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Investigation on Microwave mass-thermal effect of Nano-ZrO₂ Powders by Microwave Pyrolysis

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Abstract: Nano-ZrO₂ powders (about 200 nm) were successfully prepared by microwave pyrolysis with the heating temperature of 700°C. Zirconium oxychloride (ZrOCl₂·8H₂O) and ammonia (NH₃·H₂O) were selected as the original materials. As the mass of the raw material increases to 100 g, its pyrolysis efficiency correspondingly rises, achieving 99.88%. Microwave mass-thermal effect, which is manifested in the larger sample, more quickly heating effect, was found and it originated from the balance between the heating effect caused by microwave impedance matching and thermal diffusion of samples. Microwave mass-thermal effect make microwave pyrolysis a promising method for nano-ZrO₂ powder in large-scale industry.

Keywords: Nano-ZrO₂; microwave mass-thermal effect; impedance matching; simulation

1. Introduction

Nanoscale zirconia (nano-ZrO₂) holds considerable promise in catalysis, adsorption, and biomedicine, owing mainly to its high specific surface area, tunable structural properties, and abundant surface functional groups [1-3]. In particular, ultrafine ZrO₂ nanoparticles with a narrow particle-size distribution exhibit excellent sintering behavior and biocompatibility, making them especially suitable for biomedical applications such as dental materials [4]. Hydrothermal and gas phase method are

commonly employed for the synthesis of nano-ZrO₂ powders ^[5-7]. However, these conventional approaches often entail high energy consumption. Hassan et al. successfully synthesized lanthanide-doped ZrO₂ nanoparticles by hydrothermal synthesis, demonstrating good colloidal stability, environmental friendliness, and efficient heating performance ^[8]. In a comparative study of ZrO₂ nanoparticles prepared by gas-phase and liquid-phase methods ^[9-12], M. K. Baek reported that ultrafine ZrO₂ powders produced by chemical vapor deposition exhibited superior sintering properties ^[13]. Nevertheless, conventional preparation methods may leave harmful precursor residues, raising concerns about biological safety^[14]. Therefore, the development of novel, simple, efficient, and environmentally benign processes for producing high-quality ultrafine ZrO₂ nanoparticles is urgently needed.

Microwave heating, as an emerging alternative to conventional heating methods, has been widely applied in materials synthesis, chemical reactions, and mineral processing ^[15-17]. Its growing adoption is mainly attributable to its distinctive heating characteristics, including selective and volumetric heating, low pollution, and rapid response ^[18-20]. Microwave energy is delivered directly to the reactants, thereby reversing the direction of certain migration potential gradients that arise during conventional heat transfer ^[21-23]. As a result, volumetric heating with rapid and uniform temperature rise is achieved, promoting the formation of microstructures composed of small, uniformly distributed particles ^[24,25]. This effect has been particularly evident in the microwave sintering of zirconia ceramics. Zhang et al. demonstrated that microwave heating provides higher heating efficiency and stronger selectivity than conventional muffle furnaces ^[26]. Li et al. further highlighted the advantages of microwave pyrolysis for nano-ZrO₂ synthesis, showing that it is more effective than traditional heating methods in producing ultrafine ZrO₂ powders ^[27]. Nevertheless, the scalability of microwave heating and its underlying mechanisms, particularly the microwave thermal mass effect, still require further investigation.

In this work, ultrafine ZrO₂ nanoparticles were synthesized by microwave pyrolysis using zirconyl chloride and aqueous ammonia as precursors. The effects of pyrolysis temperature and precursor mass on the phase composition and microstructure of the resulting ZrO₂ nanoparticles were systematically investigated by X-ray diffraction (XRD) and scanning electron microscopy (SEM). The findings were further validated through experimental observations and computational simulations.

2. Experimental

Zirconium oxychloride octahydrate ($\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$, analytically pure) with 0.4 mol/L were used as initial liquid. Ammonia ($\text{NH}_3 \cdot \text{H}_2\text{O}$, analytically pure) with 0.8 mol/L was used as precipitator by 10 mL/min. The precipitations were obtained by filtrated and dried at 80 °C for 24 hours. The raw materials zirconium hydroxide ($\text{Zr}(\text{OH})_4$) were obtained. The raw material was subjected to microwave pyrolysis at a series of temperatures (700 °C, 800 °C, 900 °C and 1000 °C) and different mass (30g, 50g and 100g) to obtain nano-zirconia powder. Fig.1 shows the process flow diagram of preparing zirconia powder by microwave pyrolysis.

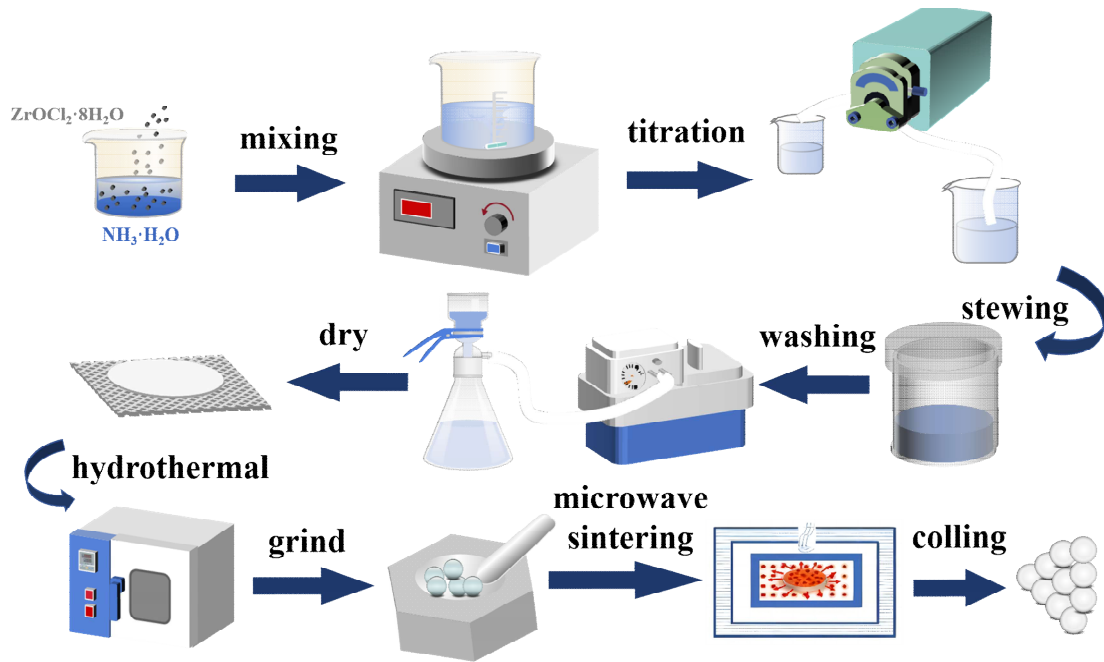


Fig. 1. process flowchart for preparing zirconia powder through microwave pyrolysis.

The phases of samples were characterized by X-ray diffraction (XRD, Rigaku, Smart Lab 3 kW). The microstructures of samples were characterized and analyzed by field emission scanning electron microscopy (SEM, JEOL, JSM 7001F). The mean grain size was determined statistically from scanning electron microscope (SEM) images, using the analysis software Nano Measurer. The measurement was conducted by measuring over 120 grains. The phase conversion was calculated by Eq. (1).

$$\eta = \frac{m-m_2}{m-m_1} \quad (1)$$

Where m is mass of raw material ($\text{Zr}(\text{OH})_4$), m_1 is mass of zirconia is obtained theoretically, m_2 is the mass of zirconia obtained after microwave pyrolysis.

In order to investigation of the thermal effect of microwave heating, simulation was used to find the electric field and thermal filed of microwave heating. The geometry model was shown in Fig 2. The electromagnetic loss of the material in an electromagnetic field was obtained using Eq. (2) ^[28]. The temperature distribution of the material in the solid heat transfer module was derived using Eq. (3). The parameters of samples that were input to the computational model were given in Table 1. Initial values were obtained through 2.45 GHz microwave frequency excitation using the four ports rectangular waveguide Transverse electric wave (TE 10-mode) and a port input power of 1500 W.

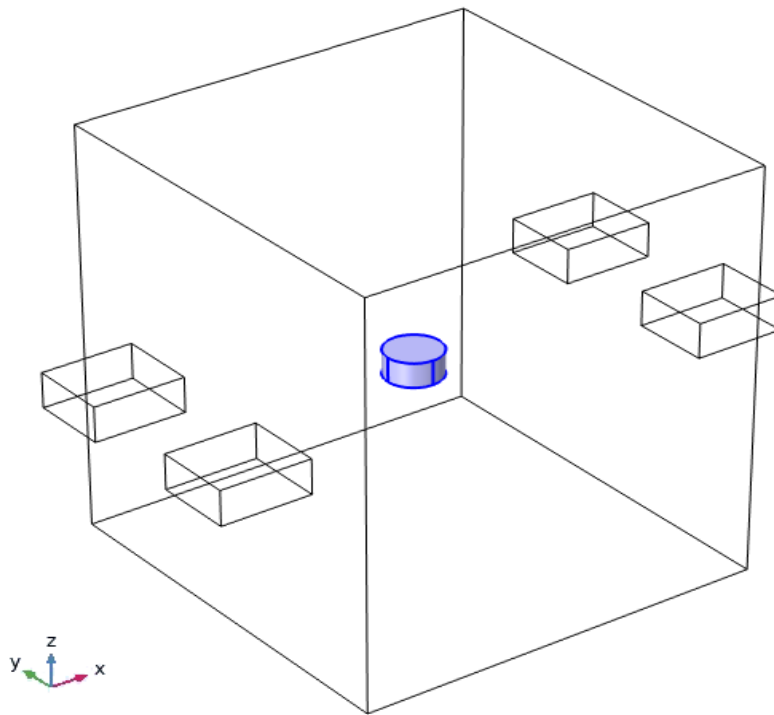


Fig 2. Simulation Model.

Table 1 Study material model input parameters

Material parameter	ZrO ₂	Unit
Relative dielectric constant (real part (ϵ'))	5.13299	1
Relative dielectric constant (imaginary part(ϵ''))	0.0495036	1
Loss tangent value ($\tan\delta = \frac{\epsilon'}{\epsilon''}$)	0.009644203	1

Relative magnetic permeability (real part (μ'))	1	1
Relative magnetic permeability (imaginary part (μ''))	0.00001	1
Electrical conductivity (σ)	0	S/m
Constant pressure heat capacity (C^p)	0.064	W/(m·K)
Bulk density (ρ)	3.7	g/cm ³

The point magnetic loss of the material in the electromagnetic field is expressed by equation (2).

$$Q_{rms} = \frac{1}{2} \{ \sigma E_{rms}^2 + \omega \varepsilon_r'' E_{rms}^2 + \omega \mu_r'' H_{rms}^2 \} \quad (2)$$

Where σE_{rms}^2 is electrical resistive loss、 $\omega \varepsilon_r'' E_{rms}^2$ is dielectric loss、 $\omega \mu_r'' H_{rms}^2$ is magnetic loss, respectively.

The temperature distribution of the material in the solid heat transfer module is derived from equation

$$\rho C_p \frac{\delta T}{\delta t} - \nabla \cdot (k \nabla T) = Q \quad (3)$$

Where ρ is density of the material, C_p is specific heat capacity at constant pressure, and k is thermal conductivity [29-32].

3. Results and Discussion

3.1 Characterization

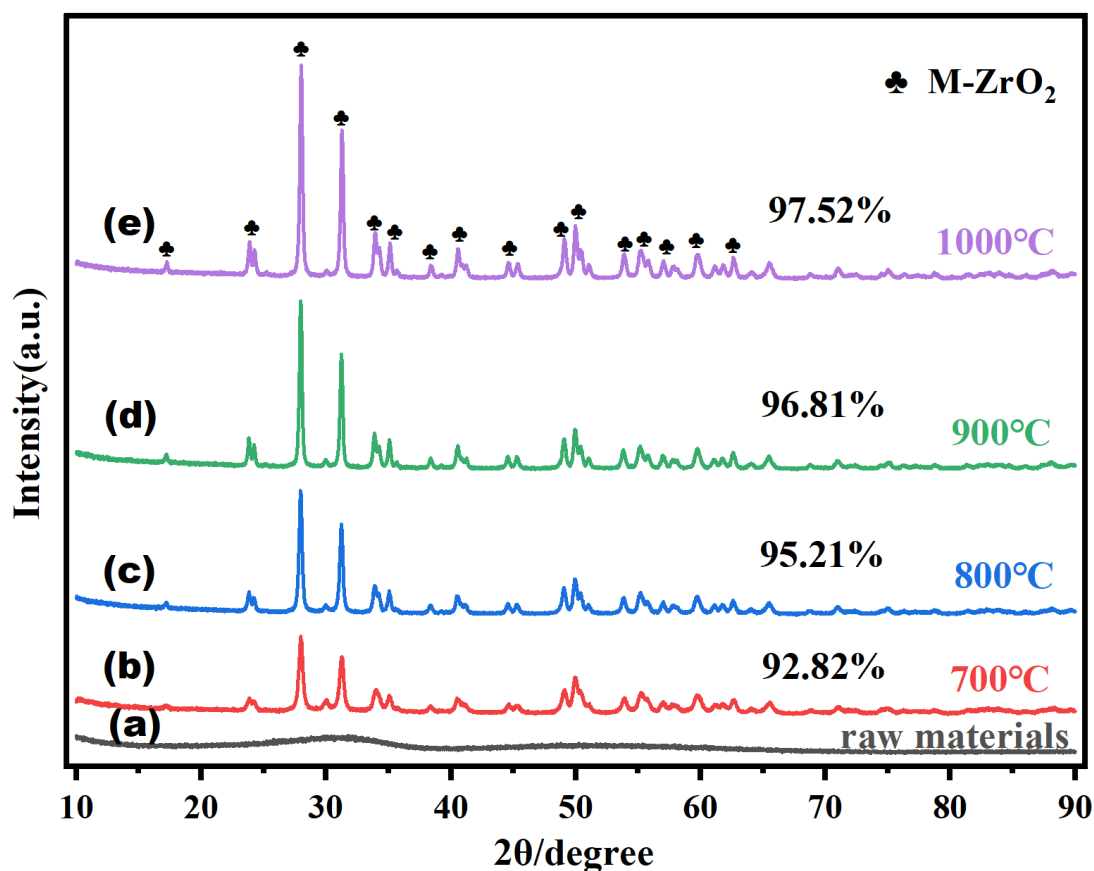


Fig. 3. XRD patterns of raw materials and samples at different pyrolysis temperatures: (a) raw materials (b) 700 °C, (c) 800 °C, (d) 900 °C, (e) 1000 °C.

Fig. 3 shows the XRD patterns of the raw materials and the samples prepared at 700 °C, 800 °C, 900 °C and 1000 °C. These results show that nano-zirconia by microwave pyrolysis are monoclinic phase, and conversion rates of samples are 92.82% (700 °C), 95.21% (800 °C), 96.81% (900 °C) and 97.52% (1000 °C), respectively. The data indicate that pyrolysis completion of raw materials correlates positively with temperature: higher temperatures result in more complete pyrolysis. Notably, all the samples, with the exception of those treated at 700°C, have undergone full pyrolysis. However, the increase in temperature leads to the sharpening and increasing of the diffraction peak of the sample. With the increase of temperature, the degree of conversion and crystallinity of the material are improved. However, the ratios of diffraction peaks in [-111] crystal plane (28.178°), [220] crystal plane (50.130°) and [011] interface crystal (24.049°) remain unchanged, indicating that the samples exhibit three-dimensional growth. The Full Width at Half Maxima of the [-111] diffraction peak

(FWHM) are 0.375 (700 °C), 0.298 (800 °C), 0.291 (900 °C) and 0.248 (1000 °C), respectively. Among the samples heated at 900 °C and 800 °C, FWHM values are similar, indicating the similarity in crystallinity.

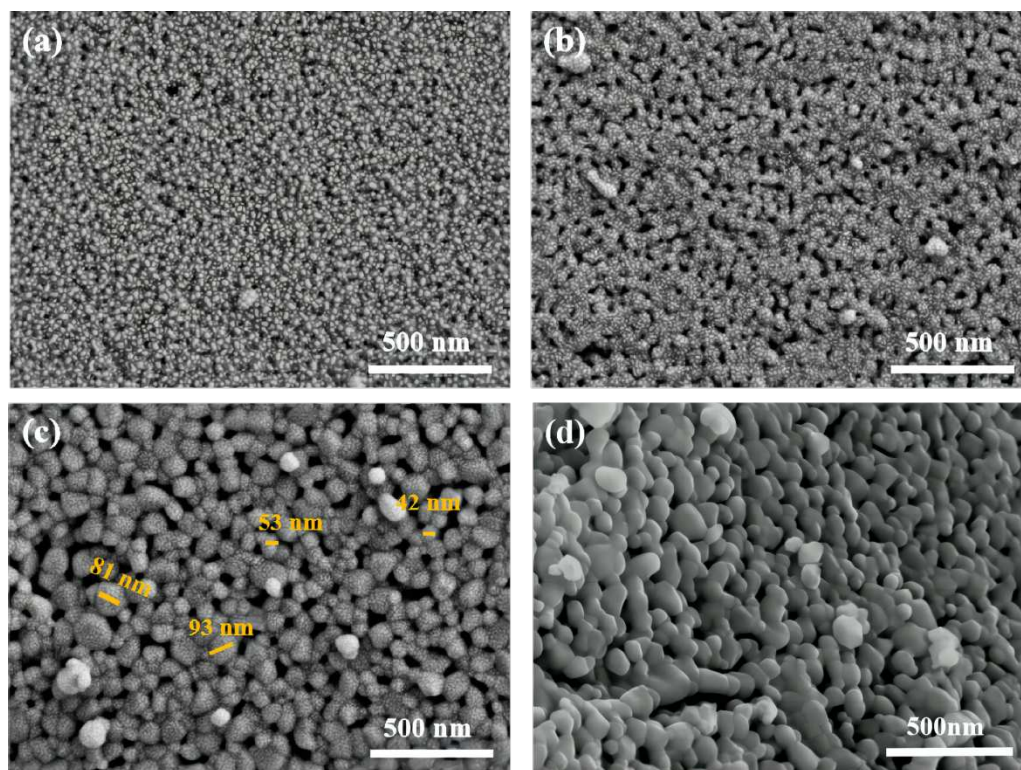


Fig. 4. SEM images of samples at different synthesis temperatures: (a) 700 °C, (b) 800 °C, (c) 900 °C, (d) 1000 °C.

Fig. 4 shows the SEM images of the samples at a series of pyrolysis temperatures (700 °C, 800 °C, 900 °C, 1000 °C), and raw materials without heating. At different pyrolysis temperatures, the particle sizes of the samples are 375 nm (700 °C), 298 nm (800 °C), 291 nm (900 °C) and 248 nm (1000 °C), respectively. Besides, the samples at a temperature of 1000 °C demonstrate pronounced hard agglomeration. This phenomenon is primarily attributed to the overburning of samples ^[33-35]. Fig. 4(a) illustrates that at a synthesis temperature of 700 °C, the particle size of the samples is approximately 50 nm, signifying relatively small dimensions. X-ray diffraction (XRD) patterns further reveal the presence of numerous unreacted raw materials within the product. From Fig. 4 (a), it can be seen that under the synthesis conditions of 700 °C, the particle size of the generated powder is small, and the particle size is about 50 nm. Combined with XRD data analysis, there are a large number of incompletely reacted raw material materials in the product. Fig. 4 (b) depicts a gradual increase in particle size for samples synthesized at 800 °C, with the microstructure progressively assuming an irregular

sphere. Fig. 4 (c) demonstrates that with the sintering temperature elevated to 900°C and 1000°C, the particle size increases and the particles adopt more regular shapes, averaging about 60 nm. These observations indicate that higher preparation temperatures facilitate the synthesis and crystal growth of the samples, resulting in more uniformly regular powder morphologies. Fig. 4 (d) illustrates that at a pyrolysis temperature of 1000°C, pronounced heavily agglomeration among particles. This agglomeration is primarily due to the electric field effects during the microwave synthesis of the powder, which induces elevated energy accumulation at particle contact points^[36]. As a result, sintering necks form between certain particles, culminating in localized hard agglomeration. Consequently, excessively high sintering temperatures impede the production of well-dispersed nano-powders^[37].

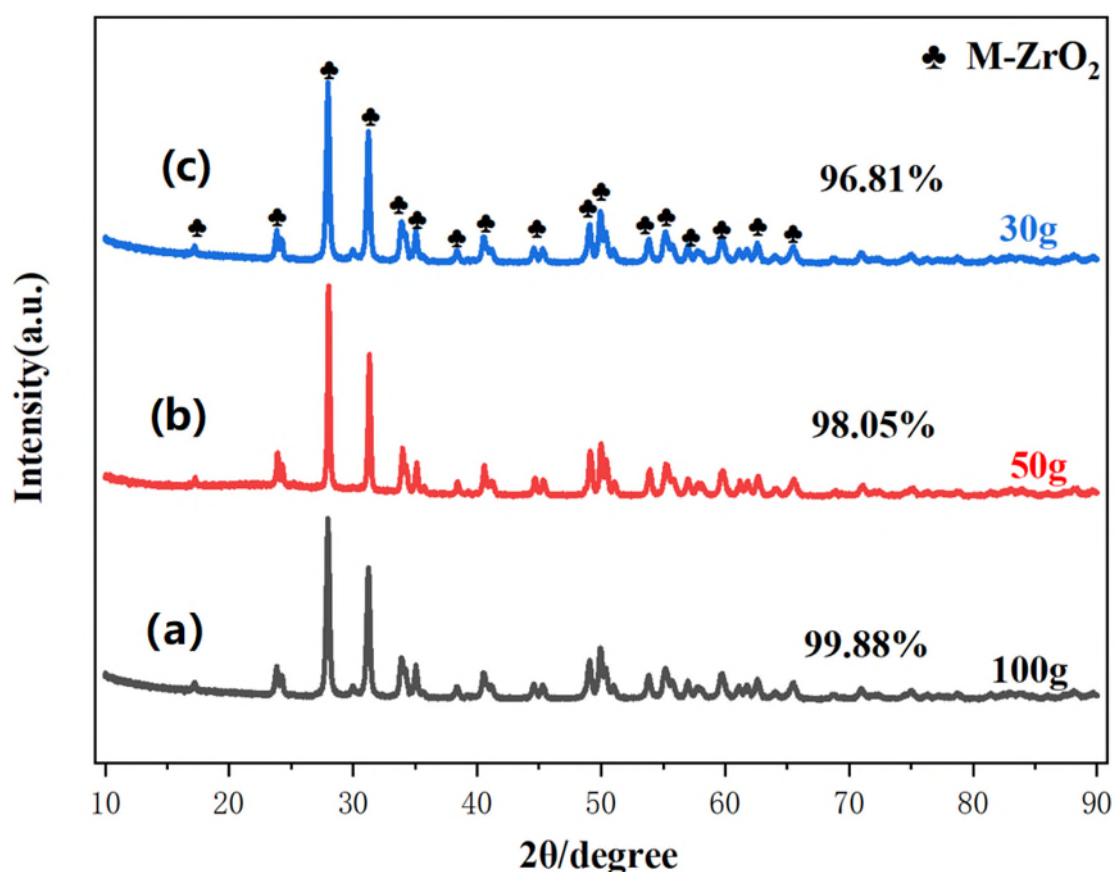


Fig. 5. XRD patterns of samples pyrolyzed to 900 °C with varying pyrolysis masses (a. 100 g, b. 50 g, C. 30 g)

Fig. 5 shows the XRD patterns of nano-zirconia samples synthesized with varying masses (30g, 50g, 100g). Obviously, the production can be completed under different synthetic mass (96.81% (30g), 98.05% (50g) and 99.88% (100g)). All samples pyrolyzed by microwave pyrolysis are composed of monoclinic phase (m-ZrO₂). When the mass is 30 g, the sample exhibits a lower diffraction peak and the FWHM values is

0.291 in [-111] crystal, indicative of reduced crystallinity. For a material mass of 50 g, the sample's diffraction peak heightens and its FWHM narrows to 0.334 in [-111] crystal, signifying a substantial increase in both crystallinity and conversion rate. For a sample mass of 100 g, there is a decrease in the diffraction peak height and the full width at half maximum (FWHM) value is 0.207 in [-111] crystal. Remarkably, the conversion rate of the sample reaches an impressive 99.81%, which may be attributed to uniform heating and overall pyrolysis^[38, 39].

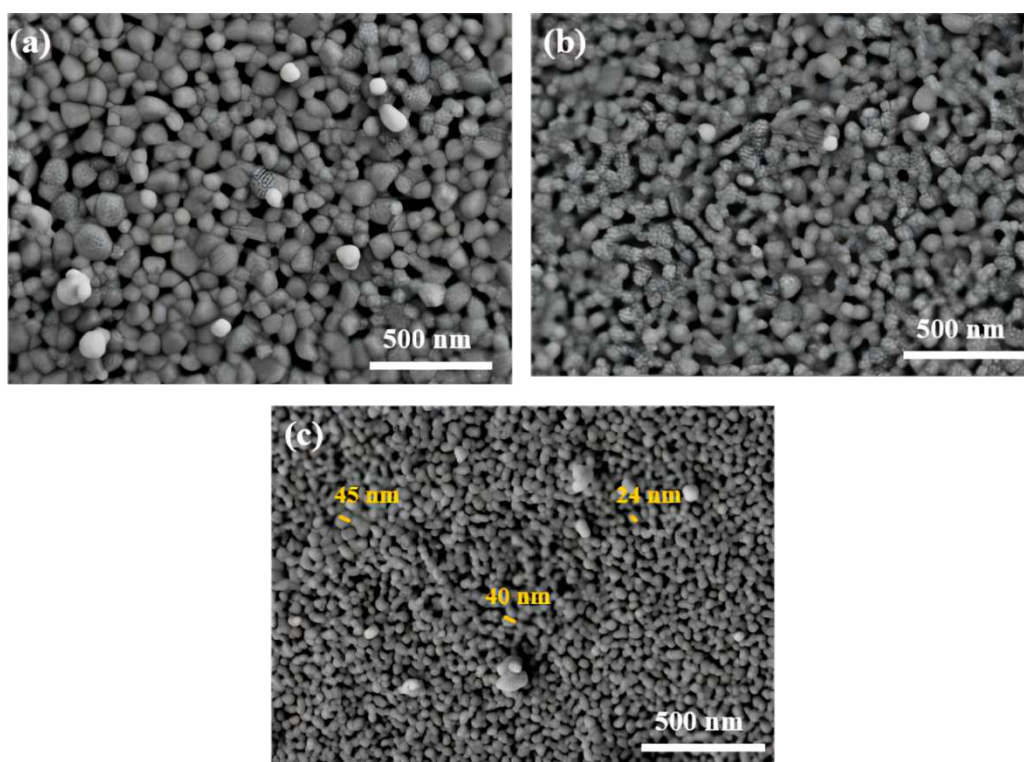


Fig.6. SEM images of samples pyrolyzed to 900°C with varying pyrolysis masses: (a) 30 g, (b) 50 g, (c) 100 g

Fig. 6 shows the morphology of nano-zirconia samples pyrolyzed with varying pyrolysis masses by microwave pyrolysis. The crystal morphology remains unchanged, consisting of spherical particles, indicating that the pyrolysis process has essentially concluded. Various levels of agglomeration phenomenon are observed.

As shown in Fig. 6 (a), the grain size about 57.40 nm for a pyrolysis mass of 30 g. Conversely, Fig. 6 (b) illustrates that with an increase mass of samples to 50 g, the grain size has notably reduced to 54.90 nm. Furthermore, it is evident that the dispersion of sphere is well. As shown in Fig. 6 (c), as the mass of the sample increases to 100 g, the grain size gets an optimal value of 21.38 nm. This phenomenon could potentially be attributed to the generation of uniform thermal field resulting from the higher mass.

3.2 Microwave Processing

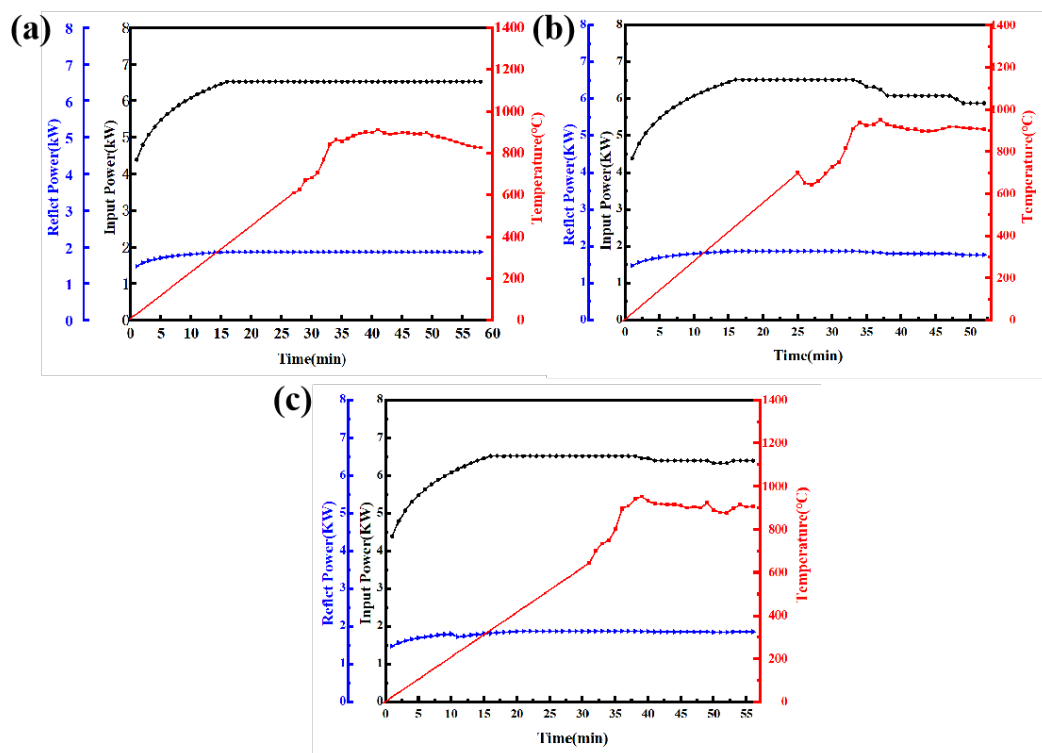


Fig. 7. Microwave heating curves of the samples with varying pyrolysis mass at 900 oC.(a) 30 g, (b) 50 g, (c) 100 g

Fig. 7 shows microwave heating curves of the samples with varying pyrolysis mass, which can be divided into three periods: preheating period, heating period, and holding period. Throughout the heating process, the sample with the mass of 30 g exhibits the higher reflection power, while the sample with the mass of 100 g shows the lower reflection power. During the preheating period of the pyrolysis process, the reflection power ratio of microwaves increases with the extension of pyrolysis time and input power of microwaves. The reflection power ratio exhibits a decrease after a duration of 14 minutes. The samples were heated to 600oC for varying durations: 35 min (30g), 26 min (50g), and 32 min (100g). During the heating period, the samples underwent a gradual temperature increase. The time required for the samples to reach a temperature of 900°C was 45 minutes (30g), 36 minutes (50g) and 33 minutes (100g). During the holding period, the reflection power ratio displayed different trends: a decrease in the reflection power ratio overall with less mass(30g), an increase in the reflection power ratio for the sample with a mass of 50g, and a relatively unchanged reflection power

ratio for the sample with higher mass(100g). The sample with a mass of 30g takes a longer time to reach 600oC, which due to heavily heat loss and poor coupling between the sample and microwave. The duration of heating is similar for all samples during the heating period. However, the sample with a mass of 100g requires more heat to reach 900°C. This indicates a better impedance matching between the 100g sample and the microwave field, resulting in increased heat generation. During the holding period, the grain growth and heat loss of the sample with a mass of 30g are obvious, requiring additional heat to maintain a constant temperature. Consequently, the reflection power ratio decreases. On the other hand, the sample with a mass of 50g shows an increase in the reflection power ratio, indicating a decrease in microwave energy utilization. Meanwhile, the reflection power ratio of the sample with a mass of 100g remains stable, indicating favorable impedance matching between the sample and the microwave field. During preheating period, the dielectric loss of ZrO_2 increases with the temperature rises. As a result, ZrO_2 exhibits efficient absorption of microwaves, whereby electromagnetic energy is converted into heat. This property is beneficial for both the nucleation and growth of ZrO_2 . As the sintering temperature continues to rise during the heating period, the dielectric loss of ZrO_2 increases, resulting in a higher conversion of microwave electromagnetic energy into heat energy. Consequently, the temperature rise of the sample depends on the dielectric loss characteristics of ZrO_2 .

3.3 Microwave mass-thermal effect

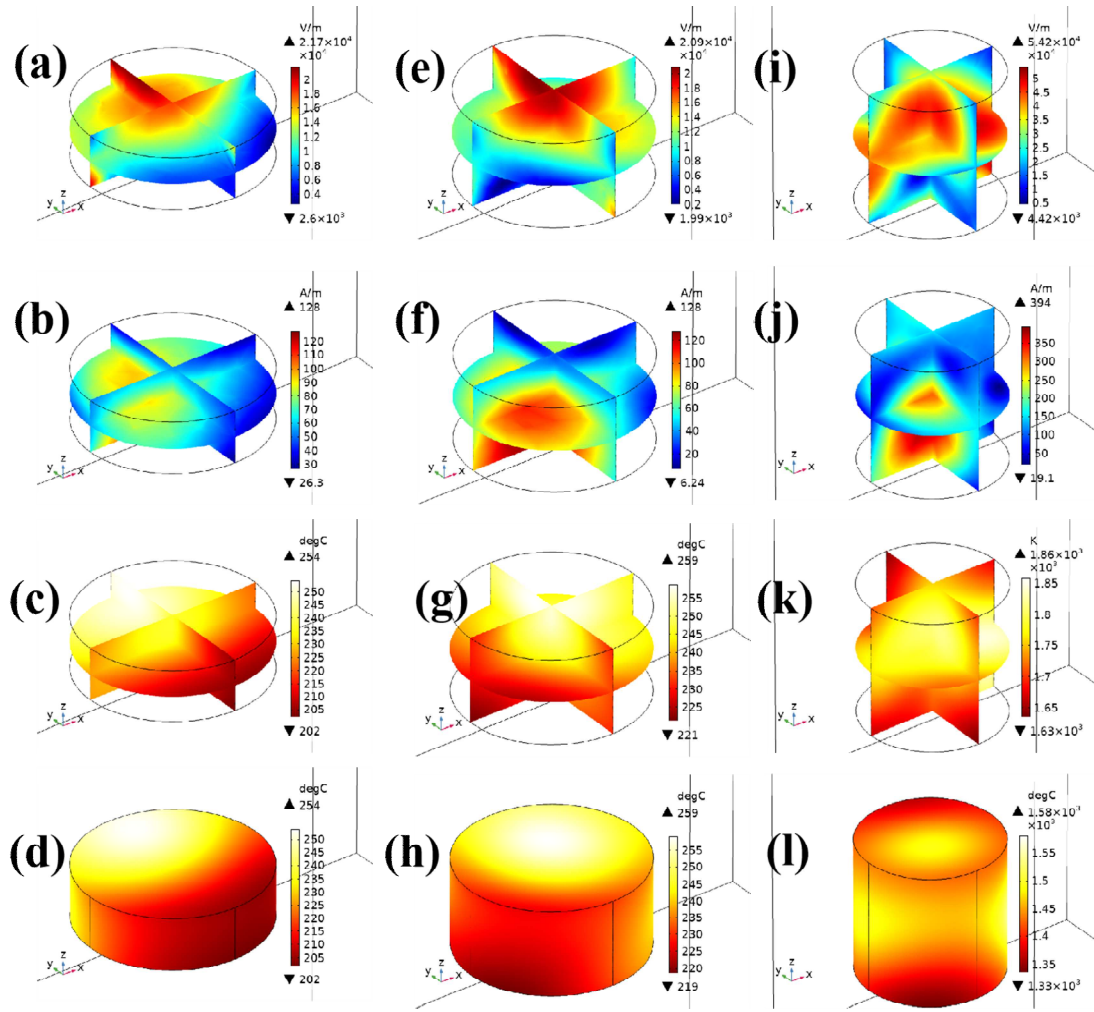


Fig. 8. the distribution of electric field, magnetic field, cross-section temperature and surface temperature of the sample by microwave heating (30 g mass (a, b, c, d), 50 g mass (e, f, g, h) and 100 g mass (i, j, k, l))

The interaction between ZrO_2 and the electric field, magnetic field, and heating field of microwave radiation was investigated using COMSOL Multiphysics software. The analysis reveals that the magnetic field has limited influence on the temperature field distribution due to the restricted permeability of zirconia. In contrast, the temperature field distribution aligns with the electric field distribution, highlighting the significant role of electric field coupling in the microwave heating of zirconia. Both the 30g and 50g samples are incapable of encompassing a complete electric field region, magnetic field region, and heating region. As a consequence, the center of the thermal field is not located at the center of the sample. This results in significant non-uniformity in temperature distribution and substantial heat dissipation. Whereas, the temperature distribution of the sample with the mass of 100g is relatively uniform due to its

favorable distribution of electric fields. As the mass increases, the central locations of the electric field and temperature field move closer to the interior of the sample, resulting in more uniform heating. This phenomenon occurs because a larger mass leads to a smaller relative specific surface area and reduces overall heat dissipation. The simulation results corroborate the experimental findings, confirming that a larger mass leads to heightened microwave efficiency during the pyrolysis process of nano-zirconia. The Fig. 9. shows the nucleation and growth process of zirconia with varying mass by microwave pyrolysis. The structure and grain size of zirconia are primarily governed by two factors: the heat loss of microwave energy and the impedance matching between the samples and the microwave source. When the raw material is 30g, the sample exhibits a large specific surface area, the conversion of microwave energy into heat is reduced, leading to higher heat loss than heat accumulation. Consequently, sample experiences a significant thermal gradient from the center to the edge. When raw material was increased to 50g, there was an increase in the conversion of microwave energy into heat, as well as a decrease in the ratio of the sample's surface area. This lower thermal gradient, from the center to the edge of the sample, was found to be advantageous in promoting the growth of zirconia grains. As the mass of the raw material increased to 100g, the ratio of the sample's surface area decreased, thereby optimizing the impedance matching between the sample and the microwave sources and contributing to the elimination of the thermal gradient temperature.

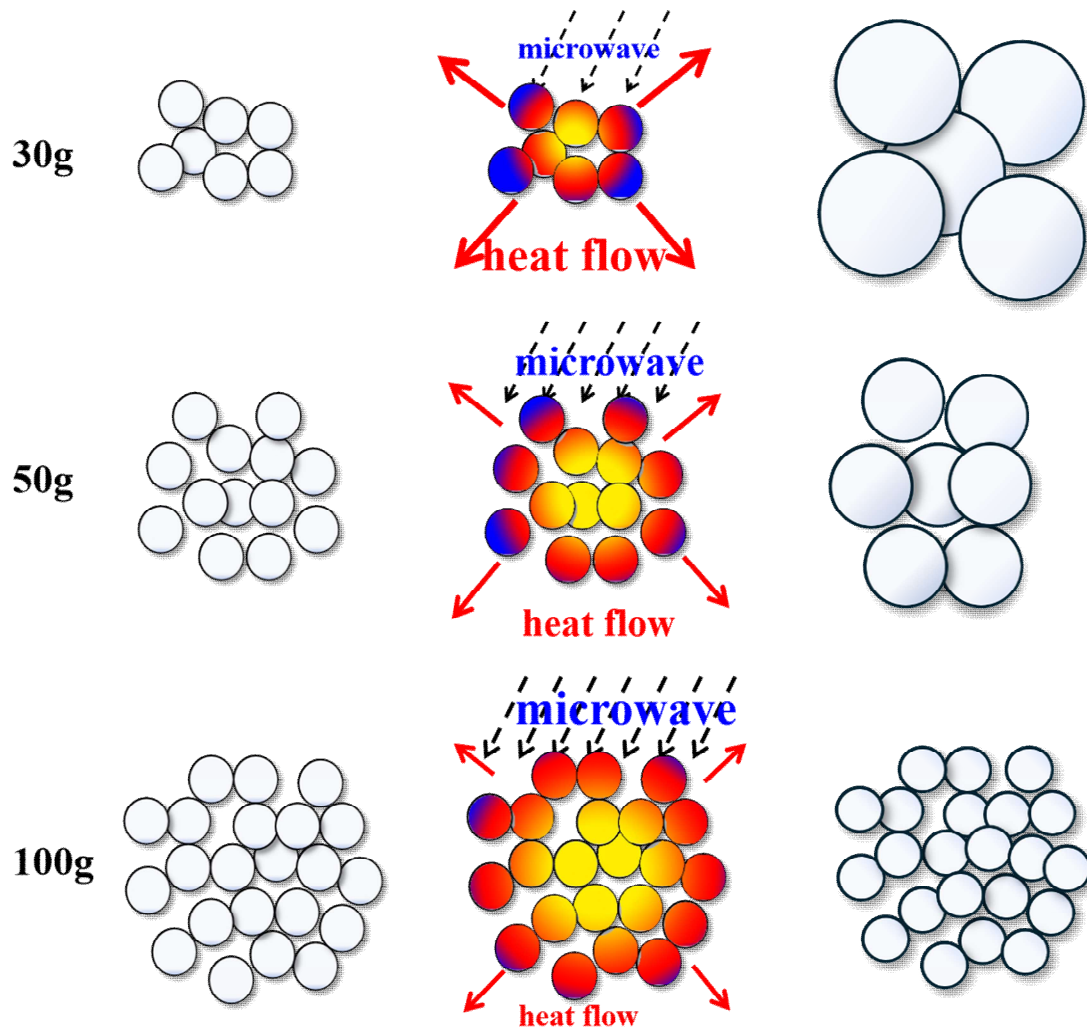


Fig. 9. Diagrammatic sketch of varying raw material mass by microwave pyrolysis

The balance of heat loss and heat generation resulting from impedance matching between the sample and the microwave sources leads to a more uniform heating process overall. As a result, the uniform temperature greatly facilitates the uniform growth of zirconia nucleation. Therefore, increasing the mass of the raw materials appropriately enhances the absorptivity of microwaves in microwave pyrolysis.

4. Conclusion

Nano-ZrO₂ powders were successfully synthesized by microwave pyrolysis over a temperature range of 700–1000 °C. The microwave method enabled complete crystallization of the precursor during the preparation of nano-zirconia at 700 °C, achieving a pyrolysis efficiency of 92.82%. When the precursor mass was increased to 100 g, the pyrolysis efficiency rose correspondingly to 99.88%. Notably, an interesting phenomenon, termed the microwave mass–thermal effect, was observed; it was

characterized by efficient and uniform heating in larger samples. Computer simulations confirmed this finding and attributed it to the balance between heat loss and heat generation resulting from impedance matching between the sample and the microwave source.

Author Contribution

Yaohong Liu: Writing – Original draft preparation, Formal analysis. **Qiancheng Gao:** Methodology, Writing - Reviewing and Editing, Supervision. **Zhaocai Zhou:** Data Curation. **Xinyuan Yang:** Data Curation. **Haojia Liu:** Writing – Original draft preparation. **Peiyue Li:** Data Curation. **Li Guan:** Methodology, Supervision. **Xinxing Liang:** Funding Acquisition. **Yongqiang Chen:** Validation. **Rui Zhang:** Supervision, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Ethics declarations

Ethics, Consent to Participate, and Consent to publish declarations

Not applicable.

Data availability

Data will be made available on request.

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