Temperature-Dependent Optical Properties of Nickel Oxide Nanostructures Prepared by Thermal Oxidation/Limited Fusion

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Abstract
This research letter explores how temperature shapes the optical properties of the nanostructured nickel oxide (NiO) layers grown on nickel substrates through thermal oxidation at 300°C, 500°C, and 700°C, showing that higher processing temperatures boost absorbance and cause a clear, stepwise red-shift in the absorption edge. Tauc plot analysis bears this out quantitatively, revealing a narrowing of the indirect band gap from 3.85 eV down to 3.58 eV as the temperature climbs. Such trend is driven by better crystallinity, larger grains, and a relaxation of quantum confinement. Altogether, these findings highlight temperature as a precise tuning knob for NiO electronic structure, opening the door to tailored designs for gas sensors and various optoelectronic devices.

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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 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, the optical properties of nickel oxide nanostructures grown on the surface of nickel substrate by the thermal oxidation/limited fusion were introduced and the effect of temperature of these properties was determined.

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 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.

3. Results and Discussion

Figure (1) shows the UV-visible absorption spectra of nanostructured NiO thin layers grown by thermal oxidation at 300°C, 500°C, and 700°C. This figure explains clearly how their electronic structure and morphology evolve. What stands out immediately is that as the temperature rises, the films absorb more and more light across both the ultraviolet and visible ranges. In the high-energy UV region (200-400nm), the strong absorption comes from fundamental band-to-band electronic transitions, where electrons jump from the O^{2-} (2p) valence band up to the Ni^{2+} (3d) conduction band. With each step up in temperature, the absorption edge shifts systematically to longer wavelengths (red-shift), which points to a narrowing of the optical bandgap. This narrowing usually goes hand in hand with larger grain sizes and better crystallinity, often explained by the Burstein-Moss effect or a relaxation of quantum confinement, as the extra thermal energy encourages NiO nanoparticles to merge and grow.

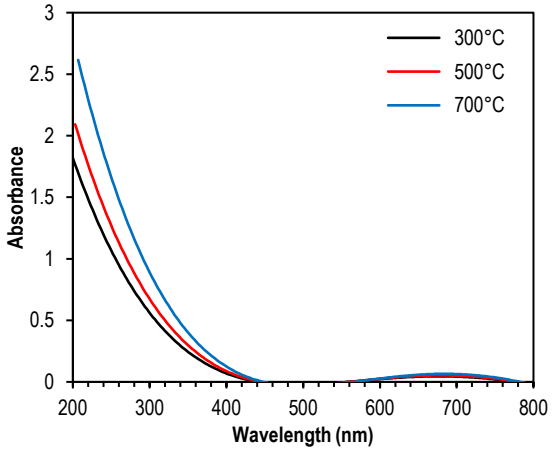

Fig. (1) Absorption spectra of nanostructured NiO layers prepared by thermal oxidation/limited fusion at different temperatures

Beyond the UV, the spectra also show a broad absorption feature centered roughly between 600 and 800 nm, reaching into the near-infrared. This secondary peak is a hallmark of p-type NiO and is typically linked to d-d transitions of Ni^{2+} ions sitting in an octahedral crystal field, or to non-stoichiometric nickel vacancies (Ni^{3+} ions). At 700°C, this visible peak becomes noticeably stronger, suggesting a greater population of these defect states or simply a thicker, more continuous oxide film formed through controlled fusion. Overall, the increase in optical density across the board highlights just how sensitive NiO is to oxidation temperature. By adjusting this single parameter, you can effectively tune charge carrier density and light-harvesting ability—both of which are critical for applications like electrochromic devices, gas sensors, and p-type transparent conducting electrodes. In short, the results confirm that turning up the processing temperature helps build a more robust nanostructured network with markedly better photon absorption.

The energy band gap (E_g) of the nanostructured NiO layers is worked out by applying the Tauc relation to the absorption data. Plotting $(\alpha h\nu)^{1/2}$ against photon energy ($h\nu$) gives information about how nanostructured NiO behaves as an indirect band gap semiconductor, and the gap itself is found by simply extending the straight-line portion of the absorption edge down to where $(\alpha h\nu)^{1/2} = 0$. What's immediately clear from the figure is a systematic shift in that intercept as the preparation temperature changes: the NiO layer made at the lowest temperature (the black curve) gives the widest band gap of roughly 3.85 eV, and as the oxidation temperature is raised to the intermediate level (red curve) and then the highest (blue curve), the band gap shrinks to about 3.70 and 3.58 eV, respectively. This steady narrowing with increasing temperature stems mainly from improving crystallinity and growing grain sizes, basically, as the extra thermal energy encourages the nanostructures to fuse together, the quantum confinement effects that normally widen the gap in very small particles start to relax.

On top of that, higher temperatures likely introduce a greater density of localized states near the band edges or subtle shifts in stoichiometry, particularly Ni^{2+} vacancies, which create tail states inside the forbidden gap and effectively lower the energy needed for electronic transitions. Having this level of control over the optical band gap through precise temperature selection is crucial when tailoring NiO layers for specific optoelectronic uses like selective UV photodetectors or hole-transport layers in solar cells, and the Tauc plots here give solid quantitative proof that thermal oxidation is a highly effective route for tuning the electronic properties of nanostructured NiO.

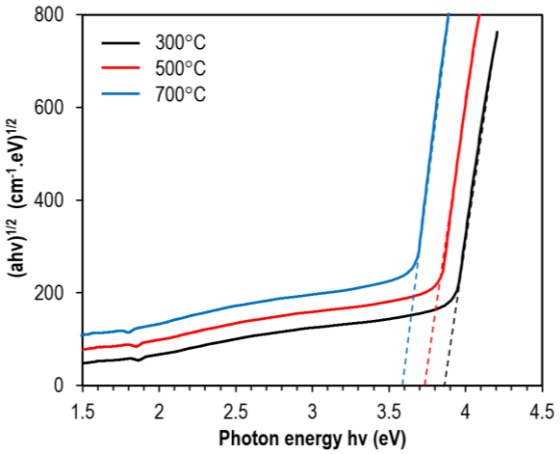

Fig. (2) Energy band gap of nanostructured NiO layers prepared by thermal oxidation/limited fusion at different temperatures

4. Conclusion

This work establishes that the optical properties of NiO nanostructures are governed first and foremost by the thermal oxidation temperature, with the jump from 300°C to 700°C delivering a major boost in photon absorption along with a systematic shrinking of the band gap, thanks to improved crystallinity and subtle shifts in stoichiometry. The results lay down a quantitative roadmap for engineering high-quality NiO layers whose electronic properties can be dialed in on demand, paving the way for optimized performance in next-generation electrochromic devices, supercapacitors, and selective UV photodetectors.

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