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Nickel Oxide Nanostructures Synthesized by thermal Oxidation/Limited Fusion Technique

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Abstract
In this study, nickel oxide (NiO) nanostructures were synthesized using a thermal oxidation/limited fusion technique. These nanostructures showed highly crystalline, phase-pure face-centered cubic bunsenite structure, with most of the growth happening along the (200) plane. A dense network of interlocking nanoparticles and polyhedral clusters, all smaller than 100 nm, were revealed, which gives the material a high surface-to-volume ratio. The composition was nearly ideal, with a nickel-to-oxygen ratio of about 1:0.94. The synthesized samples were highly transparent in the visible spectrum and had a wide indirect bandgap of 3.85 eV, just a little blue-shifted from bulk NiO as a result of quantum size effects. These results show that thermal oxidation/limited fusion is a solid method to produce robust NiO nanostructures that are well-suited for things like gas sensors, supercapacitors, and optoelectronic devices.

Keywords: Nickel oxide; Nanostructures; Thermal oxidation; Limited fusion

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1. Introduction

The thermal oxidation/limited fusion on the metal surface is one of the most effective, reliable and low-cost techniques to produce nanostructures, such as nanoparticles, nanowires, nanoplates, and nanorods [1-3]. This technique mainly depends on the control of diffusion of the metal's atoms and then their reaction with oxygen at elevated temperatures [4]. The mechanism of this technique is fundamentally based on the phenomenon of self-thermal oxidation [5]. When a plate of metal is heated in an oxygen-rich environment, a thin layer of the metal oxide starts to form on the surface. The general chemical reaction to describe this process is

$M^{2+}\ (s) + O_2\ (g) \rightarrow 2MO\ (s)$

The growth of the nanostructures occurs as a result of the mechanical stress originated from the difference in the molar volumes between metal and oxide. This induces the metal ions to diffuse through the crystalline defects or dislocations and grow outwards to reduce the pressure [6-8].

In order to produce metal oxide nanostructures of high quality, all or most parameters included in this technique must be controlled, adjusted or optimized. These parameters include temperature, heating duration, oxygen gas pressure and flow [9-13]. The relatively low temperatures may likely produce nanoparticles while the high temperatures stimulate the growth of nanowires [14]. Long heating durations increase the length and density of the produced nanostructures [15]. The oxygen gas flow rate determines the growth rate and homogeneity of the grown surface layer [16]. This technique can be used under atmospheric pressure or under vacuum as the surrounding pressure may affect the diameter of nanowires [17].

Nickel oxide (NiO) nanostructures synthesized by the thermal oxidation/limited fusion are commonly used in many practical and industrial applications. These nanostructures are used to fabricate hydrogen (H_2) and ammonia (NH_3) sensors due to the drastic surface area that can be available with good chemical and thermal stability [10-12]. They also show excellent charge storage capability to fabricate supercapacitors. As well, they can change their optical transparency when an electric potential is applied on them, which make them very beneficial for electrochromic devices [7,13,16].

In this work, nickel oxide nanostructures were grown on the surface of nickel substrate by the thermal oxidation/limited fusion technique and their characteristics were introduced.

2. Experimental Part

The nickel substrates of 99.9% purity were cleaned in acetone in an ultrasonic bath and then in ethanol in ultrasonic bath to remove the organic impurities and residuals. These substrates were then immersed in diluted hydrochloric acid (HCl) to remove the thin naturally-formed oxide layers. Such layer are unstable and cannot be employed for the aim of this work. The substrates were left in clean desiccator to completely dry. Then, they were placed inside a tube furnace, as shown in Fig. (1), and the temperature was increased from room temperature (27°C) at a rate of 10 °C/min until the final temperature is reached. This heating process may take 68 minutes to reach the final temperature (~700°C). At temperatures up to 1000°C, the process may transform from nanoscale growth to thick bulk layer as the nanoscale features are lost. Therefore, the temperature should be strictly controlled.

As soon this temperature was reached, the oxygen gas was pumped into the tube furnace at a flow rate of 50 sccm until the gas pressure reached 0.8-1 mbar. The tube is sealed for 2 hours to allow the nickel oxide nucleation to form and grow vertically on the surface before the heating is stopped and the chamber is left to cool down to the room temperature again.

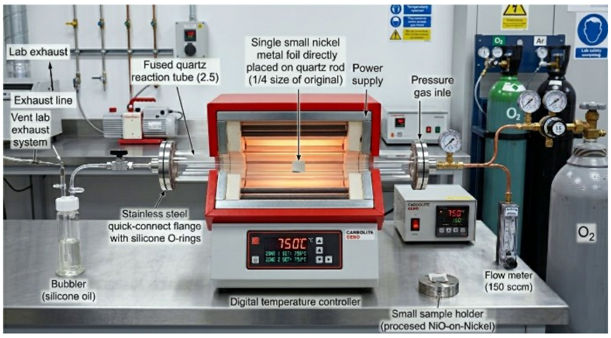


Fig. (1) The thermal oxidation/limited fusion setup to produce NiO nanostructures

3. Results and Discussion

The XRD pattern in Fig. (2) shows that the NiO nanostructures grown on the nickel substrate are clearly crystalline and phase-pure. Distinct diffraction peaks can be seen at around 37.3°, 43.3°, 62.9°, 75.4°, and 79.4°, which correspond to the (111), (200), (220), (311), and (222) planes. These are a perfect match for the face-centered cubic (FCC) rock-salt structure of bunsenite NiO, and they line up nicely with standard JCPDS data. What really stands out is how sharp and intense the (200) peak is, with a narrow full-width at half-maximum (FWHM). That tells us the material is highly crystalline and has grown especially well along that particular direction.

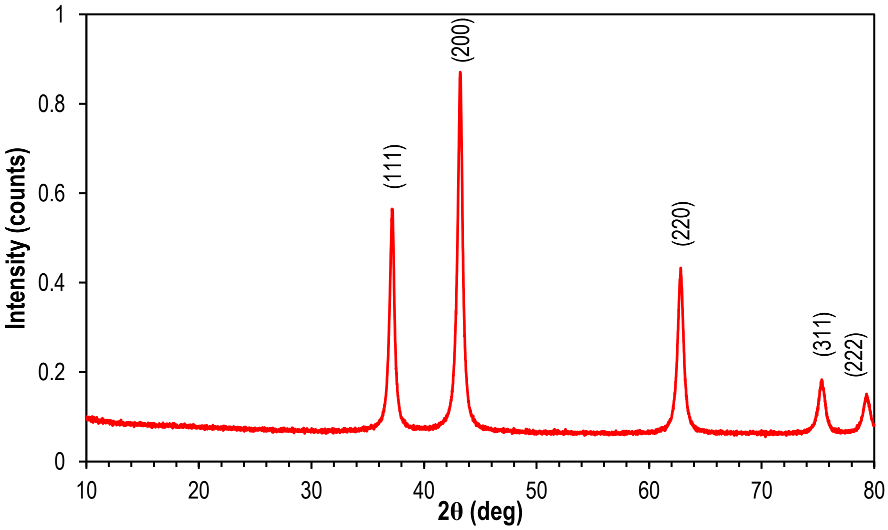


Fig. (2) XRD pattern of the NiO nanostructured sample grown on Ni substrate

Since the synthesis was done by thermally oxidizing a nickel surface, the fact that we don't see much—if any—metallic nickel peaks means a thick, continuous NiO layer has formed. Also, the peaks start to broaden at higher angles, which is a telltale sign of a nanostructured material. The crystallite size is likely in the nanometer range, something you can confirm using the Scherrer's equation. Overall, this pattern confirms that the limited fusion method works well for turning the nickel surface into a well-ordered oxide layer—exactly what you'd want for applications like gas sensing or catalysis.

This FE-SEM image shown in Fig. (3) gives us a really good look at the surface of the NiO nanostructured thin film we made by thermally oxidizing—or "limited fusion"—the nickel substrate. A dense mix of tiny, roughly spherical and polyhedral nanoparticles can be observed that have clumped together into larger, uneven clusters. The particle sizes vary quite a bit. Some are smaller than 100 nm, while others group together into clumps hundreds of nanometers across. That tells us the growth is likely driven by surface diffusion and local melting or fusing. You can even spot "necking" between neighboring particles—where heat started to fuse them together but didn't fully erase the grain boundaries. That's a good thing because it keeps the surface area high relative to volume. The porous, uneven texture comes from the fact that nickel and nickel oxide take up different amounts of space as the oxide grows—something known as the Pilling-Bedworth ratio. All these interlocking nanostructures not only stick strongly to the nickel surface but also create tons of active sites, which is great for applications like gas sensing, supercapacitors, or electrocatalysis. So overall, this thermal oxidation process turned a smooth metal surface into a disordered but continuous network of nanocrystals with a really complex, useful texture.

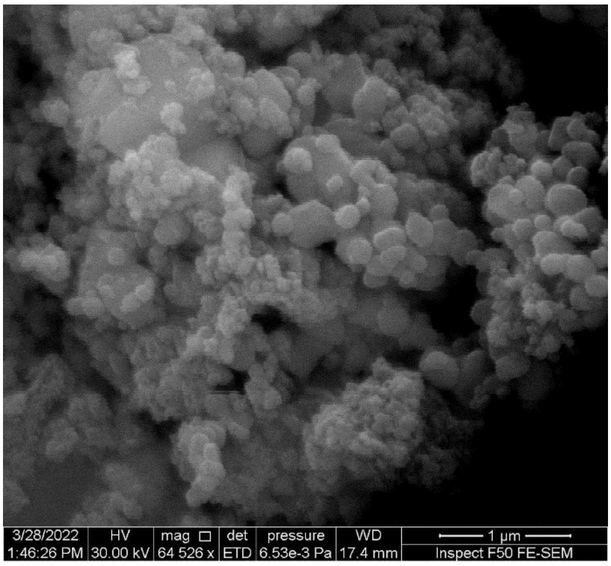
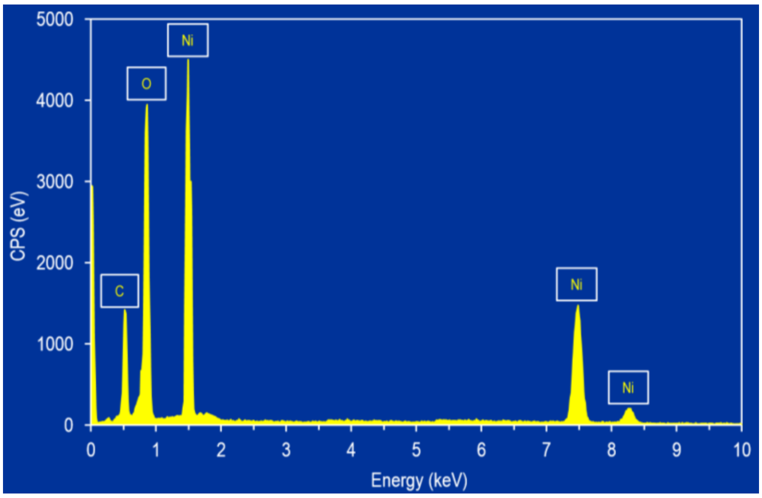


Fig. (3) FE-SEM image of the NiO nanostructured sample grown on Ni substrate

The EDX spectrum in Fig. (4) and the elemental analysis make it clear that the thermal oxidation process successfully turned the nickel substrate into a nanostructured NiO layer. The peaks for carbon (C), oxygen (O), and nickel (Ni) are clearly seen. The nickel signals are especially strong—showing up at around 0.85 keV (Lα), 7.47 keV (Kα), and 8.26 keV (Kβ) — which makes sense since nickel is the base material. But what really matters is that sharp oxygen peak at 0.52 keV, which tells us the metal surface has genuinely converted into an oxide. Looking at the atomic percentages, nickel comes in at 39.5% and oxygen at 37.3%. That gives a Ni-to-O ratio of roughly 1:0.94, which is very close to the ideal 1:1 stoichiometry of NiO. This suggests high-quality crystal growth with very few oxygen vacancies.

A big carbon signal (23.1 at.%) is seen but that's almost certainly from outside contamination — either from exposure to air or from the carbon tape typically used to mount samples for SEM/EDX imaging. It's not actually part of the NiO structure itself. The error margins are tiny, between 0.1% and 0.5%, so we can trust these numbers. Overall, both the element distribution and the weight percentages (72.3% Ni and 18.9% O) show that the limited fusion method did a great job promoting controlled oxidation. The end result is a robust NiO nanostructure that's well-suited for electronics or catalysis applications.



Element	Atomic %	Atomic % Error	Weight %	Weight % Error
C	23.1	0.5	8.8	0.2
O	37.3	0.5	18.9	0.2
Ni	39.5	0.1	72.3	0.3

Fig. (4) EDX spectrum of the NiO nanostructured sample grown on Ni substrate

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Looking at the absorption spectrum in Fig. (5) for the nanostructured NiO sample, it really tells a lot about how the material behaves with light and how well it’s put together. The clearest sign is that sharp drop in absorption right around 300 nm, which is exactly what you'd expect from NiO, a wide-bandgap p-type semiconductor. That steep edge actually hints at good crystallinity and fairly uniform nanostructures; if the slope were more gradual, you'd probably be dealing with lots of defects or disordered phases. In the ultraviolet range (<300 nm), the absorbance stays high and flat, which means strong inter-band transitions are happening—photons have enough energy to kick electrons from the valence band into the conduction band. But then, from about 400 to 800 nm — that's the visible and near-infrared—the absorbance drops to nearly zero. That kind of transparency in visible light is a big deal for things like invisible electronics, electrochromic windows, or hole-transport layers in perovskite solar cells. And the fact that there are no noticeable absorption peaks in the visible also tells us the thermal oxidation process did a good job avoiding mid-gap impurities or nickel-rich phases that would suck up unwanted light. So overall, the spectrum confirms that this NiO nanostructure has a nice wide bandgap and great optical clarity.

The Tauc plot shown in Fig. (6) is really the go-to tool when trying to figure out what kind of electronic transition you're dealing with and what the optical bandgap is for that nanostructured NiO layer. By plotting $(\alpha h\nu)^{1/2}$ against photon energy (hν), the analysis homes in on an indirect allowed transition, which makes sense given the fairly complex electronic structure of p-type NiO. If you take the linear part of the absorption edge and stretch it out until it hits the x-axis, you get a bandgap of about 3.85 eV. That's actually a bit higher than bulk NiO, which usually sits around 3.6 eV, and this slight blue shift is probably due to either quantum size effects — common in nanomaterials — or some strain coming from the thermal oxidation process on the nickel substrate. The steep slope of that linear region points to good crystalline quality and not many states hanging out near the band edges. You might also notice a small tail in the lower energy range (below 3.5 eV), which likely reflects the Urbach energy and suggests there's just a tiny bit of structural disorder or a few localized states within the bandgap. Overall, this clean, well-defined transition threshold tells us that the thermal oxidation method did a solid job producing a wide-bandgap semiconductor, perfect for things like UV optoelectronics and transparent conducting coatings.

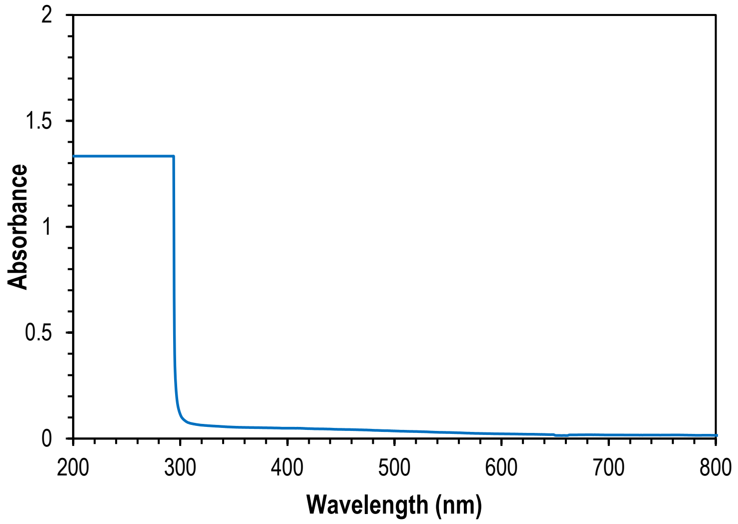

Fig. (5) Absorption spectrum of the NiO nanostructured sample grown on Ni substrate

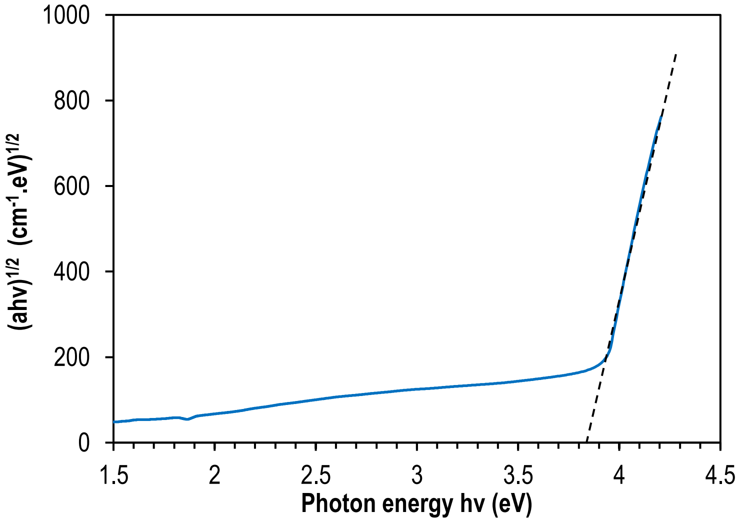

Fig. (6) Relationship between $(\alpha h\nu)^{1/2}$ with photon energy (hv) for the NiO nanostructured sample grown on Ni substrate

4. Conclusion

In concluding remarks, the thermal oxidation technique turned out to be a reliable and efficient method to synthesize functional NiO nanostructures on smooth nickel surface. This method produces high-quality, stoichiometric, and crystalline layers with a porous structure that is perfect for maximizing the number of active sites. With an optical bandgap sitting at 3.85 eV and great transparency in the visible range, these nanostructures have electronic and optical properties suitable for high-performance uses. So overall, this controlled oxidation process offers a practical, scalable route for making NiO-based components for applications like hydrogen sensors, supercapacitors, and transparent electronics.

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