

Effect of Ni/NiO particles on structure and crack propagation in zirconia based composites

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ABSTRACT

The structure formation of yttria stabilized zirconia - nickel oxide composites sintered in air and argon atmosphere were studied. It was shown that the crack propagation in 3Y-TZP ceramics greatly inhibited by creation of composite structure by addition of NiO particles and sintering at 1500°C in argon atmosphere. Prevention of formation of the monoclinic phase of zirconia was conditioned by sintering composite under argon. Increasing of K1C value was found as in sample shell as in sample core of composite, in comparison with matrix 3Y-TZP ceramics. The increasing of K1C value of obtained intergranular type of composite structure cannot be explained by crack deflection and crack bridging processes by Ni and NiO particles, respectively. The possible explanation of K1C value increasing is the intensification of phase transformation toughening of zirconia by formation of metastable tetragonal phase depleted by Y^{3+} ions. The reversibly dissociation of NiO on Ni and oxygen in neutral atmosphere provided the formation of cubic phase and Y^{3+} depleted tetragonal phase. Internal oxidation of Ni during cooling leads to formation metastable tetragonal phase in depleted Y^{3+} zirconia grains. The formation of NiO particles during cooling lead to emergence of large compressive stresses, which also increased the metastability of tetragonal zirconia grains, depleted of Y^{3+} ions. Copyright © 2014 VBRI press.

Keywords: Zirconia-nickel composites; phase transition; fracture toughness; reversible dissociation.

Introduction

Yttria-stabilized tetragonal zirconia polycrystalline (3Y-TZP) ceramic shows superior qualities compared to other materials, such as strength, toughness, hardness, and chemical stability [1, 2]. However, application of 3Y-TZP ceramics in engineering is constrained by its brittleness. A basic scientific task is to improve the crack resistance or fracture toughness of 3Y-TZP. "Transformation toughening" of zirconia is an outstanding method of enhancing of fracture toughness of zirconia-reinforced composites [3]. However, phase transformation can lead to negative results, including a loss of surface integrity. This effect often limits application of zirconia ceramic in orthopedic [4] and wear devices [5, 6]. Development of different types of composites is one widely-studied approach. Inclusions may be brittle, such as alumina, zirconia particles, silicon carbide, and mullite whiskers, or ductile metallic particles such as nickel, iron, and silver. Crack deflection and crack bridging mechanisms are also important for increasing the fracture toughness of composite materials [7, 8]. However, increased toughness of the material should not lead to a decreased strength value. Traditional zirconia-alumina composites often reflect this conflict between toughness and strength. The composite system of zirconia-nickel (nickel oxide) is rarely

used. However, we hypothesized that combining Ni and NiO in this composite system may be a compromise between plasticity and hardness of the inclusion and matrix, resulting in an increased fracture toughness value while maintaining strength on the same level.

A small number of studies have been conducted to examine the influence of Ni and NiO particles and their association with fracture toughness of monolithic 3Y-TZP ceramic materials. The Al_2O_3 -Ni (for engineering purposes) and fully stabilized zirconia-Ni/NiO (for solid oxide fuel cells) systems have been more widely studied. Previous studies showed that inclusion of nickel in an alumina and zirconia matrix led to increased fracture toughness of the alumina or zirconia ceramic material [7-12]. Investigations of the influence of NiO on sintering behavior, such as electrical and physical properties, as well as phase transitions in different zirconia materials, have been previously conducted in [13-17]. During sintering, addition of NiO can be promoted: i) stabilization a cubic phase of zirconia and ii) destabilization of tetragonal phase and formation of monoclinic phase. These two facts were investigated separately and only Delaforce and Yeomans [18] found these both phase transformations simultaneously. Formation of very large amounts of the monoclinic phase even with small quantities of NiO (0.3-2 wt%) led to sample destruction. Formation of the cubic

phase was explained based on the stabilization effects of NiO on the ZrO₂ lattice, similarly to Y₂O₃. However, Stefanic [19] and Chandra [20] studied the effect of NiO on stabilization of the tetragonal phase in pure ZrO₂ and did not identify any traces of the tetragonal phase after heating at 1100 °C. Therefore, NiO did not stabilize the tetragonal phase in ZrO₂. In our previous study [21], we found that the phase transition from the tetragonal to monoclinic phase in the 3Y-TZP-NiO composite occurred only during sintering in an air environment, while the monoclinic phase was not observed after sintering in argon environment. In the sample core, NiO grains were retained, but in a thin sample shell, metal Ni grains were formed. Formation of the complex structure increased fracture toughness of the 3Y-TZP-NiO composite.

In this study, we investigated the effect of sintering conditions on the structure and properties of the ZrO₂-3mol%Y₂O₃-NiO(Ni) composite system, as well as the effect of reversible dissociation of NiO on Ni and oxygen on the phase stability of ZrO₂. We discuss the various toughening mechanisms that can be explained our results for sintered composite material.

Experimental

Preparation methods

Zirconia nanopowders ZrO₂ – 3 mol%Y₂O₃ (3Y-TZP) were synthesized using a co-precipitation technique from ZrOCl₂·nH₂O and Y(NO₃)₃ salts. The details of this process are published elsewhere [22, 23]. The 3Y-TZP nanopowders were obtained by calcination at 700 °C for 2 hours. The preparation of the nanocomposite powders was conducted by mixing appropriate amounts of zirconia and nickel oxalate powders in distilled water using ultrasound at a frequency of 22 kHz. NiO in the composite nanopowders were obtained by the calcination of powders at 600 °C. Several powders types were prepared with compositions of 5; 7.5; 10; 15 and 20wt% of NiO. For detailed investigation in this study the composition with 10 wt% of NiO was chosen, but for understanding the trends the other composition will use if needed.

The resulting powders were uniaxially pressed at 50 MPa into rectangular specimens with dimensions of 6×6×60 mm, and samples in pellet form measuring 20 mm in diameter and 3 mm in height, and they were cold isostatically pressed at 200 MPa. Based on the results of study [21] the prepared green compacts were sintered in air at argon atmosphere at 1500 °C during 2 hours.

The specimens used for mechanical testing were machined to a bar shape measuring 3×4×45 mm, were ground with a 180-grit diamond wheel, and were subsequently polished with diamond slurries to minimize machining flaws.

Testing methods

The powders obtained after the calcinations, as well as the ceramics materials, were characterized by X-ray diffraction (XRD) employing a diffractometer DRON-3 (USSR), with CuK α radiation. For identifying of the monoclinic (M), tetragonal (T) and cubic (C) phases of zirconia, as well as Ni and NiO, the angular regions of 25°- 45° and 71°-77°

were used. The crystalline size of the powders was determined by the Scherer equation. X-ray analysis of the sintered material was performed on the polished surfaces of the samples. The fraction of monoclinic ZrO₂ on the polished surface was calculated using the equation proposed by Garvie [24]. The fitting and analysis of XRD curves were made by Powder Cell for Windows version 2.4. The powders were also studied by means of transmission electron microscopy JEM 200 A, (JEOL, Japan).

The flexural strength was determined at room temperature using a four-point flexural configuration on universal testing machine H50KT (Tinius Olsen, USA) with inner and outer spans of 20 mm and 40 mm. The indentation fracture toughness (KIC) was measured via the indentation method on Vickers hardness tester TP-7p-1, (ZIP, USSR) using 196 N force. A force of 98 N was used for hardness measurements. The hardness and fracture toughness were measured at room temperature by the Vickers indentation technique on mirror-polished surfaces with a 98 and 196 N loads, respectively. At 196 N loads the Palmquist type cracks were propagate in 3Y-TZP and composite with NiO(Ni). The fracture toughness values were calculated by Niihara equation for Palmquist type cracks [25]. The reported values for hardness, KIC and bending strength were the means of the data obtained from at least five tests. The calculated KIC values for the composites were used only as relative values to start 3Y-TZP ceramics. The density was measured using the Archimedes method. The microstructures and chemical compositions of the composites were studied by scanning electron microscopy JSM 6490LV (JEOL, Japan) with EDS analysis Inca Penta FETx3 (Oxford Instruments, UK) at polished and fractured surfaces. After removing the 100-200 μ m layer from sample surface the XRD, SEM and EDS analysis, hardness and indentation fracture toughness were measured. The 6-7 layer by layer removal was carried out for determination the influence NiO to Ni transformation on structure and properties of sintered composites.

Results and discussion

Properties and phase compositions

According to the electron microscopy and XRD data, the average particle size of matrix 3Y-TZP nanopowders was 17.5 nm. After the calcination of the nanopowder mix at 600°C, nickel oxide was formed with an average particle size of 40 nm. The 3Y-TZP -10wt%NiO powder is represented in Fig. 1. Zirconia in powders was represented by the tetragonal phase (P4m2), and NiO was represented by the cubic phase (Fm3m).

The phase composition sintered at 1500°C in air and argon environment samples are represented in Table 1. Because samples for general XRD analysis were prepared by well-known instruction for preparation of ceramic samples (grinding and polishing), the ratio between Ni and NiO composition was disturbed, then in this table only zirconia phase are represented. The change of Ni/NiO ratio with sample depth was shown on Fig. 2.

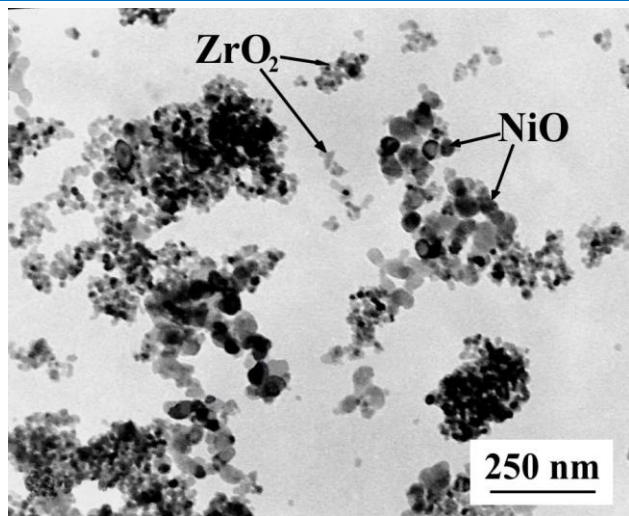


Fig. 1. TEM structure of the 3Y-TZP-10wt%NiO starting nanopowder.

Table 1. The dependence of phase composition of 3Y-TZP and 3Y-TZP – NiO composites sintered at 1500°C from NiO concentration and sintering atmosphere in sample surface layer and core (after removing more than 1 mm of sample surface).

Sintering environment	Sample composition	Phase composition, %					
		Surface layer			Sample core		
		C	T	M	C	T	M
Air	3Y-TZP	10	90	-	10	90	-
	+5wt%NiO	19	44	37	18	45	37
	+10wt%NiO	21	44	35	21	43	36
	+15wt%NiO	22	42	36	22	42	36
	+20wt%NiO	22	39	39	23	39	38
Argon	0Y-ZrO ₂	-	-	100	-	-	100
	+10w%NiO	-	-	-	-	-	-
	3Y-TZP	11	89	-	11	88	1
	+5wt%NiO	21	77	2	19	80	1
	+10wt%NiO	25	73	2	21	77	2
	+15wt%NiO	30	68	2	22	74	4
	+20wt%NiO	34	63	3	21	76	3
	0Y-ZrO ₂	-	-	-	-	-	100
	+10w%NiO	-	-	-	-	-	-

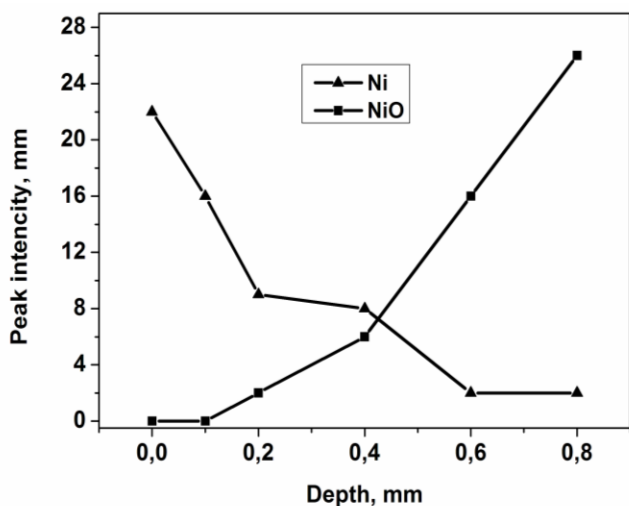


Fig. 2. The XRD data of distribution of Ni and NiO at the sample depth in 3Y-TZP – NiO composites sintered at 1500°C in argon environment.

By XRD, SEM and EDS analysis was found that phase composition depend from sintering environment and concentration of NiO. In case of sintering in argon

environment the increasing of NiO concentration lead to increasing the quantity of cubic phase in surface layer. In surface layers of samples sintered in argon the amount of cubic phase of zirconia is higher in comparison with sample core. In sample core the amount of cubic phase is stabilized on a level of $20\pm 2\%$ independently from NiO concentration. The 2-5% of monoclinic phase is conditioned by grinding and polishing procedure.

In case of sintering in air environment the huge amount of monoclinic phase are formed (35-39%). The amount of zirconia cubic phase in sample shell and core are equal and reach to a value $20\pm 3\%$. By a strange coincidence the quantity of cubic phases in zirconia samples (core and shell) sintered in air environment are equal to amount of cubic phase in zirconia samples core sintered in argon environment.

Also was found that after sintering in argon environment at the sample shell the Ni grains were formed, but layer by layer removal was shown that in the sample core at the depth 0.6-0.8 mm the NiO grains are retained. Increasing the number of grains of nickel oxide and, correspondingly, decreasing the amount of nickel metal grains in 3Y-TZP-NiO(Ni) composite occurs smoothly (Fig. 2).

The SEM analysis shown that the average grain size of composite, sintered in argon and air environment, do not depend from NiO concentration and gas atmosphere in time sintering (Fig. 3).

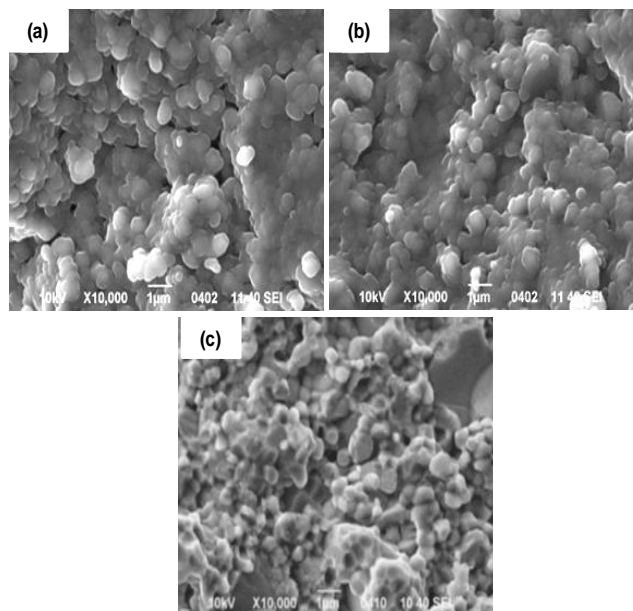


Fig. 3. SEM structures of sintered 3Y-TZP-NiO ceramic samples in an argon - a), b) and air - c) atmosphere at 1500°C: a) - without NiO, b) and c) - with 10 wt% NiO.

The hardness and indentation fracture toughness values increased with direction into the sample core (Fig. 5), consequently with increasing NiO grains instead of Ni grains. Also was found that in case of NiO the crack passes through the NiO grains and in case of Ni grains the cracks go round the inclusions. For comparison the indentation fracture toughness for matrix $ZrO_2 - 3 \text{ mol\% } Y_2O_3$ material was $6.5 \text{ MPa}\cdot\text{m}^{1/2}$. These data are in a good agreement with data published in [15-17]. We understand that the

indentation technique has a trend to increase the K_{1C} value, but we use these data in comparison with matrix material.

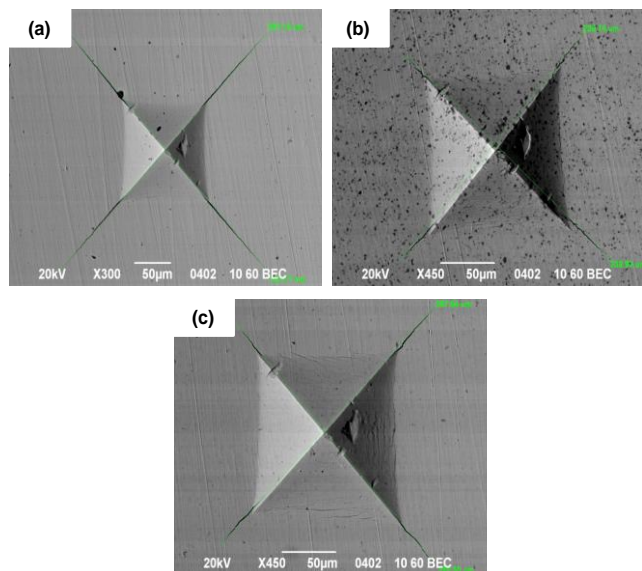


Fig. 4. SEM images of Vickers indentation and cracks at surface of sintered composite samples: (a) – matrix 3Y-TZP, (b) - 3Y-TZP+10wt% NiO sample core and (c) - 3Y-TZP+10wt% NiO sample shell.

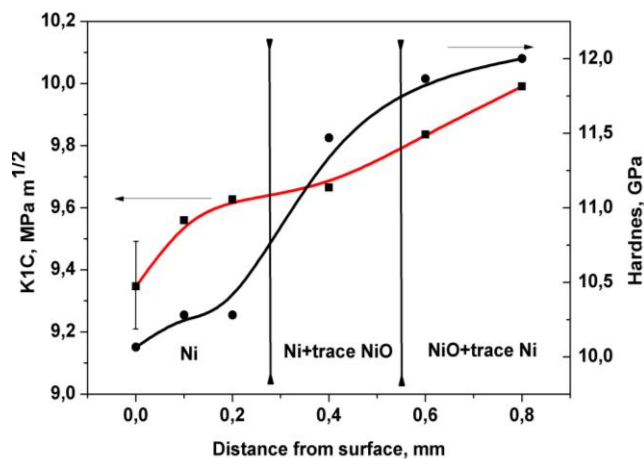


Fig. 5. The dependence of indentation fracture toughness and hardness values of sintered ZrO₂- 3mol%Y₂O₃ - NiO(Ni) composite from distance from sample surface or chemical composition.

Fracture toughness

The possible toughened mechanisms in 3Y-TZP-NiO ceramic composites [26-30] include phase transition in zirconia, crack bridging, and deflection by NiO(Ni) grains. Calculations of fracture toughness increment using equations from Li and Watanabe study [26] showed that crack bridging by NiO grains can increase the K_{1C} value by only 0.1–0.3 MPa*m^{1/2} and crack deflection by Ni grains by 0.8–1.2 MPa*m^{1/2} in comparison with matrix material 3Y-TZP. For these calculations, the Young modulus for NiO was near 100 GPa [31] and for Ni this value was 205-220 GPa [32]. We hypothesized that the crack passed through NiO grains because the Young modulus of NiO was less than that of zirconia (195-198

GPa); the crack was also around Ni grains because the Young modulus of Ni is larger than that of zirconia [33]. The calculated fracture toughness increment can be used to explain only the higher fracture toughness values due to the increased amount of filler grains (NiO or Ni) in the composites. However, the experimentally observed indentation fracture toughness data for composite materials (9.3–9.9 MPa*m^{1/2}) was larger than that for the matrix material (6.5 MPa*m^{1/2}). The sharply increased fracture toughness may be conditioned only if we assume that addition of nickel oxide affects the transformability of the tetragonal phase in 3Y-TZP.

Complex effect dopant and environment on phase composition

We divided the results obtained after sintering in argon into two groups: sample core (more than 1 mm from the surface) and sample shell (the shell within 1 mm of the surface). This division is based on the change in chemical composition. In sample shell, metal Ni grains were present, while in the sample core, NiO grains were retained.

The different behavior of phase composition of 3Y-TZP-NiO composites in time sintering in different atmospheres caused us to disturb in ability Ni²⁺ to stabilize the zirconia lattice as was shown in [18]. Any traces of tetragonal or cubic phases did not found in [19, 20], in which stabilization of the pure zirconia by NiO was studied. In our investigation, we found no traces of the tetragonal or cubic phases in sintered ceramic composites ZrO₂-10wt%NiO when using unstabilized zirconia, irrespective of the sintering atmosphere. Use the composition ZrO₂ - 1.5 mol%Y₂O₃-10wt%NiO in attempt to found formation the cubic phase with Ni²⁺ assistance did not successful also. What was responsible for these changes in the phase transformations in 3Y-TZP-NiO composites? How did the gas atmosphere affect phase stability and fracture toughness of 3Y-TZP-NiO composites?

Beginning with the analysis from the sample core, the formation of cubic and monoclinic phases occurred simultaneously during sintering of 3Y-TZP-NiO composites at 1500°C in an air environment (Table 1) agreed well with the results of a study [18]. In contrast, the monoclinic phase did not form in 3Y-TZP - NiO composites during sintering in an argon environment. The amount of the cubic phase in the sample core when sintering 3Y-TZP-NiO composites in argon were equal to the amount of cubic phase in the entire 3Y-TZP-NiO composite sintered in an air environment. Thus, the sintering environment did not affect cubic phase stabilization in the zirconia sample core in the presence of NiO.

Enrichment of zirconia grains by Y³⁺ ions passed independently on the oxygen partial pressure in the sample core, but faster in comparison with the matrix material. Between 18 and 23% of the cubic phase formed in the sample core of composites with NiO compared with 10–11% of the cubic phase in the matrix material. This can be explained by the stabilization effects of NiO on the ZrO₂ lattice, similarly to in Y₂O₃ [15-18], but NiO was enriched as grains in a cubic and tetragonal lattice (Fig. 6, Table 2).

The transformation to the monoclinic phase should not occur because the sum of stabilizers Σ ($Y^{3+}+Ni^{2+}$) was consistently larger than with only one stabilizer, Y^{3+} . However, during sintering in air, 35–39% of the monoclinic phase was formed (**Table 1**), which is a contradicting result. We included additional stabilizers and observed a larger amount of monoclinic phase. The mean grain size did not change with the addition of NiO (**Fig. 3**). Based on electronic dispersive technology data, the concentration of Y_2O_3 in tetragonal grains of zirconia in 3Y-TZP-NiO composites decreased from 5.36 wt% to 4.11 and 2.74 wt%. Tetragonal mean grain size did not increase, but the critical grain size of Y^{3+} -depleted grains decreased. Notably, when Y^{3+} content in zirconia was lower, lower amounts of nickel were also present. We hypothesized that Ni^{2+} ions do not influence the phase stabilization of zirconia, but rather help Y^{3+} ions migrate more quickly and form cubic grains. When sintering of the 3Y-TZP-NiO composite in argon, the amount of cubic phase in the sample core was 20%, but the monoclinic phase was not formed. The main reason for this was the low partial pressure of oxygen (argon atmosphere), which resulted in dissociation of NiO on Ni^{2+} and O_2^- during heating to high temperatures and cooling.

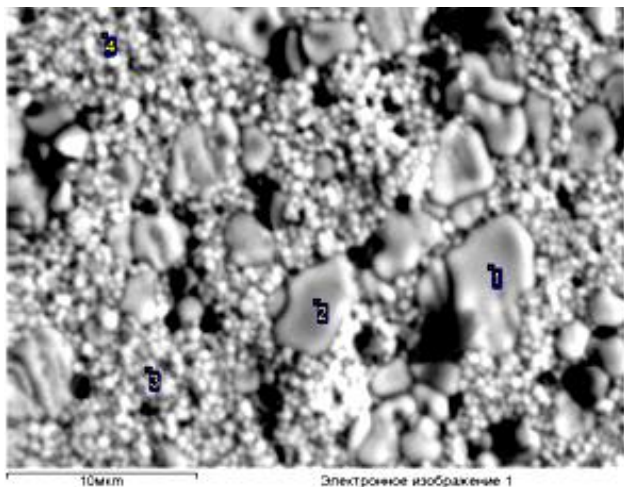


Fig. 6. The SEM image of surface of 3Y-TZP-10wt%NiO sample sintered at 1500°C in argon atmosphere. Light grey – zirconia grain, dark grey – NiO. Big grains were cubic, small were tetragonal symmetry of ZrO_2 .

Table 2. The chemical composition in sample core of 3Y-TZP-10wt%NiO composite sintered in argon environment at 1500°C. The numbers are the chemical analysis in points marked on Fig. 6.

N	Chemical composition, %			Note
	ZrO ₂	Y ₂ O ₃	NiO	
1	87.08	11.30	1.62	C-phase
2	86.37	11.61	2.02	C-phase
3	96.97	2.74	0.30	T-phase
4	94.92	4.11	0.98	T-phase

When the heating–cooling process passed through the air environment, some of the zirconia grains were enriched by Y^{3+} (cubic phase), while some grains were depleted by Y^{3+} when heating at 1500°C. During cooling, Y^{3+} -depleted grains transformed to the monoclinic phase. This is

characteristic behavior of phase transition during zirconia sintering. NiO helps to create a larger amount of Y-rich grains in the cubic phase, resulting in the formation of the monoclinic phase during cooling (**Table 1**). When 3Y-TZP-NiO composites were sintered in an argon environment, the monoclinic phase was not formed, and in Y^{3+} -depleted grains, a sufficient number of oxygen vacancies were retained. These vacancies may be created only during cooling, when the external conditions (decreasing the temperature) shift the balance in direction formation NiO grains from Ni and oxygen.

It is known that under the 1230 °C the reversibly dissociation of NiO on Ni and oxygen is passed. In a [34], it was shown that when the partial pressure of oxygen is low, NiO dissociated into Ni^{+} and O_2^- . The reduction of bulk Y-TZP-NiO is often used during solid oxide fuel cell anode preparation to reduce Ni^{2+} ions and form Ni particles within the porous ceramic matrix. This process is achieved because of the oxygen ion transfer in zirconia polycrystals [16]. Thus, extra oxygen ions can migrate through the oxygen sub-lattice of zirconia lattice and can occupy oxygen-vacant positions. This process takes place in the air condition; similarly to in argon conditions, but the equilibrium points are different. When sintering in an argon environment on the sample surfaces, the oxygen partial pressure is significantly low and oxygen ions can migrate into the zirconia lattice to the sample surface.

For NiO formation from Ni and oxygen during cooling in an argon environment, the oxygen may be taken from the residual porosity in the sample core or from the oxygen sub-lattice of zirconia, which is a good ion conductor at such temperatures. Thus, the number of oxygen vacancies in Y^{3+} -depleted zirconia grains increased and were present in a sufficient quantity for tetragonal phase retention. Additionally, the reversible dissociation in the sample core during cooling is involved in solid state and transformation of Ni ($\rho = 8.9 \text{ g/cm}^3$) to NiO ($\rho = 6.67 - 7.45 \text{ g/cm}^3$), leading to the emergence of large compressive stresses. These compressive stresses prevented the transformation of Y^{3+} -depleted tetragonal grains in the monoclinic during cooling. The metastability degree of such tetragonal grains in the 3Y-TZP-NiO composite sample volume was increased, leading to an increased effect on the stress-induced phase transformation toughening for the K1C value.

The phase and chemical transformations in the 3Y-TZP-NiO sample shell layer sintered in an argon environment were more complex. In the sample shell, metal Ni grains were formed. This supports that a reversible dissociation reaction occurred at low oxygen partial pressure. The low oxygen partial pressure on the sample surface during sintering in an argon atmosphere shifted the balance of reversible NiO dissociation toward Ni formation. The number of oxygen vacancies in the zirconia lattice in sample shell layer was sufficient to stabilize the tetragonal phase in the Y^{3+} -depleted zirconia grains and form additional cubic phase (**Table 1**). The amount of cubic phase in sample shell varied from 21 to 35% for 5–20 wt% NiO in the 3Y-TZP composite compared with 20% in the sample core. The larger amount of cubic phase may have slightly decreased the fracture toughness value of surface layers in the 3Y-TZP-NiO samples, which were sintered in

argon atmosphere, because the cubic phase shows a lower fracture toughness value compared with in the tetragonal phase.

For verification of reasons of phase stability of tetragonal phase of 3Y-TZP-NiO composites sintered in argon the annealing in air atmosphere at 1350°C was done. It was found that the monoclinic phase did not form after annealing in air of matrix 3Y-TZP sample. The oxygen vacancies caused by Y³⁺ ions remained unchanged. In case of annealing of 3Y-TZP-NiO composites sintered in argon the huge amount of monoclinic phase was formed (40-45%). This unstable behaviour of tetragonal phase confirmed our assumption that the oxygen vacancies are not formed during Ni²⁺ ions stabilization of zirconia lattice. The part of cubic and tetragonal phases which transform to monoclinic during such annealing correspond to oxygen vacancies, which formed due to influence dissociation NiO in argon atmosphere. This procedure similar to the annealing of vacuum sintered ZrO₂ samples in air. The vacancies caused by low oxygen partial pressure (vacuum) disappear during annealing in air and monoclinic phase was formed. Therefore, in our case, the oxygen vacancies formed not only because of heterovalent dopants, but also because reversible dissociation NiO in the neutral atmosphere. This indicates the simultaneous influence of the sintering environment and dopants on chemical and phase composition of 3Y-TZP-NiO composites.

Conclusion

It was found that the crack propagation in 3Y-TZP ceramics greatly inhibited by creation of composite structure by addition of NiO particles and sintering at 1500°C in argon atmosphere. Prevention of formation of the monoclinic phase of zirconia was conditioned by sintering composite under argon. The Ni grains were formed in sample shell but NiO grains retained in sample core. Increasing of K1C value was found as in sample shell as in sample core of composite, in comparison with matrix 3Y-TZP ceramics. The increasing of K1C value of obtained intergranular type of composite structure can not be explained by crack deflection and crack bridging processes by Ni and NiO particles, respectively. The possible explanation of K1C value increasing is the activation of phase transformation toughening of zirconia by formation of metastable tetragonal phase depleted by Y³⁺ ions. The reversibly dissociation of NiO on Ni and oxygen in neutral atmosphere provided the formation of cubic phase and Y³⁺ depleted tetragonal phase. Internal oxidation of Ni during cooling leads to formation metastable tetragonal phase in depleted Y³⁺ zirconia grains. The formation of NiO particles during cooling lead to emergence of large compressive stresses, which also increased the metastability of tetragonal zirconia grains, depleted of Y³⁺ ions.

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