



Production and characterization of nanocomposite thin films based on Ni matrix reinforced with SnO₂ single-crystalline nanowires for electrical contact applications



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ABSTRACT

Nanocomposite thin films based on electrodeposited Ni matrix reinforced with SnO₂ single-crystalline nanowires grown onto Si substrates by chemical vapor deposition were produced. The composites were characterized by means of scanning and transmission electron microscopy (for imaging, selected area diffraction and transmission Kikuchi diffraction), atomic force microscopy (for 3D surface profiling and roughness evaluation) and 4-point probe electrical resistivity measurements. The Ni matrices obtained were nanocrystalline in nature (41 nm crystallite mean size) even though low direct current electrodeposition was used. The topography and roughness of the samples were strongly affected by the presence of the nanowires as so was the electrical resistivity, which could be improved by Ag-coating the nanowires. A comparison with pure Ni produced in the same way is presented for determining the effects of the SnO₂ nanowires.

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1. Introduction

Electrical contact devices initiate or interrupt the flow of current in an electrical circuit through physical contact between two electrodes. They can be found in a wide spectrum of home to industrial applications with several operational demands (current, voltage, temperature, etc.). According to these, different materials are employed. One frequent alternative are composites consisting of a metal matrix reinforced with metal-oxide particles (Ag–SnO₂, Ag–Ni₂O, Ag–ZnO, etc.), yielding good resistance to arc erosion and welding given the high melting point of the oxide phase. The electrical conductivity is in turn reduced with respect to the pure metal [1–4]. These composites are mostly manufactured by powder metallurgy procedures (pressing–sintering) [5–7], but can also be prepared by electrodeposition [8].

It is the focus of our research to develop a new type of composite material with tailored microstructure and properties, which could provide enhanced resistance against electrical erosion. We intend to achieve this by replacing the micro-sized oxide particles

with 3D-networks of single-crystalline nanowires – SnO₂ in our case – chemically grown and bonded to a substrate. The traditional thermo-mechanical processing used to fabricate the composite from the powders is then no longer viable. Instead, the embedding of the nanowires within a metallic matrix would be achieved through electrochemistry metallization techniques the electroless deposition and electroplating.

The use of SnO₂ nanowire arrays is expected to provide a desirable finer dispersion of the oxide phase, thus reducing its agglomeration during arcing and welding which leads to failure of electrical contacts [9,10]. Given their single-crystalline nature, the nanowires exhibit higher thermal and electrical conductivities than the polycrystalline powders [11–13] and even metallic conduction is possible through doping [14,15]. Moreover, significantly smaller materials could be fabricated with this method, which is very important, for instance, in electromobility and microelectro-mechanical systems.

In this work, SnO₂ nanowires were obtained by chemical vapor deposition (CVD) with vapor–liquid–solid (VLS) growth [16,17]. Subsequently, they were embedded in a galvanostatically-deposited Ni matrix. The fabrication process is here described and evaluated, followed by a detailed microstructural and topographical characterization. Finally, electrical resistivity measurements are presented. The main objective of this study is to determine the

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effects of the SnO₂ nanowires on the analyzed processes and parameters. For that, a comparison with equivalent pure-Ni is performed.

2. Materials and methods

2.1. Sample fabrication

Three different types of samples conforming thin films were investigated: SnO₂ nanowires in Ni matrix (Ni–SnO₂); Ag-coated SnO₂ nanowires in Ni matrix (Ni–Ag–SnO₂); and pure Ni. All of them were set onto 0.5 × 0.5 up to 1 × 1 cm² substrates cut from monocrystalline Si wafers.

The SnO₂ nanowires were grown in a low-pressure cold-wall CVD reactor. The temperature of the precursor source containing [Sn(OrBu)₄] was maintained at 27 °C and the precursor was introduced to the reactor chamber by the applied dynamic vacuum (5×10^{-3} mbar). The deposition took place for 15 min at 750 °C. The Si substrates were previously decorated with Au nanoparticles (by sputtering), which acted as catalysts for the one-dimensional VLS growth of the nanowires.

The Ag coating of the nanowires for the Ni–Ag–SnO₂ system was done by electroless deposition [18,19]. The reaction was performed in a beaker containing 30 ml of 0.1 M AgNO₃ aqueous solution (with ammonia and ethylenediamine as complexing agents), where 3 ml of 2 M D-glucose aqueous solution (the reducing agent) were added. The reaction ran for 18 min with no external heating and under mechanical agitation. The whole Ag deposition process was done twice, consecutively. The deposits were thoroughly rinsed with deionized water after each step.

The Ni films were galvanostatically deposited [20,21]. A two-electrode galvanic cell was used, with Ni spheres acting as sacrificial anode. The electrolyte used was a commercially available solution based on Ni sulfamate, with a pH of 3.8. Each deposition was carried out at 40 °C for about 70 min under magnetic agitation, with a direct current (dc) of 10 mA (achieving current densities between 1 and 4 A/dm²). In the case of the Ni–SnO₂ and Ni systems, the respective samples were previously Au-sputtered in order to increase the electrical conductivity.

2.2. Material characterization

A FEI Helios Nanolab 600 focused ion beam (FIB)/scanning electron microscope (SEM) dual beam system was utilized for cross-sectioning and imaging the composites. The FIB consists of a Ga ion source operating at 30 kV, aligned at 52° with respect to a field emission electron gun which was used for SEM and scanning transmission electron microscopy (STEM) imaging at 5 and 30 kV, respectively. This device was also used for the preparation of transmission electron microscopy (TEM) thin slices and to obtain transmission Kikuchi diffraction (TKD) patterns for grain size analysis [22,23], using the electron backscattered diffraction module present in the microscope. The latter was done using a 30 kV voltage, 2.7 nA current and 15 nm step size. The obtained data was filtered using confidence index (CI) normalization, removing all grains with CI lower than 0.1 and also those intersected by the edges of the scanned area. A JEOL JEM-2011 high resolution TEM was operated at an acceleration voltage of 100 kV for imaging and selected area diffraction (SAD) analysis.

For the surface profiling and roughness calculation, a Veeco dimension 3000 atomic force microscope (AFM) was employed. The measurements were performed in tapping mode with 60 × 60 μm² scan size and 512 lines per scan at 0.5 Hz.

The electrical resistivity of the samples (as thin films) was investigated through a 4-point probe set-up, employing currents from 2 to 20 mA at room temperature.

3. Results and discussion

Arrays of single-crystalline SnO₂ nanowires were obtained through the CVD-VLS process, reaching up to several tens of μm in length and about 100 nm in diameter. Given the lattice mismatch between SnO₂ and the Si substrates, the one dimensional nanowire-growth proceeded randomly in multiple spatial directions, forming complex and highly intertwined networks (Fig. 1).

In the case of the nanowires treated with Ag (for the Ni–Ag–SnO₂ system), a full coating of the nanostructures was achieved (sheath/core structure) as it can be observed in Fig. 2. TEM micrographs (Fig. 3) show a continuous, pore-free interface between the SnO₂ and the Ag, indicating a good wetting between them. Through SAD at both phases, their crystalline nature could be clearly determined.

By electrodepositing Ni, the metallic matrices were assembled and the fiber-reinforced composites were finally completed. Depending on the pretreatment applied to the nanowires – Au

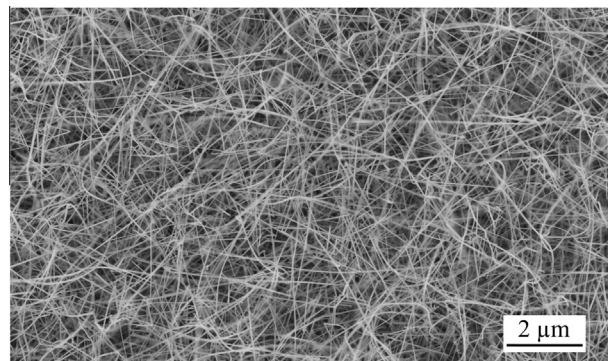


Fig. 1. SEM image of randomly oriented SnO₂ nanowire network grown onto Si by CVD.

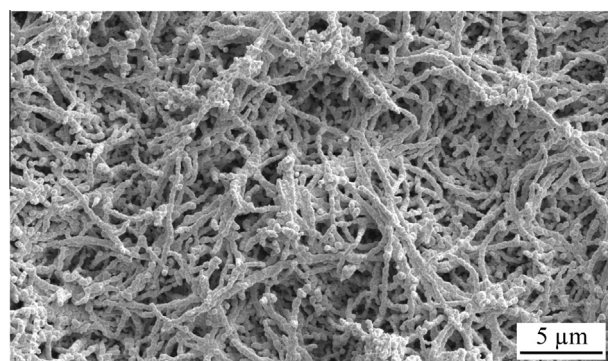


Fig. 2. SEM micrograph of SnO₂ nanowires coated with Ag by means of the electroless deposition technique.

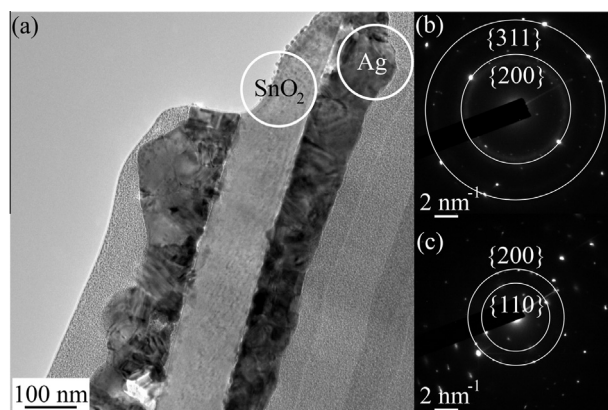


Fig. 3. (a) Bright field TEM micrograph of the cross-section of a Ag-coated SnO₂ nanowire (the circles enclose the areas from where the SAD patterns were obtained); (b) SAD pattern from Ag; (c) SAD pattern from SnO₂ nanowire.

sputtering or Ag coating – different microstructures and material properties were observed. They were also compared to those of pure Ni, which was deposited onto Si through the same process and parameters, in order to study the influence of the nanostructures on the material. The thickness of the Ni deposits ranged between 7 and 13 μm for all the systems. FIB cross-section images of each one are displayed in Fig. 4.

The composites exhibit two different regions running parallel to the substrate, which can be differentiated according to their grain structure: a fine-grained zone (A), in contact with the substrate,

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