Synthesis and characterization of NiO and NiO-Sn micro and nanostructures fabricated by a vapor-solid method

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• Introduction

Nickel oxide (NiO) is a wide band gap p-type semiconductor with good electrical, optical and magnetic properties, as well as excellent chemical and thermal stability.

Potential applicability
- Electrochemical capacitors
- Alkaline batteries
- Smart windows

Conclusions

- NiO with rock-salt structure in all cases
- Tin stays in the core of the pellet
- Ordered cavities, which confers a high surface to volume ratio
- New morphologies in this material

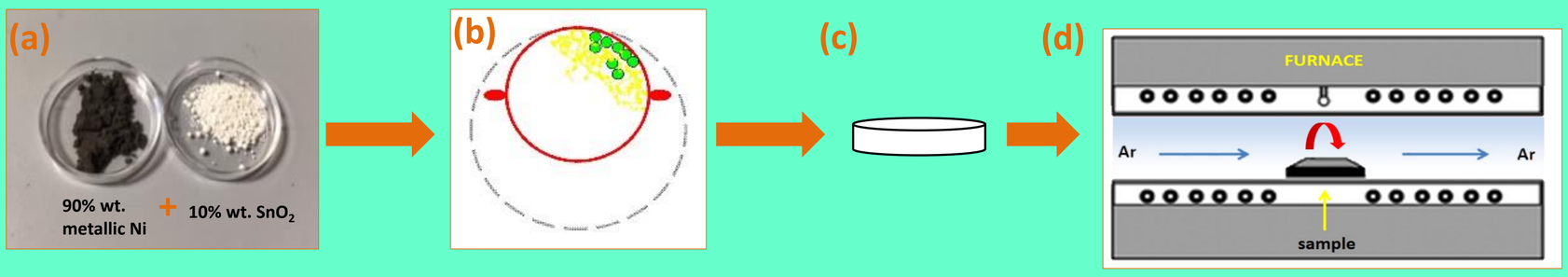
• Aims

NiO is usually synthesized in form of nanoparticles, ceramic or thin films, and less has been done in the fabrication of micro- and nanostructures with variable morphology. So, in this work, a catalyst free evaporation-deposition method, has been employed to fabricate NiO and NiO-Sn micro and nanostructures.

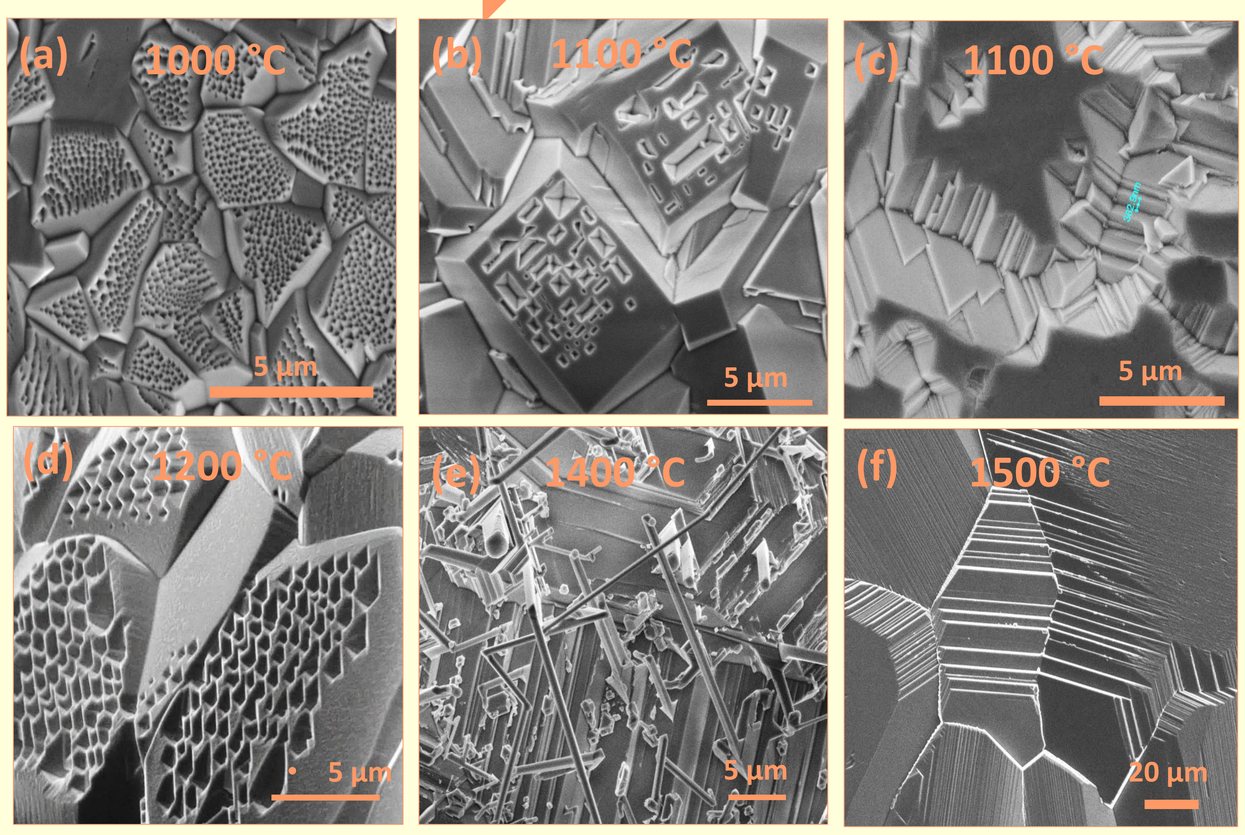
Metallic Ni	800-1500 °C	10-15 hours
Metallic Ni (90% wt.)+ SnO_2 (10% wt.)	1400 °C	10-15 hours

• Vapor-solid method

A vapor-solid method, using a controlled mixture of powders as precursor (a), has been employed in this work. The powders have been milled (b) and pressed into pellets (c). Thermal treatments were carried out under Argon flow at different temperatures (d), leading to the growth of nano- and microstructures on the surface of the treated pellets.

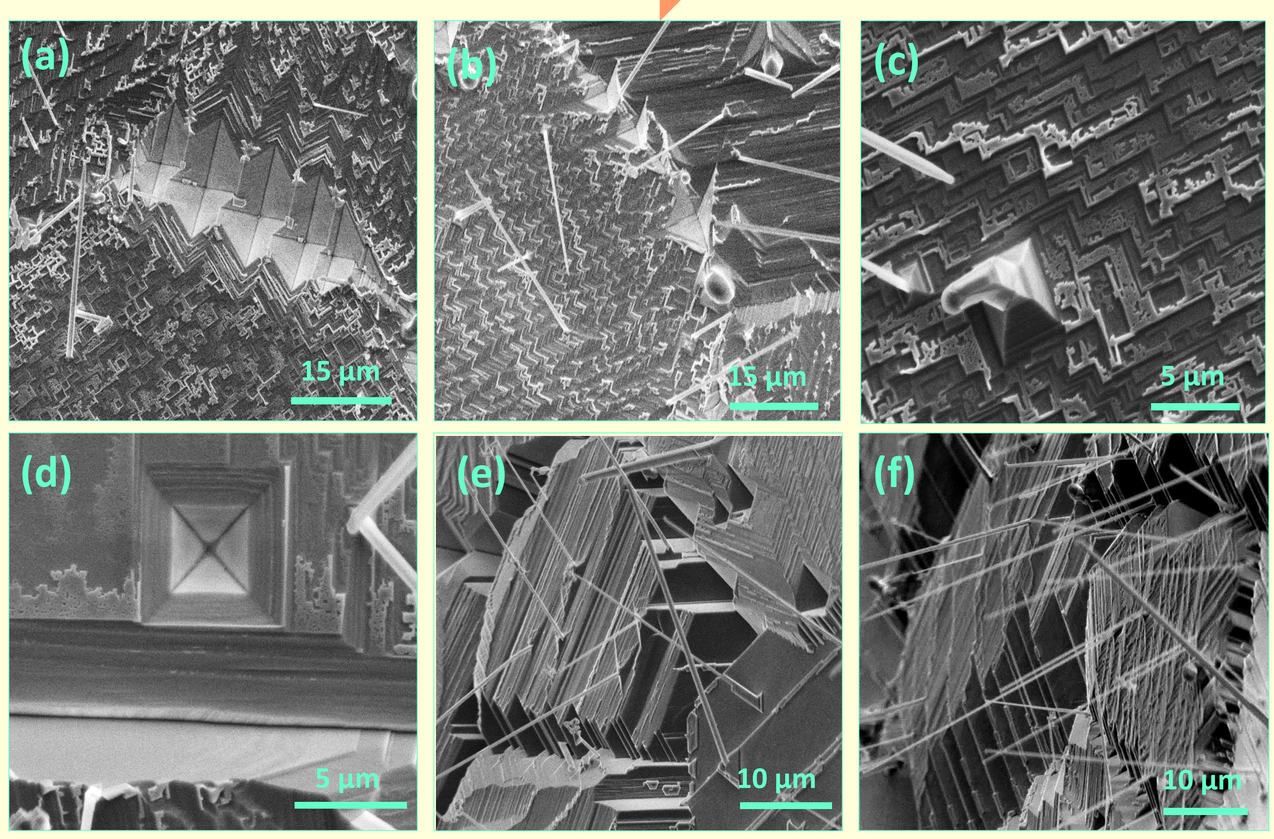


NiO morphology



- Up temperature of 1000°C large microcrystals are grown, exhibiting surfaces with big amounts of ordered hollow cavities with square sections of hundreds of nm (a,b,d).
- Some other crystals surfaces exhibit a layered structure formed by piled terraces of hundreds of nm high (c).
- At 1400°C NiO microwires (e), are also obtained mainly at the border of the pellet.
- Finally, increasing the temperature at 1500°C (f) leads to the reorganization of the surfaces into flat domains or terraced structures without microwires or cavities presence.

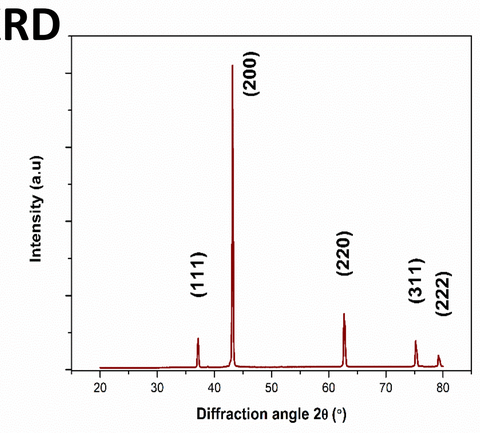
NiO + SnO_2 morphology



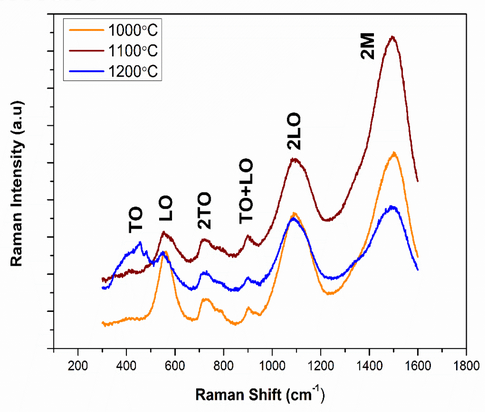
- The presence of SnO_2 assists the formation of microwires with a high aspect ratio (e,f) in a high concentration all over the surface of the pellet, not showing the characteristic morphology of NiO with ordered cavities.
- In addition to the microwires, a new two-dimensional growth, not previously observed appears (a,b,c).

NiO characterization

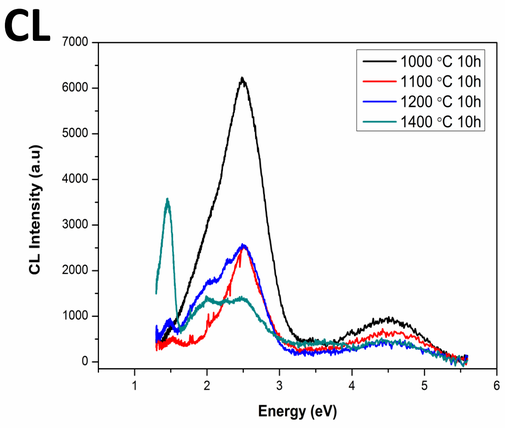
XRD



Raman



CL



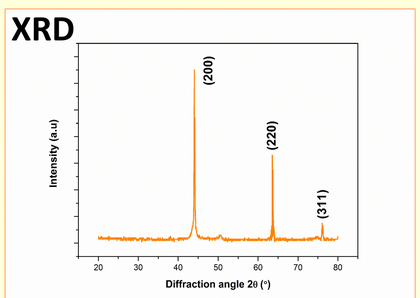
X-ray diffraction and Raman spectroscopy confirm that all samples consist of NiO with cubic rock-salt structure, and no other nickel oxides or rest from the metallic Ni precursors are observed. CL signal shows a main visible emission centered at 2,5 eV.

EDS

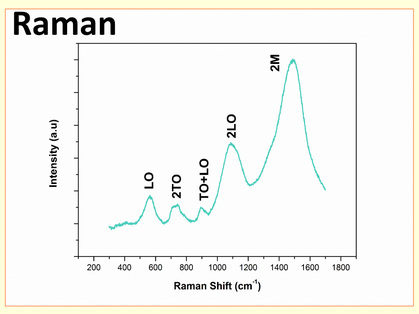
Element	% at. measured
Nickel	52,83
Oxygen	47,16

$NiO+SnO_2$ characterization

XRD

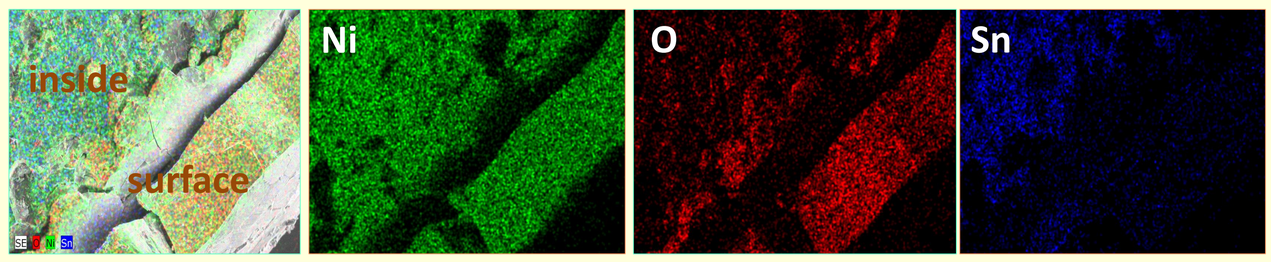


Raman



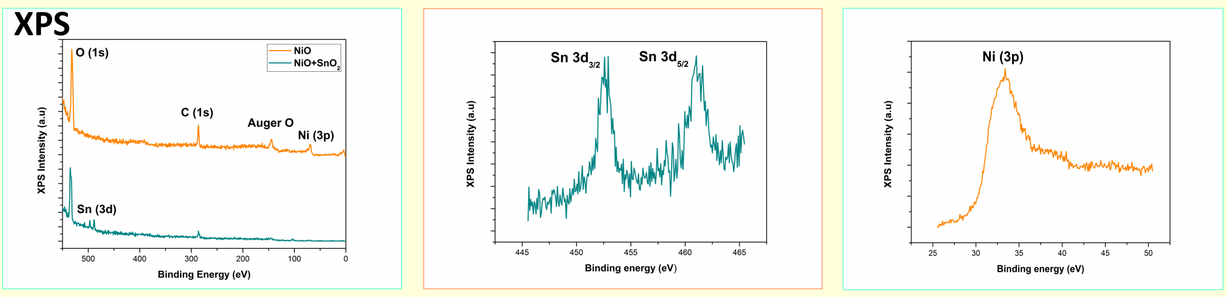
X-ray diffraction and Raman spectroscopy confirm that the sample consists of NiO with cubic rock-salt structure, not showing patterns from SnO_2.

WHERE IS Sn The presence of Sn has been confirmed by means of EDS and XPS



- Most of the tin, stays in the core of the pellet
- The presence of Sn on the surface has been confirmed by XPS

XPS



References

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