Low oxygen pressure synthesis of NdNiO$_{3-δ}$ nanowires by electrospinning

M S Medina, B N Ramirez, P M G L Ferreira, H P Huang, A Zenatti, A J C Lanfredi and M T Escote ©

Engineering, Modeling and Applied Social Sciences Center, Federal University of ABC. Av. dos Estados, 5001, Bangu, 09210-580, Santo Andre, SP - Brazil
E-mail: marcia.escote@ufabc.edu.br

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Abstract

Synthesis of RNiO$_3$ (R = rare earth) nanowires can be interesting as building blocks with potential applications in optoelectronic devices. Here, we describe the synthesis and characterization of NdNiO$_{3-δ}$ (NNO) nanowires produced by electrospinning technique via polymeric precursor solution at relatively low temperature and oxygen pressure. These NNO nanowires were characterized by x-ray diffraction (XRD), x-ray photodetection spectroscopy (XPS), Field Emission Scanning Electron Microscopy (Fe-SEM), Magnetization (M(T)) and electrical resistance (R(T)) measurements. SEM images revealed a granular nanowire microstructure of NNO nanostructures, with a distribution of nanowire diameters ranging from 50 to 150 nm. The NNO nanowires also exhibit granular characteristics with an average grain diameter of 40 nm. The x-ray diffraction patterns of the NNO nanowires indicated that these samples exhibited a high degree of crystallinity and their Bragg reflections can be indexed to an orthorhombic-distorted (Pbnm symmetry) perovskite structure. The crystalline structure seems to be slightly texturized in some Bragg directions and with a slightly strained crystallite. M(T) and R(T) measurement as a function of temperature curves show that these NNO samples present a metal-insulator (MI) transition close to $T_{MI} \sim 198$ K, which is usually observed in NNO thin films and bulk samples. The nanostructured shape and these experimental observations can be promising in designing new electronic devices using this strongly correlated oxide.

1. Introduction

One-dimensional (1D) materials have been extensively studied due to their remarkable electronic properties and potential applications in flexible, transparent electronic and optoelectronic devices. The strongly correlated perovskite oxides present very different electronic properties and could be potentially useful for these devices [1]. In particular, the RNiO$_3$ (R = Pr, Nd, Sm, etc.) compounds are a candidate for these electronic correlated systems since these compounds present a rich phase diagram and are one of the few oxides that allowed a direct correlation between the structure and the physical properties [2, 3]. These compounds can be suitable for use in glucose sensors [4], synaptic transistor devices [5], resistive random access memory devices [6, 7], photovoltaic devices [8, 9], etc.

RNiO$_3$ compounds (R ≠ La) exhibit a metal-insulator (MI) transition, in which MI temperature ($T_{MI}$) could be tuned by choice of the rare-earth ion [10]. In general, this behavior is related to the distortion of the perovskite crystal structure, which increases with the decrease in rare-earth ionic radius [11, 12]. In fact, the change in $T_{MI}$ values was attributed to the distortion of NiO$_6$ octahedrons, in particular to the Ni-O-Ni bond angle, which seems to drive the electron transfer between Ni-3d and O-2p orbitals [11–13]. RNiO$_3$ compounds (R ≠ La) crystallize in the orthorhombic-distorted perovskite structure (space group symmetry Pbnm) and exhibit an MI transition temperature at 130 K (Pr), 200 K (Nd), 400 K (Sm), and 460 K (Eu) [11, 13]. In the

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PrNiO$_3$ and NdNiO$_3$ (NNO) compounds, an antiferromagnetic order of Ni$^{3+}$ sub-lattice takes place at the same temperature as $T_{MI}$. Whereas in SmNiO$_3$ and EuNiO$_3$, this magnetic ordering occurs at temperatures below $T_{MI}$ [11].

RNiO$_3$ compounds (R = La) seem to be thermodynamically instable at typical oxide grown conditions [14]. In fact, the synthesis of these compounds requires high oxygen pressure and high temperature. Many methods have been used to obtain these compounds, including sol-gel method, hydrothermal synthesis [4], and solid-state reaction synthesis [15]. Among these compounds, NdNiO$_3$ has been widely investigated as it can be obtained in less severe temperature conditions and oxygen pressure [11, 16, 17]. To overcome these synthesis conditions, most of the works regarding RNiO$_3$ compounds have been focused on the growth of epitaxial thin films, in which the film thickness and strain can be tuned during the deposition process and modify the RNiO$_3$ properties [18–25]. The epitaxial growth of NNO thin films on single-crystalline substrates induces the film to have the same in-plane lattice parameters, distortions, lattice symmetry, octahedral tilts, and rotations. As a result, the produced thin film presents quite different physical properties from the NNO bulk [26]. Several works investigated the strain-induced by the substrate effect to control the transport properties of NNO thin films [23, 27]. Also, these thin films have been extensively studied with regard to the origin of the metal-insulator transition, the kind of insulator below $T_{MI}$ (Mott or charge transfer gap), the effect of the oxygen vacancies, and the nature of the antiferromagnetic ordering [27–31].

A primary obstacle to the direct application of these compounds is the top–down processing needed to build electronic devices. In general, this process involves etching techniques that can leave exposed surfaces, which then react with ambient surroundings, lithography process with several organic solvents, as well as other processes throughout the device development [1]. In fact, few works describe the physical properties of nanostructured NNO or other RNiO$_3$ compounds [32–34]. One study explored the transport properties of nanowires of NNO grown from a top–down process in an epitaxial thin film with different thicknesses on a LaNiO$_3$ substrate. This work describes non-conventional transport properties for the NNO nanostructure, where the transport becomes non–ohmic with a decrease in temperature [34].

These processing problems can be solved with the advancement in the nanoscale materials growth, which has been used to produce nanostructured perovskite oxides. [32, 35–41]. Such nanostructured oxides can be used as a freestanding building block not only to the technological applications cited above, but also in new optoelectronic devices (e.g., pressure sensors and new designed photovoltaic devices) [8, 9, 42].

In this context, it is important to observe that most works studying RNiO$_3$ devices are based on thin-films techniques [20, 43]. This work describes the synthesis of freestanding NNO nanowires by electrospinning methodology using relatively low temperature (800 °C) in oxygen flow. The effect of this microstructure on their microstructural and physical properties was investigated. As far as we know, this work is the first to produce NdNiO$_3$ nanowires using the electrospinning technique.

2. Experimental procedure

Sample preparation: Polycrystalline NNO nanowires were obtained using nickel(II) nitrate hexahydrate (99.999% - Sigma Aldrich) and neodymium(III) nitrate hexahydrate (99.9% - Sigma Aldrich) as a metals precursors and polyvinylpyrrolidone polymer (PVP—Sigma Aldrich, Mw = 1,300,000) to control the solution viscosity. First, the PVP was dissolved in a mixed solution of ethanol and water (1:1 in vol.) at room temperature for 12 hours. Then, a solution containing stoichiometric amounts of Nd and Ni nitrates was added to the PVP solution, with the ratio between PVP and metals being fixed at 1:1. The resultant solution was maintained under agitation at room temperature for 12 hours. Afterwards, the temperature was raised to ~100 °C to adjust the viscosity. The viscosity was measured in a digital viscometer (LVDV-IP, Brookfield) and kept at ~120 cP. The nanowires were obtained from a homemade electrospinning system at room temperature (~24 °C) and with humidity of ~60%. This system is coupled to a commercial syringe with a metallic needle and a collector (aluminum foil), both of which were connected to a high voltage source (Es30–0, Ormond Beach). The solution was electrospun using a 0.5 mm inner diameter needle with a flux rate of 1 mL h$^{-1}$, needle-collector distance of ~20 cm and 20 kV positive voltage. After the electrospinning process, the samples were dried overnight at 100 °C and then heat-treated at 350 °C with a rate of 2 °C min$^{-1}$ for 4 hours and at 800 °C for 4 hours in an oxygen atmosphere (1 atm). The heat treatment temperatures were chosen based on thermogravimetric results obtained from electrospun LaNiO$_3$ nanowires produced with a similar procedure (see figure S1 in the supplementary file) and x-ray diffraction of NdNiO$_3$ results from the literature, which indicated a crystallization process above 700 °C.[44].

Structure and microstructural characterizations: The crystalline structure of the NNO samples was studied through x-ray diffraction (XRD) on a diffractometer (Stoe, STADI-P) operating in a transmission mode,
with Mo-Kα radiation in an angular range of 2–60°, an angular step of 0.015°, and an integration time of 150 s. The morphology was analyzed on a field-emission scanning electron microscope (FESEM—JMS-6701F, JEOL).

**X-ray photoelectron spectroscopy (XPS):** The elemental composition and the valence state of Ni ions were evaluated by XPS using a spectrophotometer (ThermoFisher scientific) using Al-Kα radiation at room temperature. NdNiO₃ nanowires were deposited on carbon tape and placed in a high-vacuum chamber at pressures ∼10⁻⁹ torr. Spectra analysis was carried out with the CasaXPS software [45].

**Magnetization measurements:** The magnetic properties were performed on NdNiO₃ nanowires (m ~ 0.5 mg) inside a gelatin capsule, which were measured using an MPMS®3-SQUID magnetometer (Magnetic Properties Measurement System—Superconductor Quantum Interference Device, Quantum Design). The measurements were taken in the zero-field cooling (ZFC) and field cooling (FC) procedures from room temperature to 5 K and with an external magnetic field of 10 Oe and 100 Oe.

**Electrical transport measurements:** For the transport measurements, a large amount of NNO nanowires were dispersed on isopropanol and deposited over Au/Ti electric contact patterns previously defined by conventional lithography on SiO₂/Si(110) substrate (see details in the supplementary material). Temperature dependence of electrical resistivity was measured using this set up in a closed-cycle cryostat (CS204SL—Advance Research Science—ARS), which is coupled to a temperature controller (331, Lakeshore) and a source meter (2400C, Keithley). The measurements were taken with a dc current range of 1–10 µA and in the temperature range of 10 to 300 K.

### 3. Results and discussion

Figures 1 (a)–(c) shows the FESEM images of electrospun nanowires of NNO heat-treated at 800 °C for 4 hours in an oxygen atmosphere. These images display the fiber-like morphology of these samples with a solid cylindrical shape and a slightly rough surface. Figure 1(d) shows a histogram with the external diameters distribution of the nanowires. The nanowires present a broad distribution of diameters ranging from 50 to...
Also, FESEM images suggested that the nanowires exhibit a granular feature with an average grain size of \( \sim 40 \) nm, which can be related to the polycrystalline nature of these samples.

Figure 2 shows the XRD measured and calculated pattern for the fully crystallized NNO nanostructures after heat treatment at 800 °C in an oxygen atmosphere. This diffractogram revealed a polycrystalline sample, which crystallizes in an orthorhombically-distorted perovskite structure (Pbnm space group symmetry). The calculated x-ray data was obtained through the Rietveld method using FullProf software. From this analysis, the cell parameters were \( a \sim 5.4168(9) \) Å, \( b \sim 5.3638(1) \) Å, and \( c \sim 7.575(1) \) Å, and the cell volume \( \sim 220.1(1) \) Å\(^3\). These values are very close to those observed in bulk samples, although these cell parameters are slightly greater than the bulk cell parameters \( a = 5.3891 \) Å and \( b = 5.3816 \) Å, and \( c = 7.6101 \) Å reported in the literature \[46\]. We observed a slight decrease of the cell volume. This decrease is generally related to surface and finite-size effects. However, we believe it could also be a result of crystalline distortion derived from the initial crystallization process of these nanowires. Further, a comparison between crystal structures built in the Vesta software \[47\] with the Rietveld analysis data for the NNO nanowires and bulk sample \[46\] (see figures 2(b) and (c)) revealed slight distortions on the NiO\(_6\) octahedra.

Other features contribute to this discussion, such as the fact that the intensity of some reflection is not well fitted like the ones observed at \( 2\theta \sim 15^\circ, 21^\circ, \) and \( 26^\circ \), which belong to (020)/(112), (004), and (024)/(204) Bragg planes, respectively (see figure 2(a)). Such behavior suggests a small texture degree of these NNO nanowires in random directions. Furthermore, the base of some peaks cannot be well-fitted in \( 2\theta \sim 11^\circ, 19^\circ, \) and \( 24^\circ \). Both features indicate the presence of microstrain and microdeformation of the crystallites of these nanowires \[48\]. In fact, if we assume a pseudo-cubic structure \[49\], we obtain a medium lattice parameter \( a_{pc} \) of \( \sim 3.81 \) Å that is slightly smaller than the \( a_{pc} \) of \( \sim 3.803 \) Å usually observed in bulk samples in the literature. This result indicates that the crystalline structures of these NNO nanowires are slightly tensile strained with the similar effect of strained NdNiO\(_3\), thin films grown on SrTiO\(_3\) substrates \[21, 50\].

Similar lattice strains were observed in the crystalline structures of electrospun TiO\(_2\) nanofibers by High-Resolution Transmission electron microscopy images and x-ray data analysis \[31\]. These findings were attributed to the interaction forces that keep the grains tightly together and the surface energy originated from the nanocrystallinity of the grains. Additionally, the authors attributed a maximum surface stress for nanowires...
with diameters close to 60 nm. Therefore, we believe that the NNO nanowire grown by electrospinning in this work has similar characteristics and thus presented similar slight strain lattice.

The elemental composition and the Ni oxidation states were analyzed by XPS measurements. Figure 3(a) shows the survey and 3(b) the Ni-2p photoemission spectrum of NdNiO₃ nanowires. In the survey spectrum, it is possible to identify the expected core levels for Nd, Ni, and O atoms. From this result, the Nd/Ni ratio was calculated, and the value found was close to 1.3. This result is within range of 1.2–3.4 reported in NdNiO₃ thin films [52].

This value indicates a cationic off-stoichiometry and was related to a high Ni³⁺ content [53]. To gain insight into Ni valence, we estimated the Ni content using the total area of the Ni-2p spectrum (see figure 3(b)). The Ni-2p spectrum is characterized by two main peaks around 870 and 855 eV (with their satellites S1 and S2); these peaks correspond to the Ni - 2p₁/₂ and the Ni - 2p₃/₂ lines, respectively. The data was fitting using a Shirley background and a Lorentzian-Gaussian function.

The Neel temperature of the AF order was estimated by fitting the χ(T) curves above 200 K using the Curie-Weiss equation: \( \chi(T) = \chi_0 + \frac{T - CT_N}{C} \), where \( C \) is the Curie constant, and \( \chi_0 \) is the temperature-independent contribution of magnetic susceptibility. A preliminary analysis revealed \( T_N \) values close to 198 K; this value is similar to those reported in the literature [13].

For the electrical resistance measurements, the NNO nanowires were deposited over a gold-electric contact pattern (see figure 5(a)). The resistance versus temperature, R(T), was performed for several nanowires that were aligned between the gold tracks of the interdigital. Figure 5(b) shows a linear curve with R(T) positive slope indicating a metallic state for these NNO nanowires for temperatures varying from 300 to 200 K. Below this temperature, a sharp increase in R(T) values is observed, which indicate a metal-insulator transition temperature \( T_{MI} \) close to 198 K. This result also revealed the irreversibility between the R(T) cooling and heating curves, which is attributed to the first-order feature of the MI transition in NdNiO₃ bulk samples.
The $-d(lnR)/dt$ versus temperature curves taken during the cooling and heating processes are shown in figure 5(c). We can observe the presence of a broad and low intense peak just below the $T_{MI}$ temperature at $T_{MII} \sim 180$ K (in the heating processes) and $T_{MIC} \sim 100$ K (in the cooling process). This slight decrease of $T_{MI}$ value can be related to the structural distortions observed in the XRD data. In fact, it is reported that structural distortions due to the interface NdNiO$_3$ thin film/substrate can decrease the $T_{MI}$ values [24, 49, 54, 55]. In spite of this structural distortion in these NNO nanowires, it does not significantly affect the transport and magnetic properties of these nanowires, which make them interesting to set up new optoelectronic applications like photovoltaic and flexible sensors.
4. Conclusion

In summary, we used electrospinning, and low oxygen pressure and temperature to synthesized NdNiO$_3$ nanowires successfully. The granular nanowire microstructure of NNO nanostructures was verified through SEM images; these samples present diameters from 50 to 150 nm and grain diameter of ~40 nm. These NNO nanowires crystallize in an orthorhombic-distorted perovskite structure. The structural analysis from Rietveld refinement indicated a slight texture in some Bragg directions and a strained crystallite. The presence of both state valence of the Ni$^{2+}$ and Ni$^{3+}$ ions and the Nd/Ni ratio were verified through XPS data. The results showed a cationic off-stoichiometry with an excess of Ni content. The metal-insulator transition feature of these samples was analyzed through M(T) and R(T) measurement with a $T_{MI}$ of ~198 K. The results indicate that electrospinning is an effective technique for obtaining NdNiO$_3$ nanowires using less severe conditions. Furthermore, in this work the nanostructured shape obtained and the experimental observations reinforce the importance of these rare-earth nickel oxides for the design of new electronic devices.

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Conflicts of Interest

The authors declare that they have no conflicts of interest.

ORCID iDs

M T Escote @ https://orcid.org/0000-0003-1053-560X

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