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Fabrication and Characterization of NiO/YSZ Composite via Cold Press-Sintering Method

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ABSTRACT: Among various manufacturing processes, the use of cold press-sintering has been considered with low cost, high efficiency, and the ability to produce components with the appropriate dimensional accuracy. In this method, at least two components of the alcoholic solution used in granulation and milling steps. Removing the solutions was accompanied by energy consumption, as well as, an increment in probability of crack initiation, which may limit the widespread utilization of this method. Therefore, in the present study, the soluble component in the milling step was removed. In following just using polyvinyl alcohol as a soluble component in the granulation step, the ceramic/ceramic composite was fabricated. Doing this, samples based on NiO and YSZ were made with different weight percentages of the reinforcement component (25, 30 and 35% wt. zirconia-stabilized yttrium), and then their microstructure, density, and hardness were investigated. Microstructural studies indicate that the amount and distribution of porosity, surface quality and depth of cracks in the samples are depended on reinforcement weight. In detailed, at the surface of raw materials, no deep crack was observed. In sintered specimens, the best distribution of the gas phase was observed in NiO- 30% wt. YSZ. In addition, the radiography results provided by the above samples indicated that deep crack in the discs is not visible. Therefore, it seems that the starch as a pore former displayed the beset role via porosity distribution and lessening crack nucleation in sample having 30% reinforcement. According to microstructural and radiography results, the maximum hardness was observed in sample having the best distribution of the gas phase in matrix.

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1- Introduction

Although high temperature stability, low density, appropriate corrosion and wear resistance are significant properties of ceramics, the brittle characteristics of these fragile materials restricts their applications [1]. Therefore, optimization of ceramic materials was performed via composite processing [2]. The manufacturing of Ceramic Matrix Composites (CMCs) was done using external pressure or without it, such as warm iso-static press or slurry casting, respectively [3]. Nowadays due to CMC applications, the tendency to use new methods with high dimensional accuracy, economic efficiency, and specific geometry has increased. In the meantime, Additive Manufacturing (AM) and press-sintering methods are two approaches that have been particularly used to get attention in the development of dense, as well as, porous advanced components. It is noteworthy that, significant amounts of CMCs are produced in the porous form, such as filters and solid oxide fuel cells. Although it is possible to control the geometry and porosity distribution in the AM procedure, the manufacturing cost for laboratory-scale sample is very high and out of reach [4]. Therefore, the propensity for press-sintering processing, especially in the manufacture of porous ceramic composites, has increased [5]. One of the most usable porous ceramics also used as a solid oxide fuel cell anode is NiO/YSZ composite [6]. Researchers are endeavoring to optimize the process parameters or properties of NiO/YSZ composite via optimizing the porosity contained in the samples [7]. For example, Besra et al. [8] fabricated NiO/YSZ composite with a weight ratio of 40-60 and 50-50 by single-axis cold pressing, in order to fabricate a non-conductive composite substrate. Doing that, mixing and milling steps were performed using solutions, while starch was selected as pore former. The results show that, the density decreased by enhancing the amount of pore former. In another study, Souza et al. [9] produced 50 wt.% NiO/ 50 wt.%YSZ composite using a uniaxial press method in the presence of soluble components. The structure shows good porosity distribution and appropriate particle inter-connectivity. Subsequently, Tanhaei et al. [10] fabricated nano-composite based on NiO-YSZ produced by press-sintering method. Similar to previous work, the pore distribution was acceptable and grain growth occurred predominantly in finer reinforcing powder particles.

It is worth noting that, according to our literature review, at least two soluble components utilized during processing steps of NiO/YSZ composites, which made by press-sintering method. However, the presence of a soluble component increases the amount of energy consumed due to need for removing it before sintering [11]. In addition, the elimination of soluble components associated with the gas outflow, creates impurities in the sample, and thus increases the probability of

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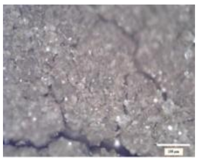
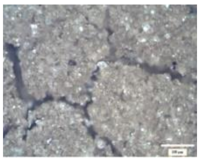
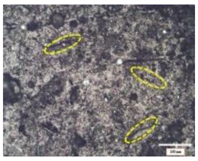
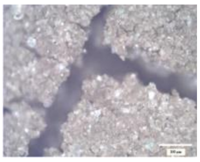
Chemical composition	Green	Sintered
NiO- 25 wt. %. YSZ		
NiO- 30 wt. %. YSZ		
NiO- 35 wt. %. YSZ		

Fig. 1. Optical micrographs of green and sintered samples

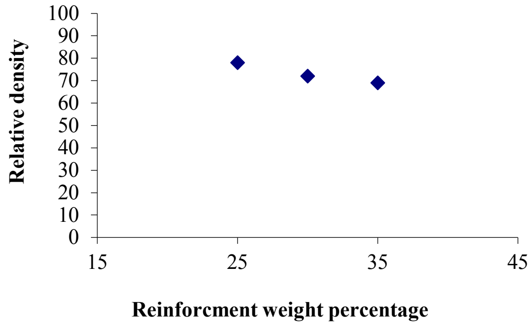


Fig. 2. Density of discs versus the weight percent of YSZ

cracking during the manufacturing process [12]. Therefore, in the present study, in order to achieve microstructure having fewer imperfections and reduce energy consumption, the minimum amount of soluble component was used during processing steps. In other words, the milling and mixing step was performed in a completely dry manner. Therefore, porous NiO-YSZ composites having different weight percentages of reinforcement were produced by dry-press-sintering technique, using starch as pore former. Considering the constant value of pore former, the pore distribution was controlled by the weight fraction of reinforcement and matrix. Then, the microstructure of pressed and sintered samples was characterized using optical and scanning electron microscopies. In addition to microstructural studies, micro-hardness test was utilized in order to determine the role of weight percent and presence of porosity on mechanical behavior of porous samples.

2- Methodology

In this research, NiO-YSZ composites having three different weight fractions of YSZ, i.e. 25, 30 and 35 wt.% were produced using powder metallurgy method. Doing that, NiO and YSZ, both having average particle size of 45 µm, and corn starch were utilized as matrix, reinforcement, and pore former, respectively. The samples fabrication done as following:

1. Dry Milling and dry mixing were performed in two steps, first: mixing and milling of NiO+YSZ and second: mixing and milling of NiO+YSZ+starch.
2. For bonding ceramic and polymer powders, granulation was done using polyvinyl alcohol (PVA), following that the pasted mixture was sieved after 24 hr.
3. Cold pressing was applied via uniaxial press and without any lubricant at 110 MPa. Green discs had 3 mm height and 17 mm diameter. Following that, samples were dried at oven for 24 hr.
4. Sintering the green discs was considered with two soaking times (30 and 120 min) with different heating and cooling gradient.

The symbol used in this study for green and sintered discs were defined as G and S, respectively. In addition, the weight percent of YSZ in each composite disc was followed by symbols.

The microstructure of samples was examined using optical and scanning electron microscopy in order to study the crack morphology and porosity distribution. Following that, the image analyzer software was utilized to study microstructural features in detail. For investigation the surface cracks, the surface of samples was grinded using abrasive papers during specific time interval, while the depths of cracked investigated using radiography. Finally, density and micro-hardness of sintered discs were studied using Archimedes and Vickers methods, respectively.

3- Results and Discussion

3- 1- Microstructural Investigation

Considering Fig.1, some separation was seen in the form of cracks and porosity in both green and sintered samples. The widths of these separation were broader in the sintered samples due to the discrepancy of thermal expansion of matrix and reinforcement materials.

In addition to optical micrographs, the results of image analyzing revealed that, the minimum and maximum separations was observed in G30 and S30 samples, whereas, the contribution of porosity was highlighted in comparison to cracks in sintered one. The radiography results of sintered discs emphasized the absence of deep cracks in S30 disc.

3- 2- Density

Fig. 2 shows that, the density of samples decreased by increasing the reinforcement contents due to the angular and plate-like morphology of raw materials, which reducing the flow-ability of powder.

3- 3- Micro-Hardness

The results show that, the value of hardness was not uniform along the diameter of the samples, i.e.; the minimum hardness observed in the center of the disc due to grain growth. In addition, the maximum hardness of samples was obtained in S30, which emphasis the low contribution of cracks in separation phase.

4- Conclusions

1. No macro-cracks, as well as, non-fractured species observed in green and sintered samples, respectively. Both subjects confirmed that this dry-press sintering method was acceptable for producing NiO-YSZ composite.

2. The absence of deep cracks emphasized shallow separations (porosity/cracks) in samples.

3. The maximum separation was observed in S30 sample due to the low contribution of cracks, which was clarified with the maximum micro-hardness.

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