

Processing and properties of Ni dispersed zirconia toughened alumina

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The combination of phase transformation of zirconia and plastic deformation of Ni metal has shown a significant improvement in toughness caused by the synergistic toughening. In the present study, the reduction process within a carbon bed, the effect of Ni content on densification, microstructure and mechanical properties of ZTA/Ni composites was investigated. Powder mixtures of Al_2O_3 , 3YSZ, and nickel nitrate as a source of Ni metal were prepared by precipitated Ni on ceramic powders to obtain ZTA/Ni composites. The composites were pressureless sintered at 1600 °C for 3 hrs within a carbon powder bed. The results showed that the addition of 5 vol.% Ni results in the highest relative density of 93% and the maximum fracture strength and toughness values of 348 MPa and $3.32 \text{ MPa.m}^{1/2}$, respectively.

Key words: Multiphase Synergistic toughening, ZTA/Ni composites, carbon powder bed.

Introduction

The exploitation of the toughening ability of zirconia has led to developing a new generation of alumina composites as zirconia-toughened alumina (ZTA) [1, 2]. ZTA is considered among advanced ceramic due to their chemical inertness, biocompatibility, hydrothermal resistance and superior mechanical properties [3-5]. For these characteristics, ZTA has been used for wide range of applications such as structural parts, cutting tools, dental restoration and biomedical implants [3,4,6-8]. The mechanical properties of alumina were also improved by the addition of dispersed metal inclusions [9-12]. Among them, Ni has been chosen as for its anti-oxidation ability [13] and relatively low melting point [12].

In order to achieve ultimate performance, two toughening agents have been added into alumina to improve its toughness [14-16]. Zirconia and Ni particles have received considerable attention due to their instant availability [17]. The transformation mechanism of zirconia has been used in combination with plastic deformation of Ni inclusions to act as synergistic toughening [18]. This point suggests that the use of two toughening agents should result in a combined toughness increment that is greater than the sum of the individual toughness increments achieved uniquely with either toughening agent [16].

Many synthesizing processes have been used to incorporate metal inclusions on ceramic powder. Practically, the choice of a powder preparation method

is to depend on the production cost and the capability of the method for achieving a certain set of desired characteristics. In view of that, the production of powders from liquid phase precipitation has been widely reported for the synthesis of ceramic matrix composites [12, 17, 19, 20]. The precipitation method is provided high purity, high uniformity, small particle size and narrow size distribution [12]. In the present study, the calcination temperature in air and the reduction processes of powder mixtures within a carbon powder bed were studied. The mechanical properties of ZTA/Ni composites in terms of various Ni content addition are evaluated.

Experimental Procedure

Materials

Alumina oxide with a purity of 99.98% ($\alpha\text{-Al}_2\text{O}_3$, Alteo, France), 3 mol% yttria stabilized zirconia (3YSZ, Torch, Japan) and Nickel (II) nitrate hexahydrate ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, Merck, Germany) was used as starting. The 3YSZ was adjusted to result in 15 vol.% of all composites.

Methods of preparation

ZTA/NiO powder mixtures were fabricated by precipitation method. A weighed quantity of nickel nitrate powder, corresponding to 0, 2.5, 5 and 7.5 vol.% Ni metal in the final composites was initially dissolved in ethanol. Subsequently, Al_2O_3 and 3YSZ powder were mixed with the above-mentioned solution and ball milled in a planetary mill for 8 hrs using Teflon bottle with Al_2O_3 balls. The planet and sun speeds were 300 and 100 rpm, respectively. The obtained solution mixtures were dried, then ground in an agate mortar and sieved to 150 mesh. Finally, they were calcined at

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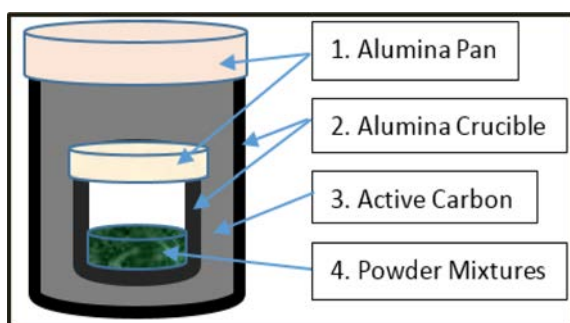


Fig. 1. The carbon powder bed schematic.

450 °C for 3 hrs in air to obtain the ZTA/NiO powder mixtures. The NiO contained powder mixtures were heated at 750 °C for 4 hrs within a carbon powder bed to reduce only NiO to metallic Ni and obtained ZTA/Ni powder mixtures. The carbon could generate a reducing atmosphere, CO. The schematic of carbon powder bed is illustrated in Fig. 1. To investigate the phase change during reduction, the powder mixtures were heated at various temperatures for 4 hrs within the carbon powder bed. The reduced powder mixtures were uniaxially pressed at 10 MPa in a round steel and followed by isostatic cold pressing (CIP) at 450 MPa for one min. The green disk-shaped samples were pressureless sintered within a carbon powder bed at 1600 °C for 3 hrs. The heating rate was fixed at 5 °C/min. The ZTA composites were also prepared with the same way.

Characterization

Phase identification of the powder mixtures was examined by X-ray diffraction (XRD) using Cu K α radiation. The thermal decomposition behavior of NiO was determined by thermogravimetric (TG) and differential thermal analysis (DTA) on STA thermal analysis device in the air up to 800 °C at a heating rate of 10 °C/min. A scanning electron microscopy (SEM) was used to microstructure analyses. The samples were ground successively up to 3000 grit silicon carbide paper and then polished with 6 and 1 μ m diamond paste. A chemical etching was carried in hot orthophosphoric acid solution (H₃PO₄, 85%) for 2.5 min. For powders examination, the powder mixtures were mixed with alcohol and subsequently dispersed using an ultrasonic bath for 15 min to avoid agglomeration.

The relative density of the sintered composites was determined using Archimedes principle (ASTM C373). The sintered disks were cut with a diamond saw and machined into rectangular bars of 3.5 mm $\times$ 3.5 mm $\times$ 24 mm. The fracture strength was determined according to ASTM D790 by the three-point bending test with a span of 18 mm. The average bending strength was obtained from measurements of four to five samples. The hardness was measured using the Vickers indentation method in accordance with ASTM C1327-99. Four to six indents were made on the polished cross-sectioned

samples using an applied load of 9.8 N (1 Kgf) during 15 seconds. The fracture toughness of the sintered samples was estimated by the Vickers indentation method under a load of 98 N (10 Kgf) during 15 seconds. Eight indents were made on polished cross-sectioned samples. The toughness (K_{IC}) was taken to be the average of eight indents and found by using Niihara expressions [21].

$$K_{IC,N} = 0.023H(c/a)^{-1.5}a^{0.5} \quad (1)$$

where H is the indentation hardness, c is the average trace length of the crack measured from the center of the indentation, and a is the half of average duration of two indent diagonals.

Results and Discussion

ZTA/NiO powder mixtures characterization

DTA-TG determination of nickel nitrate precipitated on Al₂O₃, and 3YSZ particles have been studied to determine the appropriate calcination temperature, as shown in Fig. 2. Four different endothermic peaks and three major weight loss is observed. The first is a broad peak below 180 °C and coincides with the first weight loss. It is due to the evaporation of physically adsorbed water and alcohol. The peaks 2 and 3 are located between 180 and 280 °C and accorded with the second weight loss. They correspond to the elimination of nitrate ions and formation of the nickel hydroxide Ni(OH)₂ with increasing the temperature [22]. The third weight loss from 280 to 330 °C matches the last peak and is attributed to the complete dehydration of the nickel hydroxide. Finally, a nearly stable weight is attained at temperatures above 330 °C. These results are in good agreement with the results of Esteban-Betegón et al. [20]. They precipitated nickel nitrate corresponding to 10 vol.% Ni on 3YSZ. To ensure complete decomposition and formation of NiO, the powder mixtures was calcined in air at 450 °C for 3 h. The XRD analysis of the calcined powder mixtures (not shown) indicated the presence of NiO phase together with the α -Al₂O₃, t-ZrO₂ and m-ZrO₂ phases.

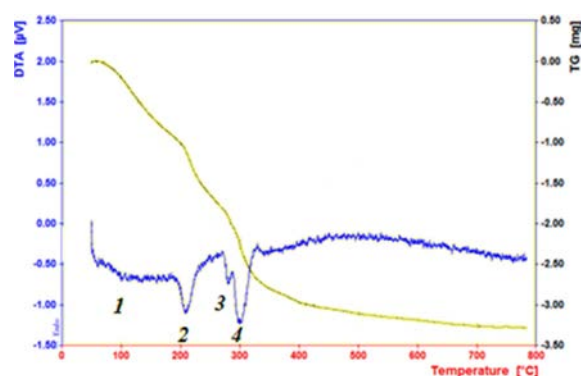


Fig. 2. DTA-TG curves for the powder mixture.

Reduction process

The key phenomenon for the synthesis process is the reduction of NiO to metallic Ni. NiO would be reduced to Ni if the oxygen pressure of the system (P_{O_2}) is lower than the stability limit of NiO ($P_{O_2(NiO)}$), which was calculated according to the next formula within the temperature (T) range of 627–1127 °C [23]:

$$P_{O_2(NiO)} = \exp\left(\frac{-563330 \pm 130}{T} + 20.42\right) \quad (2)$$

However, the thermodynamic consideration reveals that Ni can react with Al_2O_3 and O_2 to form $NiAl_2O_4$ spinel phase, whereas there is no interaction with ZrO_2 phases [20, 24]. When the formation of spinel takes place, a 4 vol.% expansion occurs with the reaction. Thus, the densification of the composites would be retarded [25]. The stability limit of the spinel formation ($P_{O_2(s)}$) was expressed by the following equation [23]:

$$P_{O_2(s)} = \exp\left(\frac{-58240 \pm 480}{T} + 17.94\right) \quad (3)$$

Similarly to Ni-NiO system, the value of P_{O_2} is necessary to be lower than $P_{O_2(s)}$ for preventing the formation of the spinel phase from 627 °C to 1127 °C. On the other hand, reduction in a carbon powder bed mainly generates carbon monoxide as:



By assuming unit activity for the solid carbon, and take a value of 1 atm for the partial pressure of CO gas, the value of equilibrium oxygen partial pressure ($P_{O_2(CO)}$) for the above reaction within the temperature range of 25–1727 °C becomes [26]:

$$P_{O_2(CO)} = \exp\left(\frac{-26868}{T} - 21.08\right) \quad (5)$$

P_{O_2} is depended directly to $P_{O_2(CO)}$. Therefore, $P_{O_2(CO)}$ should be lower than both $P_{O_2(NiO)}$ and $P_{O_2(s)}$ for providing complete reduction to Ni metal without spinel production. According to the above equations,

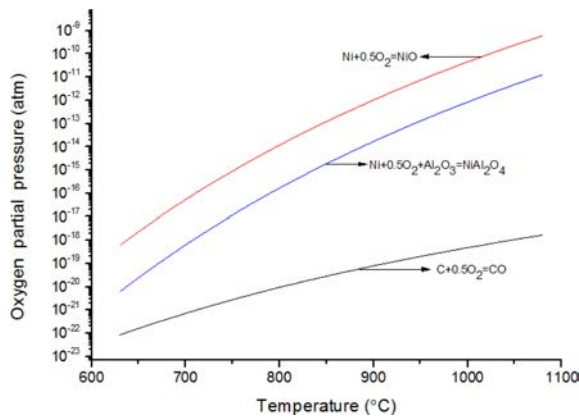


Fig. 3. Oxygen partial pressure as a function of temperature.

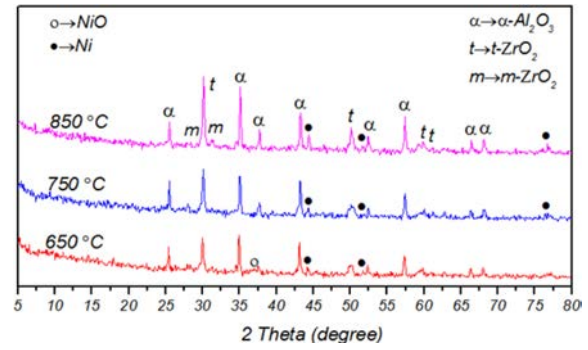


Fig. 4. The XRD patterns of the ZTA/Ni powder mixtures reduced within a carbon bed.

the value of $P_{O_2(CO)}$ within is lower than the stability limit for the both Ni-NiO and spinel formation systems, as shown in Fig. 3. Accordingly, we can estimate that CO gas generated by carbon powder can be a sufficiently high reductive atmosphere for NiO and spinel phases.

To determine the minimum reduction temperature, the ZTA/Ni powder mixtures are heated at 650, 750 and 850 °C within a carbon powder bed for 4 hrs. The XRD patterns of the heated samples are shown in Fig. 4. As expected, there is no evidence of $NiAl_2O_4$ spinel phase indicating that the XRD results confirm the thermodynamic analysis. At 650 °C, the NiO is partially reduced. Thus, both NiO and Ni phase peaks appear. The NiO phase peaks disappear completely from 750 °C, and the all Ni phase peaks are only present. Therefore, the temperature of 750 °C seems to be the lower reduction temperature of NiO within a carbon powder bed for 4 hrs in the current system.

Morphology of powder mixtures

Fig. 5 shows the morphology of the powder mixtures after calcination and reduction. Some agglomeration of Al_2O_3 particles is showed after heating at 450 °C (Fig. 5(a)). Nano-sized NiO particles were formed from nickel nitrate precursor after calcination. They dispersed on the surfaces of particles and had a roughly spherical shape. Moreover, the nano-NiO particles were agglomerated and grown during thermal decomposition. The average

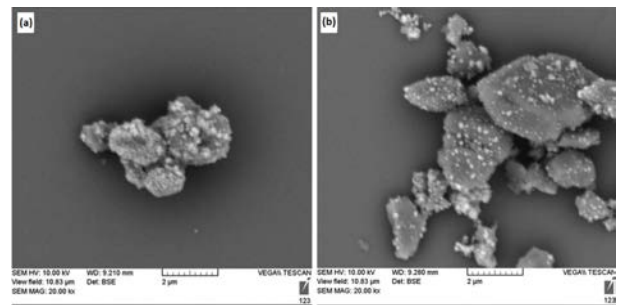


Fig. 5. SEM micrographs of powder mixtures (a) calcined at 450 °C and (b) reduced at 750 °C within a carbon bed.

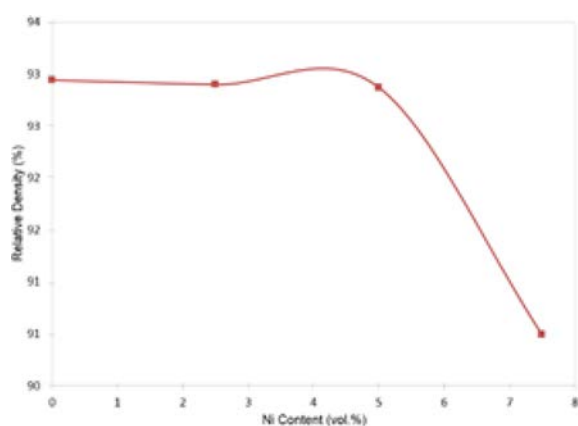


Fig. 6. The relative density of ZTA/Ni composites as a function of Ni content.

particle size of nano-NiO determined from SEM was about 90 nm. After reducing within the carbon powder bed, Ni metal is formed as revealed by XRD and predict by thermodynamic analysis. Nano-sized Ni particles were well dispersed on the surfaces of the large Al_2O_3 particles and had a roughly spherical shape (Fig. 5b). The average particle size of nano-Ni determined from SEM was about 79 nm. The size of Ni inclusion depends directly on the size of NiO before reduction [12, 27].

Effect Ni content on relative density

Fig. 6 shows the effect of the addition of different Ni content on the relative density of ZTA/Ni composites. Addition Ni up to 5 vol.% maintained the sinterability of the composites stable due to agglomeration limiting of Ni inclusions [12, 19]. However, the relative density decreased with further increasing of Ni content. Generally, the presence of Ni inclusions inhibits the composites densification because of poor wettability of Al_2O_3 by Ni melt and the thermal expansion mismatch between ceramics and Ni [16, 27, 28]. Consequently, thermal residual stresses would produce in the cooling stage that leads to forming many pores at the interfaces of Ni/ceramics. The thermal residual stresses in metal/ceramic composites are proportional to the metallic inclusions content [29]. The more Ni content at 7.5 vol.% formed larger Ni agglomerations. Thus, the interfaces became larger, and the pores amount grew significantly and that dropping the relative density of ZTA/7.5 Vol.% Ni composite [19].

Investigation of poor density

The present composites could be densified only up to 93% as sintered at 1600 °C within a carbon bed. The lower sintered density may result from thermal expansion mismatch between ceramics and Ni metal, relatively higher 3YSZ amount, transformation phase of 3YSZ and sintering in CO atmosphere. The large number of closed pores seen in Fig. 7. indicates the

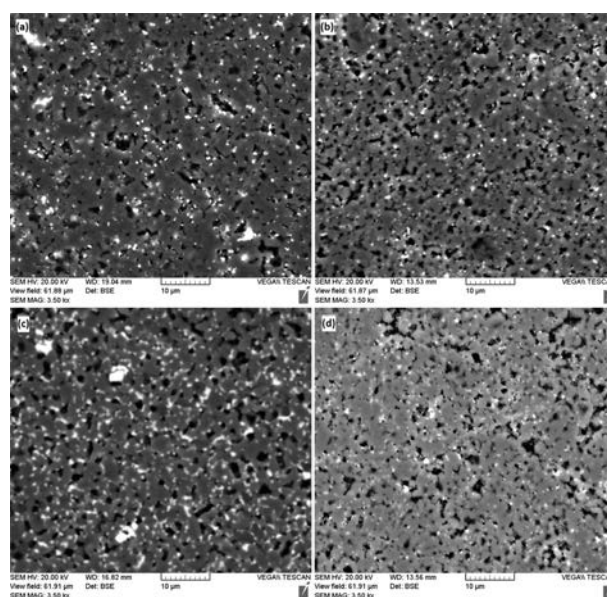


Fig. 7. SEM micrographs of the polished surfaces of ZTA/Ni containing (a) 0, (b) 2.5, (c) 5 and (d) 7.5 vol.% N.

poor density of samples. While the TEC of Ni is larger than that of Al_2O_3 and 3YSZ, internal stresses are induced within and around particles during the cooling process. In this case, $\text{Al}_2\text{O}_3/\text{Ni}$, 3YSZ/Ni and $\text{Al}_2\text{O}_3/3\text{YSZ}$ interfaces is subjected to a radial tensile stress and that causes microcracks formation [12, 30, 31]. Therefore, the closed porosities are always located at the interfaces in the composites [18, 32]. It has been reported that presence of high amount of 3YSZ as a second phase results in unequal dispersion and agglomeration of 3YSZ particles (Large white grains in Fig. 7) [5, 33]. In this case, large pores would form and the densification of composites decreases. On the other hand, the spontaneous transformation from $t\text{-ZrO}_2$ to $m\text{-ZrO}_2$ during cooling from the sintering temperature also induces simultaneously microcracks [34]. The sintering atmosphere have important effects on densification and microstructural development of monolithic alumina during sintering [35, 36, 37]. The experimental studies have indicated that Al_2O_3 could not be sintered to full density in CO or CO_2 due to the low solubility of the gas molecules trapped in the internal pores [36]. These gases cannot diffuse out as shrinkage takes place, thus accumulating and their pressure increases. Consequently, the densification of samples will be resisted and the composites could not reach full theoretical density.

Effect Ni content on microstructure

The microstructure of sintered composites with different Ni content is shown in Fig. 7. The scattering contrast results in a white feature for 3YSZ grains and grey for Al_2O_3 grains. Some 3YSZ agglomerates are found occasionally. The agglomerating of 3YSZ particles may occur because of inability of eliminating them during the powder processing step. The chemical etching

could remove some Ni inclusions from the polished surface [17]. In general, homogeneous microstructure and no abnormal grain growth of matrix were observed in all samples. Nevertheless, the dispersion of Ni inclusions within the matrix did not seem to have more effective on constraint of matrix grains. Previous researches of Chen et al. [16] and Tuan et al. [31] showed similar trends with the present study.

Effect Ni content on mechanical properties

Fig. 8 shows the hardness of ZTA/Ni composites with different Ni content. The hardness of ZTA was the highest among all samples, which attained a value of 14.8 GPa. However, the hardness dropped gradually with Ni addition and reached a value of 11.3 GPa at 7.5 Vol.% Ni. The dispersion of Ni inclusions within the composites decreases their hardness expectedly due to the softer character of the ductile inclusion. Moreover, the decreasing in hardness with increasing Ni content is in agreement with the rule of mixture. A similar tendency was showed in Refs [38, 39].

Fig. 9 shows the effect of different Ni amount addition on the fracture strength of ZTA. The strength of ZTA/Ni composites increased with Ni content and attained its highest value of 348 MPa at 5 vol.% Ni.

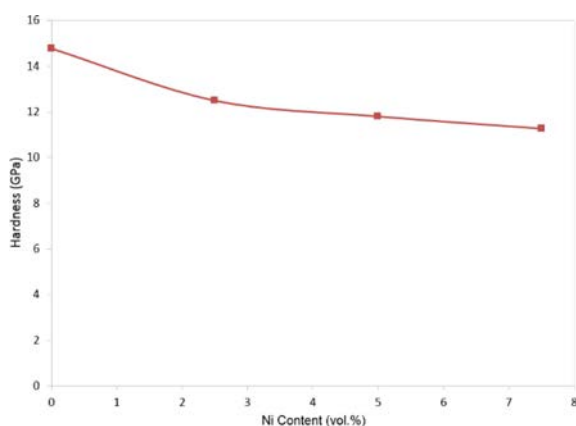


Fig. 8. The hardness of ZTA/Ni composites as a function of Ni content.

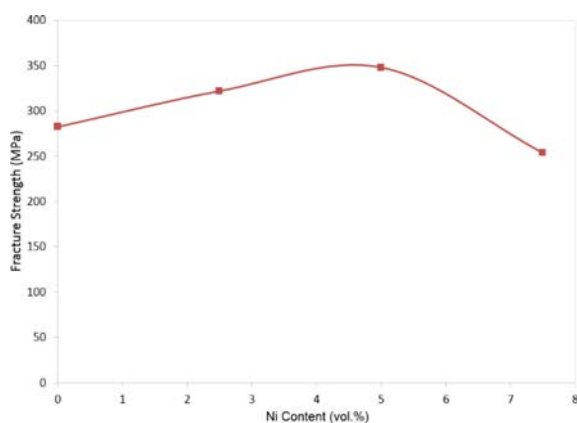


Fig. 9. The fracture strength of ZTA/Ni composites as a function of Ni content.

The increasing in strength with Ni content may be related to matrix grain refinement [12, 27]. However, with more addition of Ni up to 7.5 vol.%, the strength decreased to a value of 254 MPa. The deteriorating in strength for is due to density degradation. The higher Ni content the more Ni agglomerates would form that produce more amount of porosity and flaws [12, 40]. Chen et al. [16] reported that the strength of the $\text{Al}_2\text{O}_3/(\text{ZrO}_2 + \text{Ni})$ composites is not higher than that of $\text{Al}_2\text{O}_3/\text{ZrO}_2$ composites because of the more ability of 3YSZ to prohibit the grain growth of matrix than composites containing both 3YSZ and Ni particles. This behaviour can be attributable to the micro size of Ni inclusions. In the present study, the improvement of strength above that of ZTA composites may result from incorporation of nano sized Ni. However, the present composites was showed a relatively lower fracture strength due the porous and microcracked microstructure.

A combination of the both intergranular and transgranular fracture modes is appeared by incorporation of Ni inclusions, as shown in Fig. 10. Because of the TEC mismatch between the matrix and second phases, stresses could be induced during cooling and causes the transgranular fracture mode [40, 41]. More energy is required for crack propagation in transgranular fracture through grains than intergranular one, thus increasing the fracture strength of composites [42].

The average values of fracture toughness of ZTA/Ni composites are shown in Fig. 11. With addition of 2.5 vol.% Ni, the toughness not increased more. Yet, further addition of Ni up to 5 vol.% increased further the toughness to its highest value of $3.32 \text{ MPa}\cdot\text{m}^{1/2}$. Ashby et al. [43] proved that the toughness enhancement for the ceramic/metal composites depends strongly on the size of the metallic inclusion. Tuan et al. [25, 27] suggested

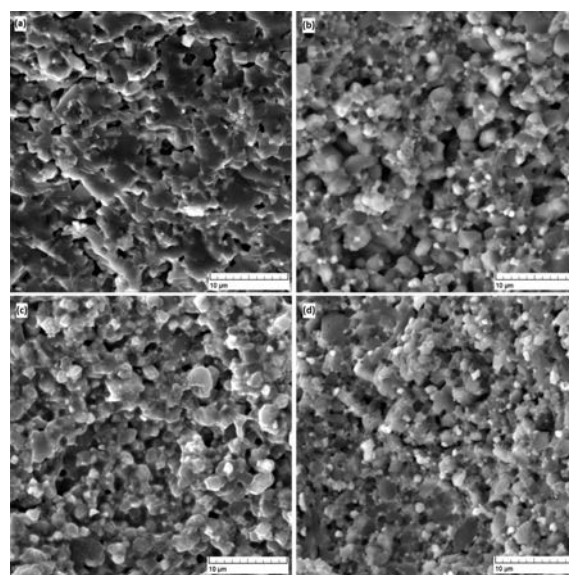


Fig. 10. SEM micrographs of the fracture surfaces of ZTA/Ni containing (a) 0, (b) 2.5, (c) 5 and (d) 7.5 Vol.% Ni.

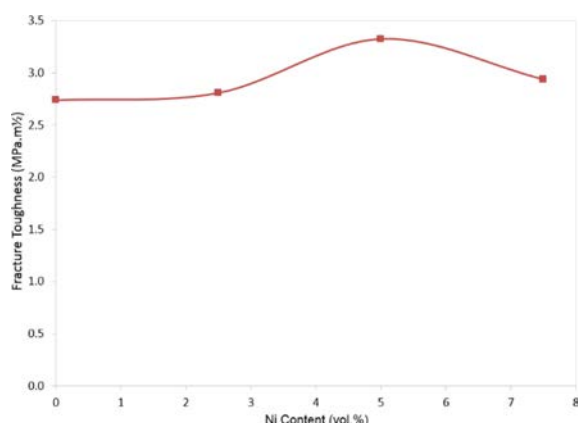


Fig. 11. The fracture toughness of ZTA/Ni composites as a function of Ni content.

that by keeping the Ni size smaller than a critical value, the toughness of $\text{Al}_2\text{O}_3/\text{Ni}$ composites was further enhanced by adding more Ni inclusions without dropping in density. Therefore, larger agglomerations of Ni arose at 7.5 vol.% Ni that results in the presence of additional microcracks and then degrades the toughness of composite [32]. The toughness for all composites value was lower than that reported in the literature due to poor density.

Conclusions

The formation of NiO found by heating at $T > 450^\circ\text{C}$ in air. NiO particles reduced to Ni metal at $T > 750^\circ\text{C}$ within a carbon powder bed with no detection of NiAl_2O_4 phase. The thermodynamic analysis confirmed with the XRD results. Pressureless sintering of $\text{Al}_2\text{O}_3/(\text{ZrO}_2+\text{Ni})$ composites at 1600°C for 3 hrs within a carbon powder bed could not reach a full, dense body, and the relative density attained its maximum value of 93% for ZTA/5 vol.% Ni composite. The poor density was mainly attributed to the effect of CO atmosphere. The maximum values of fracture strength and toughness were attained for ZTA/5 vol.% Ni composite.

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