

**MICROWAVE SINTERING OF
ZIRCONIUM STABILIZED COMPOUNDS:
PHASE EVOLUTION AND HOT CORROSION STUDY**

BY

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DHAHRAN, SAUDI ARABIA

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Requirements for the Degree of

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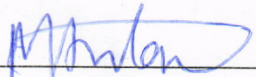
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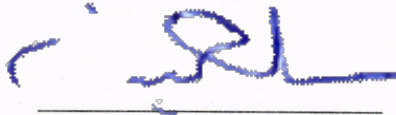
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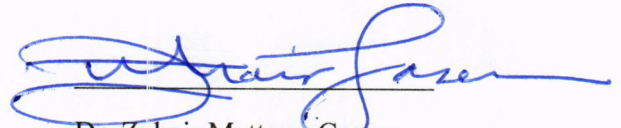
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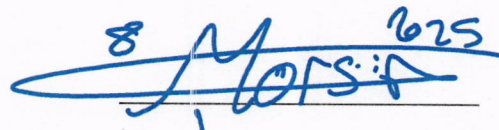
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2024

Dedicated to my parents and my sister for their endless love, support, and encouragement.

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LIST OF ABBREVIATIONS

8YSZ	:	8 wt% Ytria-Stabilized Zirconia
CSZ	:	Ceria-Stabilized Zirconia
YTiZ	:	Ytria-Titania-Zirconia
ZrO ₂	:	Zirconia
Y ₂ O ₃	:	Ytria
HMW	:	Hybrid Microwave Sintering
XRD	:	X-Ray Diffraction
SEM	:	Scanning Electron Microscopy
EDS	:	Energy-Dispersive Spectroscopy
TBCs	:	Thermal Barrier Coatings
SOFC	:	Solid Oxide Fuel Cells
HOSP	:	Hollow Oxide Spherical Process
m-ZrO ₂	:	Monoclinic Zirconia
t-ZrO ₂	:	Tetragonal Zirconia
FWHM	:	Full Width at Half Maximum
V ₂ O ₅	:	Vanadium Pentoxide
YVO ₄	:	Yttrium Vanadate

CeVO_4	:	Cerium Vanadate
TiO_2	:	Titanium Dioxide
CS	:	Conventional Sintering

ABSTRACT

Full Name : Mohammed Arif

Thesis Title : Microwave Sintering of Zirconium Stabilized Compounds: Phase Evolution and Hot Corrosion Study.

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This study explores the effects of hybrid microwave sintering (HMW) on the phase evolution, microstructure, thermal conductivity, and hot corrosion behavior of zirconium-stabilized compounds, specifically yttria-stabilized zirconia (8YSZ), ceria-stabilized zirconia (CSZ), and yttria-titania-zirconia (YTiZ). Samples were sintered at temperatures of 1350°C, 1400°C, and 1450°C to assess the benefits of HMW processing.

The results revealed that HMW sintering led to significant improvements in phase stability, achieving lower monoclinic phase content and better tetragonal phase retention across all sintering temperatures. For 8YSZ, the monoclinic phase content decreased to 4.13% at 1450°C, while CSZ exhibited a reduction to 2.6% at 1450°C. In YTiZ, optimal phase stability was achieved at 1350°C with a monoclinic phase content of 17.62%.

Thermal conductivity decreased with increasing sintering temperature, with the lowest values recorded as 0.0822 W/mK for 8YSZ at 1450°C, 0.4027 W/mK for CSZ at 1400°C, and 0.5964 W/mK for YTiZ at 1350°C. These reductions were attributed to the reduction in monoclinic phase percentage, leading to improved phase stability and refined grain structure.

Hot corrosion resistance was significantly enhanced in HMW-sintered samples, showing lower monoclinic transformation and reduced formation of deleterious secondary phases such as YVO_4 and CeVO_4 .

These findings demonstrate that hybrid microwave sintering offers superior results in terms of phase stability, microstructure, thermal performance, and corrosion resistance. This makes HMW a promising, energy-efficient alternative for processing zirconium-stabilized ceramics for high-temperature applications such as thermal barrier coatings and solid oxide fuel cells.

ملخص الرسالة

الاسم الكامل : محمد عارف
عنوان الرسالة : التلييد بالموجات الميكرويفي للمركبات المستقرة من الزركونيوم: دراسة تطور الطور والتآكل الحار
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تستكشف هذه الدراسة تأثيرات التلييد الميكرويفي الهجين (HMW) على تطور الطور والبنية الدقيقة والتوصيل الحراري وسلوك التآكل الساخن للمركبات المستقرة بالزركونيوم، وتحديدًا الزركونيا المستقرة بالإيتريا (8YSZ)، والزركونيا المستقرة بالسيرا (CSZ)، والزركونيا التيتانية التيتانية (YTiz). تم تلييد العينات عند درجات حرارة 1350 °C و 1400 °C و 1450 °C لتقييم فوائد معالجة HMW.

كشفت النتائج أن التلييد باستخدام HMW أدى إلى تحسينات كبيرة في استقرار الطور، وتحقيق محتوى طور أحادي الميل أقل واحتفاظ أفضل بالطور الرباعي عبر جميع درجات حرارة التلييد. بالنسبة لـ 8YSZ، انخفض محتوى الطور أحادي الميل إلى 4.13% عند 1450 °C، بينما أظهر CSZ انخفاضًا إلى 2.6% عند 1450 °C. في YTiz، تم تحقيق استقرار الطور الأمثل عند 1350 °C مع محتوى طور أحادي الميل بنسبة 17.62%.

انخفضت الموصلية الحرارية مع زيادة درجة حرارة التلييد، مع تسجيل أدنى القيم عند 0.0822 W/mK لـ 8YSZ عند 1450 °C، و 0.4027 W/mK لـ CSZ عند 1400 °C، و 0.5964 W/mK لـ YTiz عند 1350 °C. وتعزى هذه التخفيضات إلى انخفاض نسبة الطور أحادي الميل، مما يؤدي إلى تحسين استقرار الطور وبنية الحبوب المكررة. تم تعزيز مقاومة التآكل الساخن بشكل كبير في العينات الملبدة بـ HMW، مما يدل على انخفاض التحول أحادي الميل وانخفاض تكوين المراحل الثانوية الضارة مثل YVO_4 و $CeVO_4$.

تثبت هذه النتائج أن التلييد بالميكروويف الهجين يوفر نتائج متفوقة من حيث استقرار الطور والبنية الدقيقة والأداء الحراري ومقاومة التآكل. وهذا يجعل HMW بديلاً واعدًا وكفؤًا في استخدام الطاقة لمعالجة السيراميك المستقر بالزركونيوم للتطبيقات ذات درجات الحرارة العالية مثل الطلاءات الحاجزة الحرارية وخلايا الوقود ذات الأكسيد الصلب.

CHAPTER 1

INTRODUCTION

1.1 Background

The development of advanced materials that can withstand high temperatures is an important field of research in materials science and engineering [1]. Zirconium stabilized compounds, in particular zirconia (ZrO_2) stabilized with yttria (Y_2O_3) and other oxides (such ceria and titania), have attracted a lot of interest among these materials because of their exceptional mechanical, chemical, and thermal stability [2]. These materials are frequently employed in solid oxide fuel cell (SOFC) components, thermal barrier coatings (TBC) in gas turbines, as well as aeronautical engineering, among other high-temperature applications [3]. They are extremely valuable in industries that require great performance under harsh conditions because of their resilience to thermal shock, capacity to sustain high strength at increased temperatures, and ability to withstand corrosive environments [4].

Conventional sintering methods like sinter forging, hot pressing, hot isostatic pressing, etc. is one of the traditional processes used to manufacture these advanced ceramics. It involves exposure to higher temperatures for long duration of time, which might result in large energy consumption and inconsistencies in the material properties. Microwave sintering is a novel approach that has the potential to improve the sintering process's efficiency and results. In contrast to traditional techniques, microwave sintering generates heat internally

in the material using electromagnetic radiation. This can result in faster heating, shorter sintering times, and possibly more homogeneous microstructures. This novel method seeks to enhance the mechanical and physical properties of the sintered compounds in addition to offering energy savings [5,6].

Microwave sintering has several potential benefits, however there are few thorough investigations examining how it affects the phase evolution and hot corrosion behavior of zirconium stabilized compounds. The mechanical characteristics and stability of the material are profoundly impacted by phase evolution changes in the crystal structure and phase composition during sintering [7]. In a similar vein, knowing how hot corrosion behavior works is essential to guaranteeing the durability and dependability of these materials in the intended uses. Hot corrosion behavior entails material degradation as a result of reactions with corrosive chemicals at high temperatures [4].

By methodically examining the microwave sintering of zirconium stabilized compounds and concentrating on the development of their phase composition and resistance to hot corrosion under industrially simulated conditions, this thesis aims to acknowledge this gap. This thesis aims to identify the fundamental mechanisms guiding phase transition and corrosion processes in ceramics stabilized with zirconium that have been sintered using microwaves. In addition to advancing our understanding of these materials scientifically, this research aims to lay a strong foundation for the development of materials for high-temperature materials.

1.2 Microwaves

Microwaves are a subset of electromagnetic (EM) waves, with frequencies ranging from 300 MHz to 300 GHz. The propagation of EM waves, described by Maxwell's equations, involves electric and magnetic fields oscillating perpendicular to each other and the direction of wave travel. Microwaves occupy the spectrum between radio waves and infrared waves (figure 1.1(a)) and are widely utilized in various fields for their unique properties of energy transmission and interaction with matter. James Clerk Maxwell's seminal work on electromagnetic theory in 1864 laid the foundation for understanding how electromagnetic waves function, contributing to significant technological advancements.

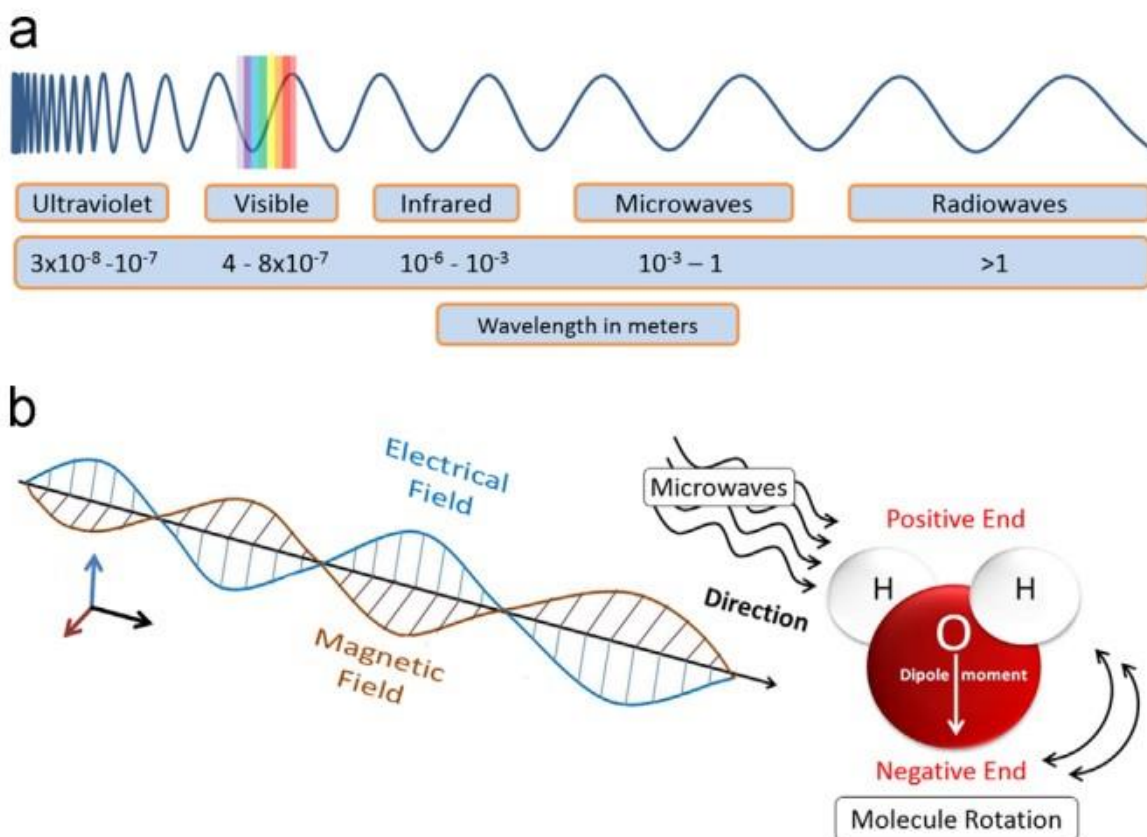


Figure 1. 1 Schematic presentation for the electromagnetic (EM) spectrum in the range around MW (a), and (b) MW interacting with a dielectric molecule (e.g. water molecule) due to its intrinsic dielectric properties [8]

The historical development of microwaves began with Faraday's discovery of electromagnetic induction in 1831, followed by Maxwell's electromagnetic field theory. Heinrich Hertz's experiments in the late 1880s confirmed the existence of electromagnetic waves, while Jagadish Chandra Bose extended these findings in the 1890s with high-frequency experiments. During World War II, the development of microwave technology accelerated due to its critical role in radar systems. The invention of the high-power cavity magnetron was a pivotal breakthrough, enabling efficient generation of microwaves for military and communication purposes.

Microwave (MW) energy processing, when effectively utilized, offers numerous advantages over traditional methods. It has been successfully applied across various fields to process materials such as coal, oxide and carbide ceramics, composites, cement, concrete, alloys, fly ash, and even for CO₂ adsorption. Additionally, MW energy has been employed in heating and sintering metals and alloys. Early research demonstrated its use for heating metals, while more recent studies have explored its application in joining metals and alloys. However, maximizing its potential requires a thorough understanding of the interactions between MW energy and materials [9].

The ability of a material to absorb microwave (MW) energy is largely determined by the amount and rate at which the energy is absorbed. The rate of MW energy absorption is typically represented as power per unit volume, which can be mathematically expressed using the following equation[8].

$$P_a = \omega \varepsilon_o \varepsilon_{eff}'' E_{rms}^2 + \omega \mu_o \mu_{eff}'' H_{rms}^2 \quad 1.1$$

where the term $(\omega \epsilon_0 \epsilon''_{\text{eff}} E_{\text{rms}}^2)$ is related to electric losses and the term $(\omega \mu_0 \mu''_{\text{eff}} H_{\text{rms}}^2)$ is related to magnetic losses. The other terms are defined as follows: ω is the angular frequency, ϵ_0 is the permittivity of free space, ϵ''_{eff} is the effective relative dielectric loss, E_{rms} is the root mean square of the internal electric field, μ_0 is the permeability of free space, μ''_{eff} is the effective relative magnetic loss, and H_{rms} is the root mean square of the magnetic field strength.

The effective relative dielectric loss can be expressed as shown below in Equation[8]

$$\epsilon''_{\text{eff}} = \epsilon''_{\text{c}} + \epsilon''_{\text{s}} + \epsilon''_{\text{d}} + \epsilon''_{\text{i}} + \epsilon''_{\text{e}} \quad 1.2$$

where ϵ''_{c} is the loss due to DC conductivity and the last four terms represent dielectric losses due to polarization mechanisms, where ϵ''_{s} is for space charge (interfacial); ϵ''_{d} is for dipolar; ϵ''_{i} is for ionic, and ϵ''_{e} is for electronic components.

Microwave (MW) energy can be absorbed by materials through either polarization or conduction mechanisms. Polarization involves the short-range displacement of charges through the formation and rotation of electric dipoles (or magnetic dipoles, when present), while conduction requires the transport of charges over a relatively longer range compared to dipole rotation. Both mechanisms contribute to absorption losses within specific frequency ranges (Figure 1.2). These absorption losses, also known as dielectric losses (ϵ''), arise from ionic conduction, which is more prominent at lower frequencies, and the rotation of permanent dipoles, which dominates at higher frequencies. As a result, the electric field generated within the material can induce ionic motion or molecular dipole reorientation. The resistance to these induced motions leads to energy losses, causing attenuation of the electric field and the generation of heat within the material. Additionally, an increase in

temperature raises the kinetic energy of the dipoles, making them more responsive to the oscillating field and shifting the absorption peaks toward higher frequencies.

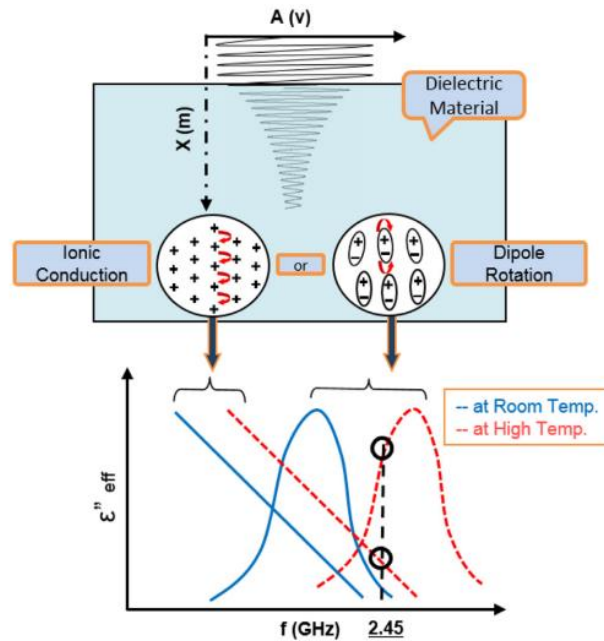


Figure1.2 The absorption mechanisms that can contribute to ϵ''_{eff} . Note that a material may be poorly absorbing MW energy at room temperature and 2.45 GHz but highly absorbing at high temperature. Terms: $x \equiv$ distance; $A \equiv$ amplitude of the electric field; $\epsilon''_{\text{eff}} \equiv$ effective dielectric loss; $f \equiv$ frequency [10]

Microwaves are a highly efficient energy source for material processing, offering advantages over conventional heating techniques. Unlike conventional technologies, which heat materials from the surface via conduction and convection, MW energy enters and interacts directly at the molecular level, resulting in uniform and quick volumetric heating. This interaction occurs through polarization, where electric or magnetic dipoles align with the oscillating MW electric field, and conduction, where long-range ionic motion generates heat. Energy absorption efficiency is determined by MW frequency, material dielectric characteristics, and electric field distribution. MW heating is very effective on polar substances, allowing for selective heating, superheating, and the potential development of novel material phases[11]. A common operating frequency of 2.45 GHz is used due to

water's strong absorption at this frequency. To overcome limitations in penetration depth, hybrid MW heating combines conventional heat sources, such as electric furnaces or susceptors, to achieve more uniform temperature distribution. This approach has shown success in enhancing microstructure and mechanical properties in materials such as zirconia-based ceramics. Despite certain limitations, MW-assisted synthesis presents a promising alternative to energy-intensive resistance heating, with growing potential for applications in materials science and biomaterials processing [10].

Microwaves have found applications in various fields. In material processing, they are used for sintering, curing, and heating of metals, ceramics, and polymers. Their ability to penetrate materials selectively enables rapid and energy-efficient heating. In communication, microwaves are used extensively in satellite transmission, wireless LAN (Wi-Fi), and Bluetooth technologies. For instance, the use of 2.4 GHz and 5 GHz frequency bands enables seamless wireless data transmission across devices. Radar systems, essential for navigation and defense, use microwaves to detect objects, track velocity, and guide navigation. In the medical field, microwaves are employed in hyperthermia treatments for cancer and as power sources in medical linear accelerators for radiology treatment.. The evolution of microwave processing of materials across different temperature ranges and timelines are shown in figure 1.2. From 1950 to 1970, microwave technology was primarily used for low-temperature applications ($T < 500^{\circ}\text{C}$) such as polymer processing, food drying, and chemical and medical applications. Between 1970 and 1999, it expanded to moderate-temperature processing ($500^{\circ}\text{C} < T < 1000^{\circ}\text{C}$), including sintering and glazing of ceramics, metal joining, and carbon nanotube (CNT) synthesis. Since 1999, microwave processing has been applied to high-temperature applications ($T > 1000^{\circ}\text{C}$), such as

sintering of high-density ceramics and metal matrix composites (MMCs), nano-material synthesis, and cladding of tungsten carbide and silicon carbide. This progression highlights how microwave technology has advanced from basic drying processes to cutting-edge applications in materials science and engineering.

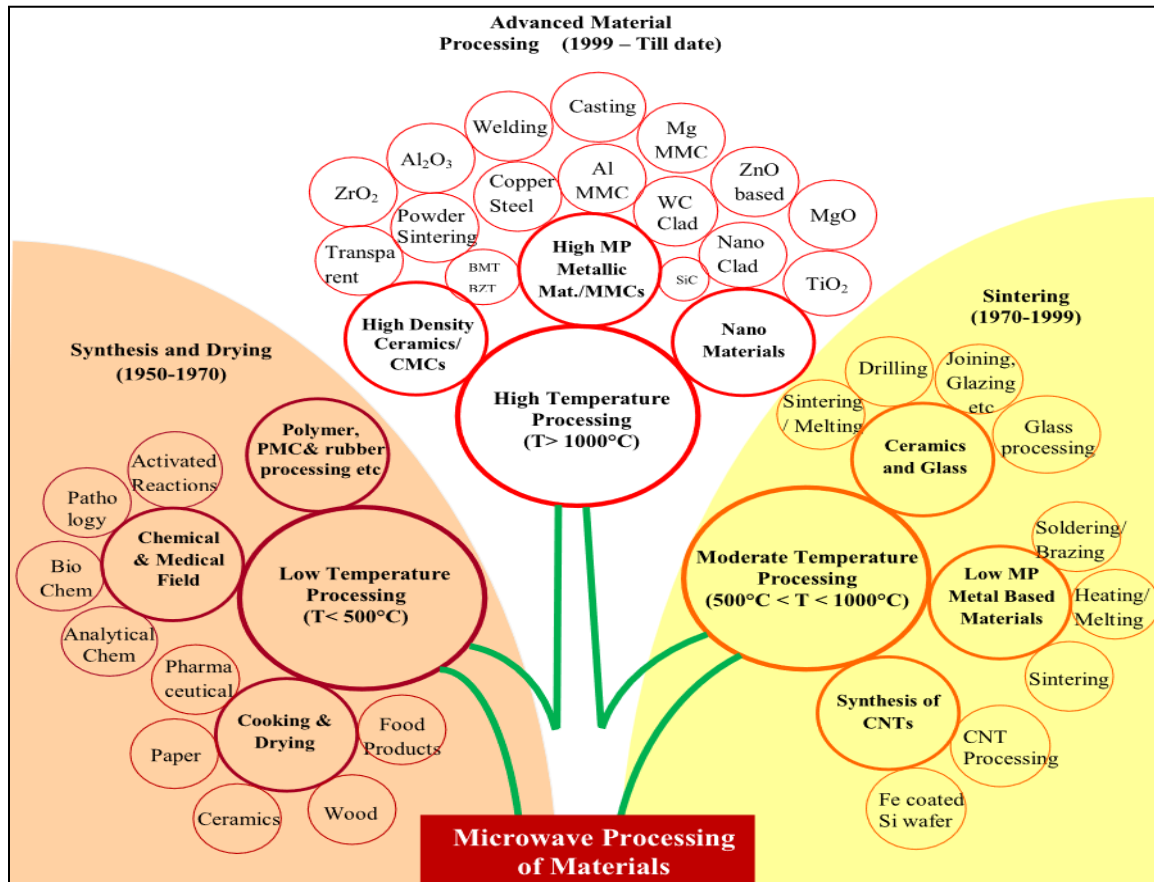


Figure 1. 3 Applications of MW Processing Materials [12]

Microwave frequency allocation is regulated to prevent interference in various applications. The industrial, scientific, and medical (ISM) frequency bands—such as 915 MHz, 2.45 GHz, and 5.8 GHz—are commonly used in everyday devices, including microwave ovens and medical equipment. The generation of microwaves is made possible through devices such as magnetrons, klystrons, and gyrotrons. Magnetrons, widely used in domestic microwave ovens and radar systems, convert electrical energy into microwave

radiation. Klystrons are employed for high-power applications such as particle accelerators and communication satellites, while gyrotrons are used for plasma heating in nuclear fusion research due to their capability to produce high-power millimeter-wave radiation

1.3 Microwave Sintering

Microwave sintering is an efficient process for material processing by which heat energy is induced within the material using electromagnetic waves. Therefore, microwave sintering is the volumetric heating and produces a more uniform temperature distribution and the densification rate in microwave heating is larger than conventional heating by conduction or convection.

For the zirconium-stabilized compound class, there is the possibility of improving phase stability and the performance of the material through hybrid microwave sintering. It will minimize hotspot conditions and gradients meaning better control of phase and density. The sintering parameters of microwave power, exposure time, and the processing atmosphere have proved distinctive advantages in modifying material properties.

In addition, microwave sintering has been effectively used on numerous ceramics and composites. However, its influence on the microstructure, phases as well as mechanical properties of zirconium-stabilized compounds is further investigated. Knowledge of these parameters is crucial to improving the microwave sintering technology for high-performance part applications.

1.4 Hot Corrosion Study

Hot corrosion is a severe form of material degradation that occurs in high-temperature environments in the presence of aggressive agents such as molten salts, oxidizing gases, and contaminants. This behavior is particularly undesirable in those materials that are subjected to severe operating conditions like gas turbines, boilers, and chemical reactors. For zirconium-stabilized compounds, which are often used in such applications, resistance to hot corrosion is a critical performance metric.

The mechanisms of hot corrosion typically involve the reaction of molten salt V_2O_5 with the material surface, resulting in the formation of non-protective scales and subsequent material degradation. Factors such as temperature, material composition, and processing conditions influence the extent of corrosion damage.

When zirconium-stabilized compounds are known for their corrosion resistance, the impact of microwave sintering on this property has not been thoroughly examined. Microwave sintering's potential to enhance microstructural uniformity and phase stability may also influence the material's resistance to hot corrosion. This study aims to bridge this knowledge gap by systematically evaluating the hot corrosion behavior of microwave-sintered zirconium-stabilized compounds.

1.5 Objectives and Approach

1.5.1 Motivation

The motivation for this study stems from the need for advanced materials that can withstand extreme environmental conditions without compromising performance. There

are already numerous promising examples of stabilized zirconium compounds that could be of great use in this respect, but their full potential remains untapped until optimized processing of the materials is discovered. Despite the advantages microwave sintering poses to industrial applications, the paucity of research on its impact on phase development and corrosion properties remains a hurdle to its application on a large scale.

Furthermore, knowledge of hot corrosion processes in materials sintered using microwave technology will help the development of materials with higher endurance and reliability. This combination makes available a new way to discuss some key questions in material science and engineering connected to the phase evolution and hot corrosion.

1.5.2 Objectives

The primary objectives of this research are:

1. To **investigate** the phase evolution of zirconium-stabilized compounds during hybrid microwave sintering.
2. To **evaluate** the hot corrosion behavior of these materials in aggressive high-temperature environments (i.e. at 900 °C).
3. To **establish** correlations between various sintering temperatures (i.e. 1350 °C, 1400 °C, and 1450 °C), their phase stability as well as corrosion resistance.
4. To **propose** an optimized sintering temperature for enhancing the performance of zirconium-stabilized compounds like YSZ, CSZ, and YTiZ.

1.5.3 Approach

The study adopts a systematic approach to achieve these objectives:

1. Experimental Design: Zirconium-stabilized compounds will be processed using hybrid microwave sintering under 1350 °C, 1400 °C, and 1450 °C.
2. Characterization: Advanced characterization techniques, such as X-ray diffraction (XRD), and scanning electron microscopy (SEM) will be employed to analyze phase composition, microstructure, and elemental distribution.
3. Thermal Conductivity: Before hot corrosion thermal conductivity was also measured.
4. Hot Corrosion Test: The sintered materials will be subjected to hot corrosion tests in simulated environments containing V_2O_5 and oxidizing gases at 900 °C.
5. Data Analysis: The results from phase analysis and corrosion testing will be correlated to identify key factors influencing material performance.
6. Optimization: Based on the findings, recommendations will be made for optimizing microwave sintering parameters to achieve the desired material properties. |

CHAPTER 2

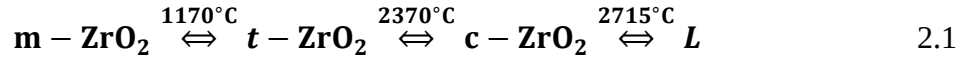
LITERATURE REVIEW

Zirconium-stabilized compounds are famous in regards to their high-temperature stability, mechanical strength, and corrosion resistance which make them useful in temperature and corrosive applications. The demand for higher performances in applications like thermal barrier coatings, solid oxide fuel cells, and aerospace has put a pace for the enhancement of the properties of these materials. Of all the processing techniques, microwave sintering has received a lot of attention because of its energy efficiency together with fast heating ability in sintering materials having improved microstructural features.

This chapter reviews the existing literature on the properties of zirconium-stabilized compounds, the importance of microwave sintering over conventional sintering, the principles and techniques of microwave sintering, phase evolution during sintering, and the influence of microwave sintering on hot corrosion behavior. The discussion emphasizes the limitations and opportunities associated with the processing of zirconium-stabilized compounds.

2.1 Zirconium-Stabilized Compounds

At room temperature (RT), zirconia (ZrO_2), a representative ceramic material, exhibits a monoclinic crystal structure that can undergo a martensitic transformation into the tetragonal phase with heating at approximately 1170°C , resulting in a volume change of about 3-4% in the unit cell [13]. Furthermore, it can be stabilized in the tetragonal phase within a temperature range of 1170°C to 2370°C [14]. Finally, it can transform into another higher-temperature cubic phase up to the melting point of ZrO_2 at $\sim 2715^\circ\text{C}$ [15] illustrated in the below equation 2.1.



The distinctive properties and applications of zirconia offer the stabilization of high-temperature phases at room temperature by the incorporation of various oxides and alloying elements, including but not limited to ceria (CeO_2), titania (TiO_2), calcium oxide (CaO), magnesia (MgO), and yttria (Y_2O_3) [16]. Yttria-stabilized zirconia (YSZ) is basically composed of two ceramics oxides, ZrO_2 and Y_2O_3 , where the latter serves as a dopant in varied quantities to stabilize the high-temperature phases at room temperature [17,18], as illustrated in Figure 2.1 of ZrO_2 - Y_2O_3 phase diagram [19]. The three (3) possible phases of zirconia with its space group information are shown in the Table 2.1.

Table 2. 1 Different phases of ZrO_2

Material	Crystal Structure	Symbol	Space group
ZrO_2	cubic (fluorite)	c- ZrO_2	Fm-3m
	tetragonal	t- ZrO_2	P4 ₂ /nmc
	monoclinic (baddeleyite)	m- ZrO_2	P2 ₁ /c

Figure 2.2 shows the thermal conductivity of various polycrystalline oxides as a function of temperature. The thermal conductivities are in the range 1 to 30 (W/m K), the values

decreasing with increasing temperature. ZrO_2 exhibits the lowest thermal conductivity among the various oxides with no significant dependence on temperature and as such, ZrO_2 -based ceramics have emerged as the first materials of choice for TBC applications. ZrO_2 in its stabilized state possessed most of these properties.

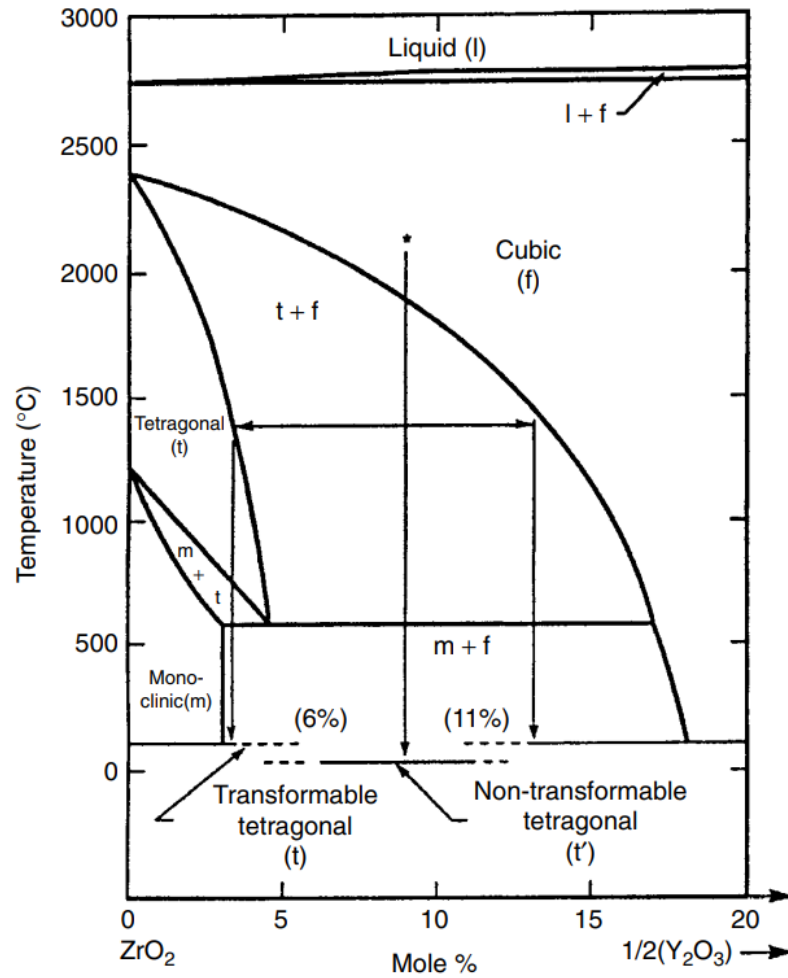


Figure 2. 1 Phase diagram of ZrO_2 - Y_2O_3 [19]

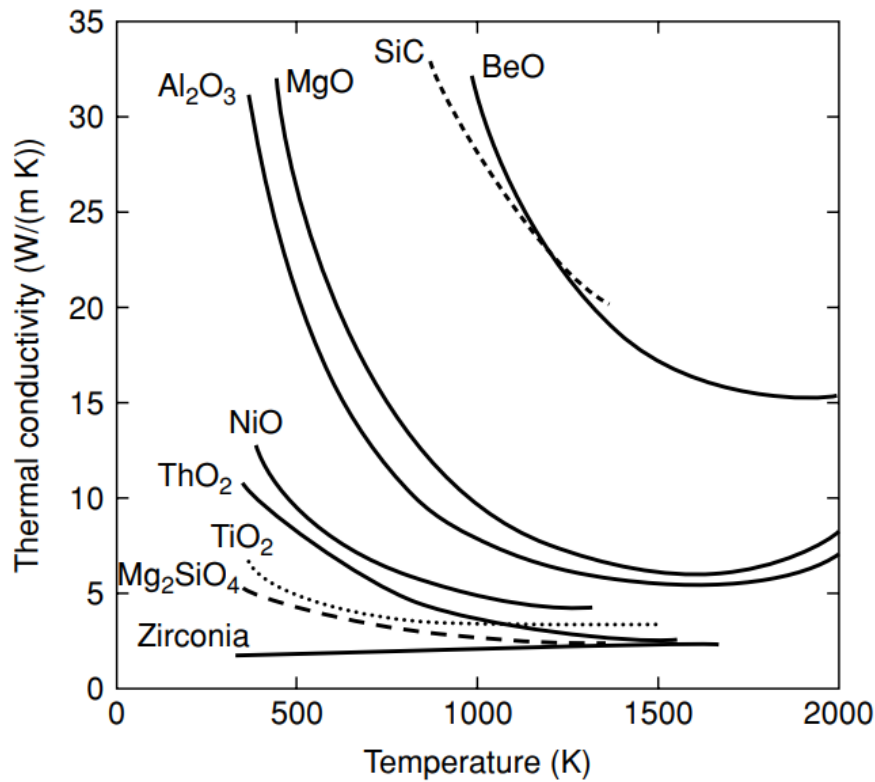


Figure 2. 2 The thermal conductivity of various polycrystalline oxides as a function of temperature [20]

2.1.1 Properties and Applications of Zirconium-Stabilized Compounds

Zirconium-stabilized compounds are renowned for their high thermal stability, resistance to phase degradation, as well as excellent mechanical properties. The addition of stabilizing agents such as yttrium improves the material's ability to withstand thermal cycling and suppress undesirable phase transformations at higher temperatures. These properties make them especially desirable for high-temperature applications. More applications are mentioned in the Table 2.2.

Table 2. 2 Applications of Zirconium based samples

Compound	Key Properties	Applications	References
Yttria-Stabilized Zirconia (YSZ)	High ionic conductivity, low thermal conductivity, excellent thermal shock resistance, high fracture toughness	Thermal Barrier Coatings (TBCs), Solid Oxide Fuel Cells (SOFCs), Biomedical implants, Oxygen sensors, Refractories	[21,22]
Ceria-Stabilized Zirconia (CSZ)	High oxygen storage capacity, excellent resistance to thermal cycling, superior fracture toughness	Catalytic converters, Structural ceramics, Dental applications, Electrochemical applications, Refractories	[23–26]
Yttria-Titania Stabilized Zirconia (YTiZ)	Enhanced corrosion resistance, improved phase stability at high temperatures, superior wear resistance	Aerospace coatings, Thermal spray coatings, Nuclear industry, SOFCs, High-temperature sensors, Wear-resistant coatings	[27–30]

2.2 Microwave Sintering

Microwave heating has its roots in Maxwell's electromagnetic theory developed in 1864. Initially, it was applied in radar and communication systems, with its potential for therapeutic uses recognized as early as the 1930s. Industrial applications gained momentum after World War II, with processes like drying rubber and textiles demonstrating the efficiency of this technology. The pivotal innovation came with Percy

Spencer's patent in 1945, which led to the development of the microwave oven, making the technology commercially available in the 1960s [31].

2.2.1 Fundamentals of Microwave Sintering

Microwave sintering is a modern material processing method which employs electromagnetic waves to heat substances via dielectric loss mechanisms. Unlike conventional sintering methods, which depend on external heat sources that heat from the surface, microwave sintering generates heat volumetrically within the material. This process results in faster and more uniform heating, significantly reducing thermal gradients and minimizing the potential for defects like warping or cracking in the sintered material [32].

The process begins with the material absorbing microwave energy, resulting in the oscillation of polar molecules or free charges within the material, so generating heat. This direct heating effect leads to uniform temperature distribution throughout the material, in contrast to traditional methods where heat is conducted from the outside in. It is represented in the Figure 2.3

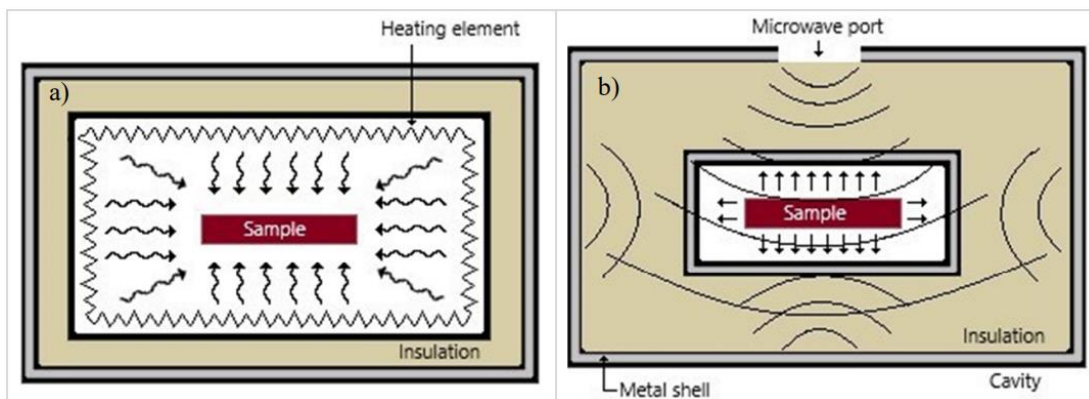


Figure 2. 3 Schematic representation of traditional sintering and microwave sintering

The efficiency of microwave sintering makes it an attractive option for materials that require high-temperature processing, like zirconium-stabilized compounds, as it helps to preserve the desired phase structure and enhances the mechanical properties of the material.

Advantages of Microwave Sintering[33]:

- **Volumetric Heating:** Achieves uniform temperature distribution, reducing the risk of thermal stresses and cracks.
- **Energy Efficiency:** Focuses energy directly on the material, minimizing wasted heat and reducing processing time.
- **Faster Processing Times:** Significantly shortens sintering cycles due to rapid heating rates.
- **Improved Material Properties:** Provides finer microstructures with reduced porosity, contributing to superior mechanical and thermal properties.

Mechanisms of Microwave Heating[33]:

- **Dipole Rotation:** Polar molecules rotate in response to the alternating electromagnetic field, generating heat due to molecular friction.
- **Ionic Conduction:** Charged particles within the material move in response to the electric field, producing localized heating.
- **Joule Heating:** Localized current paths generate resistive heat in conductive materials.

2.2.2 Types of Microwave Heating

Microwave heating can be categorized into two primary types based on the approach and material properties: pure microwave heating and hybrid microwave heating.

Pure Microwave Heating

This type of heating involves the direct exposure of materials to microwave energy. It is particularly effective for materials with high dielectric loss factors, which can absorb microwaves efficiently and convert them into heat. The energy transfer is highly efficient, leading to uniform heating and minimal energy wastage. Pure microwave heating is commonly used in applications such as food processing, chemical synthesis, and polymer curing, where materials exhibit high microwave absorption properties[34].

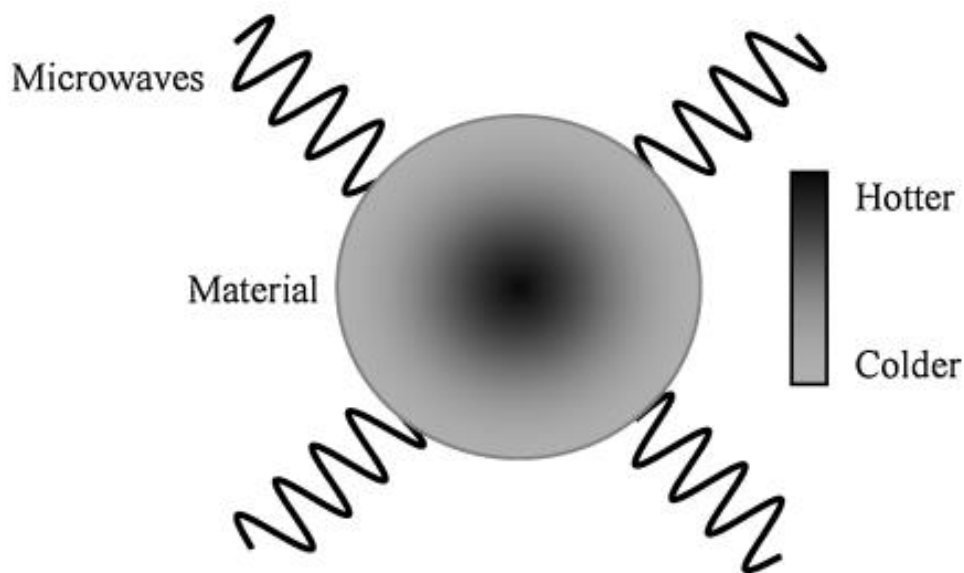


Figure 2. 4 Schematic representation of MW heating [31]

Hybrid Microwave Heating

Hybrid microwave heating combines the advantages of both microwave and conventional heating methods. In this approach, materials are simultaneously exposed to microwave energy and external heat sources, such as resistive or convective heating. This method is particularly useful for materials with low microwave absorption capabilities. The susceptor ensures consistent heating throughout the sample, addressing the challenges of uneven microwave absorption in large volumes.

The hybrid approach often enhances the physical and mechanical properties of processed materials, such as improved hardness, densification, or structural uniformity. Applications include sintering of ceramics, metal compacts, and composite materials, where achieving consistent heating is critical.

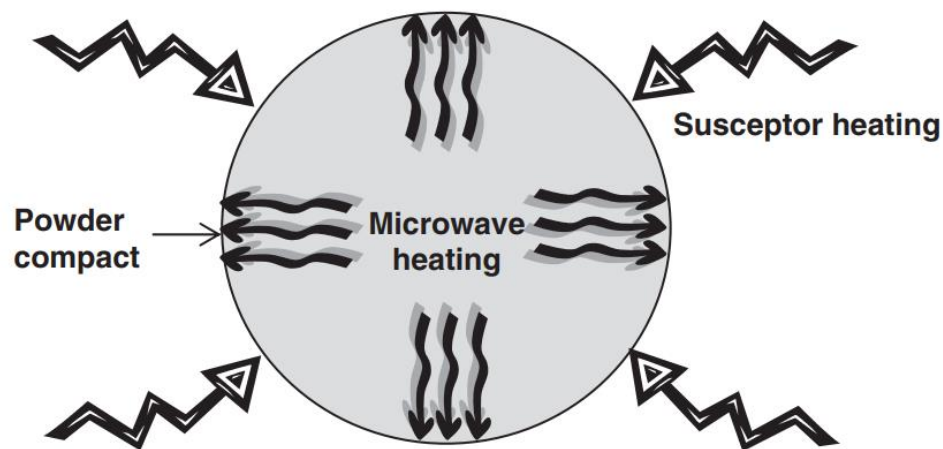


Figure 2. 5 Schematic representation of HMW heating [35]

Hybrid microwave heating (Figure 2.3) was developed to address the limitations of direct heating by integrating susceptors (materials with a high dielectric loss factor) with masking

agents. This approach involves heating the targeted substance in three phases: (1) microwave absorption, (2) conventional heating, and (3) microwave heating of the processed material. The heating methods are dictated by the properties of the target material [36].

2.2.3 Advantages of Microwave Sintering over Conventional sintering

Barchetta et al., highlights the efficiency and effectiveness of Comparing microwave sintering (MS) to conventional sintering (CS) for 3Y-TZP dental ceramics. MS at 1450°C for 15 minutes produced similar density and mechanical strength to CS at 1530°C for 2 hours, while also yielding smaller grain size. Moreover, MS significantly reduced total sintering time to 105 minutes from 481 minutes with CS, offering potential energy and cost savings without compromising material quality. This suggests that MS, followed by rapid cooling, presents a promising protocol for sintering Y-TZP dental ceramics[37].

Nightingale et al., performed a study to examine the effects of microwave heating on the densification and grain growth of samples containing 3 mol percent yttria-stabilized zirconia. The samples were compared to those that underwent conventional sintering. It was discovered that densification procedures during constant-rate heating were enhanced by microwave heating. When comparing the microwave-sintered specimens to the traditionally sintered samples, they noticed a kind of shift in the sintering process for values of less than 96% of the theoretical density, in the direction of increasing density [38]

Mazaheri et al. mentioned how the sintering technique and the conformation method (slip casting and dry pressing) affect the grain development and densification of Nano-crystalline 8 mol percent yttria stabilized zirconia nanopowder (two-step [TSS],

conventional (CS), as well as sintering assisted by microwave at two distinct rates of heating, lower rate [LMS] and higher rate [HMS]). Two-step sintering achieved nearly entirely dense bodies with an approximate grain size of 300 nm, but rapid densification using HMS resulted in a dense sample with an average grain size of 0.9 μm , compared to 2.35 μm and 2.14 μm for the LMS and CS techniques, respectively. The authors speculate that the uniform microstructure created by the rapid firing process could be the cause. [39,40].

Binner et al., by using slip casting, turned 3 mol percent yttria partly stabilized zirconia nanopowder into components, which produced lesser viscosity but higher solid content nanosuspensions. One, alongwith two stages of pressureless sintering were then conducted, utilizing hybrid-microwave heating and pure conventional heating. The materials could maintain a final mean grain size of less than 100 nm while achieving better than 99 percent of theoretical density through a two-stage sintering process. The best sample to date has a mean grain size of only 64 nm and is around 99.5 percent dense; this was achieved by using a hybrid microwave conventional technique. Most people think that using a significantly faster starting heating rate is the main benefit of the hybrid heating strategy[41].

Wroe et al., studied that microwave-assisted sintering of partially stabilized zirconia offers significant advantages over conventional methods. It allows for a substantial reduction in sintering temperature, typically by 80-100°C, while achieving slightly higher final sample densities. This improvement is ascribed to non-thermal processes that continue even in the presence of steady microwave power and quicken densification phases in the vicinity of an electrical field. Increased vacancy flux within grain borders due to the driving power of the microwave field favors grain-boundary or volume diffusion mechanisms over surface

diffusion processes, that are dominant at reduced temperatures. This preference for mechanisms with lower activation energies results in observed reductions in effective activation energy, although actual activation energies for specific diffusion processes remain unchanged. Overall, microwave-assisted sintering represents a promising approach for enhancing densification processes in zirconia ceramics through efficient vacancy flux and diffusion within grain boundaries [42].

Ramesh et al., studied that microwave sintering has demonstrated significant benefits by lowering densification temperature and improving mechanical properties and bulk density compared to conventional sintering. It is noteworthy that the sizes of the grain obtained from samples which were microwave-sintered were observed, as comparable to those obtained from conventional methods. This indicates that the primary determinant of grain size in sintered zirconia is the sintering temperature, rather than the sintering process itself. This indicates that the primary determinant of grain size in sintered zirconia is the sintering temperature, rather than the sintering process itself. Interestingly, microwave sintering yielded improved densification and mechanical characteristics at a low temperature of 1200°C, which proved to be most beneficial. Furthermore, even at higher temperatures, samples that had been microwave-sintered showed better qualities. [43].

Mago et al. studied YSZ (yttria-stabilized zirconia) and found YZT (titania-doped YSZ) to be promising as an anode material. Both materials were synthesized using conventional and microwave methods. Various techniques were used to characterize the samples. Microwave-sintered YZT showed improvements in density, phase stabilization, and hardness. Microwave processing alters sintering mechanisms, leading to superior

densification and microstructures. This technique offers advantages in time and energy efficiency for YZT synthesis [44].

CHAPTER 3

EXPERIMENTAL DETAILS

3.1 Materials

The primary powder materials utilized for this work were 8wt.% yttria-stabilized zirconia (8YSZ), ceria-stabilized zirconia (CSZ), zirconia titania yttria composite (YTiZ). In this study, 8 wt.% yttria-stabilized zirconia (8YSZ), ceria-stabilized zirconia (CSZ), and zirconia-titania-yttria composite (YTiZ) were selected due to their wide applications in high-temperature environments such as thermal barrier coatings (TBCs) and gas turbines. The 8YSZ provides excellent phase stability and thermal insulation, while the CSZ offers enhanced oxygen ion conductivity and phase stability at intermediate temperatures. Adding titania (TiO_2) to zirconia composites (YTiZ) improves mechanical strength, phase stability, and corrosion resistance [45–48]. These materials are chosen to evaluate their hot corrosion resistance, as they are frequently exposed to corrosive species, like vanadium pentoxide (V_2O_5) in practical applications. Understanding their behavior under corrosive environments is critical for optimizing material performance and durability in extreme operating conditions. Table 3.1 presents the details of the primary powders.

Table 3. 1 Details of the primary powders used

Material	Composition (wt.%)	Particle Size (μm)	Morphology
8YSZ (Metco 204 NS-G)	$\text{ZrO}_2 - 8\text{Y}_2\text{O}_3$	125	Spheroidal
Ceria-Stabilized Zirconium Oxide (Metco 205NS)	$\text{ZrO}_2 - 24\text{CeO}_2 - 2.5\text{Y}_2\text{O}_3$	125	Spheroidal
Zirconia Titania Yttria Composite (Metco 143)	$\text{ZrO}_2 - 8\text{TiO}_2 - 10\text{Y}_2\text{O}_3$	75	Spheroidal

3.2 Sintering of Zirconia Compounds via Hybrid Microwave Heating

The powders used in the study included 8 wt% Yttria Stabilized Zirconia (8YSZ), Ceria-Stabilized Zirconium Oxide (CSZ), and Zirconia Titania Yttria Composite (YTiZ), commercially known as Metco 204NS-G, Metco 205NS, and Metco 143 respectively. These powders were manufactured by Oerlikon Metco's Hollow Spherical Powder (HOSP)TM process, where the particles are heated above their melting point in ambient atmosphere, allowing surface tension to shape them into spherical particles, which are primarily utilized in applications requiring thermal barrier and/or thermal shock resistance.

Before sintering via the Hybrid Microwave (HMW) processing method, the powder was compressed using a uniaxial press machine into cylindrical disk shapes that were about 30 mm in diameter and 5 mm in height.

Prepared pellets underwent to HMW sintering using a commercial Carbolite Ltd microwave assist box furnace (model MRF 16/22/3508P1), which can achieve

temperatures up to 1600°C. This furnace utilizes a combination of free radiating heating components, utilizing 9 kW molybdenum disilicide heating elements, coupled with a microwave radiation source operating at a frequency of 2.45 GHz with 1.8 kW magnetron power. The HMW concept involves volumetric heating of the material through microwave energy, in addition to conventional heating elements, ensuring controlled sintering as illustrated in Figure 3.1.

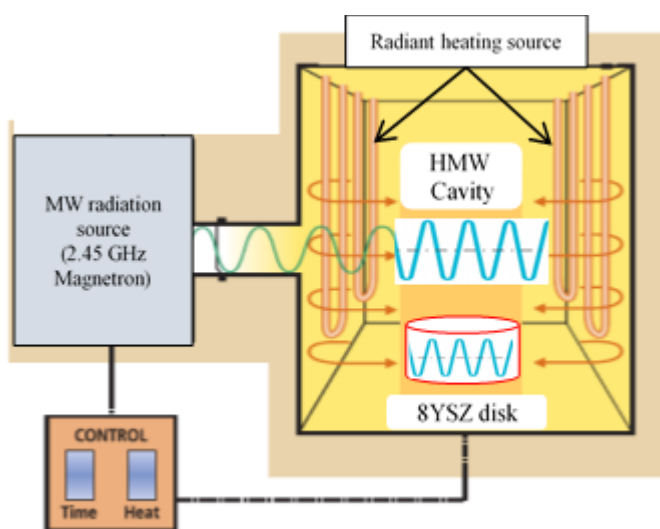


Figure 3. 1 Schematic of HMW setup with combined radiant and MW heating.

The resulting disks were sintered using HMW at three different heat-treatment temperatures: 1350°C, 1400°C, and 1450°C, with a heating rate of 50°C/min and a holding time of two hours. They were then permitted to cool to room temperature.

3.3 Characterization Techniques

A comprehensive characterization was undertaken before and after the various hot corrosion tests. The tested coupons were carefully inspected visually before a thorough examination was conducted utilizing various characterization tools for morphological

analysis, phase evolution as well as thermal conductivity. Some of the equipment that was assiduously utilized in this research are presented in Figure 3.2.

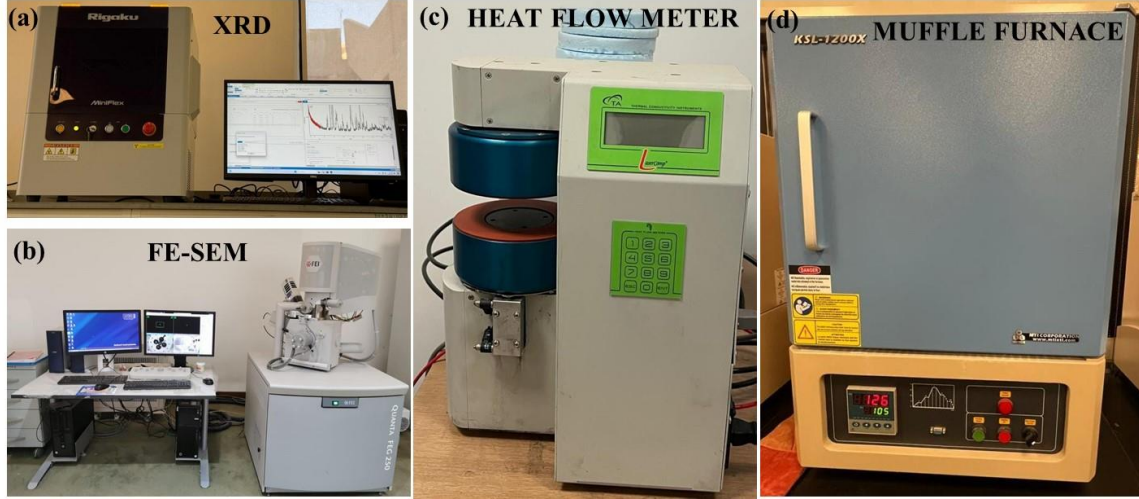


Figure 3. 2 Some of the equipment used for the work

3.3.1 X-Ray Diffraction (XRD)

One of the best methods for figuring out a material's crystal structure is XRD. XRD experiments were carried out on prepared and sintered coupons before and after hot corrosion tests using a Rigaku Miniflex X-ray diffractometer operated at 30 kV and 15 mA with Cu K α radiation of a wavelength, $\lambda = 0.15406$ nm. Scanning was performed for 2 θ values between 20° and 80° at a step size of 0.02 °/min. The volume fraction of the monoclinic phase formed for each composition was determined using the intensity of peaks at m-ZrO₂ ($\bar{1}11$), m-ZrO₂ (111), and t-ZrO₂ (101) planes as defined in equation 3.1

$$m - ZrO_2 = \frac{m(\bar{1}11) + m(111)}{m(\bar{1}11) + m(111) + t(101)} \times 100 \quad 3.1$$

This technique uses an X-ray beam with a wavelength λ generated by an X-ray tube, those radiation beams go through the sample and “diffract” from the atoms within the structure,

changing the beam's direction at an angle (θ), from the source beam. This angle is the diffraction angle and can be used in Bragg's law to determine the distance between atomic planes as per the below equation 3.2 [49]:

$$\sin\theta = n\lambda/2d \quad 3.2$$

The distance between atomic planes, d , can then be used to assess the crystal structure of the analyzed sample. Diffraction peaks observed with XRD analysis will be used to analyze the crystal structure of the characterized material and evaluate the level of crystallinity of the synthesized Zirconia-Doped samples. Scherrer's formula will be used to estimate the crystal size of particles as per the below equation 3.3 [50]:

$$d = 0.9\lambda/\beta\cos\theta \quad 3.3$$

Where d is the mean size of the crystal, λ is x-ray wavelength, β refers to the FWHM (Full width high maximum) of the peak, and θ is Bragg's angle.

Crystallite size refers to the dimensions of a single coherent crystalline domain within a material. Unlike grain size, which measures the overall size of a single grain in a polycrystalline structure, crystallite size pertains to the sub-structural regions that are free from lattice defects or dislocations. It is typically smaller than the grain size because a single grain may contain multiple crystallites due to internal imperfections, strain, or sub-grain boundaries. Crystallite size is a critical parameter in materials science as it influences a material's mechanical strength, thermal conductivity, and optical properties. It is commonly determined using X-ray diffraction (XRD) by applying the Scherrer equation, which relates peak broadening in the diffraction pattern to crystallite dimensions. This

parameter is particularly significant in nanomaterials, where crystallite size often defines the unique physical and chemical behaviors observed at the nanoscale. Smaller crystallites, for instance, can enhance properties like hardness due to the lack of long-range crystallographic continuity [51].

3.3.2 Scanning Electron Microscopy (SEM)

SEM is essential to analyze the topography and morphology of the sample's surface and will help note observations on the shape of crystals according to modified processing parameters. An image taken through SEM is composed by emitting a beam of electrons that scans the sample's surface area, generating an image with a large depth of field and a 3D appearance.

To generate images, an electron gun in the SEM machine emits a beam of electrons at the topmost level of the microscope. This beam enters the machine from the top and moves vertically through the vacuum-shielded interior. The beam passes via lenses and electromagnetic fields before focusing on the sample. The machine's detectors receive these back-scattered electrons and secondary electrons to turn them into a digital signal that is sent to a screen to form the final image. As the electron beam reaches the sample and collides with it, electrons and X-rays are released from the specimen[52]

A Field Emission Scanning Electron Microscope (FESEM FEI Quanta 250) equipped with an Energy Dispersive Spectrometer (EDS detector, 150 mm²) was employed at 20 kV to analyze the morphological characteristics of as-sintered and hot corroded samples. The Quorum coating machine (model Q150R) was employed to apply a thin layer of gold on the surface to improve conductivity.

3.3.3 Thermal Conductivity

The thermal conductivity measurement was conducted utilizing The FOX 50 Heat Flow Meter. This highly precise instrument is specifically designed for accurately determining thermal conductivity values of various materials. The measurement process involves placing the sample material between two temperature-controlled plates, and then applying a known heat flux across the sample. The FOX 50 Heat Flow Meter precisely measures the temperature gradient across the sample and the corresponding heat flux, enabling the calculation of the material's thermal conductivity. This method ensures reliable and repeatable results, essential for understanding the material's thermal properties under investigation. Additionally, the instrument's user-friendly interface and advanced data analysis capabilities facilitate efficient data collection and interpretation, contributing to comprehensive thermal characterization studies.

3.4 Hot Corrosion Test

Isothermal hot corrosion tests were used to screen the composites at 900 °C in 10 mg/cm² V₂O₅ for a total duration of 48 h. 10 minutes to stabilize before placing the crucibles containing the powder mixtures. Hot corrosion tests were carried out on the sintered samples by uniformly spreading 10 mg/cm² of V₂O₅ on the surface leaving 2 mm allowance

at the edges to avoid edge effects. The samples were then placed in an electric muffle furnace with an ambient atmosphere under a maximum temperature of 900 °C for 48 h. After the test duration, heated samples were cooled in the laboratory air and characterized using XRD analysis to estimate phase composition as well as SEM Analysis for microstructural changes.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 8 wt% Yttria-Stabilized Zirconia (8YSZ)

The following sections describe the two processes namely HMW sintering process and Hot corrosion.

4.1.1 HMW Sintering Process

Figure 4.1 displays the SEM image that demonstrates the distinctive spherical morphology of the 8YSZ particles. This spherical shape is a distinguishing characteristic feature resulting from the utilization of the Hollow Oxide Spherical Process (HOSP) during the production of the powder.

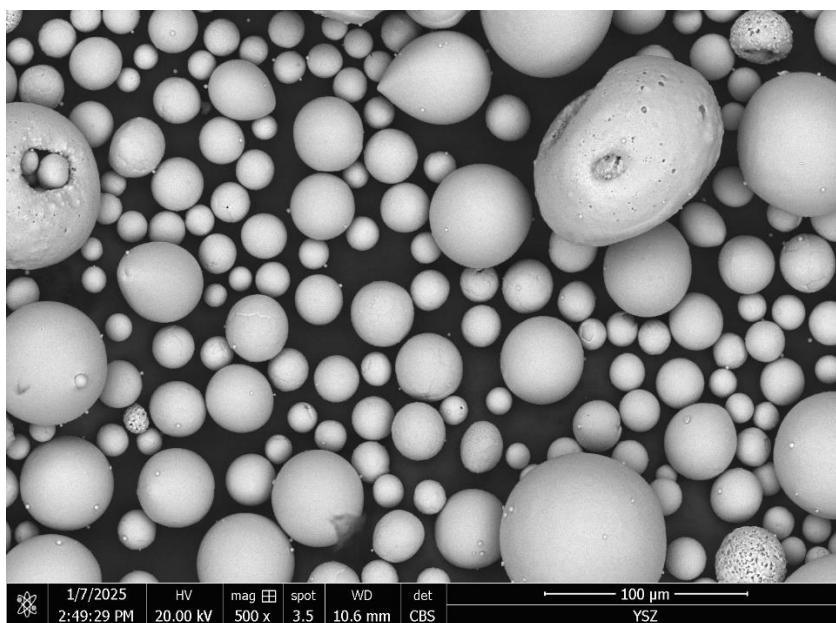


Figure 4. 1 SEM of the as-received 8YSZ spherical powder

Furthermore, the characterization of the powder as-received, to explore the impact of HMW sintering at three separate temperatures 1350 °C, 1400 °C, and 1450 °C on the powder's morphology, SEM micrographs were obtained for the samples. Figure 4.2 demonstrates the changes in the powder's morphology post the sintering process, utilizing HMW heating techniques. During sintering, a significant decrease in the primary pores among the spherical powder particles was observed in all sintered samples. Moreover, the presence of distinct grain boundaries was noted in all samples, including those subjected to a lower temperature of 1350 °C by HMW, demonstrating the effective sintering of the 8YSZ samples through HMW processing. Successful sintering transpired when the individual grains coalesced and established distinct boundaries. As HMW heat treatment temperatures rise from 1350 to 1450 °C, grain sintering becomes more pronounced, exhibiting more distinct welding between the grains. Additionally, the minor observed voids in the HMW were possibly due to spherical particle displacement or particle collapse during sintering. On the contrary, fewer microcracks were observed in the HMW at higher temperatures which can be attributed to the martensitic transformation during heating. On average, conventional heating above approximately 1300 °C, followed by cooling to room temperature, results in the transformation of the tetragonal t-phase into the monoclinic m-phase (m-ZrO₂), leading to a volume increase of approximately 3 to 5% and a significant decline in mechanical properties due to extensive microcracking [53]. The tetragonal ZrO₂ phase elasticity made it more likely than cubic and monoclinic ZrO₂ phases to produce stress concentrations and microcracks because of its higher anisotropy ratio[54]. The generation and magnitude of microcracks are markedly diminished in all HMW-treated

samples, presumably owing to the suppression of the martensitic change from tetragonal to monoclinic phases during MW heating. Consequently, HMW sintering, even at much reduced temperatures, proves effective in promoting densification while simultaneously preventing the unfavorable martensitic transition.

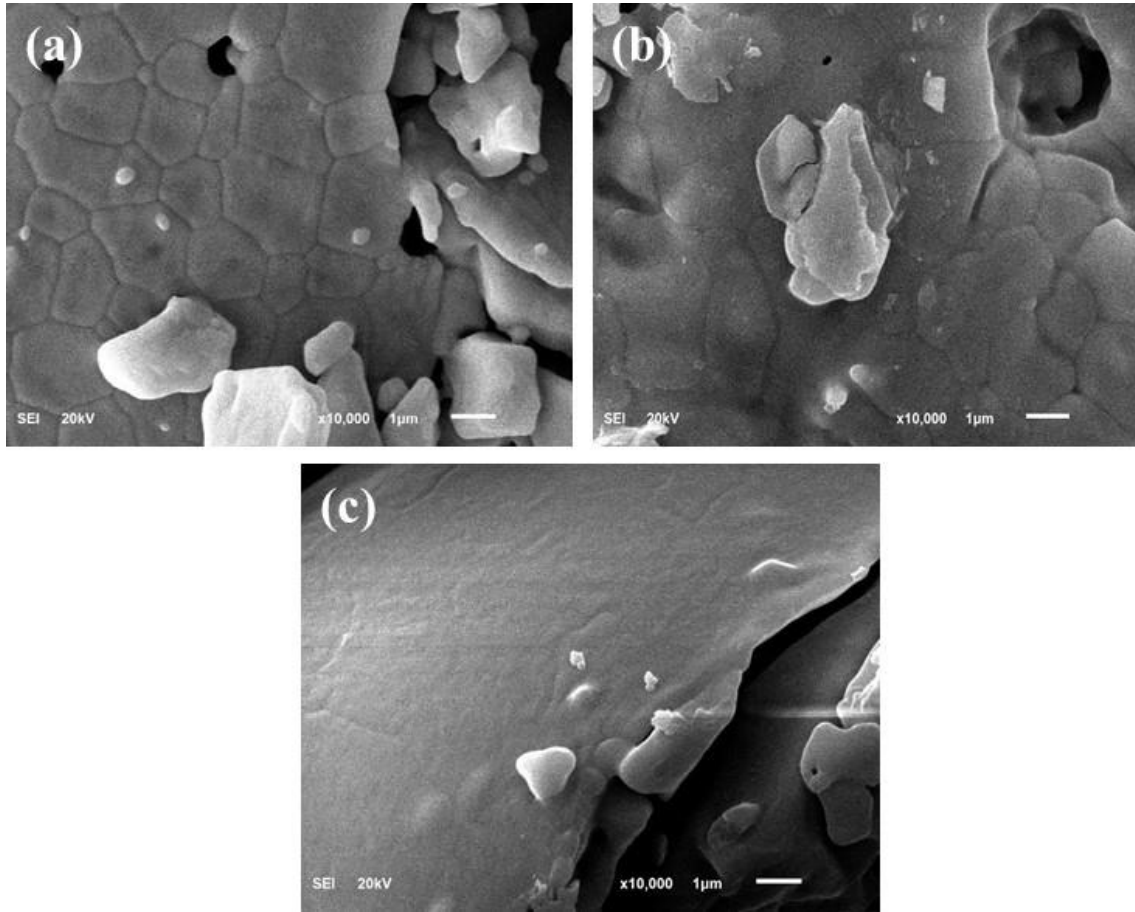


Figure 4. 2 SEM of 8YSZ sintered samples for 2 h using HMW heating at (a) 1350 °C, (b) 1400 °C, (c) 1450 °C

The XRD patterns of the as-received 8YSZ powder and its equivalent HMW-sintered samples for 2 h at three distinct temperatures offer essential insights into the structural alterations caused by varying sintering procedures and conditions. Figure 4.3 (a) illustrates the aforementioned patterns, displaying unique peak profiles and intensities. The as-received XRD pattern of 8YSZ powder exhibits the tetragonal ($t\text{-ZrO}_2$) crystal structure as

the predominant phase, accompanied by a small monoclinic (m-ZrO₂) phase. The existence of the t-ZrO₂ phase in the as-received sample is attributed to the addition of Y₂O₃ as a stabilizer, validating the chemical composition presented in Table 2.1 and aligning with previous study results. [55,56]. The major XRD peaks are indexed and identified as tetragonal crystal structures according to the ICDD PDF-card # 01-070-4432. However, the detected minor portion of the m-ZrO₂ phase suggests some local inhomogeneity within the commercially received 8 YSZ powder. This inhomogeneity may arise from its coarser microstructure in the micrometric size, leading to higher induced strains during cooling, as observed in previous studies[57,58]. Such local variations can influence the sintering process and the resultant crystallographic phases, affecting the material's properties.

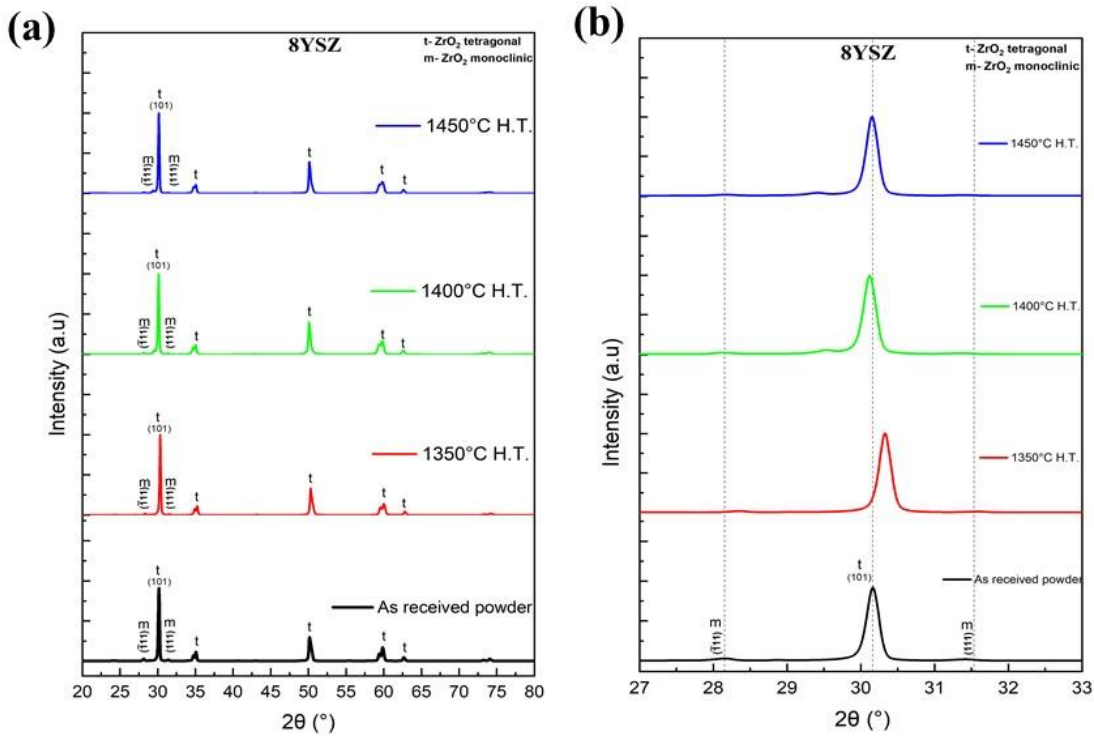


Figure 4. 3 XRD of 8YSZ as-received powder and HMW heat-treated corresponding samples at different temperatures for 2θ ranges from (a) 20° to 80° and (b) 27° to 33°.

In all sintered samples, the predominant phase identified was t-ZrO₂, exhibiting the most intense peaks at the (101) plane, along with minor distinct peaks associated with the m-ZrO₂ phase. In the HMW heat-treated samples at 1350 °C, 1400 °C, and 1450 °C, the predominance of the t-ZrO₂ phase persists, accompanied by a gradual reduction in the minor m-ZrO₂ phase as the temperature escalates from 1350 to 1450 °C, relative to the as-received sample. XRD analysis can provide extensive information regarding the types and quantities of phases in sintered 8YSZ samples at various temperatures. Figure 4.3 (b) shows XRD patterns of the as-received 8YSZ powder and its corresponding sintered samples for 2θ ranges from 27° to 33°.

Figure 4.3 (b) illustrates the XRD patterns of all samples within the (27–33°) 2θ range. The sintering temperature and method substantially affect the distinctive diffraction peak positions and lattice properties of 8 YSZ sintered samples. A little peak shift of the (101) principal diffraction peak of the t-ZrO₂ phase occurred based on the sintering temperature and technique employed. The peak shift signifies the creation of lattice stresses and/or alterations in the lattice characteristics of the t-ZrO₂ phase caused by fluctuations in sintering conditions and methodologies. The unique characteristics observed in the XRD patterns of the displayed sintered samples underscore the responsiveness of the phase type and quantity to fluctuations in sintering temperature and the employed sintering technique. The development of the monoclinic phase, influenced by the sintering process and temperature, is computed per Eq.3.1 and illustrated in Fig. 4.4. In the absence of cubic phase detection in the samples (from high-resolution scans between 70.5° and 76.5°), the volume fractions of the tetragonal and monoclinic phases were determined from the

summed peak intensities of the m-ZrO₂ (111), m-ZrO₂ (111), and t-ZrO₂ (101) planes as delineated in Eq. 1. [59] .

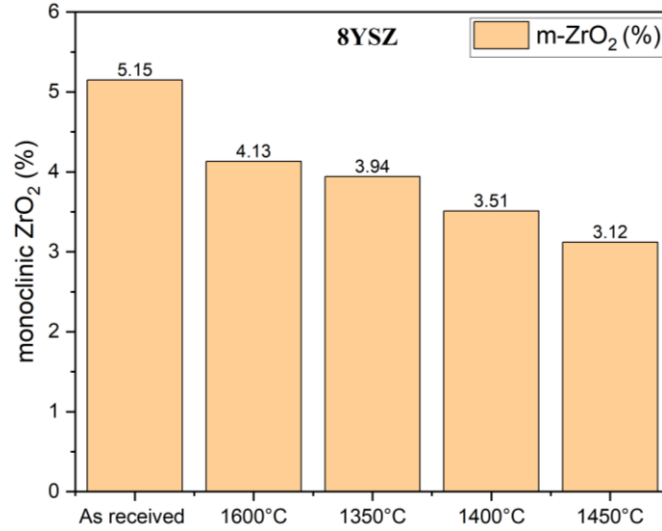


Figure 4. 4 Estimated amount of monoclinic phase of 8YSZ sintered samples.

For the HMW-sintered samples, the percentage of the m-ZrO₂ phase continuously decreases with increasing the sintering temperature, as compared to the as-received powder. The highest reduction in the m-ZrO₂ phase is occurring at the HMW sample sintered at 1450 °C. When compared to a conventionally sintered sample processed at 1600°C, the microwave-sintered sample processed at 1450°C shows a reduced monoclinic phase content. The monoclinic phase in the microwave-sintered sample is lower than that of the conventionally sintered sample about 4.13% [60]. This is due to the microwave sintering. The stabilization of the tetragonal (t-ZrO₂) phase at elevated temperatures is enhanced due to the unique heating mechanism. Microwave energy couples directly with the material at the molecular level, promoting uniform and rapid volumetric heating. This minimizes thermal gradients and ensures a more even distribution of yttria (Y₂O₃) within the zirconia matrix. The rapid and controlled heating accelerates diffusion, enabling a more

homogeneous microstructure with fewer defects. Additionally, the microwave-induced heating helps maintain oxygen vacancies more effectively, further stabilizing the tetragonal phase and preventing the transformation to the monoclinic phase. As a result, microwave sintering enhances phase stability and reduces the likelihood of undesirable phase transformations [61].

Figure 4.5 depicts the average crystallite size of the sintered samples. The mean crystallite dimensions of the HMW, calculated using the Scherrer equation. The sintered samples have average crystallite diameters ranging from 21 to 23 nm. The phenomena of grain development that happens during sintering are consistent with the reported increase in crystallite size with temperature. The coalescence of smaller crystallites into bigger domains is driven by the energy that higher sintering temperatures offer for atoms to migrate and rearrange. This is often followed by an increase in crystalline order and a decrease in flaws. The findings are consistent with prior research on comparable materials, which indicates that crystallites are forced to coarsen as temperatures increase. The Ostwald ripening mechanism explains this trend, in which smaller crystallites dissolve into larger ones to reduce the system's overall free energy [62]

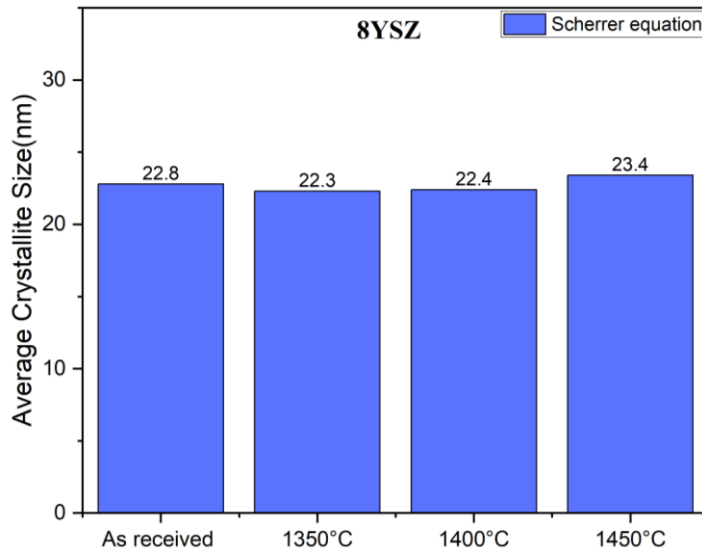


Figure 4. 5 Estimated crystallite sizes of 8YSZ sintered samples using the Scherrer equation.

Table 4.1 presents the thermal conductivity values of all sintered samples. A consistent reduction in thermal conductivity was noted for HMW samples as the sintering temperature increased at 1350°C, 1400°C, and 1450°C. Notably, both samples were conventionally sintered with nearly identical m-ZrO₂ phase composition; however, the HMW sample sintered at 1350°C exhibits the lowest level. [60]. Additionally, the HMW-sintered sample at 1450°C demonstrates the lowest thermal conductivity, due to the most significant drop in the m-phase. The volume fractions of phases in the sintered samples are a critical factor that can significantly affect the thermal conductivity results[63].

Table 4. 1 Thermal conductivity of the 8YSZ HMW heat-treated corresponding samples at different temperatures.

Material	Thermal Conductivity (W/mK)
1600°C H.T.	0.2808 [60]
1350°C H.T.	0.2780
1400°C H.T.	0.2459
1450°C H.T.	0.1316

4.1.1 Hot Corrosion

The SEM analysis of 8YSZ samples shown in Figure 4.6 sintered at 1350°C, 1400°C, and 1450°C reveals significant differences in their microstructures, phase stability, and resistance to hot corrosion. At 1350°C, the microstructure exhibits smaller grains with noticeable porosity, and the lower sintering temperature prevents the formation of YVO₄ (yttrium vanadate) due to insufficient thermal energy and limited reaction kinetics [64]. As a result, while the tetragonal phase remains present, it is partially unstable, and the sample shows poor corrosion resistance due to the porous structure, which provides pathways for corrosive agents. At 1400°C, the material shows improved grain growth and reduced porosity, and the presence of YVO₄ in the form of rod-like structures becomes evident [65]. The formation of YVO₄ (shown in equation 4.1) results from the reaction of the yttria stabilizer with vanadium-containing corrosive agents, leading to the depletion of yttria and the destabilization of the tetragonal phase. This destabilization triggers a phase transformation to the monoclinic phase, which weakens the material.



Despite this, the sample sintered at 1400°C exhibits moderate hot corrosion resistance due to its improved densification compared to 1350°C. At 1450°C, the sample achieves significant densification with large grains and minimal porosity, which enhances its overall structural integrity. However, the YVO_4 rod-like structures are not distinctly visible at this temperature, likely due to accelerated reaction kinetics and uniform phase depletion. Although the depletion of yttria still occurs, destabilizing the tetragonal phase and promoting its transformation to the monoclinic phase, the dense microstructure at 1450°C provides greater resistance to hot corrosion compared to the sample sintered at 1350°C. Overall, the sample sintered at 1450°C demonstrates the highest corrosion resistance, primarily due to its reduced porosity and enhanced densification, even though localized phase transformation remains a concern.

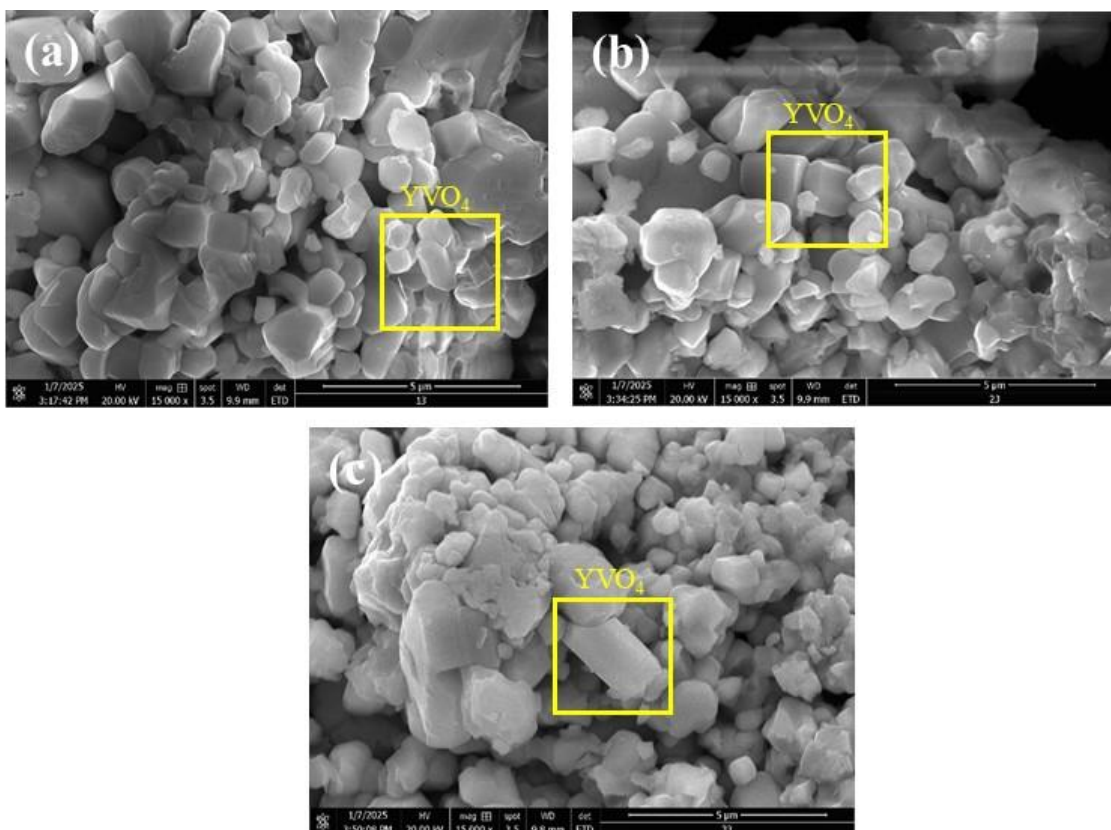


Figure 4. 6 SEM of hot corroded 8YSZ sintered samples for 2 h using HMW heating at (a) 1350 °C, (b) 1400 °C, (c) 1450 °C

The XRD patterns and phase evolution analysis of 8YSZ samples subjected to hot corrosion with V_2O_5 at different sintering temperatures (1350°C, 1400°C, and 1450°C) are shown in Figure 4.7 (a) and (b). The XRD results reveal a significant variation in the monoclinic (m- ZrO_2) and tetragonal (t- ZrO_2) phases with increasing sintering temperature. In the as-received powder (Figure 4.8), the peaks corresponding to the tetragonal phase dominate, with a minor presence of the monoclinic phase. The pronounced m- ZrO_2 peaks demonstrate that the monoclinic phase content increases dramatically to 64.59% during sintering at 1350°C (Figure 4.8). This increase indicates that the interaction with V_2O_5 causes depletion of the yttria stabilizer, destabilizing the tetragonal phase and triggering its transformation to the monoclinic phase.

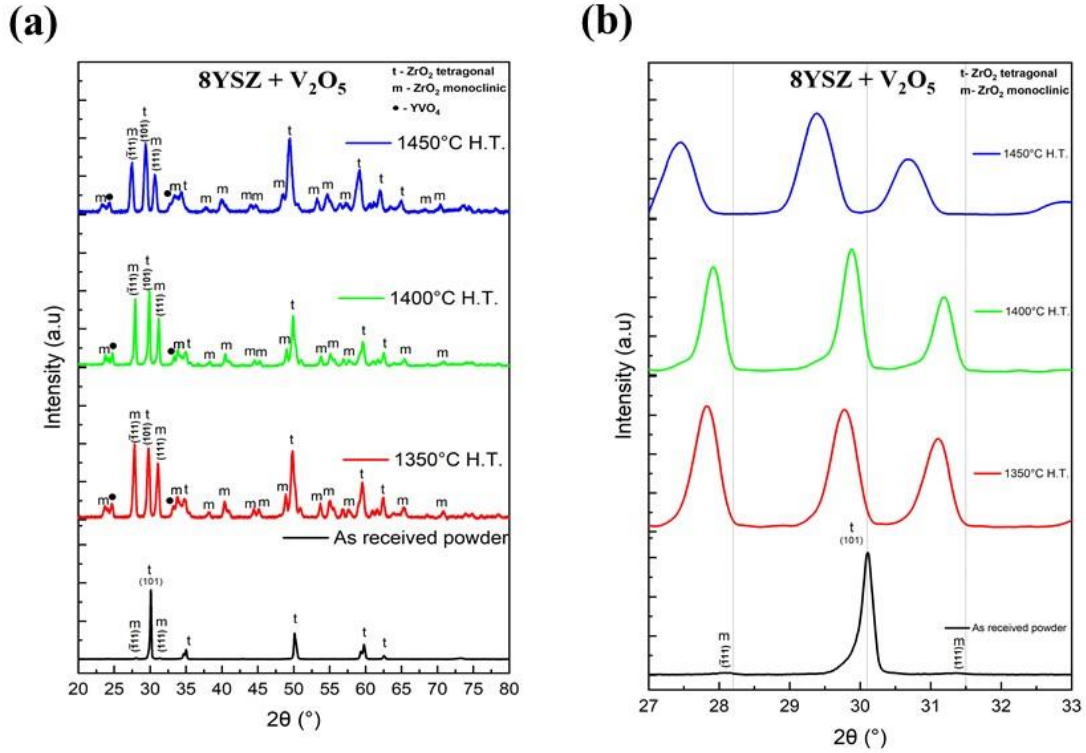


Figure 4. 7 XRD of as-received 8YSZ powder and hot corroded 8YSZ HMW heat-treated corresponding samples at different temperatures for 2θ ranges from (a) 20° to 80° and (b) 27° to 33°

At 1400°C, as shown in Figure 4.8, the monoclinic phase content reduces to 60.16% (Figure 4.8), and the tetragonal phase becomes more prominent. Additionally, the formation of YVO₄ is evidenced by the appearance of distinct rod-like structures (observed in SEM) and additional peaks in the XRD pattern, indicating the depletion of yttria due to its reaction with vanadium oxide. Nevertheless, the tetragonal phase is largely stabilized by the enhanced densification at this temperature, which leads to a lower monoclinic concentration than at 1350°C.

The shift observed in the XRD peaks, particularly for the tetragonal phase (t-ZrO₂), can be attributed to several factors arising from the interaction of 8YSZ with V₂O₅ at elevated sintering temperatures. At 1350°C, the reaction kinetics are relatively slow, and the peaks remain closer to their positions in the as-received powder, though the increase in

monoclinic (m-ZrO₂) content indicates partial destabilization of the tetragonal phase. At 1400°C, the onset of yttria depletion due to the formation of YVO₄ causes lattice strain, which results in a slight peak shift. This yttria depletion destabilizes the tetragonal phase, inducing local lattice distortions. Furthermore, the lattice repulsion forces are decreased by the creation of oxygen vacancies at this temperature, which results in minor variations in the interplanar spacing (d-spacing). At 1450°C, the reaction kinetics are enhanced, leading to further depletion of yttria and increased oxygen vacancy concentration, which causes a more pronounced shift in the tetragonal phase peaks. Furthermore, the higher thermal energy at this temperature can result in residual thermal stresses during cooling, contributing to lattice strain and peak displacement. These combined effects—yttria depletion, oxygen vacancy formation, and thermal expansion—alter the crystal lattice, causing a noticeable shift in the XRD peaks as the sintering temperature increases.

At 1450°C, the monoclinic phase content decreases further to 53.35% (Figure 4.10), the lowest among all conditions. The XRD pattern (Figure 4.7) shows sharper peaks for the tetragonal phase, indicating improved phase stability and suppression of the tetragonal-to-monoclinic phase transformation. The absence of distinct YVO₄ peaks at this temperature suggests a more uniform and distributed depletion of yttria across the material. This reduction in monoclinic content, combined with enhanced densification and grain growth, results in better structural integrity and increased resistance to hot corrosion.

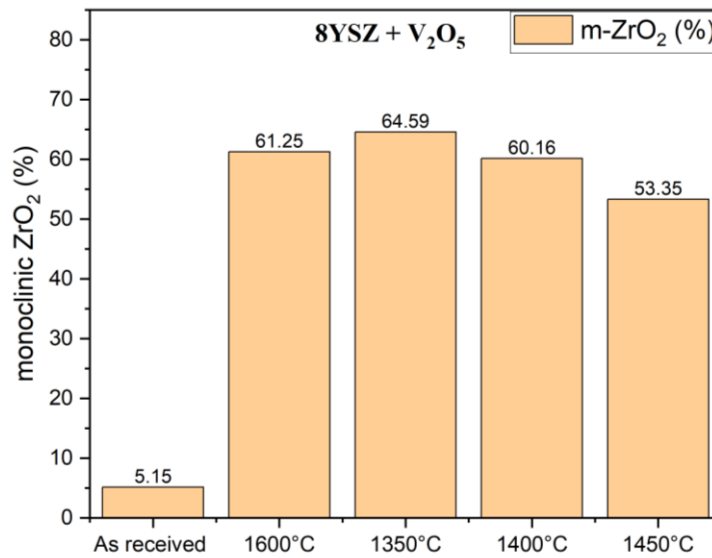


Figure 4. 8 Estimated amount of monoclinic phase of 8YSZ sintered samples after hot corrosion.

The phase evolution analysis (Figure 4.8) demonstrates that the monoclinic phase content decreases with increasing sintering temperature, from 64.59% at 1350°C to 53.35% at 1450°C. The improved tetragonal phase stability and reduced monoclinic content at 1450°C indicate that the sample sintered at this temperature possesses the highest resistance to hot corrosion, primarily due to enhanced densification, reduced porosity, and suppression of the destructive tetragonal-to-monoclinic transformation. The study [66] clearly shows that, for the conventionally sintered sample at 1600°C, the material has completely transformed into the monoclinic phase. In contrast, in our study, as the sintering temperature increases, the percentage of the monoclinic phase decreases. This is due to the microwave sintering. The stabilization of the tetragonal (t-ZrO₂) phase at elevated temperatures is enhanced due to the unique heating mechanism. Microwave energy couples directly with the material at the molecular level, promoting uniform and rapid volumetric heating. This minimizes thermal gradients and ensures a more even distribution of yttria (Y₂O₃) within the zirconia matrix. The rapid and controlled heating accelerates diffusion,

enabling a more homogeneous microstructure with fewer defects. Additionally, the microwave-induced heating helps maintain oxygen vacancies more effectively, further stabilizing the tetragonal phase and preventing the transformation to the monoclinic phase. As a result, microwave sintering enhances phase stability and reduces the likelihood of undesirable phase transformations [61]. As the microwave sintering temperature increases, the phase stability of YSZ improves significantly, even under harsh conditions like hot corrosion. The higher sintering temperatures lead to enhanced densification and grain boundary cohesion, making it more resistant to phase destabilization caused by corrosive environments [67]. This results in a material that retains its structural integrity and maintains the desired phase composition, contributing to improved durability and performance after exposure to molten salts or other corrosive agents.

4.2 Ceria Stabilized Zirconia (CSZ)

Following sections describe the two processes namely HMW sintering process and Hot corrosion.

4.2.1 HMW Sintering Process

Figure 4.9 displays a SEM image that depicts the unique spherical morphology of the CSZ particles. The spherical morphology is a characteristic feature derived by the application of the Hollow Oxide Spherical Process (HOSP) in the powder's manufacturing.

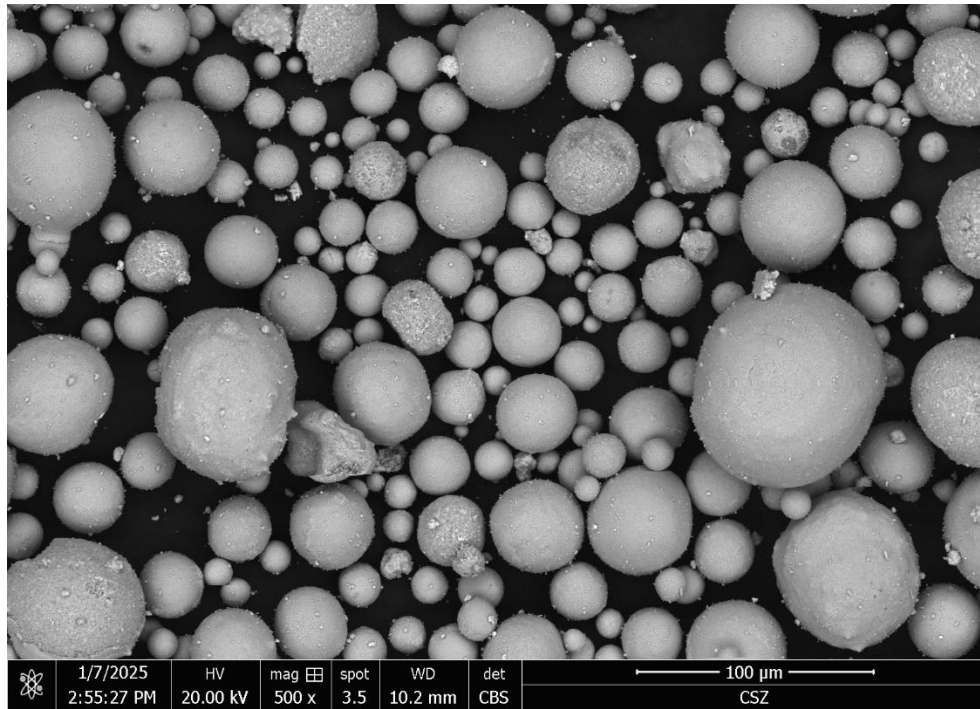


Figure 4. 9 SEM of the as-received CSZ spherical powder.

The SEM images shown in Figure 4.10 of ceria-stabilized zirconia (CSZ) samples sintered at 1350°C, 1400°C, and 1450°C using hybrid microwave sintering clearly demonstrate the influence of sintering temperature on the microstructural evolution. At 1350°C, the microstructure exhibits smaller grains with poorly defined grain boundaries and significant porosity, indicating incomplete densification due to limited thermal energy. The presence of intergranular gaps and voids indicates that particle coalescence and diffusion are still in the very early stages. As the temperature increases to 1400°C, the microstructure shows noticeable grain growth and a reduction in porosity. The grain boundaries become more defined, and the particles fuse together, reflecting improved densification and enhanced diffusion kinetics at this intermediate temperature. However, some isolated pores remain, indicating the sintering process has not yet achieved full consolidation. At 1450°C, the microstructure reveals significant grain growth with well-defined grain boundaries and minimal porosity, resulting in a dense and uniform structure. The higher thermal energy at

this temperature promotes enhanced grain boundary migration and particle coalescence, leading to near-complete densification. The transition from 1350°C to 1450°C highlights a progressive reduction in porosity and a corresponding increase in grain size, with the sample sintered at 1450°C achieving the highest degree of densification and structural uniformity. This improved microstructure at 1450°C is indicative of superior mechanical properties, thermal stability, and overall performance, making it the most suitable condition for high-performance applications.

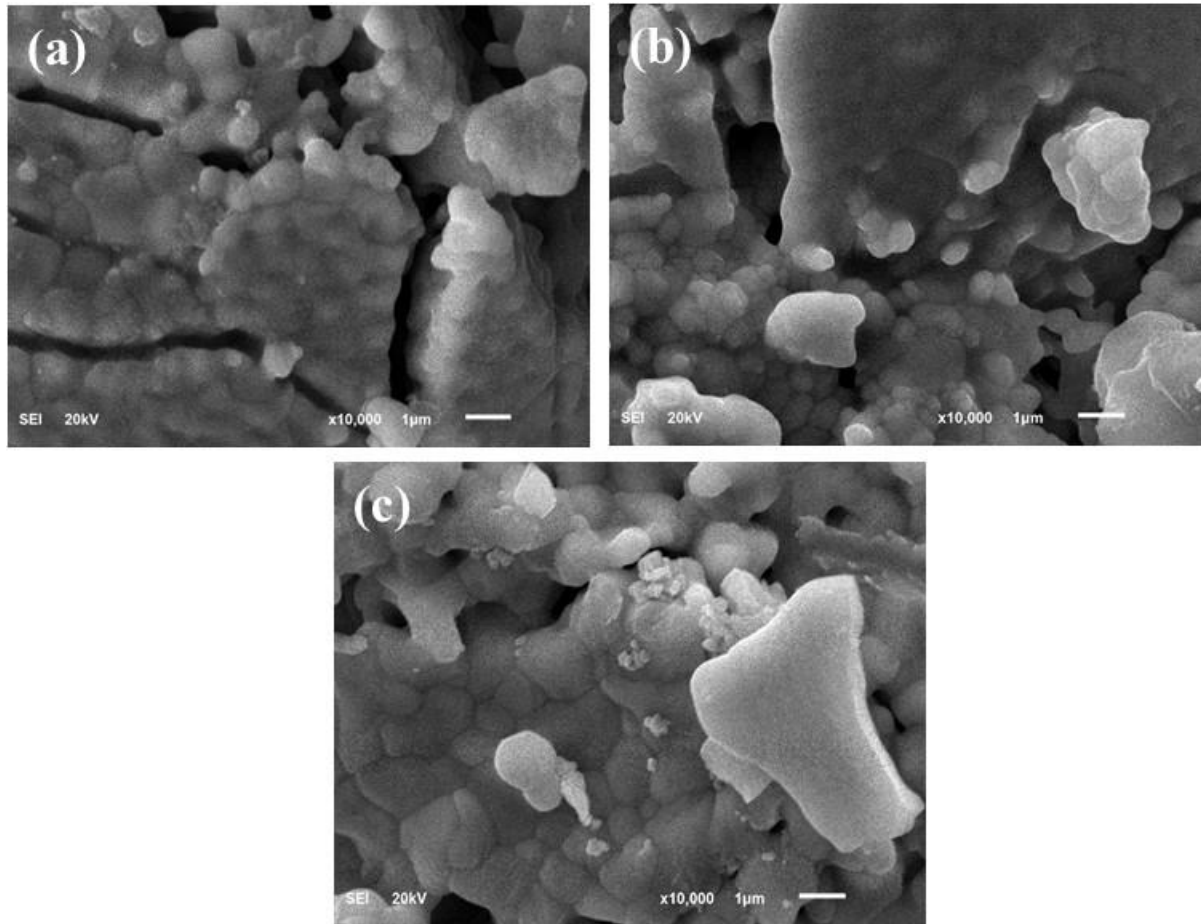


Figure 4. 10 SEM of CSZ sintered samples for 2 h using HMW heating at (a) 1350 °C, (b) 1400 °C, (c) 1450 °C

The XRD analysis and monoclinic phase (using equation 3.1) content graph of Ceria Stabilized Zirconia (CSZ) sintered at 1350°C, 1400°C, and 1450°C using hybrid microwave sintering provide detailed insights into phase evolution and peak behavior with increasing sintering temperature. The as-received powder (Figure 4.11(a)) exhibits a dominant tetragonal phase (t-ZrO₂) with a significant monoclinic phase (m-ZrO₂) content of 15.9%, as shown in the monoclinic phase content graph (Figure 4.12). Upon sintering at 1350°C, the monoclinic phase content decreases to 7.49%, with reduced intensity of the monoclinic peaks at $2\theta \approx 28^\circ$ and 31° , as observed in the XRD patterns (Figure 4.11(a)).

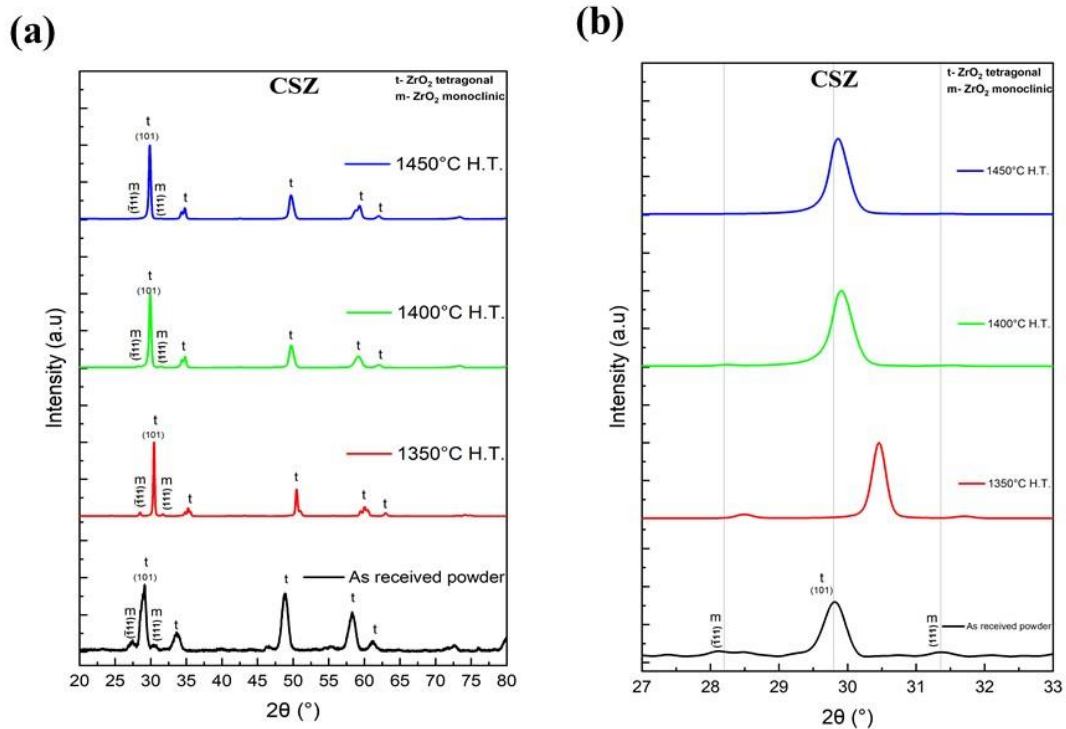


Figure 4. 11 XRD of CSZ as-received powder and HMW heat-treated corresponding samples at different temperatures for 2θ ranges from (a) 20° to 80° and (b) 27° to 33° .

At 1400°C, the monoclinic content further decreases to 3.12%, reflecting enhanced stabilization of the tetragonal phase due to improved diffusion kinetics and densification. The XRD peaks corresponding to the tetragonal phase become sharper, while a noticeable

shift in peak position is observed in the $27\text{--}33^\circ 2\theta$ range, as shown in the magnified XRD patterns (Figure 4.11(b)). This shift can be attributed to lattice strain, oxygen vacancy formation, and thermal expansion effects occurring during sintering, which slightly alter the lattice parameters of the tetragonal phase. This shift suggests the presence of compressive residual stress, which arises from thermal contraction during cooling and potential phase transformations. The sample heat-treated at 1450°C shows sharper peaks and minimal monoclinic phase content, indicating better crystallinity, phase stability, and stress relaxation [68]. Therefore, the 1450°C sample appears optimal for achieving enhanced mechanical performance and structural integrity, although excessive grain growth must be monitored to avoid brittleness.

At 1450°C , the monoclinic phase content reaches its lowest value of 2.6%, as depicted in the monoclinic phase content graph (Figure 4.12), indicating near-complete stabilization of the tetragonal phase. The XRD peaks for the tetragonal phase (Figure 4.11(a)) become sharper and more defined, confirming improved crystallinity and grain growth. The peak shift observed in the $27\text{--}33^\circ 2\theta$ range (Figure 4.11(b)) becomes more prominent, suggesting further lattice relaxation and reduction of internal stresses at this higher temperature. The significant reduction in the monoclinic phase and the peak shift highlight the lattice modifications occurring during the sintering process.

Nevertheless, the monoclinic phase is still present at this temperature as a result of the inadequate stabilization of the tetragonal structure and the limited diffusion of the grain boundary [69]. The tetragonal phase is predominantly preserved when the spheres are subjected to a temperature of 1450°C . Cerium's valency is either 3^+ or 4^+ , with Ce^{4+} being

the more stable oxidation state. The Ce^{3+} ions oxidize to produce Ce^{4+} ions throughout the breakdown process. Given the ionic radius, the likelihood of substituting Ce^{3+} ions within the ZrO_2 crystal structure at low temperatures is exceedingly unlikely; thus, the sole possibility is the creation of Ce^{4+} ions through the oxidation of Ce^{3+} ions. The tetragonal phase is favored by the substitution of Zr^{4+} ions by these Ce^{4+} ions in the ZrO_2 crystal structure. The oxalate breakdown stage may present an additional opportunity for the synthesis of the Ce_2O_3 compound. CeO_2 undergoes a sequence of oxides as the oxygen partial pressure lowers, with Ce_2O_3 being the most reduced form [70,71]. But, the X-ray diffraction patterns do not show the presence of Ce_2O_3 formations.

The strain energy linked to the zirconium ion is mitigated by the presence of these dopant ions, as evidenced by a recent study on the stability of the t-phase of zirconia by Ce^{4+} ions [72]. Nevertheless, the ceria matrix phase, which was present during heat treatment, proved to be largely successful in preventing the coarsening of the ZrO_2 particles, as seen by the small drop in peak width at the elevated temperature.

The monoclinic phase content decreases progressively with increasing temperature, while the peak shift highlights the lattice modifications and stabilization of the tetragonal phase. The sample sintered at 1450°C demonstrates the best phase stability and microstructural uniformity, making it highly suitable for applications requiring superior mechanical and thermal performance. The XRD patterns (Figures 4.11(a) and 4.11(b)) and the monoclinic phase content graph (Figure 4.12) demonstrate the influence of sintering temperature on the phase transformation behavior of CSZ.

In a study, it was observed that during conventional sintering, various mol percentages of ceria-doped yttria-stabilized zirconia (Ce-YSZ) exhibited an increase in the monoclinic phase with rising sintering temperatures[73]. This behavior contrasts with that observed in HMW Sintering, where the monoclinic phase typically decreases as the sintering temperature increases. The comparison shows that the monoclinic ZrO_2 (m- ZrO_2) content for the conventionally sintered sample at 1600°C is 13.89%[66], while for the microwave-sintered samples, it significantly decreases to 3.12% at 1400°C and 2.6% at 1450°C. This indicates that microwave sintering achieves superior phase stability with lower monoclinic content even at higher sintering temperatures, due to its uniform volumetric heating and faster sintering process, which minimize grain coarsening and internal stresses that typically promote phase transformation.

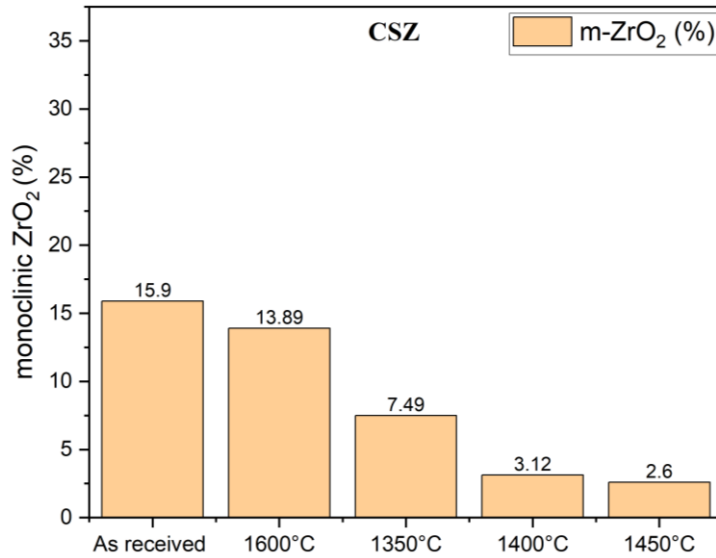


Figure 4. 12 Estimated amount of monoclinic phase of CSZ sintered samples.

The average crystallite size of CSZ sintered at 1350°C, 1400°C, and 1450°C using hybrid microwave sintering was determined using the Scherrer equation, as presented in Figure

4.13. The results demonstrate a distinct increase in the average crystallite size as the sintering temperature increases, underscoring the significance of thermal energy in the enhancement of crystallinity and the promotion of grain growth. In the as-received powder, the average crystallite size is measured as 13.84 nm, indicating fine crystalline domains. Upon sintering at 1350°C, the crystallite size increases slightly to 14.53 nm, suggesting the initial stages of grain growth and densification driven by enhanced diffusion mechanisms. At 1400°C, the average crystallite size further increases to 14.72 nm, reflecting moderate grain growth as higher thermal energy facilitates better particle coalescence and grain boundary mobility. Finally, at 1450°C, the crystallite size reaches its maximum value of 16.33 nm, indicating significant grain growth and improved crystallinity due to accelerated ion diffusion and grain boundary migration at this temperature. This trend aligns with the XRD results, where sharper and more defined peaks were observed, confirming enhanced phase stability and structural uniformity. The spectral lines' width decreases as the sintering temperature increases, which is indicative of the steady increase in crystallite size throughout the sintering process [74]. The same trend is observed for the conventional sintering process also[71].

The effectiveness of hybrid microwave sintering in promoting densification and grain development is demonstrated by the increase in crystallite size with temperature. This is particularly true at 1450°C, where the material achieves the highest crystallinity, rendering it ideal for applications that require superior mechanical and thermal properties.

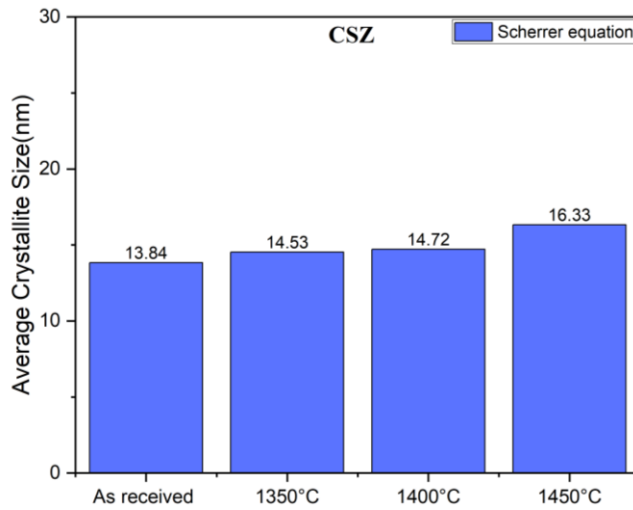


Figure 4. 13 Estimated crystallite sizes of CSZ sintered samples using the Scherrer equation

Table 4. 2 Thermal conductivity of the CSZ HMW heat-treated corresponding samples at different temperatures

Material	Thermal Conductivity (W/mK)
1600°C H.T.	1.9138 [66]
1350°C H.T.	0.5964
1400°C H.T.	0.4027
1450°C H.T.	0.4159

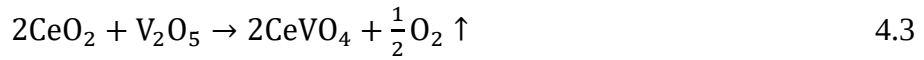
Table 4.2 illustrates the thermal conductivity values of all sintered samples. A consistent reduction in thermal conductivity was noted for HMW samples sintered at 1350°C, 1400°C, and 1450°C as the sintering temperature increased. Both samples demonstrate nearly identical values of m-ZrO₂ phase content. Moreover, the HMW-sintered sample at 1400°C and 1450°C possesses the lowest thermal conductivity, which can be attributed to the highest reduction in the m-phase. The slight increase in thermal conductivity of HMW sintered CSZ at higher temperatures may result from improved grain boundary connectivity

and reduced porosity during sintering, which temporarily enhances phonon transport. Additionally, point defect interactions and minor electron contributions can offset typical phonon scattering, leading to this anomaly [75,76]. The volume fractions of phases in the sintered samples are considered a crucial component that can substantially influence the thermal conductivity values. The thermal conductivity of the traditionally sintered sample at 1600 °C is 1.9138 W/mK, significantly higher than that of the microwave sintered sample [66]. The decrease in thermal conductivity at higher sintering temperatures in hybrid microwave sintering is primarily due to phase stability issues. Elevated temperatures can induce partial destabilization of the ceria-stabilized zirconia matrix, leading to phase transitions or the formation of secondary phases. These phase changes introduce lattice distortions and defects that increase phonon scattering, thereby reducing the material's ability to conduct heat effectively [77].

4.2.2 Hot Corrosion

The SEM images of Ceria Stabilized Zirconia (CSZ) samples sintered at 1350°C, 1400°C, and 1450°C after hot corrosion reveal distinct microstructural features influenced by the sintering temperature. At 1350°C, the microstructure shows significant porosity and poorly defined grain boundaries, with smaller, less consolidated grains. The presence of voids and intergranular spaces indicates incomplete densification, providing evidence of early-stage sintering. At 1400°C, the microstructure exhibits noticeable grain growth and a reduction in porosity compared to the sample sintered at 1350°C. The grain boundaries are better defined, and the particles appear more consolidated, although some localized voids and cracks are still visible. Initial signs of secondary phase formation can be observed, suggesting chemical interactions occurring during the corrosion process. At 1450°C, the

microstructure shows significant grain growth and a highly dense structure with minimal visible porosity. Notably, rod-like structures[78], indicative of YVO_4 and CeVO_4 , are observed, pointing to the formation of secondary phases as a result of chemical reactions shown below.



The grains at this temperature appear larger, with well-defined grain boundaries, indicating extensive grain boundary migration and particle coalescence at higher sintering temperatures[79]. Overall, the microstructural evolution from 1350°C to 1450°C highlights a progressive improvement in densification, grain growth, and the appearance of secondary phases such as YVO_4 at elevated temperatures.

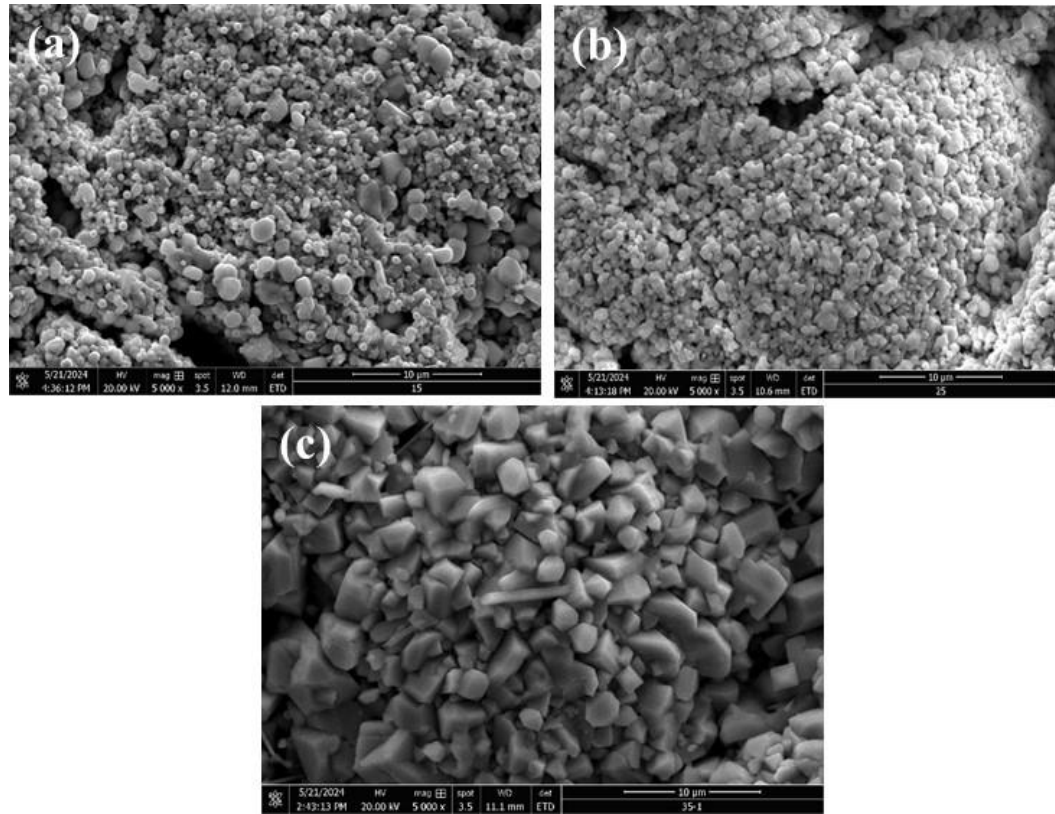


Figure 4. 14 SEM of CSZ sintered samples for 2 h using HMW heating at (a) 1350 °C, (b) 1400 °C, (c) 1450 °C

The XRD patterns and monoclinic phase content graph of Ceria Stabilized Zirconia (CSZ) samples subjected to hot corrosion after hybrid microwave sintering at 1350°C, 1400°C, and 1450°C reveal critical insights into phase composition and structural changes. In the as-received powder (Figure 4.15(a)), the XRD peaks predominantly correspond to the tetragonal phase ($t\text{-ZrO}_2$) with minor peaks corresponding to the monoclinic phase ($m\text{-ZrO}_2$). The monoclinic phase content is 15.9% (Figure 4.16), reflecting the baseline stability of the as-received CSZ sample.

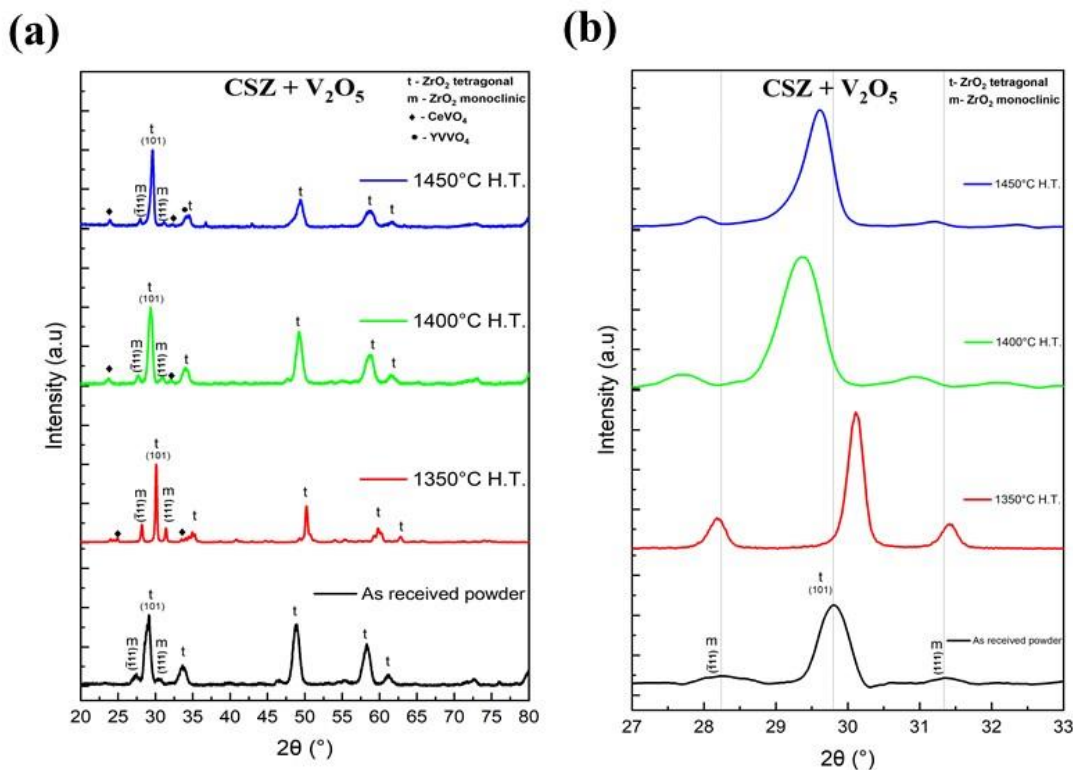


Figure 4. 15 XRD of as-received CSZ powder and hot corroded CSZ HMW heat-treated corresponding samples at different temperatures for 2θ ranges from (a) 20° to 80° and (b) 27° to 33°

After hot corrosion at 1350°C , the XRD patterns (Figure 4.16) display a significant increase in the intensity of monoclinic phase peaks at $2\theta \approx 28^\circ$ and 31° , indicating a transformation from the tetragonal to the monoclinic phase. The monoclinic phase content increases to 29.57% (Figure 4.16), the highest among the sintered samples. This sharp increase can be attributed to the reaction of the CSZ matrix with vanadium pentoxide (V_2O_5), which depletes ceria (CeO_2), destabilizing the tetragonal phase and promoting the destructive phase transformation to the monoclinic phase [78]. The incomplete densification at this lower sintering temperature further accelerates the interaction between the corrosive species and the material [80].

At 1400°C, the XRD patterns indicate a partial stabilization of the tetragonal phase, as observed by the reduced intensity of monoclinic peaks compared to the 1350°C sample. The monoclinic phase content decreases to 19.37% (Figure 4.16), reflecting improved phase stability. This decrease can be attributed to enhanced densification and grain growth at 1400°C, which reduces porosity and limits the pathways for corrosive species to interact with the material. However, secondary phase peaks corresponding to CeVO_4 and YVO_4 begin to emerge, indicating the reaction between ceria and vanadium-based corrosive agents. These phases signify the progressive depletion of the stabilizing agents, which could still contribute to localized destabilization of the tetragonal phase.

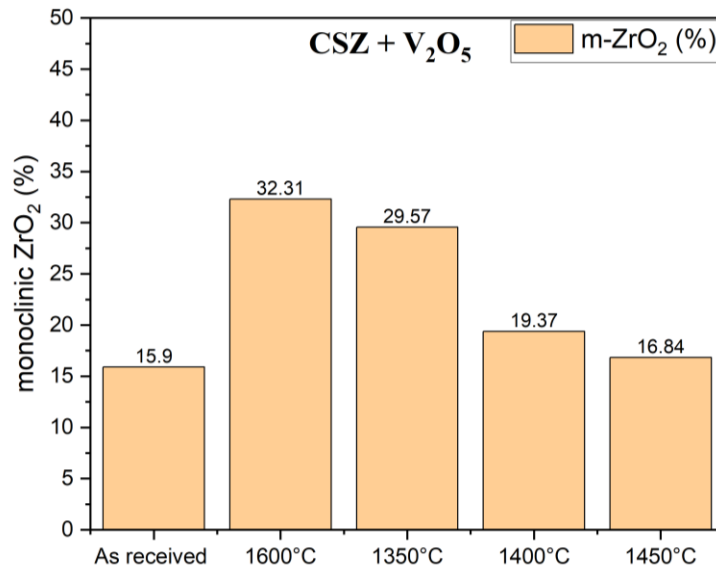


Figure 4. 16 Estimated amount of monoclinic phase of CSZ sintered samples after hot corrosion.

At 1450°C, the XRD patterns (Figure 4.15(a)) show further stabilization of the tetragonal phase, with sharper and more defined peaks and reduced intensity of monoclinic reflections. The monoclinic phase content is minimized to 16.84% (Figure 4.16), approaching the as-received value. The higher thermal energy at 1450°C promotes

significant grain growth and improved densification, which enhances the structural integrity of the material and reduces the susceptibility to phase transformation. However, peaks corresponding to YVO_4 and CeVO_4 are more distinct at this temperature, suggesting advanced chemical interactions between the yttria-ceria stabilizers and V_2O_5 . The formation of these secondary phases is further confirmed by the rod-like YVO_4 structures observed in the SEM analysis.

The monoclinic phase content increases significantly at 1350°C due to incomplete densification and increased phase transformation under hot corrosion. At 1400°C , partial stabilization of the tetragonal phase is achieved, but secondary phases such as CeVO_4 and YVO_4 begin to form. At 1450°C , the monoclinic phase content is minimized, and the tetragonal phase shows improved stability due to enhanced densification and grain growth. However, the formation of secondary phases, particularly YVO_4 and CeVO_4 , remains evident, indicating ongoing chemical reactions during hot corrosion at elevated temperatures. It is evident that when compared to the study[78] conducted on conventionally sintered samples at 1400°C , which showed a value of 48.9%, and the HMW sample exhibited a value of 19.37%. Also It is evident that when compared to the study conducted on conventionally sintered samples at 1600°C , which showed a value of 32.31%, and the HMW sample exhibited a value of 19.37% at 1450°C . Furthermore, this value gradually decreases as the sintering temperature increases. This clearly shows how HMW sintering affects the sample.

4.3 Yttria-Titania-Zirconia (YTiZ)

Following sections describe the two processes namely HMW sintering process and Hot corrosion.

4.3.1 HMW Sintering Process

Figure 4.17 displays a SEM picture depicting the unique spherical morphology of the YTiZ particles. The spherical morphology is a characteristic feature arising from the application of the Hollow Oxide Spherical Process (HOSP) in the powder's manufacturing.

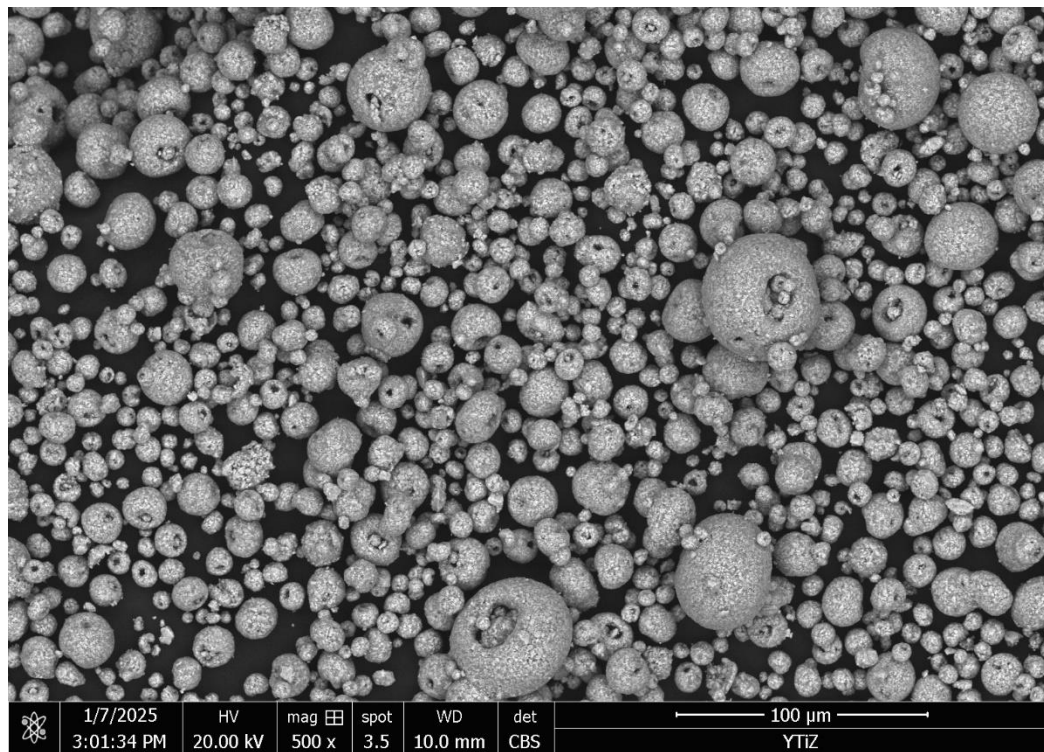


Figure 4. 17 SEM of the as-received YTiZ spherical powder.

The SEM images of microwave-sintered Yttria Titania Zirconia (YTiZ) composites at 1350°C, 1400°C, and 1450°C reveal clear differences in microstructure concerning grain size, porosity, and densification. At 1350°C, the microstructure shows small and uniform

grains with well-distributed porosity, indicating partial densification and controlled grain growth, which ensures a stable and reliable structure. At 1400°C, the grain size increases noticeably, suggesting enhanced grain boundary migration and improved densification, though some isolated voids and agglomerates remain, reducing the uniformity. In contrast, at 1450°C, the microstructure exhibits extensive grain growth and near-complete densification, with minimal porosity but irregular and excessively large grains indicative of over-sintering. This uncontrolled grain growth can compromise the mechanical performance of the material due to reduced grain boundary area. Overall, the microstructure at 1350°C achieves the best balance between grain size and porosity, avoiding excessive grain growth while maintaining structural uniformity and stability, making it the optimal sintering temperature for improved phase stability and mechanical performance.

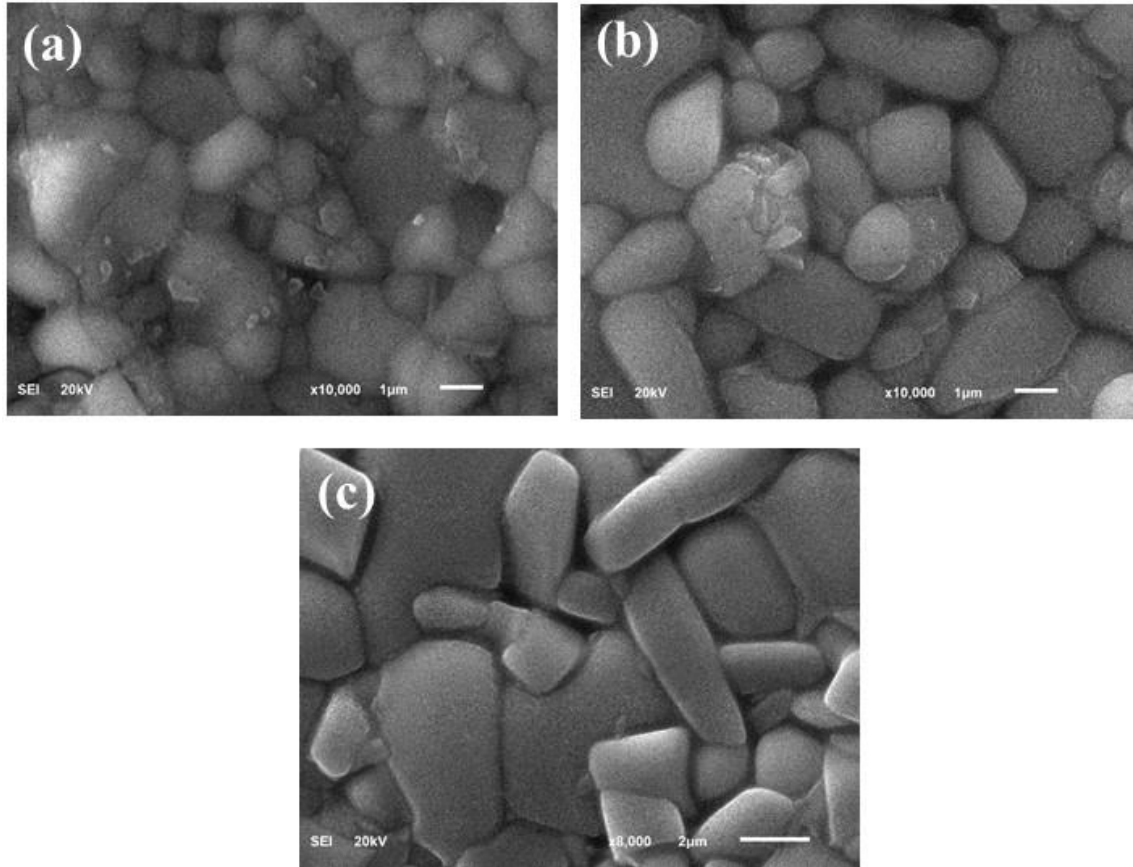


Figure 4. 18 SEM of YTiZ sintered samples for 2 h using HMW heating at (a) 1350 °C, (b) 1400 °C, (c) 1450 °C

The XRD analysis and monoclinic phase content graph of Yttria Titania Zirconia (YTiZ) composite sintered at 1350°C, 1400°C, and 1450°C reveal significant variations in phase composition and stability. In the as-received powder (Figure 4.23), the XRD peaks are dominated by the monoclinic phase (m-ZrO₂), with the monoclinic phase content quantified as 100% (Figure 4.25). This indicates that the material in its as-received state is fully monoclinic and lacks tetragonal phase stabilization.

Upon sintering at 1350°C, the XRD patterns (Figure 4.19(a)) show a significant transformation of the monoclinic phase to the tetragonal phase (t-ZrO₂), as indicated by the appearance of intense peaks corresponding to the tetragonal phase at $2\theta \approx 30^\circ$. The monoclinic phase content decreases sharply to 17.62% (Figure 4.25), which is the lowest

among the sintered samples. This indicates that the tetragonal phase remains the most stable at 1350°C, with minimal phase transformation to monoclinic zirconia. Additionally, no significant peaks corresponding to secondary phases such as ZrTiO_4 are observed, suggesting better phase purity and stability at this sintering temperature.

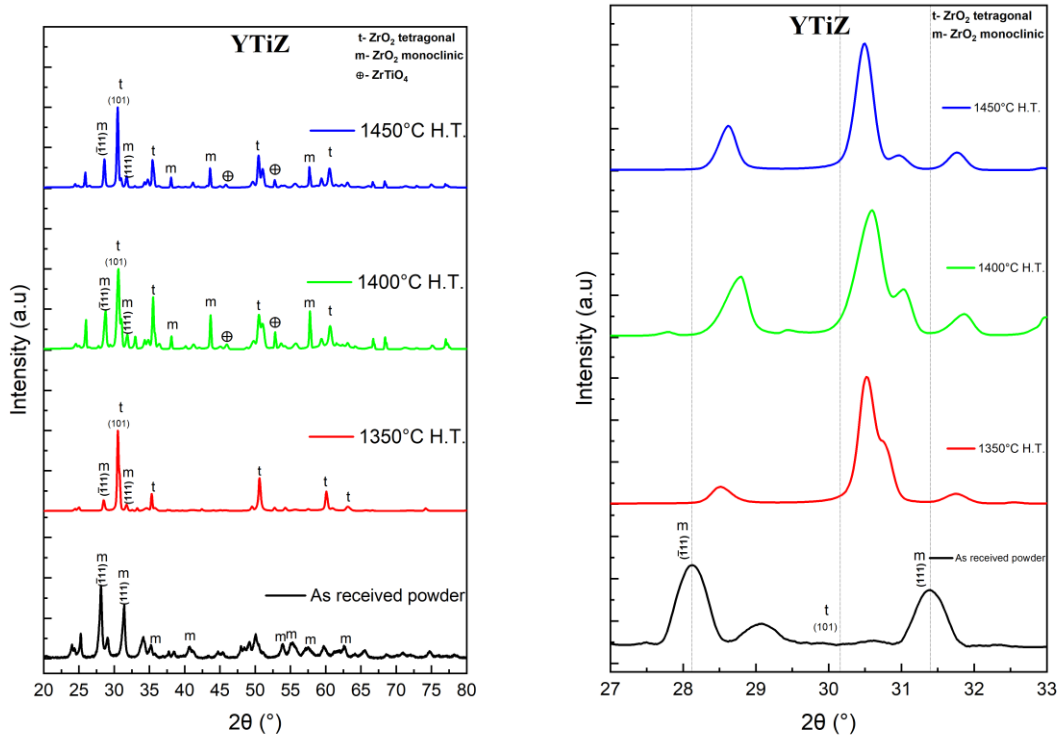


Figure 4. 19 XRD of YTiZ as-received powder and HMW heat-treated corresponding samples different temperatures for 2θ ranges from (a) 20° to 80° and (b) 27° to 33°

At 1400°C, the XRD patterns indicate a partial destabilization of the tetragonal phase, as evidenced by the increased intensity of monoclinic phase peaks at $2\theta \approx 28^\circ$ and 31° . The monoclinic phase content rises to 39.72% (Figure 4.25), suggesting a reversal of the stabilization process observed at 1350°C. This increase in monoclinic content may be attributed to localized stresses or interactions within the material, which promote the tetragonal-to-monoclinic phase transformation. Additionally, peaks corresponding to

ZrTiO₄ appear in the XRD patterns, indicating the formation of a secondary phase due to the reaction between zirconia and titania at this elevated temperature [81].

At 1450°C, the XRD patterns show further stabilization of the tetragonal phase compared to 1400°C, as the monoclinic phase content decreases to 35.01% (Figure 4.20). However, the presence of well-defined ZrTiO₄ peaks remains evident, indicating an increased degree of interaction between zirconia and titania at this higher sintering temperature [82]. While the material shows improved densification and crystallinity at 1450°C, the formation of secondary phases and the persistence of monoclinic zirconia suggest the overall phase. The sample sintered at 1350°C demonstrates the best phase stability, with the lowest monoclinic phase content (17.62%) and minimal formation of secondary phases like ZrTiO₄. At higher sintering temperatures of 1400°C and 1450°C, the stabilization of the tetragonal phase is partially reduced due to the increased monoclinic phase content and the formation of secondary phases. These observations suggest that 1350°C is the optimal sintering temperature for achieving better phase stability and purity in the Yttria Titania Zirconia composite. A similar trend is observed for the conventionally sintered samples. In one study, the structure remains stable at 1300°C, but at temperatures around 1400°C and 1500°C, the formation of ZrTiO₄ and an increase in the monoclinic phase were noted[81]. The chart compares the monoclinic zirconia (m-ZrO₂) content in YTiZ samples under conventional (1600°C) and hybrid microwave sintering (1350°C, 1400°C, 1450°C). The conventional sample retained 78.87% m-ZrO₂, while microwave-sintered samples showed significantly reduced values: 17.62% at 1350°C, 39.72% at 1400°C, and 35.01% at 1450°C. The lowest m-ZrO₂ content was achieved at 1350°C, demonstrating that hybrid

microwave sintering, even at lower temperatures, is more effective at stabilizing zirconia phases than conventional sintering.

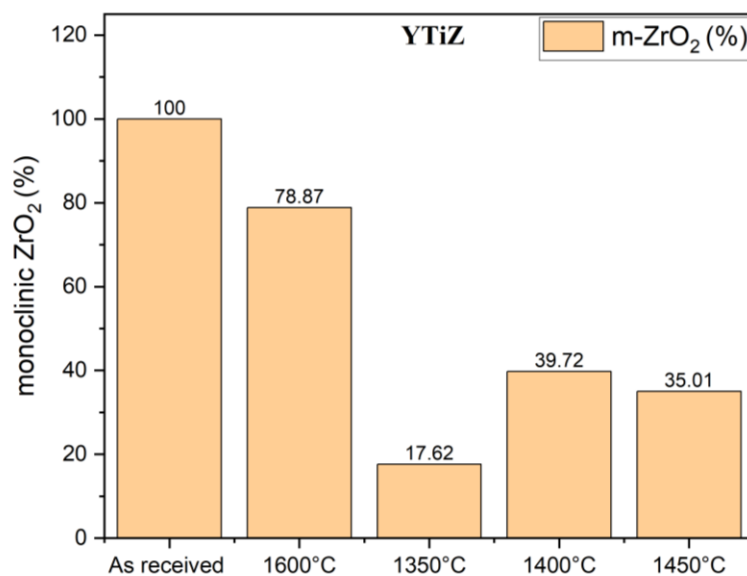


Figure 4. 20 Estimated amount of monoclinic phase of YTiZ sintered samples

The average crystallite size of Ytria Titania Zirconia (YTiZ) composites sintered at 1350°C, 1400°C, and 1450°C using microwave sintering, as calculated using the Scherrer equation, reveals a distinct trend with increasing sintering temperature. In the as-received state, the crystallite size is measured as 10.06 nm, indicating a very fine nanocrystalline structure. Upon sintering at 1350°C, the crystallite size increases significantly to 26.96 nm, reflecting the onset of grain boundary migration and particle coalescence facilitated by microwave energy, which promotes densification while maintaining a relatively fine microstructure. At 1400°C, the crystallite size decreases slightly to 23.5 nm, which could be attributed to the onset of secondary phase formation or localized constraints on grain growth due to stress buildup within the material during sintering. With a further increase

to 1450°C, the crystallite size decreases to 21.98 nm, suggesting that grain growth is partially inhibited, likely due to the formation of secondary phases such as ZrTiO_4 , which restricts further crystallite coalescence. This trend indicates a balance between grain growth and phase stabilization at higher sintering temperatures. Overall, the significant crystallite growth at 1350°C, followed by a controlled reduction at higher temperatures, highlights the effect of microwave sintering on the evolution of the microstructure, with 1350°C showing the most pronounced crystallite size growth while maintaining structural uniformity.

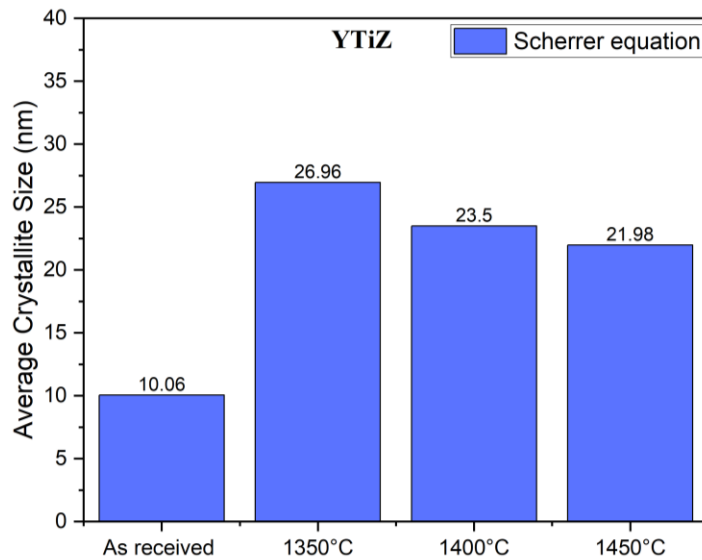


Figure 4. 21 Estimated crystallite sizes of YTiZ sintered samples using the Scherrer equation

Table 4. 3 Thermal conductivity of the YTiZ HMW heat-treated corresponding samples at different temperatures

Material	Thermal Conductivity (W/mK)
1600°C H.T.	1.5888
1350°C H.T.	0.2415
1400°C H.T.	0.2841
1450°C H.T.	0.2875

Table 4.3 presents the thermal conductivity values of all sintered samples. For HMW samples, a consistent rise in thermal conductivity was noted with the elevation of the sintering temperature for samples sintered at 1350°C, 1400°C, and 1450°C. The sample having less monoclinic m-ZrO₂ phase content shows lower thermal conductivity. The thermal conductivity of ceria-stabilized zirconia decreases significantly in hybrid microwave-sintered samples compared to conventional sintering at 1600°C (1.5888 W/m·K). The values for microwave sintering are much lower: 0.2415 W/m·K at 1350°C, 0.2841 W/m·K at 1400°C, and 0.2875 W/m·K at 1450°C. The lowest conductivity at 1350°C indicates that microwave sintering enhances phase stability and creates microstructural changes, such as defects and reduced grain boundary density, which scatter phonons and reduce heat transfer efficiency. Furthermore, the HMW-sintered samples at 1400°C and 1450°C exhibit the lowest thermal conductivity, attributable to the most significant drop in the m-phase. The volume fractions of phases in the sintered samples are considered a crucial component that can substantially influence the thermal conductivity values [63].

4.3.2 Hot Corrosion

The SEM analysis of the Yttria Titania Composite after hot corrosion for samples heat-treated at 1350°C, 1400°C, and 1450°C reveals notable differences in their microstructures shown in Figure 4.22. In the 1350°C sample, well-defined rod-like TiO₂ structures are prominently embedded within the surrounding zirconia grains, suggesting effective crystallization and integration of TiO₂ during heat treatment [83].

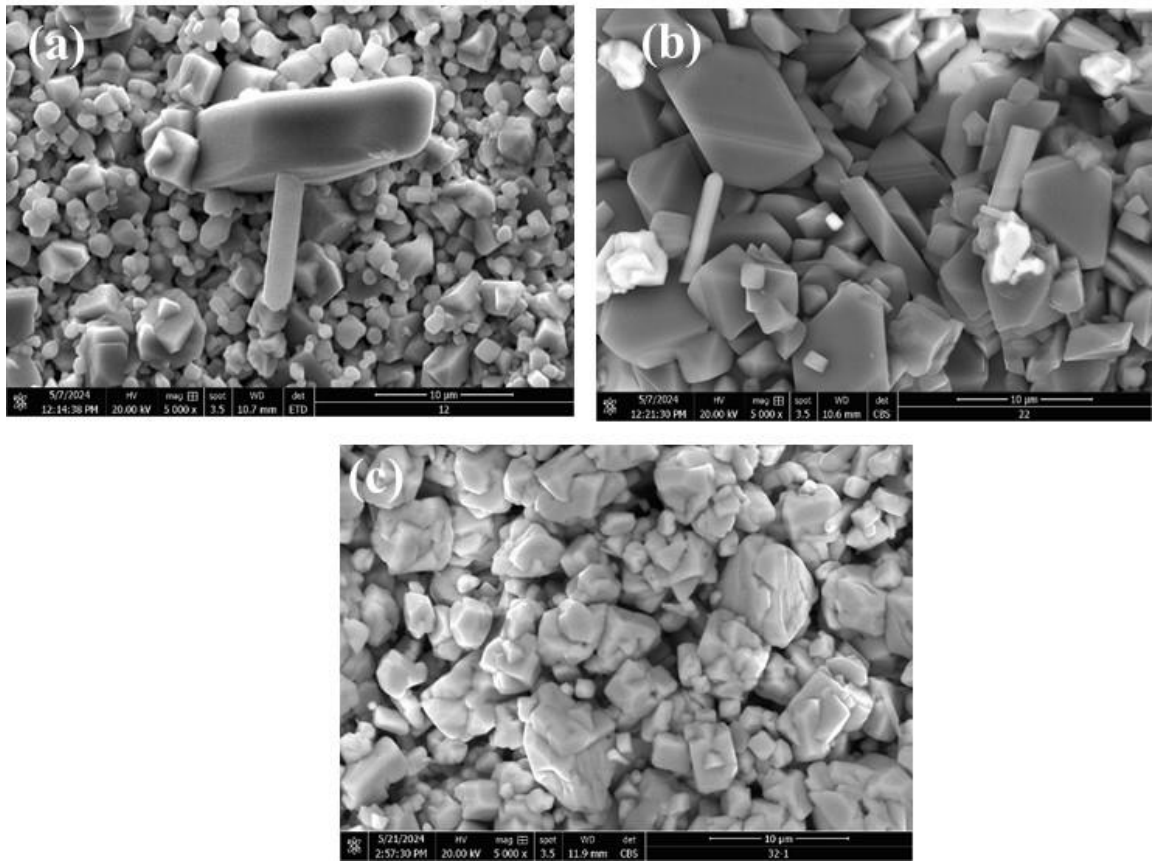


Figure 4. 22 SEM of hot corroded 8YTiZ sintered samples for 2 h using HMW heating at (a) 1350 °C, (b) 1400 °C, (c) 1450 °C

This microstructure exhibits tight packing of zirconia particles and good reinforcement from TiO₂ rods, enhancing mechanical strength and resistance to hot corrosion. In the 1400°C sample, fewer and less-defined TiO₂ rods are present, accompanied by larger,

tetragonal zirconia grains, indicating partial grain growth and less effective TiO_2 crystallization. This may reduce the overall structural integrity of the composite. The 1450°C sample shows densely packed zirconia grains with a uniform size distribution, but distinct TiO_2 rods are absent, likely due to excessive grain growth or dissolution of TiO_2 at this higher temperature [84]. While the zirconia grains are well-packed, the lack of visible TiO_2 reinforcement may limit the composite's mechanical performance. Therefore, the 1350°C heat-treated sample demonstrates the most favorable microstructure, with well-integrated TiO_2 rods providing enhanced durability and resistance to hot corrosion.

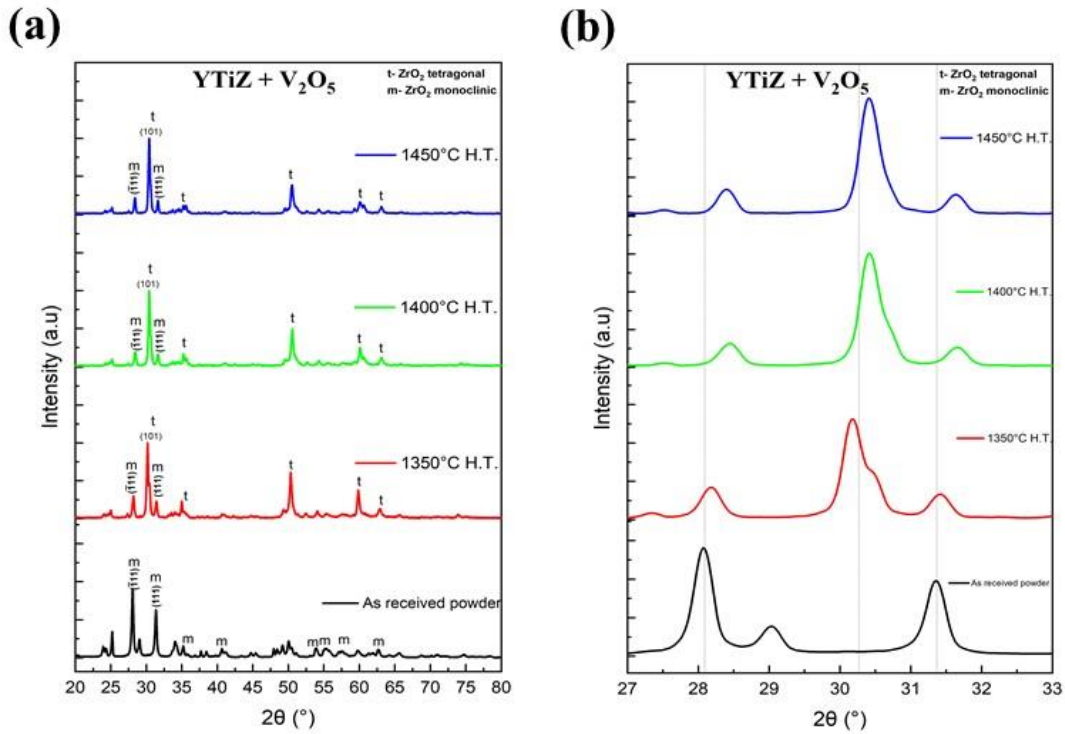


Figure 4.23 XRD of YTiZ as-received YTiZ powder and hot corroded YTiZ HMW heat-treated corresponding samples at different temperatures for 2θ ranges from (a) 20° to 80° and (b) 27° to 33°

The XRD study of the Ytria-Titania Composite after hot corrosion offers important information on the phase transformations caused by various heat treatment temperatures. The as-received powder primarily consists of monoclinic zirconia (m-ZrO_2), as indicated

by the dominance of monoclinic peaks in the XRD patterns (Figure 4.23(a)). This phase, while stable at room temperature, is prone to phase transformation under thermal and mechanical stresses, making it less suitable for applications requiring high durability and corrosion resistance.

Upon heat treatment at 1350°C, a significant reduction in the monoclinic phase content is observed, dropping to 27.71%. This can be seen clearly in the XRD pattern for the 1350°C sample (Figure 4.23(a)) and the detailed zoomed-in region (Figure 4.23(b)). The lowest monoclinic content among all the samples analyzed indicates a successful transformation to the tetragonal zirconia (t-ZrO₂) phase. The tetragonal phase is known for its higher toughness and improved resistance to crack propagation, making it advantageous for applications involving thermal cycling and hot corrosion[85]. The stabilization of the tetragonal phase at this temperature shows that 1350°C is the ideal heat treatment temperature for balancing phase transformation and preventing excessive grain development.

At 1400°C, the monoclinic content increases to 36.28%, as shown in the corresponding XRD pattern (Figure 4.23(a)). This indicates partial destabilization of the tetragonal phase. The detailed peak analysis (Figure 4.23(b)) reveals the presence of monoclinic peaks that are more pronounced than in the 1350°C sample. This suggests that while tetragonal stabilization is achieved, the higher temperature may lead to a slight reversion to the monoclinic phase.

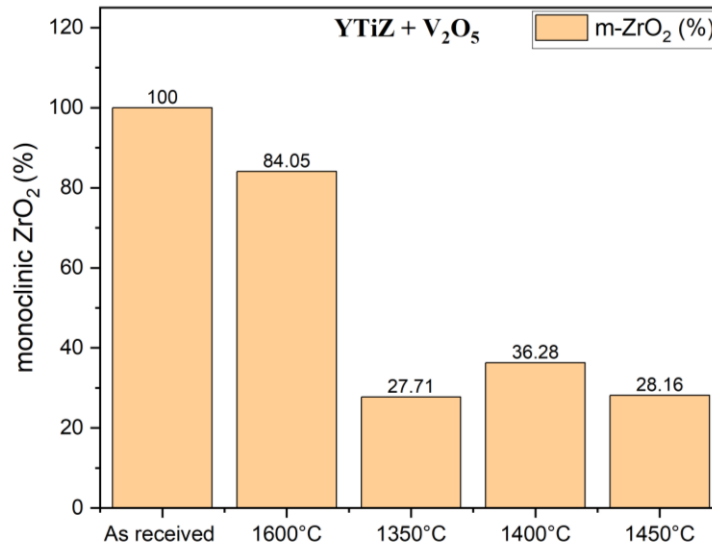


Figure 4. 24 Estimated amount of monoclinic phase in YTiZ sintered samples after hot corrosion.

Heat treatment at 1450°C results in a monoclinic content of 28.16%, which can be observed in Figures 4.23(a) and 4.23(b). This shows better stabilization of the tetragonal phase compared to the 1400°C sample. However, the minimal difference in monoclinic content between the 1350°C and 1450°C samples does not justify the increased energy expenditure and grain coarsening risk associated with the higher temperature. Figure 4.24 shows a bar chart summarizing the monoclinic phase composition across all samples, which demonstrates this tendency. The chart compares monoclinic zirconia (m-ZrO₂) content samples under conventional (1600°C) and hybrid microwave sintering (1350°C, 1400°C, 1450°C). The conventional sample retained 84.05% m-ZrO₂, while the microwave-sintered samples showed significant reductions: 27.71% at 1350°C, 36.28% at 1400°C, and 28.16% at 1450°C. The lowest m-ZrO₂ content was observed at 1350°C, indicating that the addition of V₂O₅ enhances phase stabilization, with hybrid microwave sintering being more effective at reducing the monoclinic phase even at lower temperatures.

Based on these observations, the 1350°C heat-treated sample emerges as the most effective condition for phase stabilization. It achieves the lowest monoclinic content, maximizing the tetragonal phase fraction while maintaining cost and energy efficiency. This improved heat treatment improves the material's structural stability and resistance to hot corrosion, making it an ideal alternative for advanced applications requiring long-lasting ceramic composites.

CHAPTER 5

CONCLUSION

This study investigated the effects of hybrid microwave sintering (HMW) on the phase evolution, microstructure, thermal conductivity, and hot corrosion behavior of zirconium-stabilized compounds, specifically 8YSZ, CSZ, and YTiZ. The findings revealed significant improvements in material performance with HMW compared to conventional sintering (CS). The key findings for each sample, including comparisons with conventionally sintered samples where applicable, are summarized below:

5.1 8 wt% Yttria-Stabilized Zirconia (8YSZ)

- HMW sintering effectively sinters 8YSZ at lower temperatures (1450°C) compared to conventional sintering (1600°C), demonstrating energy efficiency.
- The HMW-sintered samples are free from microcracks, unlike conventionally sintered samples, which suffer from microcracks due to martensitic phase transformation and volumetric expansion (3–5%).
- HMW processing significantly suppresses the transformation of the tetragonal (t-ZrO₂) to monoclinic (m-ZrO₂) phase.
- The reduction in the monoclinic phase content enhances the mechanical properties and stability of the material.
- Enhanced diffusion of Yttrium ions and the generation of oxygen vacancies during HMW sintering contribute to phase stability and prevent phase transformations.

- The structural and thermal conductivity differences between HMW and conventional samples underscore the sensitivity of 8YSZ to sintering methods and parameters.
- HMW samples exhibited better hot corrosion resistance due to improved densification, which showed more pronounced microcracking and phase transformation.
- HMW processing offers superior microstructural refinement, improved phase stability, and defect suppression, making it a promising technique for sintering zirconia-based ceramics.

5.2 Ceria-Stabilized Zirconia (CSZ)

- HMW-sintered CSZ exhibited improved phase stability and grain coalescence across different sintering temperatures.
- The tetragonal phase was retained effectively with a steady reduction in monoclinic phase content as the sintering temperature increased.
- The highest phase stability was observed at 1450°C, confirming the effective stabilization of the CSZ structure.
- The refined microstructure and stabilized phases highlight the suitability of CSZ for high-temperature applications.

5.3 Yttria-Titania-Zirconia (YTiZ) Composite

- The YTiZ composite processed through HMW demonstrated superior phase retention and structural stability.

- The optimal phase stability was observed at 1350°C, where the tetragonal phase content was maximized.
- The addition of titania enhanced the stabilization of the tetragonal phase, mitigating phase transformation.
- The reduction in monoclinic phase content with increasing sintering temperature underscores the effectiveness of HMW in maintaining a uniform and stable structure.
- These results establish YTiZ as a promising material for applications in harsh thermal and corrosive environments.

These results demonstrate that hybrid microwave sintering offers significant advantages over conventional sintering in enhancing the performance, phase stability, and corrosion resistance of zirconium-stabilized ceramics, making them suitable for high-temperature applications such as thermal barrier coatings as well as solid oxide fuel cells. Among all the samples, CSZ sintered at 1450°C demonstrated the highest phase stability both after sintering and post-hot corrosion testing. The significant retention of the tetragonal phase and the lowest monoclinic phase content highlight the efficiency of hybrid microwave sintering (HMW) in achieving superior phase stabilization. The unique heating mechanism of HMW ensures uniform energy absorption and rapid diffusion, which effectively suppresses undesirable phase transformations even under aggressive high-temperature and corrosive conditions. This indicates that HMW sintering is an efficient processing method for enhancing the performance of CSZ in demanding applications.

5.4 Future Work and Recommendations

- Investigate the effects of varying microwave sintering parameters such as power levels, sintering time, and holding temperatures to enhance phase stability and microstructural properties.
- Conduct hot corrosion experiments in diverse environments such as Na_2SO_4 and mixtures of Na_2SO_4 and V_2O_5 to evaluate corrosion resistance under different conditions.
- Perform a comparative analysis by replacing hybrid microwave sintering (HMW) with full microwave sintering (FMW) to assess differences in material properties, phase stability, and performance.
- Replace isothermal hot corrosion tests with cyclic hot corrosion studies to simulate real-world thermal cycling and evaluate the durability of materials under fluctuating thermal and chemical stresses.
- Incorporate advanced techniques such as Transmission Electron Microscopy (TEM), X-ray Photoelectron Spectroscopy (XPS), and Raman spectroscopy for detailed microstructural and chemical analysis
- Implement computational modeling and simulation of hot corrosion processes to predict degradation mechanisms, optimize material compositions, and enhance corrosion resistance.

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Conferences and Publications:

1. Morsi M. Mahmoud, Nestor Ankah, **Mohammed Arif**, Zuhair M. Gasem, “Sintering of Yttria Stabilized Zirconia using Hybrid Microwave Processing”, Arabian Journal of Science and Engineering (AJSE), 2024.
2. Morsi M. Mahmoud, **Mohammed Arif**, Zuhair M. Gasem, “Hybrid Microwave Sintering of Yttria Stabilized Zirconia”, Proceedings of SCOPE, 2023.