

**Electrical Behavior of Nickel Coated YSZ Cermet Prepared by
Electroless Coating Technique**

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Abstract

Nickel – yttria stabilized zirconia (Ni/YSZ) cermet has been prepared by coating YSZ particles with metallic nickel using electroless coating technique. Concentration of nickel was varied between **7.23** and **64.99% by weight**. Bulk samples were prepared using these nickel coated YSZ powders by uniaxial pressing followed by sintering in the temperature range 1200 – 1350°C with a soaking time of 2-6 h. A thorough investigation on the electrical characteristics of the samples has been performed and an attempt has been made to study the effect of starting YSZ particle size, matrix density on the temperature dependence of conductivity of the cermet. Samples prepared by this technique shows metallic conductivity at a Ni concentration as low as **27.04% by weight**. A detailed microstructural investigation of the samples is also reported.

PACS No: 81.05.Mh

Keywords: Ni/YSZ cermets; solid oxide fuel cells; Electrical conductivity; Microstructure

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1. Introduction

Solid oxide fuel cells (SOFC) are getting importance as energy conversion systems due to their high efficiency, modularity in design and environmentally friendly nature. The most popular anode material for solid oxide fuel cell (SOFC) is nickel yttria stabilized zirconia (Ni-YSZ) cermet [1-3]. In order to achieve the best performance of anode cermet, there are three main factors to be considered. First of all, anode should have high electrical conductivity to reduce the ohmic loss. Next, it should have enough electrochemical activity to reduce the activation polarization that is related with the electrochemical reaction at anode. And lastly, it should have proper microstructural condition to reduce the concentration polarization, which is related with the diffusion of the reactant or product of the electrode reaction. These factors are necessary to obtain the best performance of the anode and partial fulfillment of those conditions is not sufficient for the proper operation of SOFC. For example, for higher electrical conductivity, higher Ni content is the best choice but; higher Ni content leads to instability of microstructure due to large Ni coarsening and thermal expansion coefficient mismatch. On the other hand, for lower concentration polarization, high porous composite is better but one cannot guarantee the proper mechanical and electrical properties. Open porosity is required for the electrode to supply fuel and for removal of reaction products. The nickel particles forming a percolative network have a large catalytic activity and are responsible for transporting electrons from the electrode reaction site to the current collector. The addition of YSZ is necessary to support the nickel particles, to inhibit coarsening by sintering into larger particles at the usual operating temperature of an SOFC, and to give

the cermet a thermal expansion coefficient close to that of other cell components [4-7]. Literatures on Ni/YSZ cermet synthesis [8-10] aims to uniform and homogeneous distribution between the Ni and YSZ phases in the matrix. However cermets prepared by these techniques behave as biphasic composite and shows conductivity percolation at **44.38 – 49.70 % by weight** Ni depending on the process parameter. However such a high amount of Ni meets the conductivity requirement of SOFC anode along with the porosity requirement (35-40%) for lowering concentration polarization. It has higher thermal expansion coefficient among the other cell components.

In the present investigation, a conceptual microstructure of the Ni/YSZ was proposed wherein attempts has been made to reduce the thermal expansion coefficient of the cermet anode by lowering the Ni content without compromising its electrical conductivity and porosity. It is well established that the metallic conductivity of Ni/YSZ cermet (above percolation threshold) is achieved by the formation Ni-to Ni chain within the cermet matrix [1-4]. Taking into this fact under consideration in this study an attempt has been made to cover the YSZ surface by metallic nickel following electroless technique. The amounts of Ni require to completely covering the YSZ surface will depend on the YSZ particle size. Increasing the YSZ particle size one can decrease the amount of Ni content for complete covering the YSZ surface and hence can be able to reduce its thermal expansion coefficient. In this investigation Ni/YSZ cermet material has been prepared by coating YSZ powder with metallic nickel via an electroless technique [11] and a systematic study has been carried out to determine the minimum possible nickel concentration required to have sufficient electrical conductivity. Effect of YSZ

particle size and matrix density on the electrical properties and microstructure of Ni-YSZ cermets have also been studied.

2. Experimental

YSZ powders of two different particle sizes were used: (a) TZ – 8Y (8 mole % yttria stabilized zirconia) grade powder from TOSOH Corporation, Japan in the as received condition having average particle size (d_{50}) of 0.4 μm (henceforth designated as YSZ1) and (b) abovementioned TZ – 8Y powder calcined at 800°C for 12 hours followed by grinding to yield YSZ particles of average particle size (d_{50}) of 0.85 μm (henceforth designated as YSZ2). The particle size of the YSZ powders was measured by SediGraph 5100, Micromeritics, USA. Both YSZ1 and YSZ2 powders were coated with metallic nickel following electroless technique. For this purpose, YSZ powder surfaces were sensitized using SnCl_2 and PdCl_2 solution. The sensitized powders were then coated with metallic nickel using a patented electroless bath [12]. The detailed deposition behavior of nickel and the other processing parameters were reported elsewhere [11]. Nickel content of the coated powder was varied from **7.23-64.99% by weight** in the cermet. The coated powders were then pressed in the form of a rectangular bar of dimension 15mm x 3mm x 2mm following conventional pressing. These pressed pellets were sintered in air in the temperature range 1200°C-1300°C for 4h when NiO-YSZ was formed. The air-sintered pellets were then reduced under mixed atmosphere of hydrogen and argon at 1000°C for 2h to convert NiO back to metallic nickel. Porosity of the cermets was measured by water displacement method using Archimedes' principle. Electrical conductivity of the cermets was measured by four-probe technique using a 7-digit multimeter (HP 3458A). Unfluxed platinum paste was used as contacts for electrical measurements. For each sample,

measurement was carried out at different temperatures in the range from ambient to 1000°C. For microstructural study, a few representative samples were examined under optical microscopy (Letiz – Ortholux Pol BK) and scanning electron microscopy (LEICA S440).

3. Results and Discussion

3.1 Microstructure

The SEM fractographs of **49.70 weight %** nickel prepared with YSZ1 powder is presented in Fig. 1 wherein, (a) represents the same before reduction and (b) represents that after reduction. It is clear from the micrograph that an increase in porosity of the samples occurs upon reduction. This is obvious due to the fact that upon reduction, NiO in the air sintered samples is converted to metallic Ni. This conversion is associated with an increase in porosity due to loss of oxygen as well as the crystallographic change. However, the two different phases namely NiO or Ni and YSZ are not clearly resolved from the microstructure.

Optical microstructure of polished surface of **14.14-49.70 weight %** sintered Ni/YSZ cermets prepared with YSZ2 powder is presented in Fig. 2. The gray particle in the micrographs represents YSZ grains, while the white rings around it represent the Ni present in the cermet. This nickel ring formation around the YSZ particles confirms the coating of the metallic nickel by the electroless technique. As the nickel content in the cermet increases, the coating thickness also increases. It has been found that the complete coverage of the YSZ surface with metallic nickel takes place at **27.04 weight %** nickel. As a result of that all the samples containing **27.04 weight %** or more Ni shows metallic

behavior. The increase in nickel thickness with increasing nickel content is clearly visible from the micrographs.

In order to study the nickel distribution within the samples, SEM microstructure under back scattered mode along with Ni mapping of the polished surfaces was performed. A representative back scattered micrograph and the corresponding Ni distribution of the samples containing **49.70 weight %** nickel prepared with both YSZ1 and YSZ2 are presented in Fig. 3 (a & b) and Fig. 3 (c & d) respectively. The nickel distribution in the samples prepared with YSZ1 powder shows a very fine and uniform distribution of nickel over the particle surface. However the formation of nickel ring around the YSZ particles is clearly indicated for samples prepared with YSZ2 powder.

3.2 Temperature dependent electrical conductivity of Ni coated YSZ cermet

The resistivity of metallic nickel vary with temperature as $\rho = 6.8 \mu\Omega \text{ cm} + 0.0359 \mu\Omega \text{ cm K}^{-1}\text{T}$ [13] whereas that for YSZ the dependence is expressed as $\rho = 0.003 (\Omega \text{ cm}) \exp (10300 \text{ K/T})$ [14]. Temperature dependent electrical conductivity of this cermet system was found quite interesting. The cermet prepared different techniques [17-21] shows metallic behavior for those samples contains more than **38.85 weight %** Ni, whereas the samples containing less than **38.85 weight %** Ni shows non metallic behavior. However, the samples prepared by electroless coating technique have a slightly different behavior. Cermets prepared in this technique shows metallic behavior for the samples containing nickel content as low as **27.04 weight %** and samples containing less than **27.04 weight %** Ni shows non metallic behavior.

The temperature dependent conductivity of the Ni/YSZ cermet prepared by electroless nickel coating on YSZ2 powder containing **14.14 and 27.04 weight % Ni** is represented in Fig.4. It can be seen that the conductivity of the cermet prepared with **14.14 weight % Ni** is low and it increases with temperature, whereas that containing **27.04 weight % Ni** is relatively high and decreases with increase with temperature. The conductivity of the cermet containing **14.14 weight % Ni** is 1.84×10^{-4} S/cm at 400°C and 0.25 S/cm at 1000°C. On the other hand the conductivity of the cermets containing **27.04 weight % Ni** is 215 S/cm at 400°C and 126 S/cm at 1000°C. The conductivity temperature data for samples containing **14.14 weight % Ni** were best fitted with an exponential growth equation and that containing **27.04 weight % Ni** was best fitted with an linear equation (solid lines in Fig.4). The exponential increase in conductivity in the samples containing **14.14 weight % Ni** is dominated by an activated process similar to that of YSZ (16-18). On the other hand the linear decrease in conductivity with temperature is indicative of metallic conduction. From the microstructure (Fig.2) it can be seen that the Ni coating on the YSZ grains is not uniform and it contains some bare YSZ surface for samples containing **14.14 weight % Ni**. Hence in these samples the conductivity is predominated by the YSZ to YSZ paths presents in the samples. On the other hand complete coverage of YSZ surface by Ni was observed for samples containing **27.04 weight %** or more Ni (Fig.2). This Ni rings on the YSZ surface provides the conduction path for the samples containing **27.04 weight % Ni** resulting metallic conduction in this samples. The same behavior is observed for the YSZ1 coated samples.

The temperature dependent electrical conductivity of the cermets (containing **27.04-64.99 weight %** nickel and sintered at 1300°C for 4 hours) is represented in Fig.5 (a and b), wherein (a) shows the variation in electrical conductivity with temperature for samples prepared with YSZ1 powder and (b) represents the results for samples prepared with YSZ2 powder. In the former case, the conductivity measured at 1000°C varies in the range 127 – 449 S/cm and it is 542 – 3048 S/cm for the latter case for nickel content **27.04-64.99 weight %**. For all the case, the conductivity decreases with increasing temperature indicating metallic behavior. The conductivity of the cermets increases with increasing nickel content. Most promising feature is that these Ni/YSZ sample containing **27.04 weight %** nickel shows metallic behavior. This is much lower than the literature value of **38.85 weight %** nickel for observation of the metallic behavior of the cermet [16-18].

For a particular **weight %** of Ni, the conductivity value for a cermet prepared with YSZ1 powder is lower than that prepared with YSZ2 powder. In general, the conductivity of the cermets having a particular volume percent nickel prepared by coating coarse YSZ powder was found higher than that prepared by coating fine YSZ powder. This is due to the fact that in the former case, owing to the fineness of the YSZ powder, the surface area of the powder to be coated is large requiring larger quantity of nickel to cover the surface, which in turn decreases the coating thickness. This nickel coating thickness seems to be the controlling parameter for the increased conductivity of the cermets. In order to explain the above behavior a simple theoretical calculation was done to calculate the nickel coating thickness on YSZ particle assuming uniform coating and spherical particle size of YSZ as follows:

Let r is the starting YSZ particle size and t is the thickness of Ni coating on it when V_{Ni} is the volume fraction of Ni phase. If V_{YSZ} be the volume fraction of YSZ phase present in the cermet. Then $V_{Ni} + V_{YSZ} = 1$

Volume of YSZ particle is given by $(4/3) \pi r^3$

Volume of Ni coating is given by $(4/3) \pi (r + t)^3 - (4/3) \pi r^3$

Total volume of solid = $(4/3) \pi (r + t)^3$

Volume fraction of Nickel phase (V_{Ni}) = $\{(4/3) \pi (r + t)^3 - (4/3) \pi r^3\} / \{(4/3) \pi (r + t)^3\}$

Volume fraction of YSZ phase (V_{YSZ}) = $((4/3) \pi r^3) / \{(4/3) \pi (r + t)^3\}$

$$V_{Ni} / V_{YSZ} = \{(4/3) \pi (r + t)^3 - (4/3) \pi r^3\} / ((4/3) \pi r^3) = 3 (t/r) + 3 (t/r)^2 \quad (1)$$

Table 1 shows the variation of nickel coating thickness on YSZ particle with **weight** percent of Ni calculated using equation 1.

The above calculation of coating thickness (table 1) shows that for a particular volume percent of nickel, the coating thickness increases with increasing YSZ particle size. Hence for the same volume fraction of nickel cermets prepared by coating YSZ2 powder have thicker nickel coating in comparison to that with YSZ1 powder. Since conductivity path arises from nickel-nickel contact, an increase in coating thickness increases the cross sectional area of the conductivity path resulting in higher conductivity.

3.3 Effect of initial particle size of YSZ on temperature dependent electrical conductivity

The usual porosity requirement for SOFC anode application is around 35% for easy diffusion of fuel and reactants [1-3]. The temperature dependent conductivity of the

49.70 weight % Ni/YSZ cermets having 35% porosity prepared by electroless nickel coating of YSZ1 and YSZ2 powder is given in Fig. 6. For the same cermet porosity the conductivity of the cermet at 1000°C prepared with YSZ2 powder is found to be 2321 S/cm while that prepared with YSZ1 powder is 1095 S/cm. Similar results are observed at other temperatures also. In general, the conductivity of the cermets having a particular volume percent of nickel prepared by coating YSZ2 powder is found to be higher than that prepared by coating YSZ1 powder. As shown earlier (table 1), for a particular volume percent of nickel, the coating thickness increases with increasing YSZ particle size. Hence for the same volume fraction of nickel, cermets prepared by coating YSZ2 powder have thicker nickel in comparison to that with YSZ1 powder. Since conductivity path arises from nickel-nickel contact, the conductivity increases with an increase in coating thickness. Similar trend is observed for all cermets containing **27.04-64.99 weight %** nickel prepared by coating either YSZ1 or YSZ2 powder.

3.4 Conductivity of Ni/YSZ cermet having equal porosity at 1000°C as a function of nickel content and YSZ particle size

The electrical conductivity of the samples containing **7.24-64.99 weight %** nickel measured at 1000°C is plotted in Fig.7 as a function of nickel concentration and YSZ particle size having same matrix porosity. A series of S-shaped curves are obtained where each curve correspond to a different YSZ particle size. All these plots exhibit a sharp increase in conductivity at around **27.04 weight %** nickel, corresponding to the electrical continuity/discontinuity transition point of the dispersed nickel phase in contradiction to the literature value of **38.85 weight %**. This variation apparently looks similar to that of biphasic composite system [19-23]. However, in this case the system

will not behave as a biphasic system due to the very nature of the synthesis technique used. In this technique we have coated the YSZ powder surface by metallic nickel following electroless technique. The microstructure (Fig.2) also dictates the feature of complete coverage of the bear YSZ surface at **27.04 weight % Ni**. Hence in this case the electroless coated YSZ particles will behave as if they are metallic particles. Based on the calculation given in Table 1 it was found that the coating thickness increases with nickel content. Hence with increase in Ni content in the cermet the conducting path cross sectional area increases. This leads to an increase in conductivity of the cermet with Ni content. The sharp change in conductivity can be explained from the point of view of attainment of a reasonable coating thickness with increase in Ni content on the YSZ surface which can impart the metallic behavior in the sample. For a fixed matrix density, the conductivity was found to increase with increasing YSZ particle size. At a constant nickel loading the coating thickness increases with increasing YSZ particle size (Table 1) resulting in higher conductivity of the samples prepared with YSZ2 powder.

3.5 Temperature dependent electrical conductivity of Ni/YSZ cermet as a function of porosity

Fig. 8 represents the temperature dependence of electrical conductivity as a function of matrix porosity of Ni/YSZ cermet containing **49.70 weight % Ni**. The samples were prepared using YSZ2 powder. Matrix porosities were changed by varying the sintering conditions in the temperature range 1200 to 1350°C with a soaking time in the range 2-6h. The conductivity of the cermet at 1000°C is found to increase from 2250 – 2320 S/cm corresponding to a decrease in porosity from 44% - 35%. Corresponding to any particular nickel content and temperature an increase in matrix porosity resulted in a

decrease in the conductivity. A decrease in porosity within the sample provides improved particle-to-particle contact between the nickel particles resulting in a higher conductivity of the samples. Similar nature is also observed for cermets containing **27.04-64.99 weight %** nickel prepared with either YSZ1 or YSZ2 powder.

3.6 Electrical conductivity at 1000°C as a function of nickel content and porosity

The electrical conductivity of nickel/YSZ cermet is strongly dependent on its nickel content. The electrical conductivity of the samples containing **7.24-64.99 weight %** nickel measured at 1000°C is plotted in Fig. 9 as a function of nickel concentration for different matrix porosities. A series of S-shaped curves, each corresponding to different matrix porosity is obtained. In this case also, all these plots exhibit a sudden increase in conductivity at around **27.04 weight %** nickel. For a fixed matrix density, the conductivity is found to increase with increasing YSZ particle size. At a constant nickel loading the coating thickness increases with increasing YSZ particle size resulting in higher conductivity. The conductivity also increases significantly with decrease in matrix porosity due to better particle-to-particle contact between the nickel particles. This is also supported by the observed decrease in conductivity with matrix porosity (Fig. 8).

4. Conclusions

1. Porous Ni/YSZ cermets are electronically conducting at Ni contents greater than **27.04 weight %** of total solids. Below **14.14 weight %** nickel the conductivity falls to that of the ionically conducting zirconia matrix.
2. The conductivity percolation thresholds for these cermets are as low as **14.14-27.04 weight %** nickel.

3. Large size YSZ particles are better suited for this particular application.

5. Acknowledgements

Financial help from MNES (Govt. of India) is gratefully acknowledged. The authors also acknowledge the help rendered by Mr. B. Dolui of this institute in obtaining the optical micrographs.

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Figure captions

- Fig.1 SEM fractographs of **49.70 weight%** Ni/YSZ cermet (a) before reduction (b) after reduction showing porosity change.
- Fig.2 Optical micrographs of Ni/YSZ cermet containing (a) **27.04 weight %** (b) **38.85 weight %** and (c) **49.70 weight %** Ni prepared with YSZ2 powder. Magnification 400X.
- Fig.3 SEM micrographs in back scattered mode of the cermets prepared with (a) YSZ1, (b) YSZ2 powder and corresponding nickel mapping of the cermet surfaces (c) and (d).
- Fig.4 Temperature dependent electrical conductivity of Ni/YSZ cermet containing **14.14 and 27.04 weight %** nickel prepared by electroless coating YSZ2 powders.
- Fig.5 Temperature dependent electrical conductivity of Ni/YSZ cermet containing **27.04-64.99 weight %** nickel prepared by electroless coating different YSZ powders (a) YSZ1 (b) YSZ2.
- Fig.6 Temperature dependent electrical conductivity of Ni/YSZ cermet: effect of starting YSZ particle size.
- Fig.7 Electrical conductivity of Ni/YSZ cermet at 1000°C as a function of nickel content: effect of starting YSZ particle size.
- Fig.8 Temperature dependent electrical conductivity of Ni/YSZ cermet: effect of porosity.
- Fig.9 Electrical conductivity of Ni/YSZ cermet at 1000°C as a function of nickel content: effect of porosity.

Table 1 Variation of Ni coating thickness on YSZ particles with Ni loading

Nickel content (weight %)	Coating thickness (μm) on	
	YSZ $d_{50} = 0.4 \mu\text{m}$	YSZ $d_{50} = 0.85 \mu\text{m}$
07.24	0.0068	0.0145
14.14	0.0140	0.0305
27.04	0.0310	0.0658
38.85	0.0505	0.1070
49.70	0.0585	0.1240
59.71	0.1060	0.2250
64.99	0.1850	0.3110

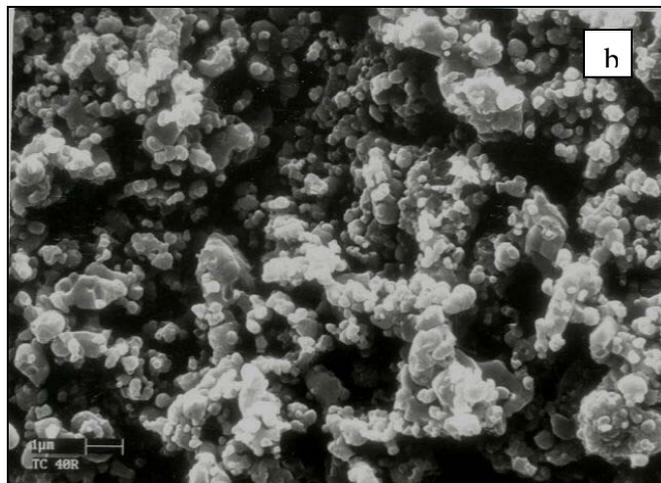
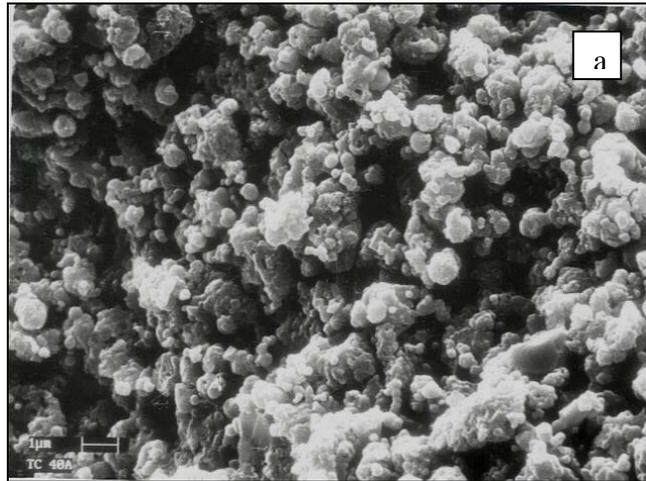


Fig.1 SEM fractographs of **49.70 weight % Ni/YSZ cermet** (a) before reduction (b) after reduction showing porosity change.

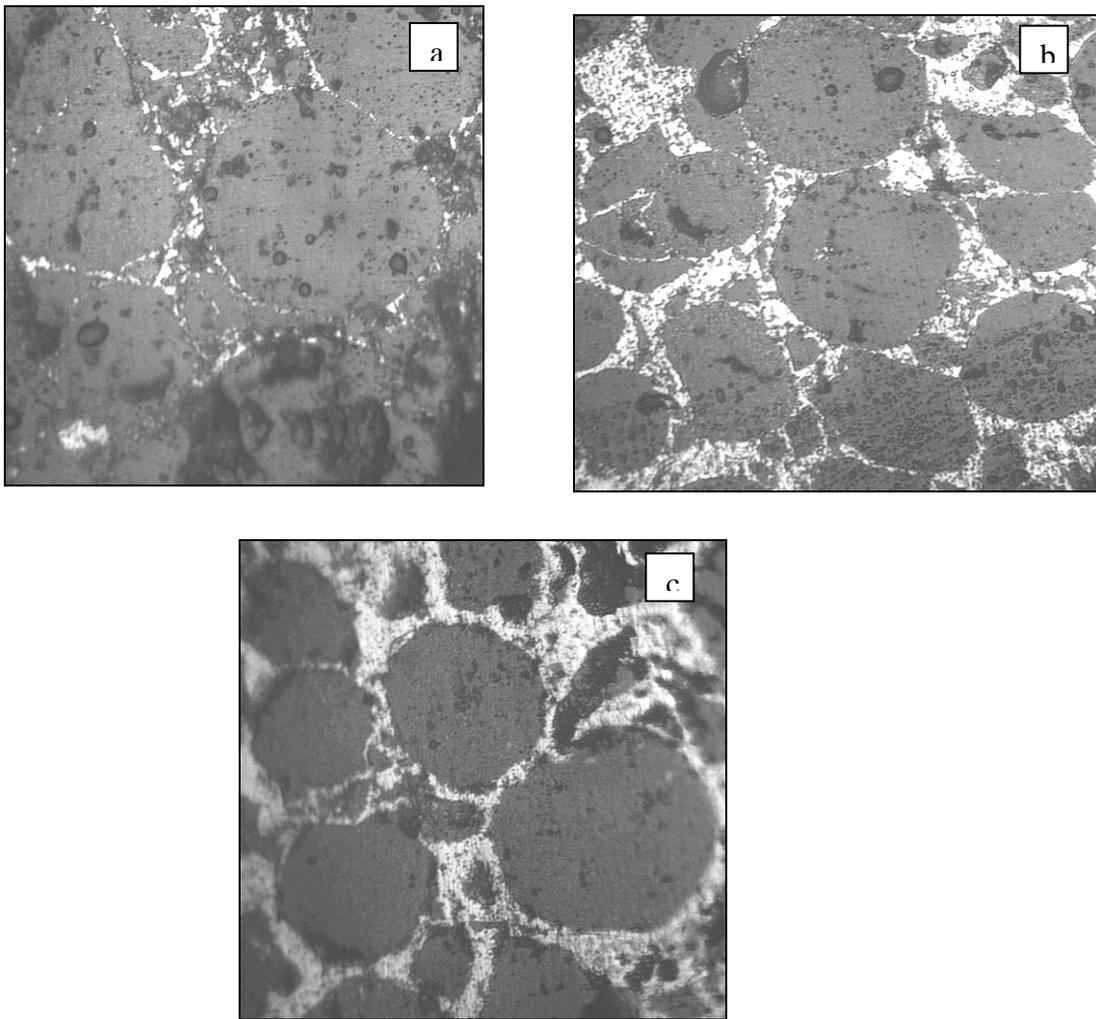


Fig.2 Optical micrographs of Ni/YSZ cermet containing (a) **27.04 weight %** (b) **38.85 weight %** and (c) **49.70 weight %** Ni prepared with YSZ2 powder. Magnification 400X.

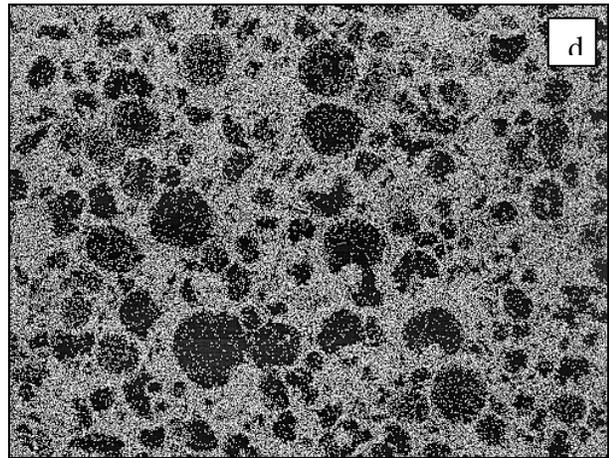
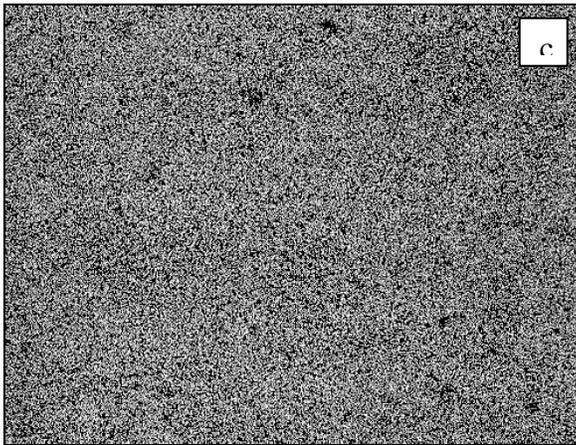
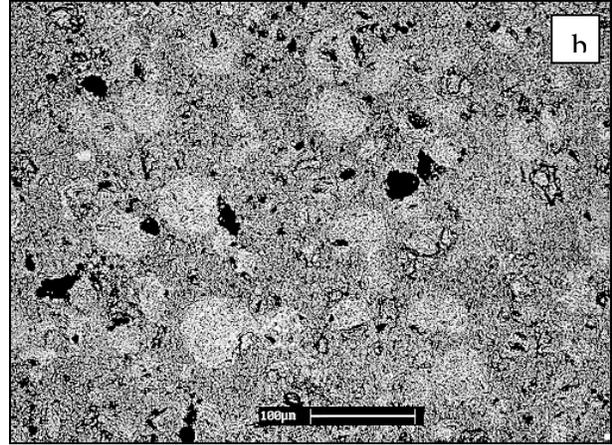
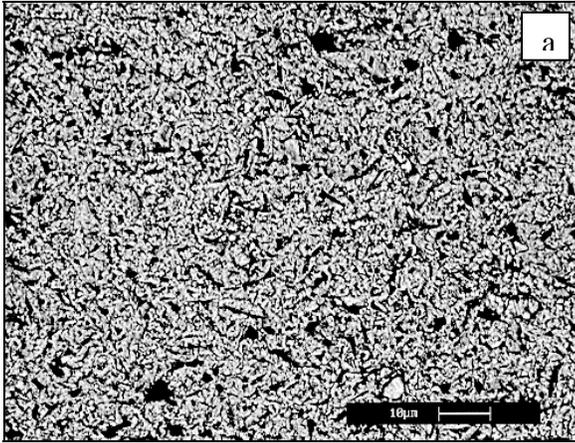


Fig.3 SEM micrographs in back scattered mode of the cermets prepared with (a) YSZ1, (b) YSZ2 powder and corresponding nickel mapping of the cermet surfaces (c) and (d).

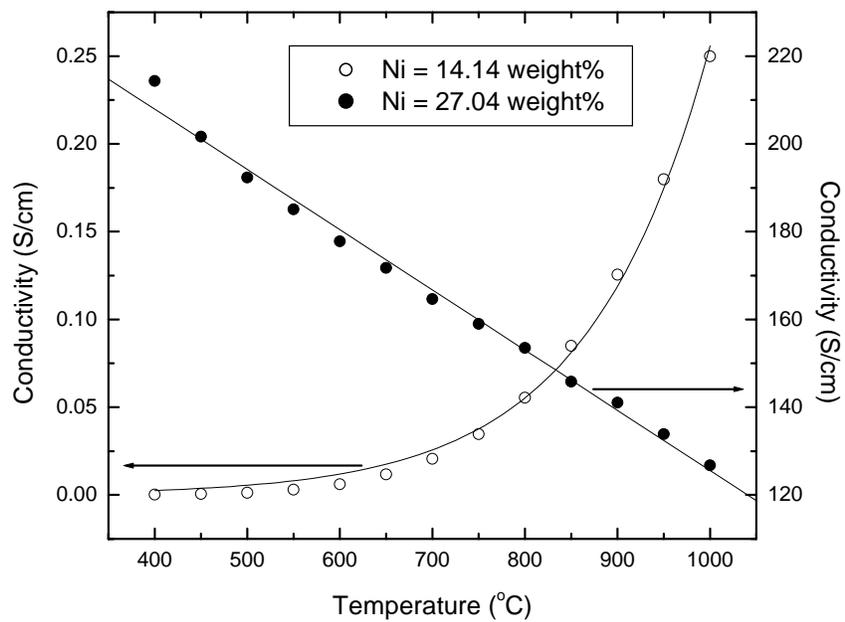


Fig.4 Temperature dependent electrical conductivity of Ni/YSZ cermet containing **14.14 and 27.04 weight %** nickel prepared by electroless coating YSZ2 powders.

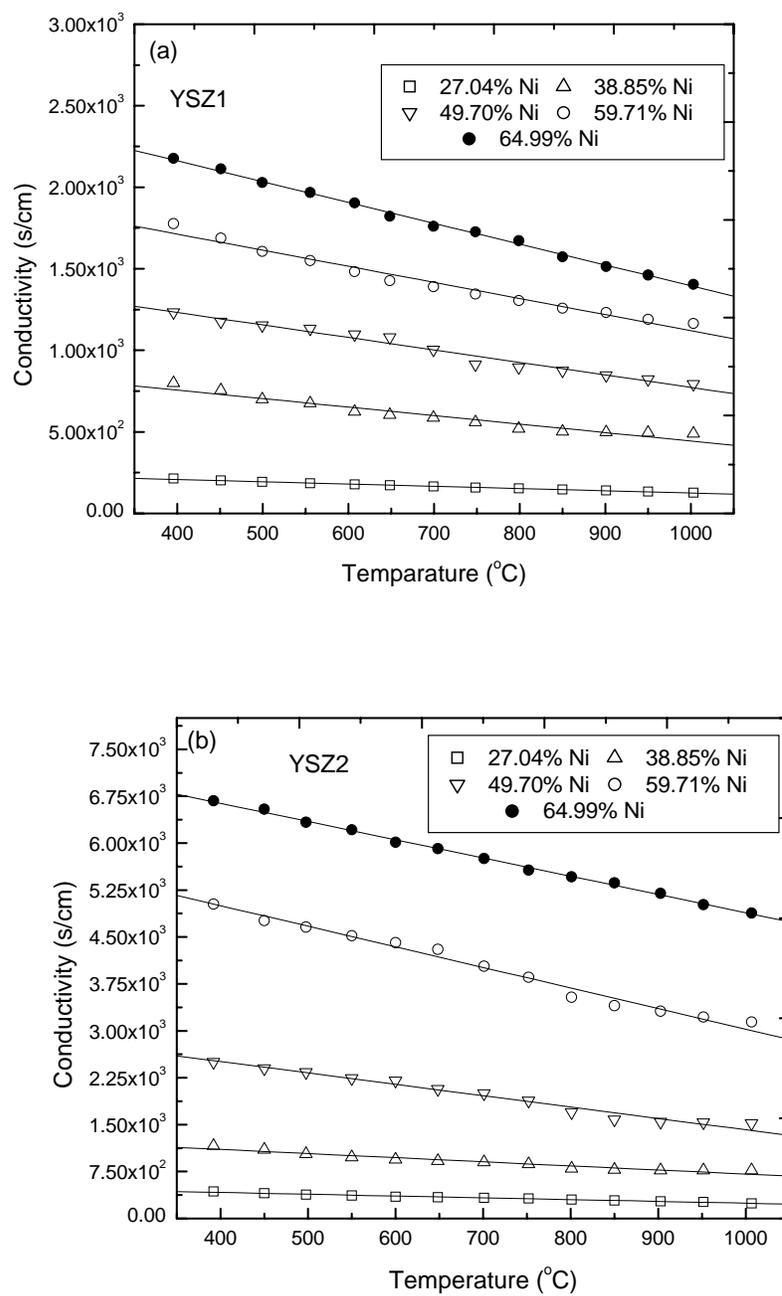


Fig.5 Temperature dependent electrical conductivity of Ni/YSZ cermet containing **27.04-64.99 weight %** nickel prepared by electroless coating different YSZ powders (a) YSZ1 (b) YSZ2.

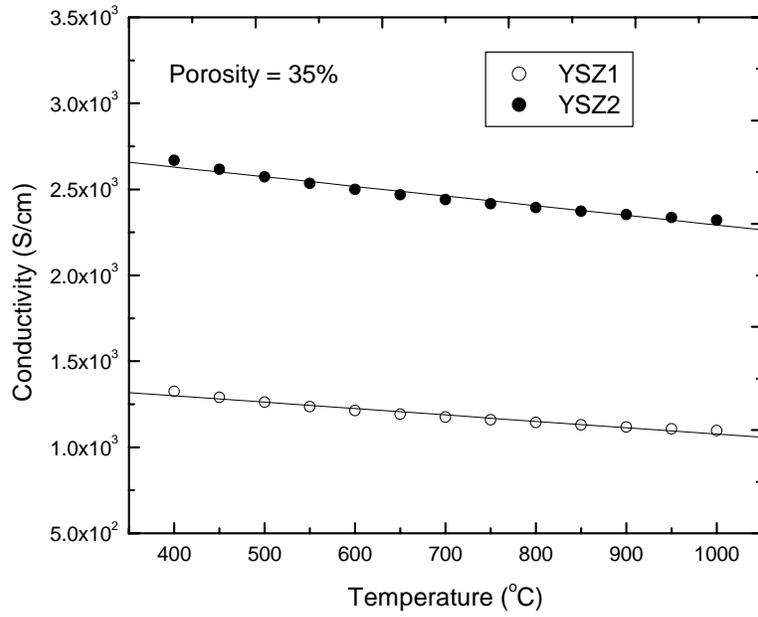


Fig.6 Temperature dependent electrical conductivity of Ni/YSZ cermet: effect of starting YSZ particle size.

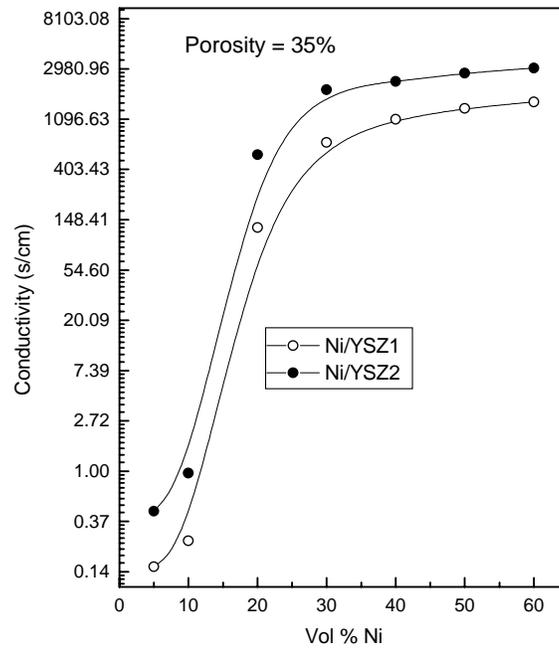


Fig.7 Electrical conductivity of Ni/YSZ cermet at 1000°C as a function of nickel content: effect of starting YSZ particle size.

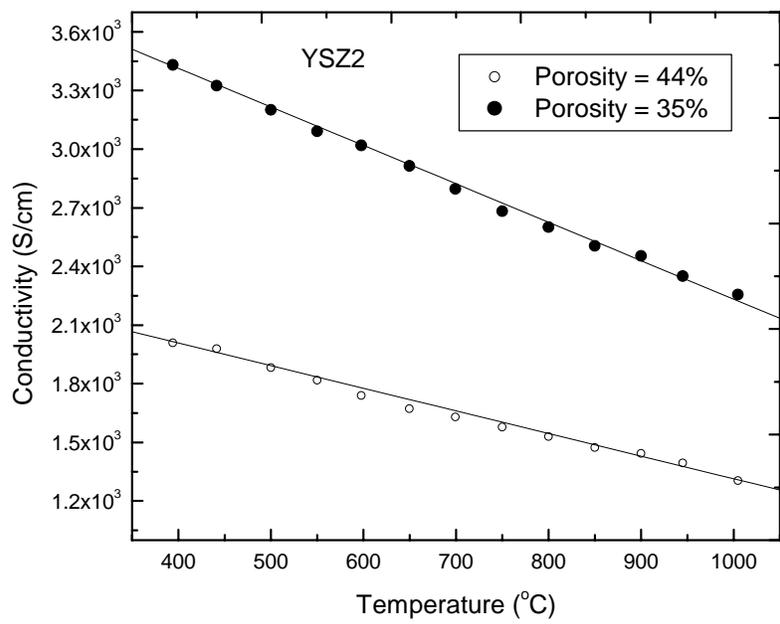


Fig.8 Temperature dependent electrical conductivity of Ni/YSZ cermet: effect of porosity.

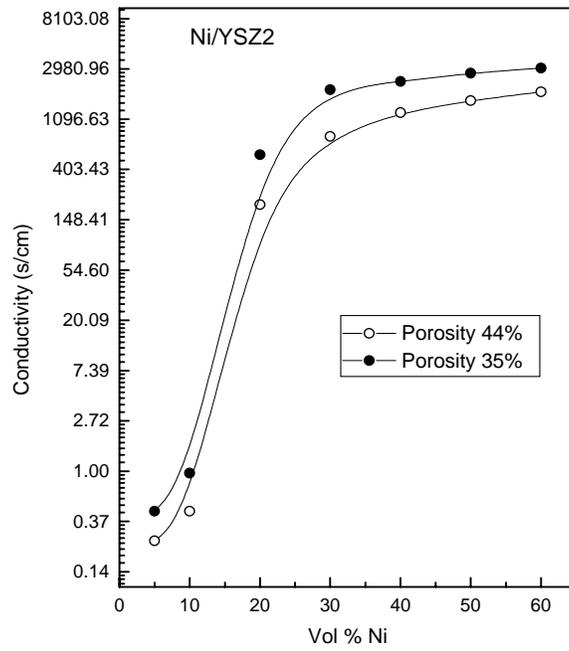


Fig.9 Electrical conductivity of Ni/YSZ cermet at 1000°C as a function of nickel content: effect of porosity.