SYNTHESIS OF NiO-8YSZ NANOPOWDERS BY CO-PRECIPITATION METHOD AND ITS STRUCTRUAL AND MICROSTRUCTURAL CHARACTERIZATION

A THESIS SUBMITTED IN PARTIAL FULFILLMENT
OF THE REQUIREMENTS FOR THE DEGREE OF

Bachelor of Technology
in
Ceramic Engineering

By
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Department of Ceramic Engineering
National Institute of Technology
Rourkela
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Under the Guidance of
Prof Bibhuti B. Nayak

Department of Ceramic Engineering
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CERTIFICATE

This is to certify that this thesis entitled, "**SYNTHESIS OF NiO-8YSZ NANOPOWDERS BY CO-PRECIPITATION METHOD AND ITS STRUCTRUAL AND MICROSTRUCTURAL CHARACTERIZATION**" submitted by Mr. **DEEPAK PATGIRI** in partial fulfillments for the requirements for the award of Bachelor of Technology Degree in Ceramic Engineering at National Institute of Technology, Rourkela is an authentic work carried out by him under my guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University / Institute for the award of any Degree or Diploma.

Date:

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6th May 2011 **DEEPAK PATGIRI**

CONTENTS

ABSTRACT

This project is mainly based on the effect of synthesis route on the NiO-8YSZ samples prepared by co precipitation method by varying the weight percentage of NiO.

The fuel cell has tremendous future prospect not because of its high efficiency and low emission, and also for their modular and distributed nature and zero-noise pollution. This may solve our future requirement, as it plays an essential role in any future hydrogen fuel economy. YSZ has very good conductivity of O^{2-}, the presence of Ni enhances the electronic conductivity. The composite of NiO-8YSZ is used to prepare Solid Oxide Fuel Cell's (SOFC) anode part. This allows us to do further study to improve the sample for SOFC using co precipitation method.

Our objective in this present project include the synthesis of NiO-8YSZ using co precipitation method by varying the weight percentage of NiO as 0, 20, 30, 40 percentage respectively and study its structural and microstructural properties. It was studied that the calcine temperature of the samples comes around ~800 ^{o} C. The next part of the project is all about finding out the crystallite size and the lattice parameter of samples using XRD data and the SEM images, the SEM image shows that the samples are porous in nature.

Apparent porosity decreases with increase in wt % of NiO. Bulk density increases with increase in wt % of NiO. This helps us to synthesize sample best suitable for various applications.

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Chapter 1

GENERAL INTRODUCTION

Introduction

From the previous studies it was found that Nickel/yttria stabilized zirconia (Ni/YSZ) cermet has some promising aspect as anode material for solid oxide fuel cells (SOFCs). The electrochemical property of Ni/YSZ anode is strongly dependent on the microstructure [1].

For application in solid state ionic devices such as solid oxide fuel cell electrodes and flow sensors, the incorporated metallic nickel or nickel oxide particles in yttria stabilized zirconia(YSZ) has made it possible. It can also be used as thermal barrier coating (TBC) for reducing the mismatch strains between the metallic structure and its coating. NiO/Ni is chosen as second phase because of its low cost, stability over a wide range of temperatures, limited solubility in solid solution and there is no reaction of products or intermediate phases.[2]

1.1Structure and properties of zirconia

ZrO_2 exhibits Polymorphism, and it shows three different polymorphic forms. Those are Monoclinic, cubic and tetragonal. The structure exhibited by Cubic ZrO_2 is a fluorite structure with Zr atoms being coordinated to eight oxygen atoms, the tetragonal structure is a distorted fluorite structure, whose XRD analysis indexes it to a face centered tetragonal cell and the monoclinic structure also referred as baddeleyite structure. The phase transformation from monoclinic-phase to tetragonal-phase or cubic-phase is reversible because of this high temperature polymorphs do not retain on cooling back to room temperature. The structure of pure ZrO_2 is monoclinic and this is at room temperature and undergoes monoclinic→ tetragonal and tetragonal→ cubic phase transitions at 1173°C and 2370°C respectively[3,4].

1.2Yittria stabilized zirconia

It has been found that the high temperature ZrO_2 forms can be stabilized at room temperature by addition of a small amount of oxides as MgO, CaO, Y_2O_3, CeO_2 etc..[5-6]

Eight-mole percent yttria stabilized zirconia (8YSZ) is a proven electrolyte material for Solid Oxide Fuel Cells The 8YSZ used in anode need to be sintered to full density at temperature as low as possible since the co-sintering of anode support and YSZ layer at high temperature results in warpage. It is well known that a crystalline ceramic powder are highly reactive and may be sintered to their near theoretical density (TD) at relatively low temperatures. [7]

1.3 Solid Oxide Fuel Cell

The SOFC is more of like a combustion engine, as the fuel has the energy source as chemical fuel, as a battery it converts the chemical energy directly into electrical energy. The advantage of SOFC here is the conversion goes on without a messy and relatively inefficient combustion step. The fuel cell has tremendous future prospect not because of its high efficiency and low emission, but for their modular and distributed nature and zero-noise pollution. This may solve our future requirement, as it plays an essential role in any future hydrogen fuel economy. SOFC provide high energy conversion efficiency due to their direct conversion of fuels electric power at high temperature in a co-generation system, compared to that of proton exchange membrane fuel cells (PEMFC) and direct methanol fuel cells (DMFC).[8-9]

1.4 Applications of NiO-8YSZ nanopowders

For application in solid state ionic devices such as solid oxide fuel cell electrodes and flow sensors, as YSZ has very good conductivity of O^{2-}, the presence of Ni enhances the electronic conductivity. The composite of NiO-8YSZ is used to prepare Solid Oxide Fuel Cell's (SOFC) anode part. As it is a very good conductor of O^{2}-, Ni-YSZ is used as oxygen sensors. For reducing the mismatch between the metallic surface and coatings Ni-YSZ is used as a thermal barrier coating (TBC). [2]

1.5 Organization of project report

Preliminary introduction about NiO-8YSZ and their application were discussed in chapter 1.Chapter 2 provides a detailed discussion of literature on synthesis of NiO-8YSZ and their properties. The main objective of the present work, which is based on the literature survey, is presented towards the end of chapter 2. In chapter 3, the synthesis and characterization techniques used in the present work are described in detail. Chapter 4 describes about the thermal, micro structural study of the nano-powders and the pellets prepared. Finally, conclusions of this project work are given in Chapter 5.

Chapter 2

LITERATURE REVIEW

2.1 Synthesis and microstructural properties of NiO-8YSZ nanopowders

Synthesis of NiO-8YSZ can be done by different methods like sol–gel, co-precipitation, spray drying, spray pyrolysis, freeze drying, combustion synthesis, auto ignition process, polymer precursor synthesis and oxalate precipitation [10-13].

When the yttria stabilized zirconia are impregnated with nickel oxide as an inclusion, this made possible for the development in solid state ionic devices.[14] The purpose of YSZ matrix; it supports the nickel particles, inhibits the coarsening by sintering during SOFC operation and gives a thermal expansion coefficient close to other cell components.[15] The co precipitation route helps to obtain high sinter-reactive NiO-YSZ powders with controlled chemical composition; because of this it is the most important method. It’s important for its simplicity and low cost.[2]

Now a day’s an energy conversion system SOFC is becoming a great interest due to their higher energy efficiency and environmental friendliness. [14]. Pratihar et al. [16] reported that the powder preparation technique plays vital role in microstructural parameters.

NiO and YSZ have an easier processing as a mixture rather than that of a Ni metallic and an YSZ. Then the desired ceramic-metal composite is obtained by in situ reduction of already processed NiO-YSZ composite. It is shown that NiO–YSZ powder can be prepared by sol gel-combustion method, because of its simple processing technique and it helps in the formation of nano-crystalline powders. Combustion synthesis involves heating of the concentrated metal nitrates or oxy-nitrates solution with suitable fuel. Porosity observed with sintering time for the NiO-YSZ after reduction was larger than before reduction. [17]

Synthesis of nano-sized NiO/YSZ composite particles through co precipitation method provided fine as well as homogenous porous structure. The microstructure of the NiO/YSZ composite particles changes drastically as we vary the pH of the solution; this might be attributed to difference of precipitation and growth behaviors of the hydroxides.

For use in SOFC as anode, 8YSZ must be highly thin film of thickness varying from 5-20 mm [18]. Warpage occurs in YSZ layer because of Co-sintering at high temperature. [7]

Because of coating precipitation synthesis process the morphology of anode fabricated from these precipitated composite particles shows a long term SOFC operation. This is mostly because of the YSZ grains on the Ni network which successfully prevents the grain growth of Ni at high temperature. There is a formation of a continuous network structure by YSZ while

Ni particles were dispersed homogeneously in YSZ network. This shows that the Coating precipitation method, successfully, helps in controlling the sintering of Ni and also prevents its agglomeration more effectively than manual mixing method. [20]

2.2 Summary of Literature

Summary of the literature survey are:

(1) There are different routes as discussed in the literature part for preparation of NiO-8YSZ.

(2) Co-precipitation method was found most suitable.

(3) The calcination temperature lies between 600^0C-800^0C.

(4) The microstructure of the NiO/YSZ composite particles changes drastically as we vary the pH of the solution.

(5) The Coating precipitation method, successfully, helps in controlling the sintering of Ni and also prevents its agglomeration and it’s more effective than the manual mixing method.

(6) The powder preparation technique plays vital role in microstructural parameters.

2.3 Objective of the present studies

In this paper we limited and focussed ourselves on the synthesis of NiO-8YSZ powders only, and studied the change in structure, microstructure, thermal properties, porosity and density with the variation of wt% of NiO.

(1) Synthesis of NiO-8YSZ nano powders with varying wt% of Ni, using the salts of Ni, Zr, and Y_2O_3 powder. In this process, NaOH was used for gelation and precipitation.

(2) Thermal analysis (DTA, DSC) of the precursors thus formed up to 1000˚C and analyzing the results in comparative manner.

(3) Calcinations of the precursors thus formed at 800˚C for 1hr and study XRD for comparative - structural analysis, crystallite sizes of the phases, vol % NiO.

(4) Making pellets of the powders and sintering at 1200˚C for 2hr, taking SEM micrographs, measuring density and analyzing the results in comparative manner.

Chapter 3

EXPERIMENTAL WORK

Introduction

Co-precipitation technique is one of the important techniques for synthesizing nano-size ceramic materials. In this work, nano size NiO-8YSZ has been prepared through co-precipitation techniques. Several different characterization techniques have been used to study the properties of nano NiO-8YSZ powders. In this chapter, the synthesis and characterization techniques are described in detail.

3.1Synthesis of pure 8YSZpowder

3.1.1 Experimental setup

Salts used for preparation were Y_2O_3 and $ZrOCl_2.8H_2O$.Calculation was made to get different amount of salt for different wt % of NiO in 8YSZ.The percentage are 0, 20, 30 and 40 wt % of NiO w r t 8YSZ.

Nanosize zirconia powders were prepared through co-precipitation technique by using NaOH (Sodium Hydroxide). A solution of Y_2O_3 was made by adding few ml of H_2O and HNO_3 was added to dissolve it completely with constant stirring and heating. And an aqueous solution of $ZrOCl_2.8H_2O$ was prepared from high purity salt by adding 30ml of H_2O. NaOH solution was prepared by adding 25ml of H_2O to 5g of NaOH pellets. The Zr-salt solution was added to the Y_2O_3 solution leaving them to mix homogeneously. The Zr-salt solution is highly acidic with a pH of ~ 0.3. On the other hand the NaOH solution has a highly basic pH of ~ 10. The NaOH solution is added drop wise to the solution till ph-7 with constant stirring. Then the solution was kept for some-time to mix homogeneously with the help of constant stirring. The calculated weight of salts can be seen from Table 3.1

3.2. Synthesis of pure NiO-8YSZ powder

3.2.1 Experimental setup

NiO-salt solution was prepared by adding 10ml of water to $NiCl_2.6H_2O$ salt. This solution was added to the precursor prepared earlier of Y_2O_3 salt solution and Zr salt solution. Again NaOH was added drop wise to the solution till ph-13 with constant stirring. The precipitated was allowed to settle down and it was decanted several times using hot water to get ph up to 7. The precipitated was dried in oven and dried sample was grinded with the help of mortar and pestle.

Table 3.1 Calculated weight of salts used

Composition	$ZrOCl_2.8H_2O$ (g)	Y_2O_3 (g)	$NiCl_2.6H_2O$ (g)
0:100	22.56	1.37	-
20:80	18.05	1.10	6.37
30:70	15.79	0.96	9.56
40:60	13.53	0.82	12.75

3.3 General characterization

3.3.1 Thermal

The thermal analysis of NiO-8YSZ was done using thermogravimetric and differential scanning calorimetric (TG-DSC) and the rate of heating was 10 °C/min in the thermal analyzer (Model STA 4096, NETZSCH, Germany)

3.3.2 X-ray diffraction

For the study of phase analysis the sample were calcined at 800^{o}C and sintered at 1200 ^{0}C and the analysis was made with the help of X-ray diffractometer Philips PANalytical (Model: PW 1830diffractometer, Netherland) with Cu Kα radiation. The scanning was made from 20^{o}-80^{o} for obtaining the intensity and the scanning time period was 25min.

3.3.3 Scanning Electron Microscope

Microstructural features were studied using Scanning Electron Microscope (JSM 6480 LV JEOL, Japan). For this the samples were palletized and were sintered at 1200 ^{0}C for 2 hrs.

3.3.4 Particle size measurement

Size of the particles is usually obtained with the help of scanning electron microscopy (SEM).The size corresponds to the mean value of the crystalline domain size of the particles is determined from the X-ray line broadening using Debye-Scherrer formula with correction factor as given below,

$D_x = \frac{0.9\lambda}{\beta \cos\theta}$ Where $\beta = \sqrt{\beta^2_{sample} - \beta^2_{s\tan dard}}$ Where D_X is average crystalline size, λ is the X-ray wavelength used, β the angular line width of half maximum intensity and θ the Bragg’s angle in degree. Standard value of β is taken as the angular line width of half maximum intensity for polycrystalline SiO_2 material.

3.3.5 Density and apparent porosity

Bulk density is the weight per unit volume of the material including the volume of open pore space. Direct Volume Measurement Method was used to measure bulk density, which is generally used for deeply branded and irregular materials, gives more accurate results than the direct measurement method.

Bulk density of the material is given by the relation

$$\text{Bulk Density} = \frac{D}{W-S} \times \text{density of liquid medium}$$

Liquid medium in our case is kerosene with density of 0.78–0.81 g/cm^3.

Where, D, W, S stands for dry weight, soaked weight and suspended weight respectively of the sample.

Apparent porosity is the percentage relationship between the volume of the pore space and the total volume of a material. Evacuation method was used to measure it.

$$\text{Apparent Porosity} = \frac{W-D}{W-S} \times 100(\%)$$

Chapter 4

RESULTS AND DISCUSSION

Introduction

This chapter describes the thermal, structure, microstructure, porosity and density analysis of NiO-8YSZnanopowdersprepared through precipitation by hydrolysis technique using NaOH.

4.1Thermal behavior of as-prepared 8YSZ and NiO-8YSZ nanopowders

Fig. 4.1 (a) and (b) show the Thermal Gravimetric (TG) – Differential Scanning Calorimetric (DSC) curves of as-prepared 8YSZ and 40 wt % NiO-8YSZnanopowdersprepared through co-precipitation route using NaOH respectively.

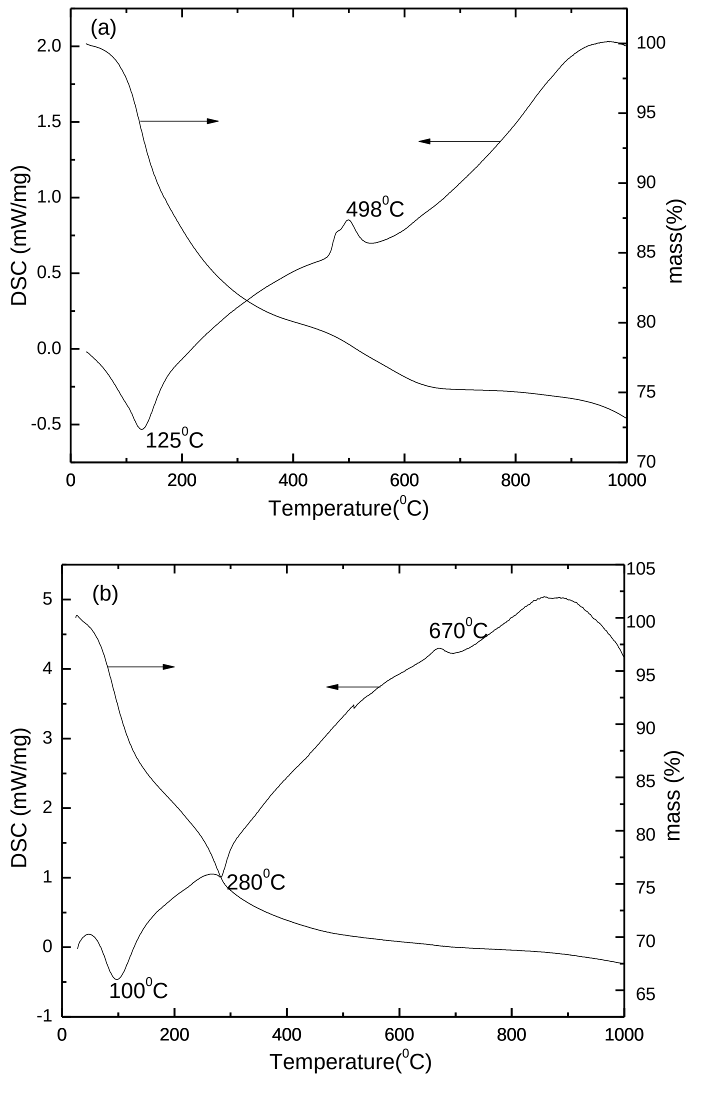


Fig. 4.1 (a) and (b): TG-DSC of as-prepared 8YSZ and 40 wt % NiO-8YSZ nanopowders prepared though precipitation route using NaOH.

The TG curve shows two stages of major weight loss. One stage ranges from room temperature to 400 °C. In the DSC plot there are an endothermic peak at 125 °C for 8 YSZ and two endothermic peaks at 100 °C and 280 °C for 40 wt % NiO-8YSZ samples. The endothermic peaks are associated with the loss of absorbed and or adsorbed water. The exothermic peak at 498 °C (for 8YSZ sample) and 670 °C (for 40 wt % NiO-8YSZ sample) is probably due to crystallization of cubic zirconia (c-ZrO_2) by the important weight loss of ~ 5 %. The TG curve shows that major weight losses (about 25% 0-30 % weight loss) were associated with both endothermic and exothermic effects. It was confirmed from the thermal results that with addition of 40 wt % NiO, the crystallization temperature shifts from 498 °C to higher temperature of around 670 °C.

4.2 Structural properties of NiO-8YSZ nanopowders

The presence of various phases, crystallite size, and lattice parameter as well as cell volume were determined from the X-ray diffraction pattern. Fig. 4.2 (a) shows the X-ray diffraction pattern of the dried as-prepared 8YSZ powders after calcination at 800 °C for 1h.

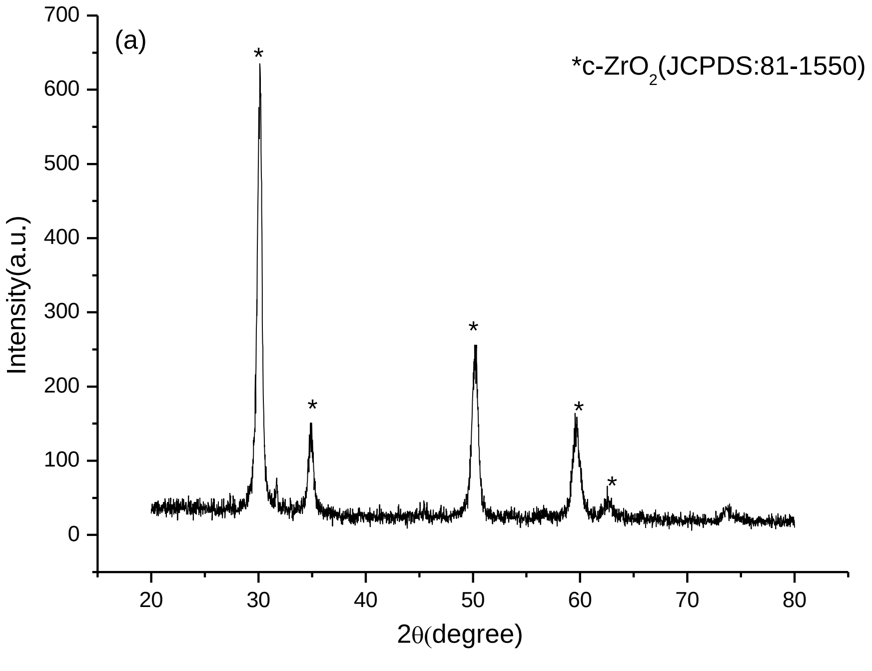


Fig. 4.2 (a) XRD pattern of 8YSZ nanopowders calcined at 800 °C.

Fig. 4.2 (b) shows the X-ray diffraction pattern of the dried as-prepared 20 wt % NiO-8YSZ powders after calcination at 800 °C for 1h.

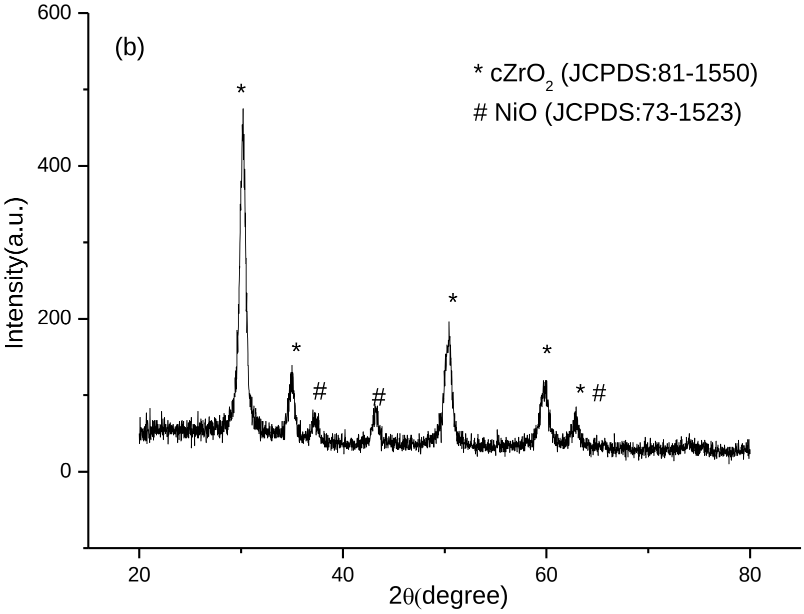


Fig. 4.2 (b) XRD pattern of 20 wt % NiO-8YSZ nanopowders calcined at 800 °C.

Fig. 4.2 (c) shows the X-ray diffraction pattern of the dried as-prepared 30 wt % NiO-8YSZ powders after calcination at 800 °C for 1h.

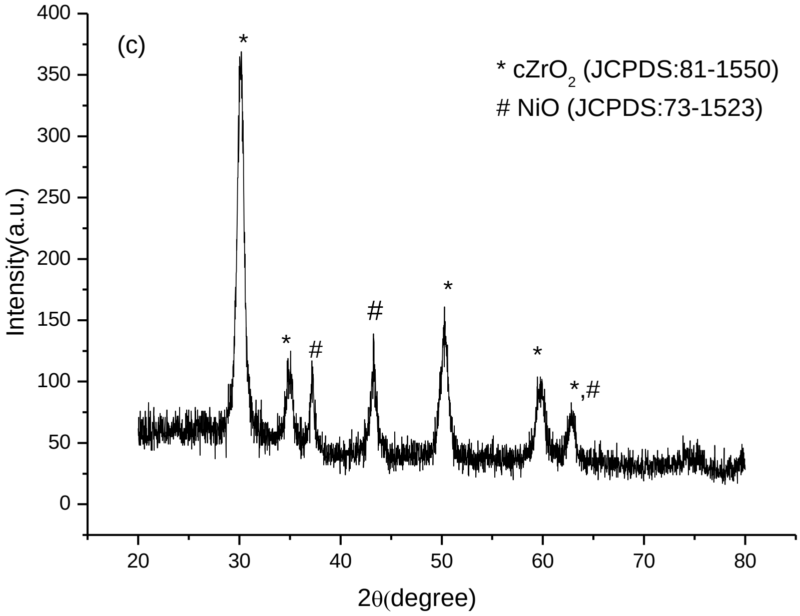


Fig. 4.2 (c) XRD pattern of 30 wt % NiO-8YSZ nanopowders calcined at 800 °C.

Fig. 4.2 (d) shows the X-ray diffraction pattern of the dried as-prepared 40 wt % NiO-8YSZ powders after calcination at 800 °C for 1h.

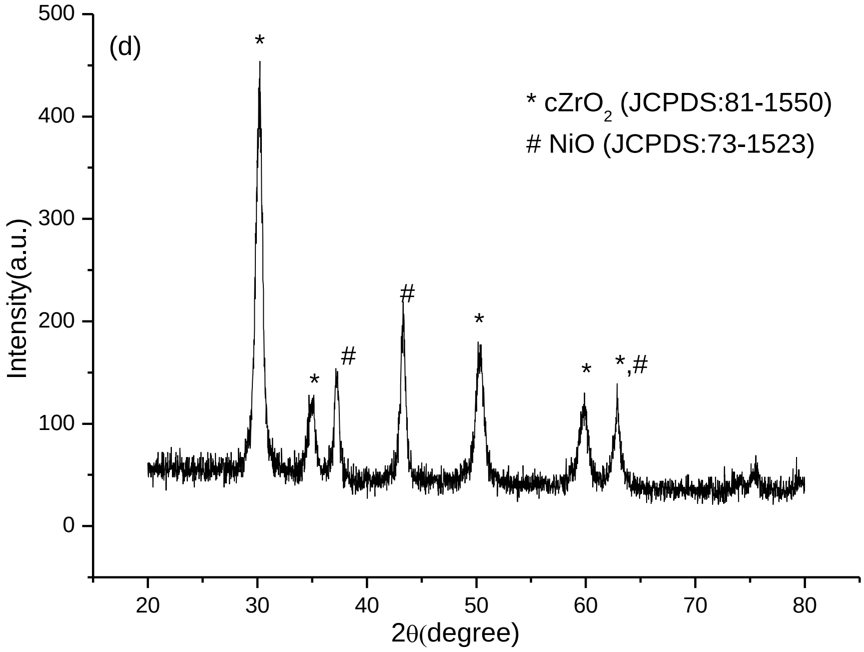


Fig. 4.2 (d) XRD pattern of 40 wt % NiO-8YSZ nanopowders calcined at 800°C.

All the peaks are assigned to either c- ZrO_2 or NiO phase as confirmed from the JCPDS files as mentioned in the figures. With increase in wt % of NiO, the peak intensity at 43.29 ° increases. The crystallite size, lattice parameter and cell volume of 8YSZ and NiO-8YSZ powder samples calcined at 800 °C are given in Table 4.1. The crystallite size of c-ZrO_2 for 8YSZ sample was found to be around 18 nm. With addition of NiO, the crystallite size decreases to 12 nm.

Table 4.1: Crystallite size, lattice parameter and cell volume of 8YSZ and NiO-8YSZ samples.

Sample	Crystallite size of c-ZrO_2 (nm)	Crystallite size of NiO (nm)	Lattice parameter(Å) and cell volume (Å^3) of c-ZrO_2	Volume % of NiO phase
8YSZ	18	-	5.138/135.615	-
20 wt% NiO-8YSZ	14	16	5.117/133.965	7.54
30 wt% NiO-8YSZ	12	21	5.125/134.636	3.09
40 wt% NiO-8YSZ	12	19	5.121/134.85	2.27

Fig. 4.3 (a) shows the X-ray diffraction pattern of the sintered (at 1200 °C for 2h) 8YSZ pellets.

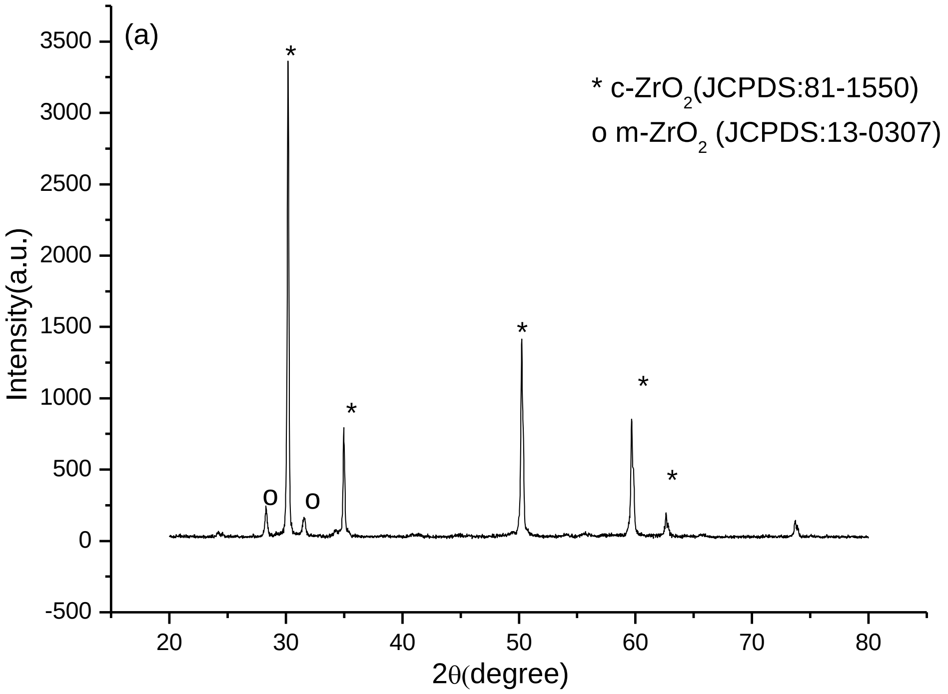


Fig. 4.3 (a) XRD pattern of 8YSZ sintered pellets.

Fig. 4.3 (b) shows the X-ray diffraction pattern of the sintered (at 1200 °C for 2h) 20 wt % NiO- 8YSZ pellets.

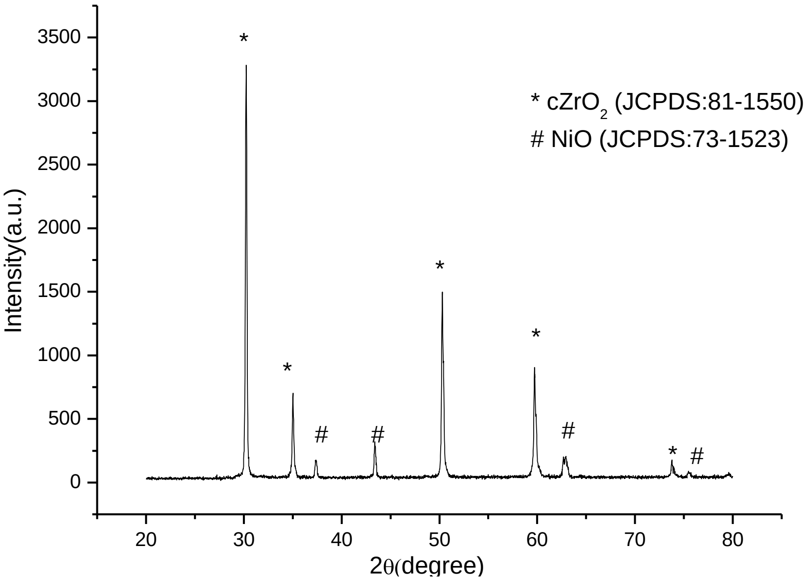


Fig. 4.3 (b) XRD pattern of 20 wt % NiO-8YSZ sintered pellets.

Fig. 4.3 (c) shows the X-ray diffraction pattern of the sintered (at 1200 °C for 2h) 30 wt % NiO- 8YSZ pellets.

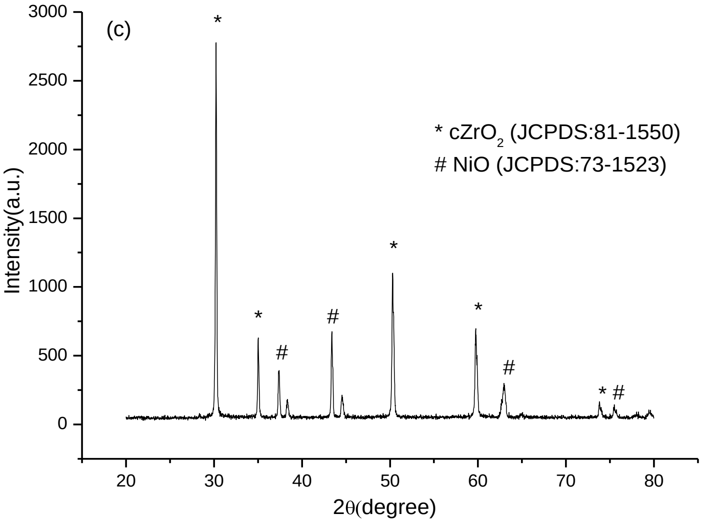


Fig. 4.3 (c) XRD pattern of 30 wt % NiO-8YSZ sintered pellets.

Fig. 4.3 (d) shows the X-ray diffraction pattern of the sintered (at 1200 °C for 2h) 40 wt % NiO- 8YSZ pellets.

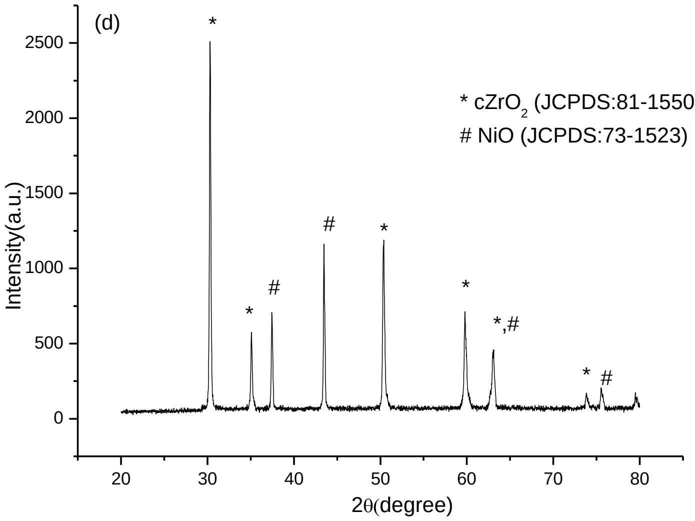


Fig. 4.3 (d) XRD pattern of 40 wt % NiO-8YSZ sintered pellets.

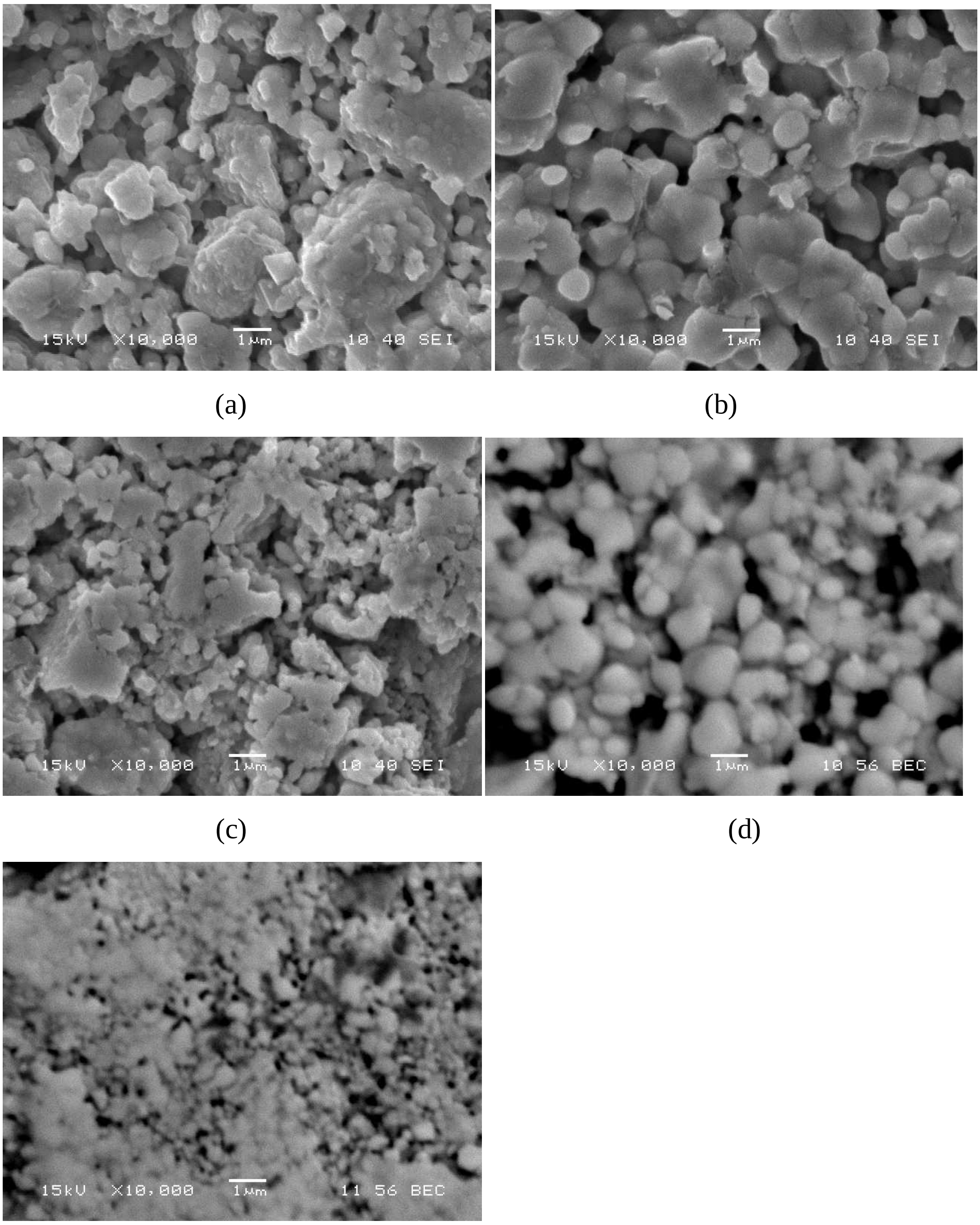
All the peaks are assigned to either c- ZrO_2, m-ZrO_2 or NiO phase as confirmed from the JCPDS files as mentioned in the figures. With increase in wt % of NiO, the peak intensity at 43.29 ° increases. The crystallite size, lattice parameter and cell volume of 8YSZ and NiO-8YSZ sintered samples are given in Table 4.2. The crystallite size of c-ZrO_2 for 8YSZ sample was found to be around 56 nm. With addition of NiO, the crystallite size is not varies appreciable.

Table 4.2: Crystallite size and cell volume of 8YSZ and NiO-8YSZ samples.

Sample	Crystallite size of c-ZrO_2 (nm)	Crystallite size of NiO (nm)	Volume % of NiO phase
8YSZ	56	-	-
20 wt% NiO-8YSZ	49	44	11.55
30 wt% NiO-8YSZ	55	43	4.39
40 wt% NiO-8YSZ	46	48	2.22

4.3 Microstructural properties of NiO-8YSZ nanopowders

Scanning electron microscopy (SEM) gives the information about the size, shape and agglomeration behavior of NiO-8YSZ pellets. Fig. 4.4 (a), (b) (c) and (d) show the microstructural SEM secondary and backscattered images of 20 wt % NiO, 30 wt % NiO and 40 wt % NiO (SE and BSE images) sintered pellets, respectively. The particle size is nearly spherical and agglomerate in nature.



(a) (b)

(c) (d)

(e)

Fig. 4.4 SEM images of (a) 20 wt % NiO-8YSZ, (b & d) 30 wt % NiO-8YSZ and(c and e) 40 wt % NiO-8YSZ sintered pellets.

4.4 Density and porosity of NiO-8YSZ pellets

Apparent porosity and bulk density of NiO-8YSZ pellets are given in Table 4.3.

Table 4.3: Apparent porosity and bulk density of 8YSZ and NiO-8YSZ samples.

Sample	Dry wt (g)	Soaked wt (g)	Suspended wt (g)	AP(%)	BD(g/cm^3)
8YSZ	0.53	0.5728	0.4551	36.36	3.65
20 wt % NiO-8YSZ	0.56	0.5970	0.4997	38.03	4.66
30 wt % NiO-8YSZ	0.58	0.6087	0.5139	30.27	4.96
40 wt % NiO-8YSZ	0.58	0.6096	0.5162	31.69	5.03

Apparent porosity measured by *evacuation method* decreases with increase in amount of NiO (wt %). While bulk density measured by *direct volume determination method* increases with increase in addition of NiO (wt %).

Chapter5

CONCLUSIONS

Four different samples of NiO-8YSZ were prepared by co-precipitation method. The composition used for the DSC, TGA, XRD, BD, AP and SEM analysis were 20:80, 30:70, 40:60, 0:100 of NiO:8YSZ respectively. The following conclusions were made after the characterization:

1. There are two endothermic peaks at 100 °C and 280 °C for 40 wt % NiO-8YSZ samples. The endothermic peaks are associated with the loss of absorbed and or adsorbed water.
2. With addition of 40 wt % NiO, the crystallization temperature shifts from 498 °C to higher temperature of around 670 °C.
3. Gel formation was found around pH □ 5-6.
4. The intensity of NiO increases with increase in amount of NiO (wt %).
5. Apparent porosity decreases with increase in wt % of NiO.
6. Bulk density increases with increase in wt % of NiO.

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