - 200°C), (40°C - 100°C), respectively. The thermal stability plateau, as shown in **Figure 3(a)** which corresponds to (nPS/Ni) sample, is larger than the thermal stability plateau of nPS, as shown in **Figure 3(b)**. Complete combustion of the porous silicon above 600°C yields the nPS content; on the other hand, above 900°C the nanocomposite (nPS/Ni) is composted. So, we get the improving of the thermal stability of the nPS powder to facilitate using it in many applications of the thermal insulation process. There is equality in the decreasing of weight percentage in two samples, as shown in **Figure 3(a)** and **Figure 3(b)**, noticeable, the improvement of the thermal stability until 900°C. A significant mass loss of approximately 2 % to 3% was observed within 100°C to 450°C for nPS, as mentioned above. While, gradual mass loss (1% to 3%) can be observed within 100 to 600°C for (nPS/Ni) nanocomposite. This result indicates that the nanocomposite powders were able to maintain thermal stability within temperatures (600°C to 900°C) and sintering temperature (700°C).

4. Conclusions

Metal deposition into the pores of nPS, it is a versatile material which is utilizable in various fields, is also a suitable host material for the deposition of metals as Ni. In the presented work, mainly Ni is discussed. Ni can be precipitated in structures, whereas the diameters correspond to the pore diameter, as reported by the authors in previous works. Thermal stability is one of the critical parameters, which plays a significant role in thermal energy storage applications.

The nanocomposite (nPS/Ni) is more stable than (nPS); the ordered porous structure of silicon was cooperated with Ni element, which enhance the thermal stability, then the nanocomposite (nPS/Ni) begin in decomposed at temperatures as high as 900°C. This impact on the surface chemistry and functional groups present but does not appear to induce significant structural reorganization of nanocomposite (nPS/Ni) network.

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