

Article

Enhanced Thermal Stability of Ni@TiO₂ Core-Shell Nanoparticles

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Abstract

Ni@TiO₂ core-shell nanoparticles were synthesized by magnetron sputtering and their structure verified by HRTEM and EDS analysis. The thermal stability of these particles was investigated using in situ TEM annealing and compared with that of pure Ni nanoparticles. While pure Ni particles sinter at 450 °C and exhibit significant growth at 800 °C, Ni@TiO₂ nanoparticles remain stable up to 700 °C, with the sintering onset between 700 and 800 °C. A simple thermal-mismatch model was applied to explain the stabilizing effect of the TiO₂ shell, demonstrating that differences in thermal expansion between Ni and TiO₂ generate interface stresses sufficient to crack the shell after the amorphous–rutile transformation. The TiO₂ coating effectively delays Ni coalescence by 250 °C relative to bare Ni, highlighting its role as a protective shell against high-temperature sintering.

Keywords: magnetron sputtering; in situ transmission electron microscopy; high-temperature annealing; rutile transformation; nanoparticle sintering

1. Introduction

Nanotechnology has significantly broadened its capabilities in numerous fields over the last decade by creating functional systems at the nanoscale, primarily in the form of nanoparticles [1]. Notably, nickel nanoparticles (NiNPs) have attracted significant interest due to their distinctive magnetic, chemical, and physical properties, including high reactivity, operational simplicity, biocompatibility, bacterial resistance, cost-effectiveness, abundance, anti-inflammatory effects, and environmental compatibility [2]. These properties offer a potential for their applications in a variety of fields, including catalysis [3], production of batteries [4], applications in optical switches [5], direct immobilization of biomolecules via magnetic properties [6], and sintering additives in coatings, plastics, and fibers [7,8].

Nickel nanoparticles are prone to agglomeration due to their high surface energy [9]. This agglomeration can reduce effectiveness in applications where high surface area is critical, such as catalysis and sensor technologies. NiNPs sintering is particularly problematic at elevated temperatures [10,11], which are often required in industrial processes. The particles lose their distinct nanoparticle properties, effectively diminishing their catalytic activity, primarily due to changes in their surface area [12] and magnetic properties [13] as their magnetic domain structures change.

A variety of strategies have been developed to address these issues, including surface modification [14,15] and the incorporation of nanoparticles into or onto stable matrices [16]. For instance, carbon-coated Ni nanoparticles remain ferromagnetic up to 127 °C [17]. Additionally, Ni@SiO₂ nanoparticles can withstand high temperatures up to 700 °C [18].



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Alumina-coated NiO nanopowders also show a significant enhancement in catalytic activity at elevated temperatures ranging from 700 to 800 °C [19], compared to their uncoated counterparts.

Ni–TiO₂ nano-heterostructures have been widely investigated for applications such as photocatalysis, hydrogen sensing, and lithium-ion batteries [20–26]. Reported systems include Ni nanoparticles dispersed on TiO₂ thin films and nanotube structures [20,21], as well as composite Ni/TiO₂ nanoparticles [22–25], in both of which TEM characterizations reveal that TiO₂ surrounds or decorates Ni particles without forming a fully encapsulating shell. More recently, core–shell Ni@TiO₂ nanoarchitectures have been developed [26] using a hydrolytic coating method. However, structural characterizations show porous TiO₂ shells.

While suitable for electrochemical applications, these porous shells provide limited resistance to thermally activated sintering. In contrast, fully encapsulating TiO₂ shells effectively suppresses Ni coalescence, and in situ TEM heating experiments allow direct visualization of stress evolution, shell rupture, and subsequent diffusion-driven collapse of the core–shell structure. In this study, the impact of an amorphous titanium dioxide (TiO₂) shell on the sintering behavior of nickel (Ni) nanoparticles is explored. Magnetron sputtering was used to fabricate Ni@TiO₂ core–shell nanoparticles, employing two planar magnetron sources for deposition. In situ transmission electron microscopy (TEM) was used to directly observe and analyze sintering behavior. This observation enabled a comparative analysis of the behavior of pure Ni nanoparticles and their Ni@TiO₂ core–shell counterparts.

2. Materials and Methods

The system for producing core–shell nanoparticles comprised a primary gas aggregation cluster source (GAS) for producing Ni NPs and a secondary chamber for coating them with a Ti film. The primary GAS featured a water-cooled aggregation chamber, 100 mm in diameter, and a DC planar magnetron, 81 mm in diameter, equipped with a 1.5 mm thick Ni target. The corresponding working pressure was 48 Pa. The secondary chamber contained another DC magnetron with an 81 mm diameter and a 4 mm thick Ti target (purity 99.2%). Both magnetrons operated under constant current, using Ar (purity 99.996%) as the working gas. Since the depositions were carried out under an Ar atmosphere, both the Ni cores and the Ti shell were deposited in a metallic form. After removal from the vacuum chamber and exposure to ambient air, the Ti shell spontaneously oxidizes due to its high affinity for oxygen. The encapsulation of the Ni core by the Ti/TiO₂ shell limits the direct oxygen access to the core. More details about the experimental setup can be found in the work by Hanus et al. [27].

The nanoparticles were sputtered on a glass substrate. The TEM samples were prepared by dropping the nanoparticles, suspended in methanol, onto a grid with a lacey carbon support film. A transmission electron microscope JEOL 2200FS (JEOL Ltd., Akishima, Tokyo 196-8558, Japan), operating in TEM and STEM (scanning transmission electron microscopy) modes, with bright-field (BF), high-angle annular dark field (HAADF) (JEOL Ltd., Akishima, Tokyo 196-8558, Japan), and secondary-electron (SE) (JEOL Ltd., Akishima, Tokyo 196-8558, Japan) detectors, was used. The annealing was performed in a Gatan (Gatan, Inc., 5794 W Las Positas Blvd, Pleasanton, CA 94588, USA) in situ heating holder under the intrinsic high-vacuum conditions of the TEM column (base pressure $\sim 10^{-7}$ – 10^{-9} mbar). Selected-area electron diffraction (SAED) patterns were analyzed using the JEMS software package (version 4.8731U2020b19) [28].

For the simulation part, polycrystalline rutile shells were constructed using AtomsK (version beta-0.11.2) [29]. The shells were stabilized at 700 °C, while a spherical indenter placed inside the empty shell expanded at a rate of 2 nm/s. All simulations were performed

using the LAMMPS software (version 23 Jun 2022—Update 3) [30], Ti–O interactions were modeled with the Matsui–Akagoi potential [31], which has been widely applied to TiO₂ polymorphs [32]. The results were visualized and analyzed using OVITO (version basic-3.14.1) [33].

3. Results

3.1. Pure Ni Nanoparticles

The characterization of Ni nanoparticles (Figure 1) revealed a mean diameter of approximately 9 nm. The diffraction pattern analysis confirmed that the nanoparticles possess a face-centered cubic (fcc) structure. Additionally, energy-dispersive X-ray spectroscopy (EDS) maps indicated that the nanoparticles have a Ni core surrounded by a thin oxidized shell.

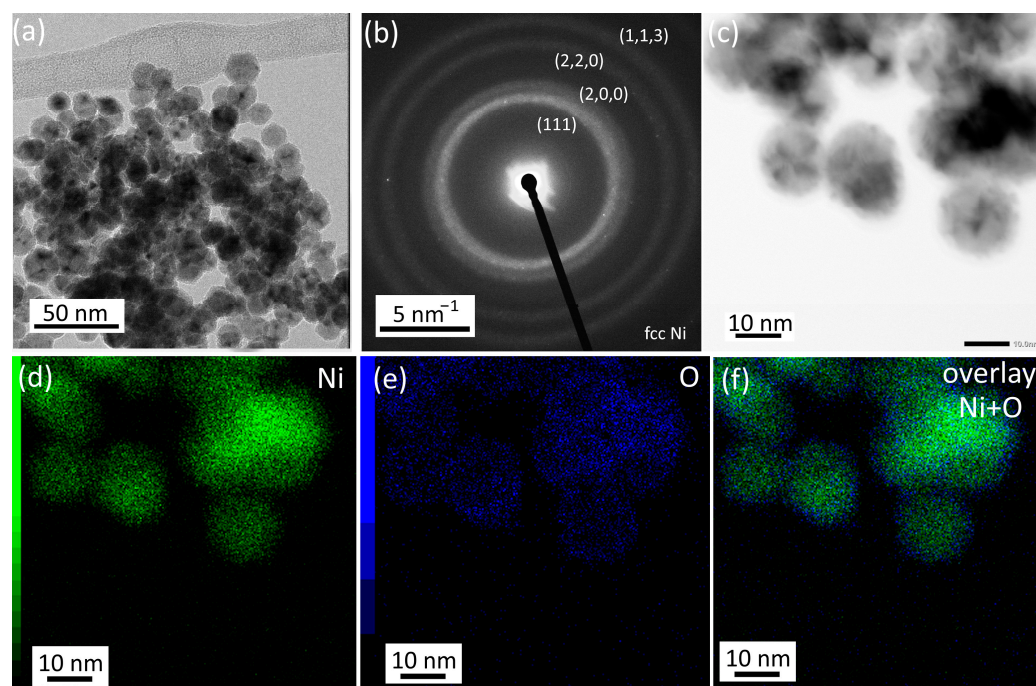


Figure 1. Pure Ni nanoparticles: (a) TEM BF, (b) TEM diffraction pattern, (c) STEM BF, (d–f) EDS maps (Ni green, O blue).

The particles were annealed to 100 °C, then in 50 °C increments to 850 °C. Each temperature was held for 5 min. The annealing process and its effects on an agglomerate of NiNPs are documented in Figure 2. The particles maintained their original shape up to 400 °C. At 450 °C, significant sintering commenced, as evidenced by the necking between neighboring nanoparticles and the coalescence of smaller particles into larger ones. This coalescence increased the average nanoparticle size to 13 nm. These results are consistent with other studies, which report substantial sintering of Ni nanoparticles with mean sizes of 9 nm and 20 nm at around 450 °C [11,34]. A further notable change in the shape of the nanoparticles occurred at 800 °C, when they grew to diameters ranging from 15 to 30 nm.

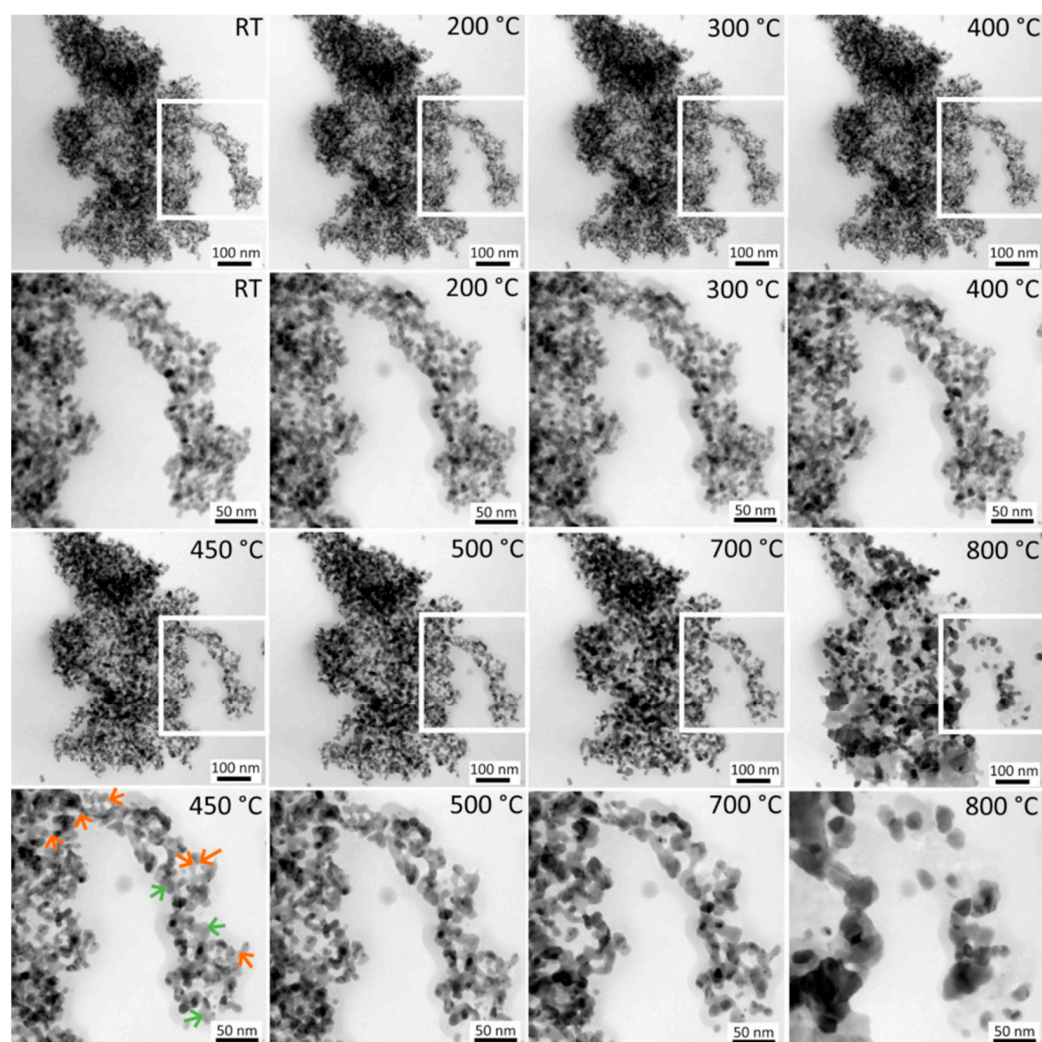


Figure 2. Annealing of the pure Ni nanoparticles, BF STEM images. The details of the area in the white rectangle are shown under the image for each temperature. Orange arrows indicate necking of the particles, while newly formed particles with increased size are indicated by green arrows.

3.2. Core–Shell NiTi Nanoparticles

The core–shell structure of nanoparticles, consisting of a nickel core coated with titanium using magnetron sputtering, was confirmed by STEM analysis (Figure 3). The nanoparticles have a mean diameter of 24 nm, with a core diameter close to 20 nm and a shell thickness of 2 nm. The HAADF detector showed a brighter core in the image, indicating that the atoms in the core are heavier. This finding aligns with the EDS analysis, which clearly distinguishes the Ni core from the Ti shell (Figure 3d,e). Additionally, oxidation of the Ti shell was observed (Figure 3f).

Figure 4 shows HRTEM images of the structure of the nanoparticles. The face-centered cubic (fcc) structure of the core is confirmed by Fast Fourier Transform (FFT) images, which were compared to JEMS-simulated diffraction images in several orientations. No crystalline structure was detected in the shell, indicating that the titanium oxide (TiO₂) shell is amorphous.

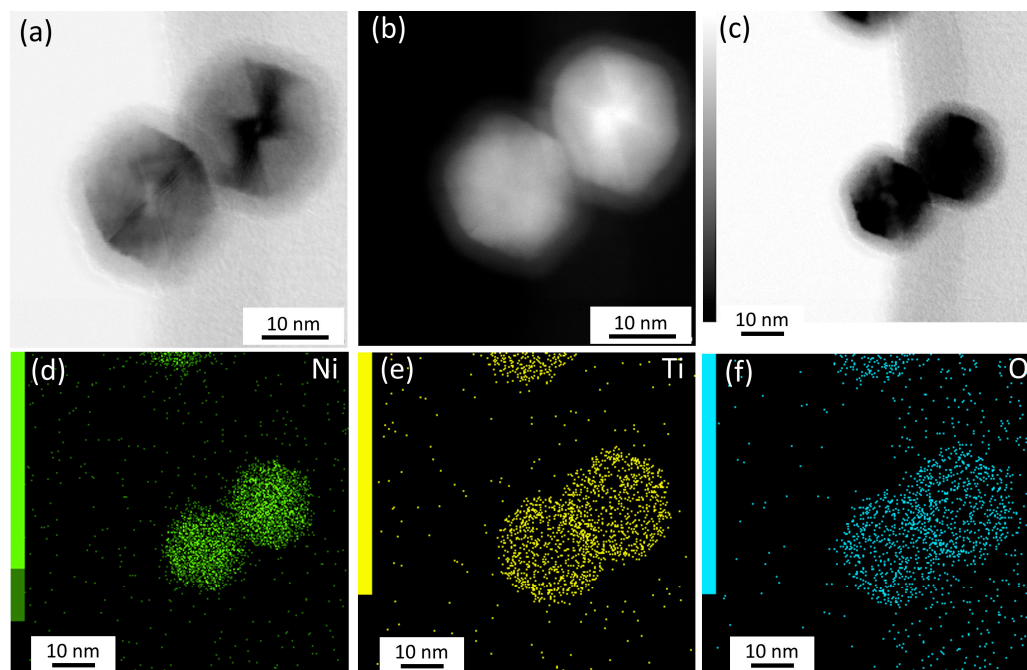


Figure 3. TEM images of NiTi nanoparticles: (a,b) TEM BF, (c) STEM BF, (d) STEM HAADF, (e) STEM BF—image of EDS map area, (d–f) EDS analysis (Ni green, Ti yellow, O blue).

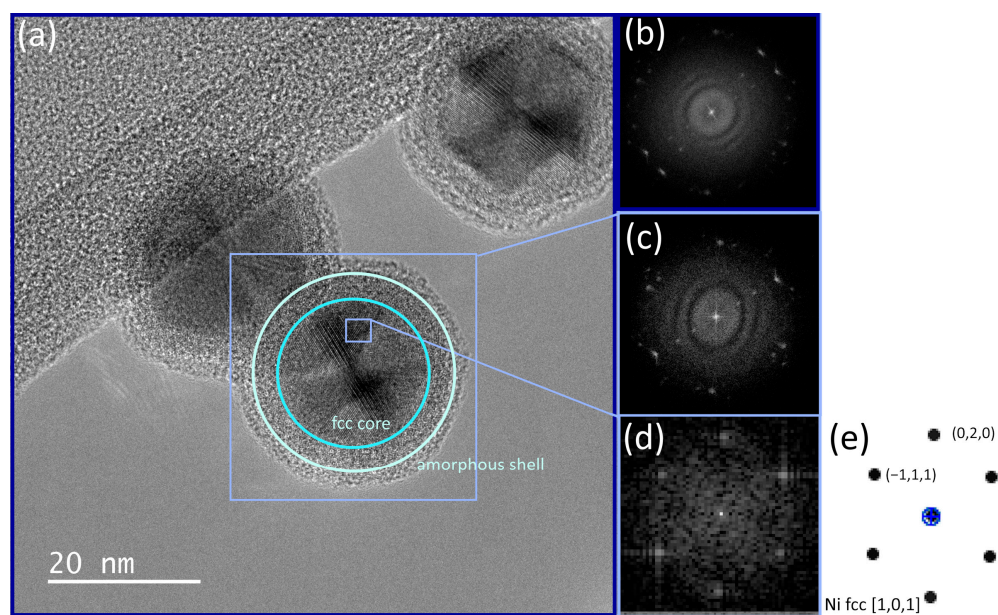


Figure 4. (a) HRTEM image of core-shell Ni@TiO₂ nanoparticles: (b–d) FFT images are taken from respective areas inside the blue rectangles, (e) simulated diffraction patterns of fcc Ni in the corresponding orientation consistent with the FFT image in (d). The core-shell structure of the particles is indicated.

Figure 5 shows the annealing process of an agglomerate of core-shell Ni@TiO₂ nanoparticles. Bright-field and secondary-electron detectors were used to capture the projection and surface of the agglomerate. The agglomerate remains stable up to 700 °C, with no visible changes in particle shape, size, or surface characteristics. Initial sintering occurs between 700 °C and 800 °C, with considerable necking and particle merging observed at 800 °C. The particles further grow as the temperature increases to 900 °C.

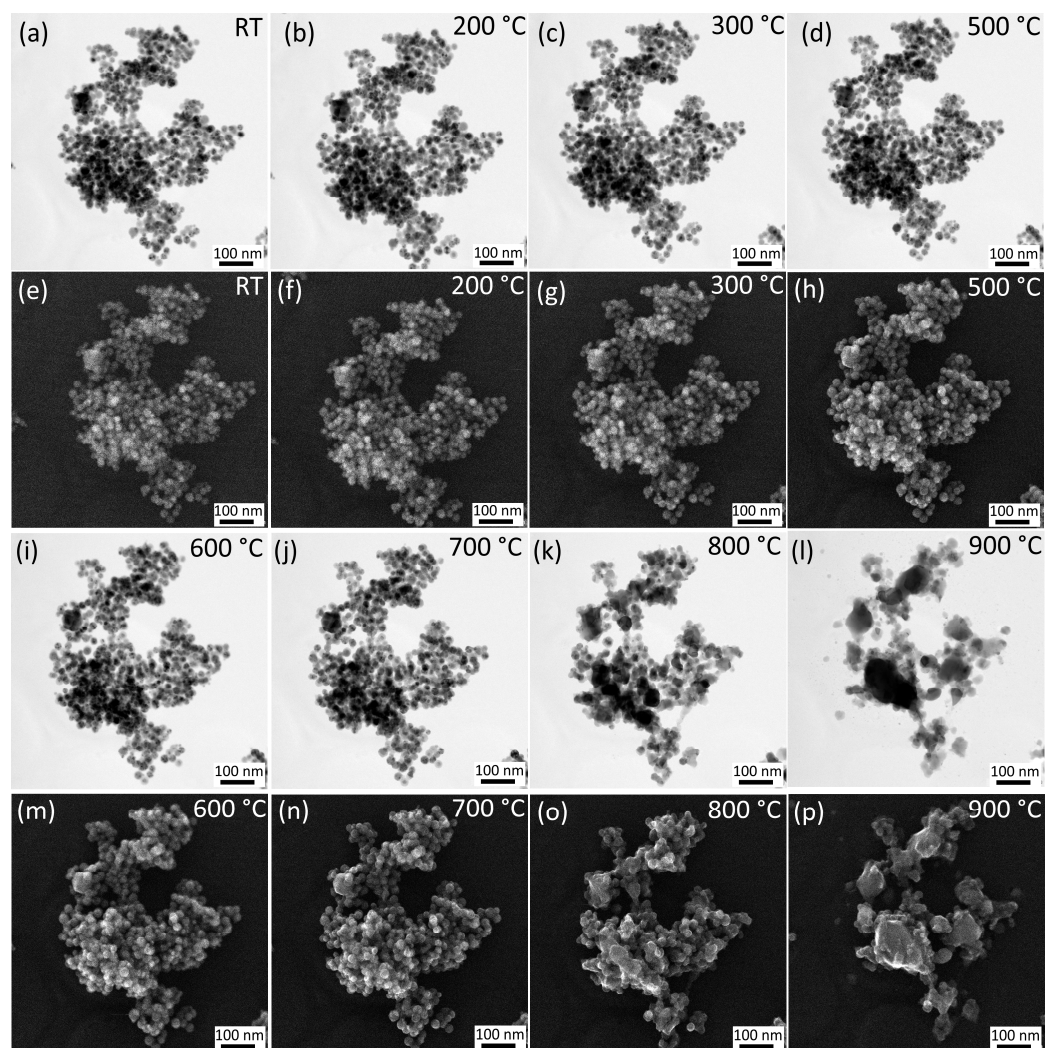


Figure 5. Annealing of Ni@TiO₂ core shell nanoparticles in the temperature range RT–900 °C: (a–d) and (i–l) STEM BF, (e–h) and (m–p) STEM SE detector.

The analysis of the particles formed after the sintering of Ni@TiO₂ core–shell nanoparticles (Figure 6) reveals a separation of Ni and Ti components. EDS analysis of the samples cooled to RT confirmed that newly formed particles, with diameters reaching up to 100 nm, are primarily composed of Ni. The HAADF image shows that the TiO₂ shells have merged into the background, forming a thinner TiO₂ layer.

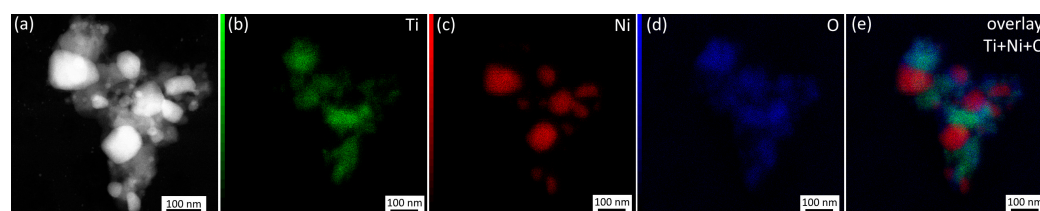


Figure 6. Images of a representative nanoparticle cluster after annealing: (a) STEM HAADF, (b–d) EDS maps of different elements (Ti, Ni, O), (e) overlay of the EDS maps (Ti green, Ni red, O blue).

Figure 7 presents a detailed analysis of the core–shell evolution at elevated temperatures. HRTEM images reveal that the initially amorphous shell begins to crystallize between 500 and 600 °C, with lattice spacings indicative mainly of the rutile phase. A detailed analysis of the diffraction pattern from a larger area (Figure 7e) confirms the presence of nickel along with a rutile and minor brookite shell.

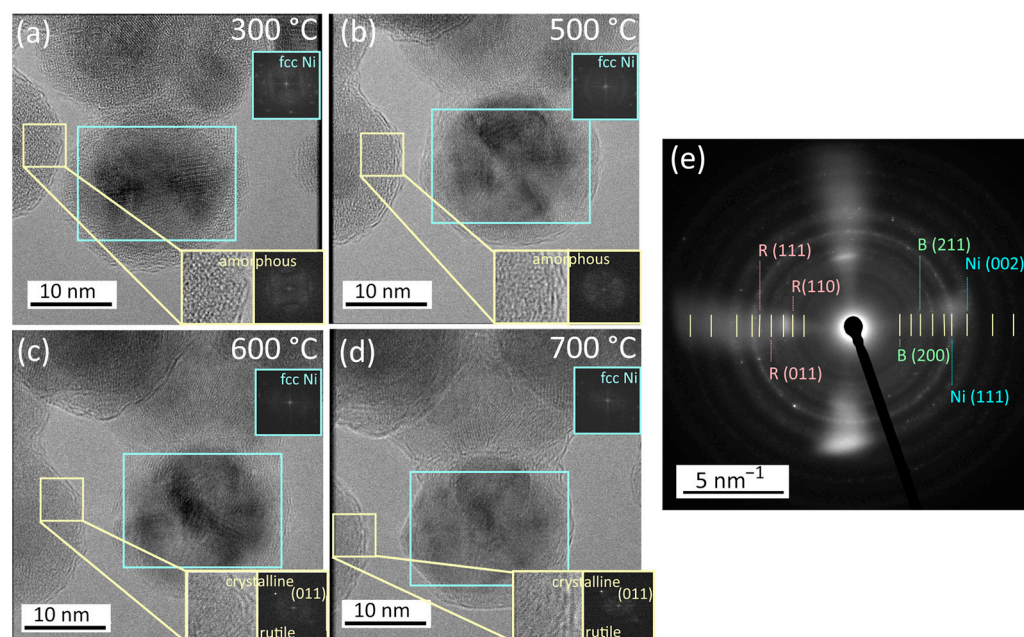


Figure 7. (a–d) HRTEM images of particles annealed between 300 and 700 °C, (e) SAED pattern at 700 °C (pink-rutile, green-brookite, blue-fcc Ni).

4. Discussion

The in situ TEM experiments show that Ni@TiO₂ particles remain morphologically stable up to ~700 °C, with the shell failure and the Ni coalescence emerging between 700 and 800 °C. This stabilization window aligns with earlier reports that TiO₂ coatings substantially suppress Ni sintering. Specifically, TiO₂-coated Ni powders/compacts prepared by wet chemistry exhibit markedly reduced sinterability and require temperatures ≥ 150 –200 °C higher than bare Ni to achieve comparable densification [35]. Likewise, for Ni@TiO₂ ‘nanocapsules’, thermomechanical and short-dwell annealing data show that, relative to bare Ni (shrinkage onset ~657 °C), the TiO₂ shell shifts the observed sintering onset by ~200–300 °C [36].

For tens-of-nanometer Ni cores with few-nanometer shells, TiO₂ performs on par with SiO₂ and Al₂O₃ prepared by atomic layer deposition in delaying coalescence into the high-temperature window. In our case (~2 nm TiO₂ on ~20 nm Ni), sintering is held off until ~700–800 °C. With a comparably thin alumina overcoat, an ~4 nm Al₂O₃ layer on Ni/SiO₂ confines Ni growth and maintains stability during high-temperature operation (700–800 °C range), attributed to the overcoat physically restraining Ni migration [37]. Likewise, thin-shell Ni@SiO₂ core-shell particles developed for high-temperature solar absorbers remain morphologically stable at ~700 °C, indicating a similar level of thermal suppression with silica at a few nm thickness [18]. Altogether, when shell thickness is kept in the ~2–4 nm band, and Ni cores are ~10–30 nm, the onset temperature for visible sintering clusters is around ~700–800 °C across TiO₂, SiO₂, and Al₂O₃, with exact thresholds shifting with shell porosity, continuity, and atmosphere.

Sintering of pure Ni and TiO₂ nanoparticles within this temperature range is generally considered to be a process controlled by surface diffusion [10,38]. The onset of sintering observed between 700 and 800 °C occurs well below the temperatures typically required for TiO₂ nanoparticles to sinter [39–41]. In the experiment, the formation of large Ni particles was observed without interaction with the TiO₂ shell. This observation suggests that the Ni was trapped in the cores, and sintering was possible after the deterioration of the TiO₂ shell. This suggestion is supported by the HRTEM images captured at elevated temperatures (Figure 8), which show that shell deterioration is already evident at several locations,

exposing the Ni core. Notably, the TiO_2 shell is observed to crystallize into rutile before the appearance of visible cracks; detailed HRTEM images demonstrating the crystalline shell prior to rupture are provided in the Supplementary Materials (Supplementary Figure S1).

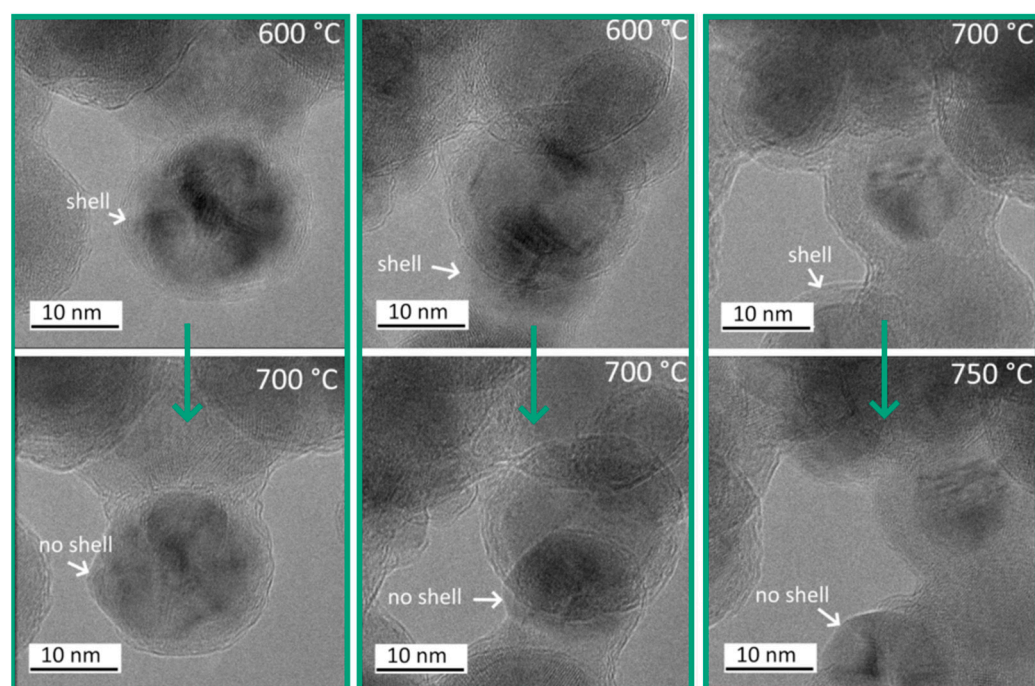


Figure 8. HRTEM images of representative areas at temperatures between 600 and 700 °C. The images are arranged in pairs corresponding to the same region (linked by green arrows): the upper image in each pair shows the particle with the shell, while the lower image shows the identical area after shell removal (indicated by white arrows).

The deterioration is therefore caused by the combination of mechanical rupture of the shell due to thermal expansion differences and consequent diffusion-based shrinking of the ruptured shell to minimize surface energy. Once the shell integrity is compromised, rapid Ni migration occurs. At these elevated temperatures (700–800 °C), surface diffusion dominates mass transport [11]. The exposed Ni atoms migrate along particle surfaces and through interparticle contact regions. Because Ni–Ni metallic bonding is energetically favored over Ni– TiO_2 interfacial contact, Ni atoms preferentially diffuse toward other Ni domains. Ni nanoparticles thus coalesce into larger, denser metallic particles, reaching ~100 nm in size. Simultaneously, the fractured TiO_2 shell undergoes diffusion-driven retraction and coarsening to minimize its own surface energy. As a result, the morphology is characterized by spatial separation of large Ni particles and coarsened TiO_2 regions (Figure 6).

The rupture of the shell due to mechanical stress at elevated temperatures was further examined using molecular dynamics simulations (Figure 9), where the TiO_2 shell was modeled in the rutile phase. The amorphous-to-rutile transformation observed experimentally around ~600 °C was not explicitly simulated due to time-scale limitations inherent to MD. Consequently, temperature-dependent changes in elastic properties associated with the phase transition were not directly included. The model therefore provides a simplified mechanical description but captures stress evolution due to thermal-expansion mismatch at the crack-initiation temperature (700 °C). At a core expansion of approximately 2 Å, corresponding to the end of the elastic regime, the stress reaches about 100 MPa. With further expansion, the stress increases to approximately 250 MPa at around 9 Å, where the onset of a large, visible crack and the opening of the shell are observed. In the intermediate

expansion range between these two values, local damage in the form of small microvoids within the shell is observed. These defects do not fully penetrate the shell but are accompanied by irregular thinning of the shell and changes in surface morphology. In the presence of the nickel core, such defects are expected to enhance diffusion pathways locally prior to complete shell failure.

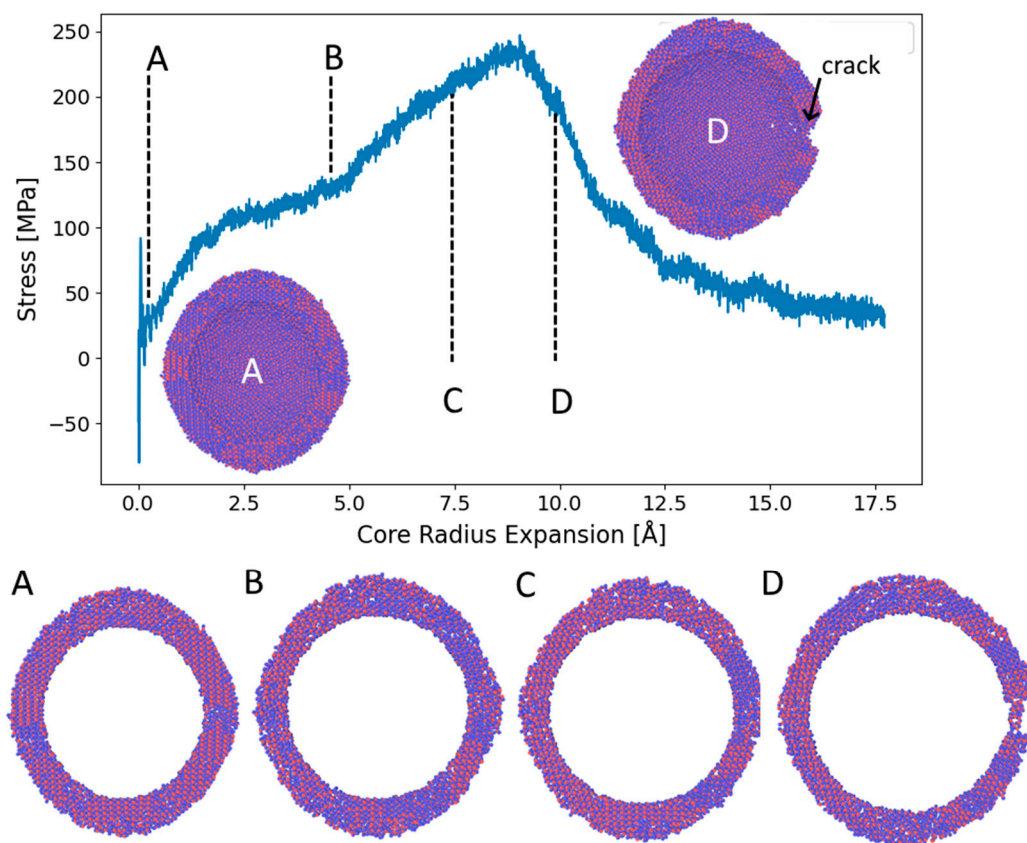


Figure 9. Average shell stress as a function of core radius expansion for a 10 nm large shell with 2 nm thickness. Visualizations of the shell (Ti atoms—red, O atoms—blue) sliced in half at the initial state and after the appearance of a visible crack are shown (A,D). 5 nm thick slices are shown for 4 different radius values (A–D).

The stress at the core–shell interface can be evaluated using a simple model based on the differences in thermal expansion coefficients of *Ni* and *TiO*₂. At room temperature, it is assumed that a contact layer exists between the *Ni* core and the surrounding *TiO*₂ shell. Notably, the linear thermal expansion coefficient α of nickel is greater than that of *TiO*₂ (α_{TiO_2} in the range $(8.4\text{--}11.8) \times 10^{-6} \text{ K}^{-1}$ and $\alpha_{\text{Ni}} = (12\text{--}13.5) \times 10^{-6} \text{ K}^{-1}$) [42,43]. This difference in thermal expansion increases tension in the contact layer as temperature rises. The shell thickness influences the thermo-mechanical response of the core–shell system. In a thermally mismatched spherical core–shell system, the maximum tensile hoop stress occurs at the inner shell surface, regardless of the shell thickness. However, increasing shell thickness enhances mechanical robustness and fracture resistance by increasing structural constraint and reducing sensitivity to local defects. In the present work, MD simulations comparing 1 nm and 2 nm shells (Supplementary Figure S2) demonstrate that the 2 nm shell exhibits clearly enhanced mechanical robustness. While thicker shells may further improve mechanical confinement and diffusion resistance, a systematic thickness-optimization study was beyond the scope of this work.

The maximum pressure that causes volumetric deformation of the *Ni* core and contraction of the *Ni* side of the contact layer can be approximated as the tensile strength of

TiO₂. Then, the deformation ε_{Ni} , which leads to the total deformation observed in the inner part of the TiO₂ layer at the point of fracture, can be calculated using Equation (1).

$$\varepsilon_{Ni} = \frac{1}{3} \cdot \frac{\Delta V}{V} = \frac{1}{3} \cdot \frac{\sigma}{B_{Ni}}. \quad (1)$$

We assume that the mechanical properties, namely the Young's modulus E_{TiO2} (value in the range 230–288 GPa), and the bulk modulus of Ni B_{Ni} (value in the range 162–200 GPa) [44,45], remain constant across the temperature range considered. Additionally, we assume that the linear coefficients of thermal expansion remain constant. The interface pressure $\sigma(T)$ as a function of temperature T with initial temperature T_0 can then be estimated using Equation (2).

$$\sigma(T) = \frac{(\alpha_{Ni} - \alpha_{TiO2}) \cdot (T - T_0)}{\frac{1}{E_{TiO2}} + \frac{1}{3B_{Ni}}} \quad (2)$$

On the nanoscale, elastic constants may deviate from bulk values. Using the bond energy model of Pandey and Kumar [46], the bulk modulus of Ni nanoparticles decreases only slightly (from 180 to 170 GPa for 20 nm), suggesting minimal size effect. Young's modulus is also reduced in thin films, but even for shell thicknesses <5 nm, it remains above ~80% of the bulk value [47,48].

The temperature dependence of the interface pressure computed from the average values of the material constants is shown in Figure 10. The pressure limit for crack formation (250 MPa) is reached at around 600 °C, which is close to the temperature at which the first cracks were observed in the experiment. Despite the simplicity of the model and the absence of explicit phase transformations, the estimated cracking temperature is in reasonable agreement with experimental observations.

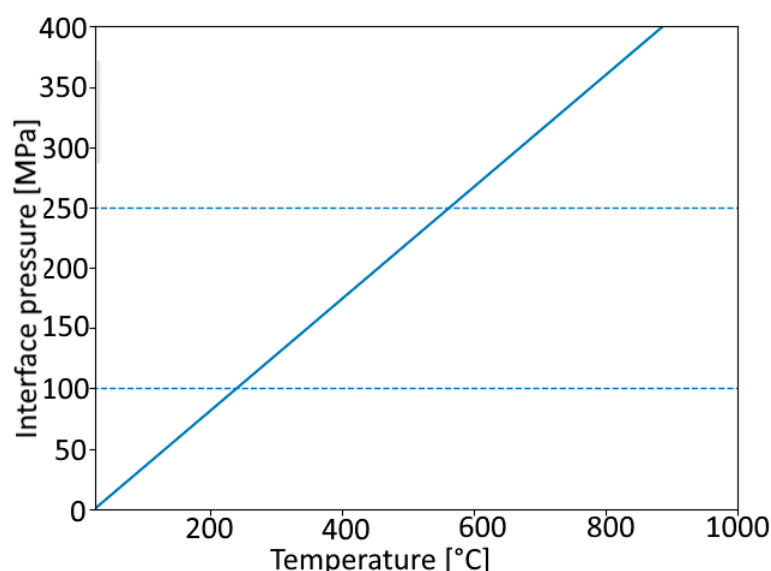


Figure 10. Interface pressure estimated from Equation (2) for average values of constants ($\alpha_{TiO2} = 8.4\text{--}11.8 \times 10^{-6} \text{ K}^{-1}$, $\alpha_{Ni} = 12\text{--}13.5 \times 10^{-6} \text{ K}^{-1}$, $E_{TiO2} = 230\text{--}288 \text{ GPa}$, $B_{Ni} = 162\text{--}200 \text{ GPa}$). The stress required for the shell cracking, as estimated from the MD simulation (Figure 9), is highlighted by horizontal lines (100–250 MPa).

It should be emphasized that this analytical model intentionally neglects transformation-induced stresses associated with the amorphous-to-rutile transition and assumes temperature-independent elastic constants. Possible elastic softening at elevated temperatures and microstructural evolution of the TiO₂ shell are therefore not explicitly accounted for. Conse-

quently, the calculated interface pressure and corresponding cracking temperature should be regarded as order-of-magnitude estimates rather than exact predictions. According to Equation (2), the interface stress is inversely proportional to the effective elastic compliance of the shell–core system. A lower Young’s modulus of amorphous TiO₂ would reduce the calculated interface stress at a given temperature. Modeling the shell as rutile likely provides an upper bound on interfacial stress prior to crystallization. However, in our experiments, the amorphous-to-rutile transformation occurs around 600 °C, whereas shell cracking occurs at 700–800 °C. Therefore, the failure regime corresponds to a shell that is already largely crystalline.

5. Conclusions

In this study, the sintering behavior of Ni@TiO₂ core–shell nanoparticles was investigated, with a focus on how the TiO₂ shell affects their stability and sintering resistance. Compared to pure Ni nanoparticles that began to sinter at 450 °C, the Ni@TiO₂ nanoparticles remained stable up to 700 °C, with initial sintering observed between 700 °C and 800 °C. After shell rupture, the Ni cores coalesce into larger Ni particles, confirming that the shell acts as a confinement barrier up to its mechanical failure. Molecular dynamics simulations and a thermal-mismatch model indicate that the interfacial stresses induced by the thermal expansion difference between Ni and TiO₂ are sufficient to drive shell cracking once the amorphous shell crystallizes to rutile. Overall, the TiO₂ shell effectively suppresses sintering, extending the operational stability of Ni nanoparticles by ~250 °C and offering a promising route for designing thermally robust core–shell nanostructures.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/met16020225/s1>, Figure S1: HRTEM images of Ni@TiO₂ nanoparticle shells at elevated temperatures prior to crack formation: (a) 700 °C and (b) 600 °C. The TiO₂ shell is crystalline, with the rutile phase confirmed by FFT analysis; Figure S2: Average shell stress as a function of core radius expansion for a 10 nm Ni core with TiO₂ shell thicknesses of 1 nm and 2 nm.

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References

- Altammar, K.A. A review on nanoparticles: Characteristics, synthesis, applications, and challenges. *Front. Microbiol.* **2023**, *14*, 1155622. [CrossRef]
- Tailor, G.; Chaudhary, J.; Jandu, S.; Chetna; Mehta, C.; Yadav, M.; Verma, D. A review on green route synthesized nickel nanoparticles: Biological and photo-catalytic applications. *Results Chem.* **2023**, *6*, 101195. [CrossRef]
- Bibi, I.; Kamal, S.; Ahmed, A.; Iqbal, M.; Nouren, S.; Jilani, K.; Nazar, N.; Amir, M.; Abbas, A.; Ata, S.; et al. Nickel nanoparticle synthesis using *Camellia sinensis* as reducing and capping agent: Growth mechanism and photocatalytic activity evaluation. *Int. J. Biol. Macromol.* **2017**, *103*, 783–790. [CrossRef]

4. Cheng, Y.; Guo, M.; Zhai, M.; Yu, Y.; Hu, J. Nickel nanoparticles anchored onto Ni foam for supercapacitors with high specific capacitance. *J. Nanosci. Nanotechnol.* **2020**, *20*, 2402–2407. [[CrossRef](#)]
5. Reena Mary, A.P.; Suchand Sandeep, C.S.; Narayanan, T.N.; Philip, R.; Moloney, P.; Ajayan, P.M.; Anantharaman, M.R. Nonlinear and magneto-optical transmission studies on magnetic nanofluids of non-interacting metallic nickel nanoparticles. *Materials* **2011**, *22*, 375702. [[CrossRef](#)] [[PubMed](#)]
6. Barsan, M.M.; Enache, T.A.; Preda, N.; Stan, G.; Apostol, N.G.; Matei, E.; Kuncser, A.; Diclescu, V.C. Direct immobilization of biomolecules through magnetic forces on Ni electrodes via Ni nanoparticles: Applications in electrochemical biosensors. *ACS Appl. Mater. Interfaces* **2019**, *11*, 19867–19877. [[CrossRef](#)]
7. Hill, D.; Barron, A.R.; Alexander, S. Comparison of hydrophobicity and durability of functionalized aluminium oxide nanoparticle coatings with magnetite nanoparticles: Links between morphology and wettability. *J. Colloid Interface Sci.* **2019**, *555*, 323–330. [[CrossRef](#)] [[PubMed](#)]
8. Jaji, N.; Lee, H.L.; Hussin, M.H.; Akil, H.M.; Zakaria, M.R.; Othman, M.B.H. Advanced nickel nanoparticles technology: From synthesis to applications. *Nanotechnol. Rev.* **2020**, *9*, 1456–1480. [[CrossRef](#)]
9. Nik Roselina, N.R.; Azizan, A. Ni nanoparticles: Study of particle formation and agglomeration. *Procedia Eng.* **2012**, *41*, 1620–1626. [[CrossRef](#)]
10. Tsyganov, S.; Kästner, J.; Rellinghaus, B.; Kauffeldt, T.; Westerhoff, F.; Wolf, D. Analysis of Ni nanoparticle gas phase sintering. *Phys. Rev. B* **2007**, *75*, 045421. [[CrossRef](#)]
11. Bajtošová, L.; Kihoulou, B.; Králík, R.; Hanuš, J.; Cieslar, M. Nickel nanoparticles: Insights into sintering dynamics. *Crystals* **2024**, *14*, 321. [[CrossRef](#)]
12. Wang, H.; Lu, J. A review on particle size effect in metal-catalyzed heterogeneous reactions. *Chin. J. Chem.* **2020**, *3*, 1422–1444. [[CrossRef](#)]
13. Issa, B.; Obaidat, I.M.; Albiss, B.A.; Haik, Y. Magnetic nanoparticles: Surface effects and properties related to biomedicine applications. *Int. J. Mol. Sci.* **2013**, *14*, 21266–21305. [[CrossRef](#)]
14. Patange, M.; Biswas, S. Structural stability and magnetic properties of Ni nanoparticles with an in situ formed surface stabilization layer of graphitic carbon. *Mater. Res. Bull.* **2024**, *169*, 112509. [[CrossRef](#)]
15. Wang, T.; Zhou, Q.; Wang, X.; Zheng, J.; Li, X. MOF-derived surface modified Ni nanoparticles as an efficient catalyst for the hydrogen evolution reaction. *J. Mater. Chem. A* **2015**, *3*, 156–164. [[CrossRef](#)]
16. Manukyan, A.S.; Mirzakhanyan, A.A.; Badalyan, G.R.; Shirinyan, G.H.; Sharoyan, E.G. Preparation and characterization of nickel nanoparticles in different carbon matrices. *J. Contemp. Phys.* **2010**, *45*, 132–136. [[CrossRef](#)]
17. El-Gendy, A.A.; Ibrahim, E.M.M.; Khavrus, V.O.; Krupskaya, Y.; Hampel, S.; Leonhardt, A.; Büchner, B.; Klingeler, R. The synthesis of carbon-coated Fe, Co, and Ni nanoparticles and an examination of their magnetic properties. *Carbon* **2009**, *47*, 2821–2828. [[CrossRef](#)]
18. Ding, D.; Wei, W.; He, X.; Ding, S. Thermally stable Ni@SiO₂ core-shell nanoparticles for high-temperature solar selective absorber. *Sol. Energy* **2021**, *228*, 413–417. [[CrossRef](#)]
19. Baktash, E.; Littlewood, P.; Schomäcker, R.; Thomas, A.; Stair, P.C. Alumina-coated nickel nanoparticles as a highly active catalyst for dry reforming of methane. *Appl. Catal. B* **2015**, *179*, 122–127. [[CrossRef](#)]
20. Solis-Cortazar, J.C.; Zamudio-Torres, I.; Rojas-Blanco, L.; Perez-Hernandez, G.; Arellano-Cortaza, M.; Castillo-Palomera, R.; Espinosa de los Monteros, A.; Ramirez-Morales, E. Photoresponse enhancement in TiO₂ thin films by incorporation Ni and Cr nanoparticles using sol-gel method. *J. Mater. Sci. Mater. Electron.* **2022**, *33*, 7668–7678. [[CrossRef](#)]
21. David, T.M.; Gnanasekar, K.I.; Wilson, P.; Sagayaraj, P.; Mathews, T. Effect of Ni, Pd, and Pt Nanoparticle Dispersion on Thick Films of TiO₂ Nanotubes for Hydrogen Sensing: TEM and XPS Studies. *ACS Omega* **2020**, *5*, 11618–11626. [[CrossRef](#)]
22. Wu, Y.; Wu, X.; Zhang, L. Preparation and microstructure characterization of Ni/TiO₂ nanocomposite. *China Particuol.* **2003**, *1*, 262–265. [[CrossRef](#)]
23. Chen, W.; He, Y.; Gao, W. Electrodeposition of sol-enhanced nanostructured Ni–TiO₂₂ composite coatings. *Surf. Coat. Technol.* **2010**, *204*, 2487–2492. [[CrossRef](#)]
24. Majerič, P.; Rudolf, R.; Anžel, I.; Bogović, J.; Stopić, S.; Friedrich, B. Synthesis of NiTi/Ni–TiO₂ composite nanoparticles via ultrasonic spray pyrolysis. *Mater. Tehnol.* **2015**, *49*, 75–80. [[CrossRef](#)]
25. Bouchabou, M.; Rives López, J.M.; Román Martínez, M.C.; Lillo-Rodenas, M.A. Evaluating H₂ production by ultraviolet-induced water splitting over (Cu or Ni)–TiO₂ nanoparticle photocatalysts. *ACS Appl. Nano Mater.* **2025**, *8*, 19945–19955. [[CrossRef](#)]
26. Chen, L.; Yu, H.; Zhu, D.; Liu, S.; Zhang, L.; Sun, J.; Zhao, Z.; Li, Q.; Chen, G.; Li, Q. Designing electron/ion dual-phase conductor Ni@TiO₂ for high-performance lithium-ion storage: Combining insertion and space charge mechanism. *Appl. Phys. Lett.* **2024**, *124*, 133901. [[CrossRef](#)]
27. Hanuš, J.; Vaidulych, M.; Kylián, O.; Choukourov, A.; Kousal, J.; Khalakhan, I.; Cieslar, M.; Solař, P.; Biederman, H. Fabrication of Ni@Ti core-shell nanoparticles by modified gas aggregation source. *J. Phys. D Appl. Phys.* **2017**, *50*, 475307. [[CrossRef](#)]

28. Stadelmann, P.A. EMS—A software package for electron diffraction analysis and HREM image simulation in materials science. *Ultramicroscopy* **1987**, *21*, 131–145. [\[CrossRef\]](#)
29. Hirel, P. Atomsk: A tool for manipulating and converting atomic data files. *Comput. Phys. Commun.* **2015**, *197*, 212–219. [\[CrossRef\]](#)
30. Thompson, A.P.; Aktulga, H.M.; Berger, R.; Bolintineanu, D.S.; Brown, W.M.; Crozier, P.S.; In't Veld, P.J.; Kohlmeyer, A.; Moore, S.G.; Nguyen, T.D.; et al. LAMMPS: A flexible simulation tool for particle-based materials modeling at the atomic, meso, and continuum scales. *Comput. Phys. Commun.* **2022**, *271*, 108171. [\[CrossRef\]](#)
31. Matsui, M.; Akaogi, M. Molecular dynamics simulation of the structural and physical properties of the four polymorphs of TiO₂. *Mol. Simul.* **1991**, *6*, 239–244. [\[CrossRef\]](#)
32. Reinhardt, A. Phase behavior of empirical potentials of titanium dioxide. *J. Chem. Phys.* **2019**, *151*, 064505. [\[CrossRef\]](#)
33. Stukowski, A. Visualization and analysis of atomistic simulation data with OVITO—The Open Visualization Tool. *Model. Simul. Mater. Sci. Eng.* **2009**, *18*, 015012. [\[CrossRef\]](#)
34. Matsuno, M.; Bonifacio, C.; Thron, A.; Rufner, J.; Holland, T.; van Benthem, K. In situ sintering of Ni nanoparticles by controlled heating. *Microsc. Microanal.* **2011**, *17*, 524–525. [\[CrossRef\]](#)
35. Tomoshige, R.; Kato, A.; Nagashima, K. Sinterability of TiO₂-coated fine Ni powder. *Ceram. Int.* **2006**, *32*, 221–222. [\[CrossRef\]](#)
36. Li, X.-Y.; Wei, G.-L.; Shen, N.-R.; Wang, Y.-L.; Dong, X.-L.; Jung, Y. Synthesis and characterization of Ni@TiO₂ nanocapsules for RF-MLCC electrodes via DC arc plasma method. *Mater. Sci. Eng. B* **2025**, *311*, 117861. [\[CrossRef\]](#)
37. Kim, S.; Armutlulu, A.; Liao, W.-C.; Hosseini, D.; Stoian, D.; Chen, Z.; Abdala, P.M.; Copéret, C.; Müller, C.R. Structural insight into an atomic layer deposition (ALD) grown Al₂O₃ layer on Ni/SiO₂: Impact on catalytic activity and stability in dry reforming of methane. *Catal. Sci. Technol.* **2021**, *11*, 6496–6507. [\[CrossRef\]](#)
38. Rahbar, H. Sintering rate of nickel nanoparticles by molecular dynamics. *J. Phys. Chem.* **2023**, *127*, 6802–6812. [\[CrossRef\]](#)
39. Buesser, B.; Gröhn, A.J.; Pratsinis, S.E. Sintering rate and mechanism of TiO₂ nanoparticles by molecular dynamics. *J. Phys. Chem. C* **2011**, *115*, 10983–10991. [\[CrossRef\]](#) [\[PubMed\]](#)
40. Kobata, A.; Kusakabe, K.; Morooka, S. Growth and transformation of TiO₂ crystallites in an aerosol reactor. *AIChE J.* **1991**, *37*, 347–354. [\[CrossRef\]](#)
41. Seto, T.; Shimada, M.; Okuyama, K. Evaluation of sintering of nanometer-sized titania using an aerosol method. *Aerosol Sci. Technol.* **1995**, *23*, 183–200. [\[CrossRef\]](#)
42. Kirby, R.K. Thermal expansion of rutile from 100 to 700 K. *J. Res. Natl. Bur. Stand. A* **1967**, *71A*, 363–369. [\[CrossRef\]](#) [\[PubMed\]](#)
43. Touloukian, Y.S. *Thermal Expansion: Metallic Elements and Alloys*; Springer: New York, NY, USA, 1975.
44. Wachtman, J.B., Jr.; Tefft, W.E.; Lam, D.G., Jr. Elastic constants of rutile (TiO₂). *J. Res. Natl. Bur. Stand. A Phys. Chem.* **1962**, *66A*, 465–471. [\[CrossRef\]](#)
45. Ledbetter, H.M.; Reed, R.P. Elastic properties of metals and alloys, I. Iron, nickel, and iron–nickel alloys. *J. Phys. Chem. Ref. Data* **1973**, *2*, 531–618. [\[CrossRef\]](#)
46. Pandey, V.; Kumar, M. Development of size- and shape-dependent model for bulk modulus from bulk to nanoscale. *Physica B Condens. Matter* **2022**, *629*, 413617. [\[CrossRef\]](#)
47. Lu, H.; Meng, X. Correlation between band gap, dielectric constant, Young's modulus, and melting temperature of GaN nanocrystals and their size and shape dependences. *Sci. Rep.* **2015**, *5*, 16939. [\[CrossRef\]](#)
48. Rawat, K.; Goyal, M. Young's modulus and vibrational frequency dependence on shape and size in nanomaterials. *Mater. Today Proc.* **2021**, *42*, 1633–1637. [\[CrossRef\]](#)

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