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Recovery of monoclinic phase of VO_2 nanoparticles from oxidized samples: Assessment of reusability and optimization of re-annealing conditions

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ABSTRACT

Vanadium dioxide (VO_2) nanoparticles in the monoclinic (M) phase exhibit a reversible metal–insulator transition (MIT) at ~68 °C, underpinning thermochromic applications such as smart windows and thermal sensors. However, prolonged atmospheric exposure causes progressive surface oxidation that converts V^{4+} to V^{5+}, leading to phase degradation and loss of thermochromic function. This study demonstrates that oxidized VO_2 nanopowders can be fully recovered by thermal re-annealing in an inert argon atmosphere. Starting from VO_2(M) nanoparticles synthesized via a hydrothermal route, controlled oxidation under ambient conditions produced a degraded sample rich in V_2O_5. Systematic re-annealing at 450–600 °C for 2–3 h was investigated to identify the optimal recovery conditions. XRD, Raman spectroscopy, and UV-Vis-NIR transmittance measurements confirm complete phase restoration and recovery of thermochromic switching performance equivalent to that of the as-synthesized material, establishing a practical strategy for reusing degraded VO_2 nanopowder stocks.

Keywords: *Vanadium dioxide, smart window, transition material, thermochromic recovery.*

1. INTRODUCTION

Vanadium dioxide (VO_2) is a polymorphic transition-metal oxide capable of adopting several crystal structures-monoclinic (M), VO_2(A), VO_2(B), and VO_2(D) - despite sharing the same chemical composition [1] - [3]. The differences among these polymorphs arise from distinct VO_6 octahedral arrangements within the crystal lattice, giving each phase unique structural and electronic properties (Figure1).

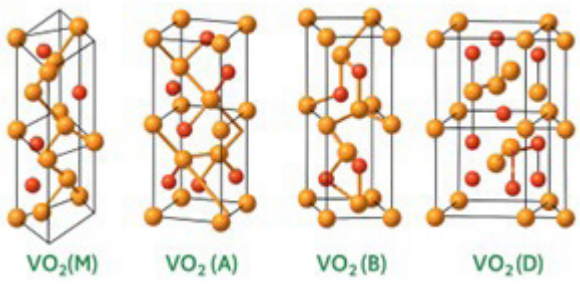

Figure 1. Crystal lattice structures of the four VO_2 polymorphs: VO_2(M), VO_2(A), VO_2(B), and VO_2(D). Orange: V atoms; red: O atoms.

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Among these, monoclinic VO_2(M) is thermodynamically stable at room temperature and is of exceptional importance owing to its reversible semiconductor-metal phase transition near 340 K (~68 °C), first reported by Morin [4]. In the monoclinic state, vanadium atoms form paired dimers along the c-axis, opening a band gap and yielding semiconducting behavior. Upon heating, dimerization disappears, the density of states near the Fermi level increases sharply, and metallic conductivity is recovered [4] - [5]. This structural reorganization also reverses the optical response: below the transition temperature, VO_2 transmits near-infrared (NIR) radiation; above it, the metallic phase strongly reflects NIR light [6] - [7]. This reversible electro-optical switching underpins thermochromic smart windows, thermal sensors, and optical limiters.

A variety of synthesis strategies have been developed for VO_2, including physical vapor deposition, magnetron sputtering, sol–gel, and hydrothermal methods [8] - [9]. Hydrothermal synthesis is particularly attractive because it permits direct control of crystal phase, particle size, and morphology through adjustment of temperature, pH, and redox atmosphere [10] - [11]. Typically, a V^{5+} precursor such as V_2O_5 is reduced to V^{4+} under hydrothermal conditions, yielding metastable VO_2(B) or VO_2(A) that is subsequently annealed to phase-pure VO_2(M) in an inert atmosphere [10] - [12].

Despite extensive research, the storage stability of synthesized VO_2 nanopowders remains a practical challenge. Even in nominally sealed environments, prolonged ambient exposure causes gradual surface oxidation of V^{4+} to V^{5+}, forming V_2O_5-rich surface layers that broaden the MIT, reduce thermochromic contrast, and impair switching performance [1] - [13]. The nanoscale morphology of these particles accelerates this degradation relative to bulk material owing to high surface-to-volume ratios. Recovery of degraded VO_2 nanopowders by thermal re-annealing in inert or reducing atmospheres has been proposed [12] - [14], yet systematic optimization of re-annealing conditions for nanopowder specimens remains sparse in the literature.

In the present work, we synthesized VO_2(M) nanoparticles by a hydrothermal route, intentionally oxidized them under ambient conditions, and conducted systematic re-annealing in argon across temperatures of 450–600 °C and durations of 2–3 h. XRD, Raman spectroscopy, and UV-Vis-NIR transmittance measurements are used to track phase evolution and verify restoration of thermochromic function. Our results identify 500 °C for 2 h as the optimal condition, demonstrating a straightforward, cost-effective protocol for reusing degraded VO_2 nanoparticle batches.

2. EXPERIMENTAL SECTION

2.1. Materials

Vanadium pentoxide (V_2O_5, ≥ 98%), concentrated sulfuric acid (H_2SO_4, 98%), hydrazine hydrate ($N_2H_4 \cdot H_2O$, 50–60%), sodium hydroxide (NaOH, ≥ 95%), polyvinylpyrrolidone (PVP, MW = 40,000 g/mol), and absolute ethanol (99.6%) were used as received. Deionized (DI) water was used throughout.

2.2. Synthesis of VO_2(M) nanoparticles

VO_2(M) nanoparticles were prepared via a four-step hydrothermal procedure illustrated in Figure2.

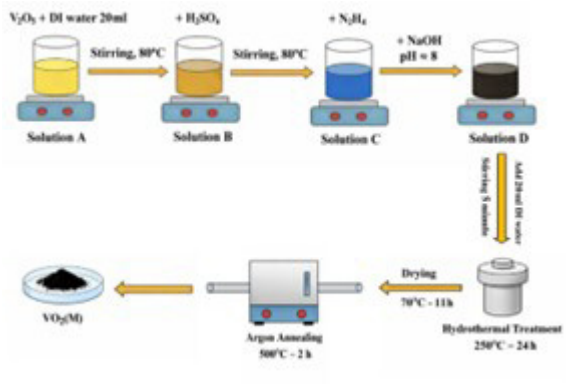

Figure 2. Schematic of the hydrothermal synthesis route for VO_2(M) nanoparticles, from V_2O_5 precursor dissolution to argon annealing.

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Step 1: Precursor preparation: 0.9 g of V_2O_5 was dispersed in 20 mL DI water under stirring at 60 °C (Solution A, yellow suspension). Then 1.5 mL of H_2SO_4 was added dropwise at 80 °C to give a deep-yellow solution (Solution B).

Step 2: Reduction and pH adjustment: 0.5 mL of N_2H_4 was introduced dropwise, raising the temperature to 90–100 °C and yielding a blue solution (Solution C, pH ≈ 0.8-1). NaOH (2.3 g) was then added to adjust pH to ~8, producing a dark-brown suspension (Solution D). The color evolution is shown in Figure 3.

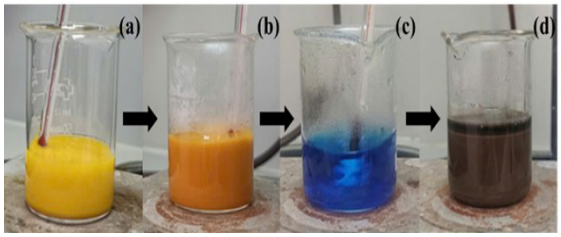

Figure 3. Optical photographs of the precursor at each synthesis step: (a) yellow V_2O_5 suspension; (b) deep-yellow Solution B; (c) blue Solution C after N_2H_4 reduction; (d) dark-brown Solution D after pH adjustment.

Step 3: Hydrothermal treatment: the precursor was transferred to a 25 mL Teflon-lined stainless-steel autoclave and heated at 250 °C for 24 h (5 °C min^{-1}).

Step 4: Post-treatment and annealing: the black precipitate was washed twice with DI water and once with ethanol, dried at 70 °C for 11 h, then annealed at 500 °C for 2 h in flowing Ar (10 °C min^{-1}) to obtain phase-pure VO_2(M).

2.3. Controlled oxidation

Synthesized VO_2(M) powders were stored in an open beaker under ambient laboratory conditions (~25 °C, relative humidity 60–70%) for a controlled period (1 week) to simulate real-world aging. Color changes were recorded photographically.

2.4. Recovery by re-annealing

Oxidized powders were re-annealed in a tube furnace under continuous Ar flow. Stage 1 (temperature optimization): re-annealing at 450, 500, 550, and 600 °C for 2 h. Stage 2 (duration optimization): re-annealing at 500 °C for 2 h and 3 h. All heating ramps were 10 °C min^{-1}.

2.5. Characterization

The crystal structure of the samples was determined by X-ray diffraction using a D2 Phaser diffractometer (Bruker, Rheinstetten, Germany) equipped with a Cu Kα radiation source (λ = 1.54 Å). Raman spectra were acquired using a Horiba XPlora Plus spectrometer (Horiba, Villeneuve d'Ascq, France) with a 532 nm laser excitation. The surface morphology was examined using a JSM-IT200 scanning electron microscope (JEOL, Japan). Thermochromic performance was evaluated by UV-Vis-NIR transmittance spectroscopy over a range of 300 – 2000 nm using a JASCO V-770 spectrophotometer (JASCO, Hachioji, Japan) equipped with a temperature-controlled stage during heating and cooling cycles between 30 and 100 °C.

3. RESULTS AND DISCUSSION

3.1. As-synthesized VO_2(M) nanoparticles

The hydrothermal synthesis and Ar annealing steps produced a jet-black powder (Figure 4, right), in stark visual contrast to the yellow V_2O_5 precursor (Figure 4, left). The color change reflects successful $V^{5+} \rightarrow V^{4+}$ reduction and VO_2(M) phase formation.

Figure 4. Optical photographs of the yellow V_2O_5 precursor powder (left) and the as-synthesized black VO_2(M) nanoparticle powder (right).

The UV-Vis-NIR transmittance spectra of original sample (Figure 5) show a pronounced divergence between the 30 °C (semiconducting) and 100 °C (metallic) curves in the NIR region (800 - 2000 nm), confirming active

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thermochromic switching through the MIT at ~68 °C [15]. This large NIR transmittance contrast is the key functional signature of high-quality VO_2(M).

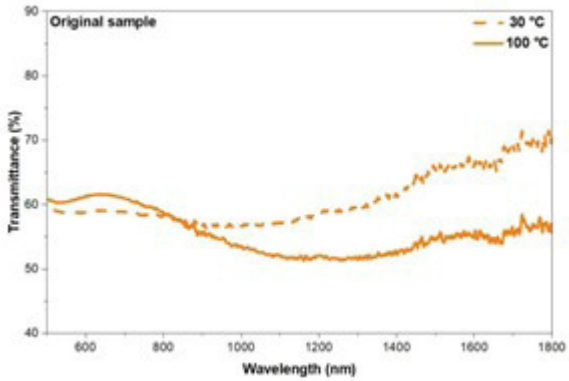

Figure 5. UV-Vis-NIR transmittance spectra of as-synthesized VO_2(M) film (Original sample) at 30 °C (dashed line) and 100 °C (solid line), showing clear thermochromic NIR switching.

3.2. Oxidation behavior under ambient conditions

When VO_2(M) powder was stored in an open beaker under ambient air (Figure 6), a progressive color transformation was observed over the course of days to weeks: black → bluish-black → moss green → dark moss green (Figure 7a). This sequence mirrors the gradual oxidation of V^{4+} to V^{5+} and accumulation of V_2O_5-like surface species. The SEM image in Figure 7b reveals that re-annealing at 500°C for 3 hours resulted in a mixed morphology, consisting predominantly of nanoparticles with a partial formation of nanorods.

Figure 6. Storage experiment: empty reference beaker (left) and VO_2 powder sample after ~one week of ambient air exposure (right).

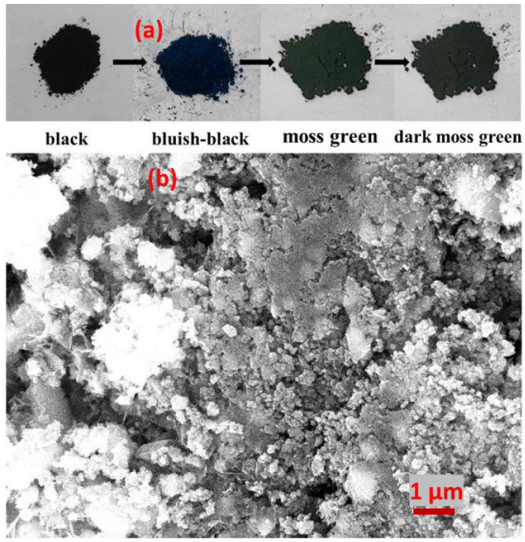

Figure 7. Time-dependent color evolution of VO_2 powder during ambient oxidation: black → bluish-black → moss green → dark moss green (a) and SEM image of the recovery sample annealed at 500°C in 3h.

XRD analysis of the oxidized sample (Figure 8) reveals emergence of V_2O_5 reflections alongside diminished VO_2(M) peaks, confirming phase transformation rather than mere surface contamination [16]. The original sample (orange) displays only sharp VO_2(M) reflections, while the oxidized sample (cyan) shows clear additional peaks at positions characteristic of V_2O_5 at 2 theta poisitions of 26.2°, 31°, 32.4°, 41°, 52° and 62°. Nevertheless, the persistence of lower-intensity peaks at 27.9°, 37°, 55°, 65° and 70° indicates a residual VO_2(M) component. Furthermore, the diffractogram suggests the secondary presence of the VO_2(B) phase as an impurity.

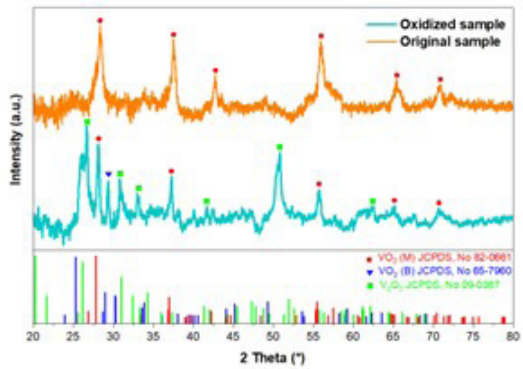

Figure 8. XRD patterns of the original VO_2(M) (orange) and oxidized sample (cyan) compared with reference stick patterns for VO_2(B) (blue), V_2O_5 (green) and VO_2(M) (red). Dashed lines mark key VO_2(M) peak positions.

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The optical consequences are decisive: Figure 9 shows UV-Vis-NIR spectra of the oxidized sample at 30 and 100 °C. The near-identical curves confirm complete abolition of thermochromic switching [17]. This finding is consistent with reports that even partial V^{5+} contamination can substantially suppress MIT sharpness, particularly in nanoparticle systems with high surface-to-volume ratios.

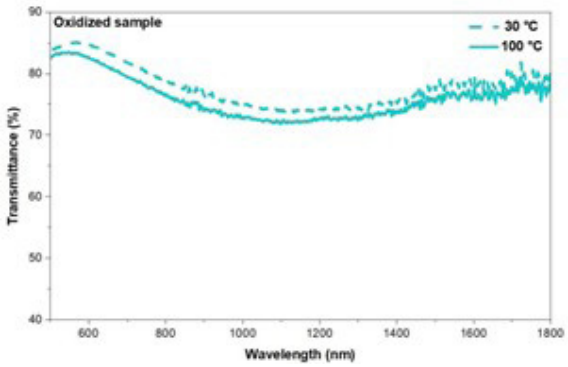

Figure 9. UV-Vis-NIR transmittance spectra of the oxidized sample at 30 °C (dashed line) and 100 °C (solid line). Near-identical curves confirm complete suppression of the thermochromic MIT.

Raman spectroscopy further corroborates the phase loss (Figure 10). The oxidized sample displays a featureless, monotonically rising background with no discernible VO_2(M) phonon modes, consistent with a V_2O_5-dominated surface that lacks the characteristic V-V dimerization modes of the monoclinic lattice.

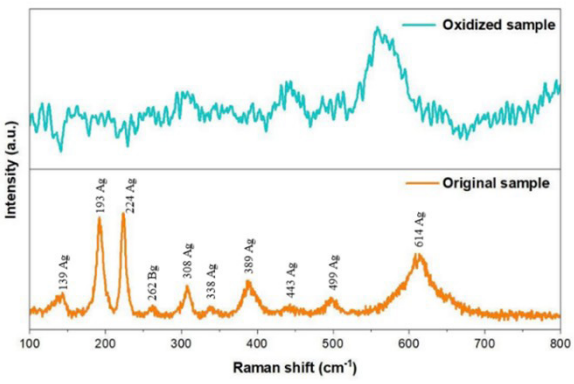

Figure 10. Raman spectrum of the oxidized VO_2 sample versus synthesized one. The featureless background and absence of VO_2(M) modes confirm complete disruption of the monoclinic lattice.

3.3. Recovery of VO_2(M) by thermal re-annealing

Re-annealing in Ar establishes a low oxygen partial pressure environment that suppresses re-oxidation of intermediate phases, enabling the thermally driven, stepwise lattice oxygen release sequence $V_2O_5 \rightarrow V_3O_5 \rightarrow V_4O_9 \rightarrow V_6O_{13} \rightarrow VO_2$ to proceed without introducing unwanted dopants or secondary chemical reactions [18]. The phase evolution was tracked visually (Figure 11) and by XRD (Figure 12).

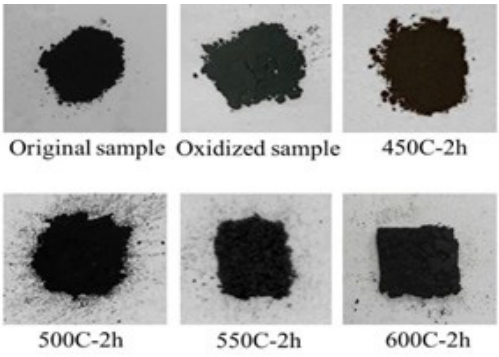

Figure 11. Optical photographs comparing original VO_2(M), oxidized sample, and re-annealed samples at 450, 500, 550, and 600 °C (2 h each). The 500 – 600 °C samples recover the characteristic black color.

In Figure 12, re-annealing at 450 °C (black trace) yields broad, poorly resolved peaks with poor agreement to the VO_2(M) reference - indicating incomplete reduction. At 500 °C (pink trace), a clean pattern matching the VO_2(M) reference (red sticks) is obtained with no V_2O_5 or VO_2(B) impurities. At 550 °C and 600 °C, additional peaks inconsistent with VO_2(M) appear, attributable to minor VO_2(B) or over-reduced secondary phases. [18]

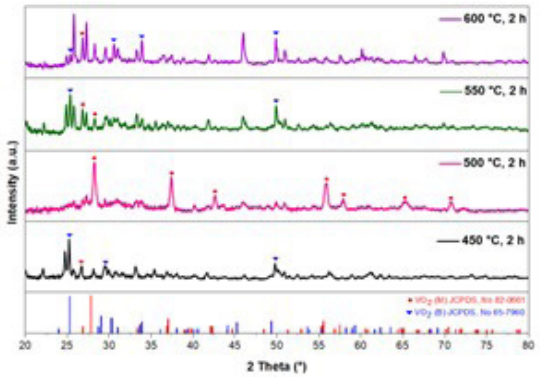

Figure 12. XRD patterns of samples re-annealed at 450 °C (black), 500 °C (pink), 550 °C (dark green), and 600 °C (purple), with VO_2(B) (blue) and VO_2(M) (red) reference patterns.

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Raman spectroscopy of the 500 °C for 2 h sample, Figure 13 confirms the reappearance of sharp VO_2(M) phonon modes at ~224 and ~614 cm^{-1}, with additional features at ~139, ~193, ~262, ~308, ~338, ~389, ~443, and ~499 cm^{-1}-all characteristic of the monoclinic lattice and V-V dimerization. No V_2O_5 Raman bands are visible, providing unambiguous spectroscopic confirmation of complete phase recovery.

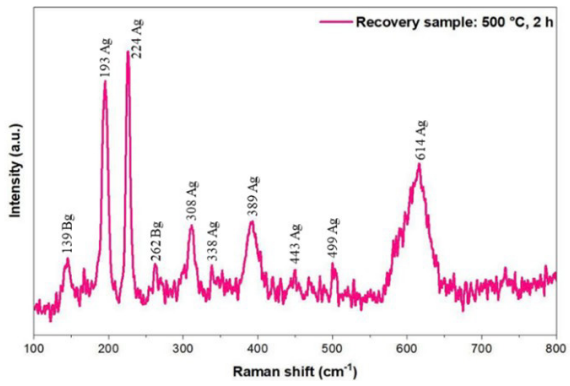

Figure 13. Raman spectrum of recovered VO_2(M) (500 °C for 2 h). Characteristic VO_2(M) modes at ~224 and ~614 cm^{-1} are clearly restored.

3.4. Optimization of re-annealing duration

Comparing re-annealing at 500 °C for 2 h versus 3 h reveals a critical narrow processing window (Figure 14). The 2 h sample (left, pink) yields a phase-pure VO_2(M) XRD pattern with no extraneous reflections. Extending the duration to 3 h (right, purple) introduces VO_2(B) peaks (highlighted by dashed lines), indicating the onset of over-reduction or phase instability under prolonged thermal treatment. Therefore, 2 h is the optimal duration-sufficient for complete reduction yet short enough to avoid unwanted phase evolution.

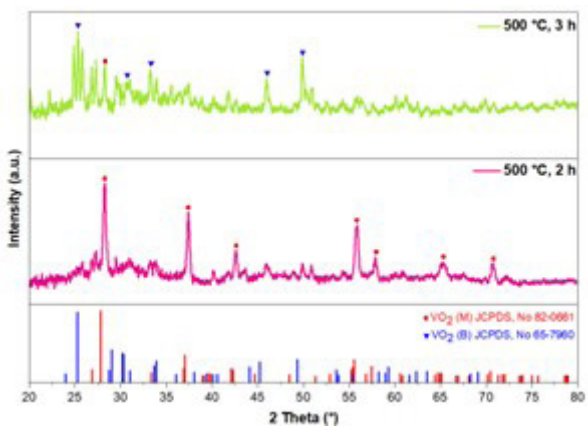

Figure 14. XRD comparison of re-annealing at 500 °C for 2 h (pink, left) and 3 h (lime green, right). The 3 h sample shows VO_2(B) impurity peaks (dashed lines), confirming 2 h as optimal.

3.5. Verification of thermochromic performance restoration

Figure 15 presents UV-Vis-NIR data for the optimally recovered sample (500 °C for 2 h). The spectra show a pronounced divergence between the 30 and 100 °C curves in the NIR region, fully restoring the thermochromic switching behavior of the original material. The recovered NIR contrast and switching temperature (~68°C) are quantitatively comparable to those of the original as-synthesized sample, with transmittance differences at 1800 nm of approximately 15% and 12.5% (~83.33%) for the original and re-annealed samples, respectively.

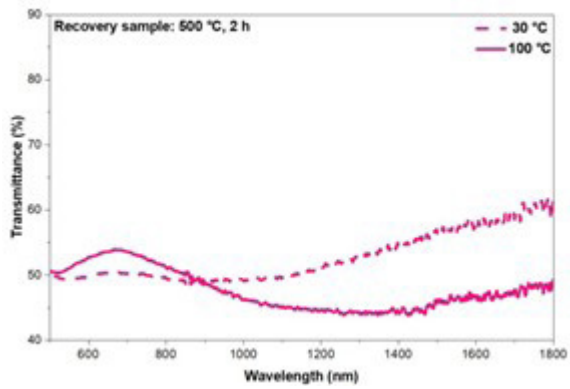

Figure 15. Characterization of recovered VO_2(M) (500 °C for 2 h): UV-Vis-NIR transmittance spectra at 30 °C (dashed line) and 100 °C (solid line) confirming full thermochromic recovery.

The complete restoration of the MIT - a property exquisitely sensitive to stoichiometry, lattice order, and surface chemistry - confirms that re-annealing at the optimal condition drives thorough phase recovery, extending from the nanoparticle surface into the bulk. This result is consistent with previous reports on thin-film VO_2 systems where post-synthesis reduction treatments restored MIT sharpness and thermochromic contrast [2], [14], [19], [20]. The practical significance is considerable:

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VO_2 nanoparticle synthesis requires multi-step procedures, specialized equipment, and expensive precursors. Rejuvenating oxidized stocks via a single 2-hour Ar annealing step - identical to the original synthesis annealing - eliminates the need to discard and resynthesize degraded material, reducing cost, waste, and synthesis time.

4. CONCLUSION

This study has systematically demonstrated the feasibility of recovering phase-pure monoclinic VO_2(M) nanoparticles from ambient-oxidized nanopowders through thermal re-annealing in argon. Progressive atmospheric oxidation converted V^{4+} to V^{5+}, yielding V_2O_5 - rich phases that completely suppressed the MIT, as evidenced by XRD, Raman spectroscopy, and UV-Vis-NIR transmittance. Systematic re-annealing experiments across 450 – 600 °C and 2-3 h identified 500 °C for 2 h in Ar as the optimal recovery condition. Under these conditions, XRD and Raman spectroscopy confirmed complete VO_2(M) phase restoration, and UV-Vis-NIR measurements verified quantitative recovery of thermochromic performance equivalent to the original as-synthesized nanoparticles. These results provide a practical, scalable protocol for extending the operational lifetime of VO_2 nanopowder batches, with direct implications for reducing material waste and cost in thermochromic device fabrication.

DECLARATION OF CONFLICTS OF INTEREST

The authors declare that this research has no conflicts of interest.

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TẠP CHÍ KHOA HỌC
TRƯỜNG ĐẠI HỌC QUY NHƠN

Phục hồi pha tinh thể đơn tà của hạt nano VO_2 từ mẫu bị oxy hóa: đánh giá khả năng tái sử dụng và tối ưu điều kiện nung phục hồi

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TÓM TẮT

Các hạt nano vanadium dioxide (VO_2) pha đơn tà (M) có khả năng chuyển đổi pha giữa kim loại - bán dẫn (MIT) thuận nghịch tại ~68 °C đã mở ra các ứng dụng nhiệt sắc như cửa sổ thông minh và cảm biến nhiệt. Tuy nhiên, việc phơi nhiễm trong môi trường khí quyển gây ra hiện tượng oxy hóa bề mặt, chuyển đổi V^{4+} thành V^{5+}, dẫn đến suy giảm chất lượng pha tinh thể hoặc thay đổi thành phần pha làm mất chức năng nhiệt sắc. Trong nghiên cứu này, các hạt nano VO_2 bị oxy hóa cho thấy khả năng được phục hồi hoàn toàn bằng cách nung lại trong môi trường khí trơ. Từ các hạt nano VO_2(M) được tổng hợp bằng phương pháp thủy nhiệt, quá trình oxy hóa được thực hiện trong điều kiện môi trường không khí tạo ra mẫu có chất lượng tinh thể bị suy giảm và chứa thành phần V_2O_5. Để khảo sát khả năng hồi phục pha tinh thể VO_2(M), quá trình nung lại được khảo sát một cách hệ thống từ 450 - 600 °C trong 2-3 giờ, kết quả cho thấy nung ở điều kiện tại nhiệt độ 500 °C trong 2 giờ là điều kiện phục hồi tối ưu. Phép phân tích XRD, phổ Raman và đo phổ truyền qua UV- Vis-NIR cho thấy VO_2(M) khôi phục hoàn toàn pha và hiệu suất nhiệt sắc gần tương đương với vật liệu ban đầu.

Từ khóa: *Vanadium dioxide*, *cửa sổ thông minh, vật liệu chuyển pha, hồi phục tính chất nhiệt sắc.*

**Tác giả liên hệ chính.*
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