

## Y<sub>2</sub>O<sub>3</sub> and Nd<sub>2</sub>O<sub>3</sub> co-stabilized ZrO<sub>2</sub>-WC composites

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**Abstract** Y<sub>2</sub>O<sub>3</sub> + Nd<sub>2</sub>O<sub>3</sub> co-stabilized ZrO<sub>2</sub>-based composites with 40 vol% WC were fully densified by pulsed electric current sintering (PECS) at 1350 °C and 1450 °C. The influence of the PECS temperature and Nd<sub>2</sub>O<sub>3</sub> co-stabilizer content on the densification, hardness, fracture toughness and bending strength of the composites was investigated. The best combination of properties was obtained for a 1 mol% Y<sub>2</sub>O<sub>3</sub> and 0.75 mol% Nd<sub>2</sub>O<sub>3</sub> co-stabilized composite densified for 2 min at 1450 °C under a pressure of 62 MPa, resulting in a hardness of 15.5 ± 0.2 GPa, an excellent toughness of 9.6 ± 0.4 MPa.m<sup>0.5</sup> and an impressive 3-point bending strength of 2.04 ± 0.08 GPa. The hydrothermal stability of the 1 mol% Y<sub>2</sub>O<sub>3</sub> + 1 mol% Nd<sub>2</sub>O<sub>3</sub> co-stabilized ZrO<sub>2</sub>-WC (60/40) composites was compared with that of the equivalent 2 mol% Y<sub>2</sub>O<sub>3</sub> stabilized ceramic. The double stabilized composite did not degrade in 1.5 MPa steam at 200 °C after 4000 min, whereas the yttria stabilized composite degraded after less than 2000 min. Moreover, the (1Y,1Nd) ZrO<sub>2</sub>-WC composites have a substantially higher toughness (~9 MPa.m<sup>0.5</sup>) than their 2Y stabilized equivalents (~7 MPa.m<sup>0.5</sup>).

### Introduction

Yttria stabilized tetragonal ZrO<sub>2</sub> polycrystalline (Y-TZP) ceramics are favourable because of their high toughness [1]. Addition of about 40 vol% of electro-conductive carbide or nitride ceramic phases to a Y-TZP matrix allows to

further improve the hardness, toughness and strength and enables electrical discharge machining (EDM) as a cost-effective machining technique for producing complex ceramic parts [2, 3]. Amongst the investigated ZrO<sub>2</sub>-based composites with WC, TiN, TiCN, TiC, TiB<sub>2</sub> and NbC as secondary phase [4–7], ZrO<sub>2</sub>-WC [8–10] shows the best combination of hardness, strength and toughness. The reciprocating sliding wear resistance against WC-Co of EDM ZrO<sub>2</sub>-WC (60/40) composites is also reported to be higher than for ZrO<sub>2</sub>-based composites with 40 vol% TiN or TiCN [2]. Earlier reports on ZrO<sub>2</sub>-WC composites revealed that the optimum composition from a mechanical point of view as well as EDM behaviour contain 40 vol% of WC phase in combination with an Y<sub>2</sub>O<sub>3</sub> stabilizer content of 2 mol% [8–10].

The main drawback of ZrO<sub>2</sub> is the possible degradation in aqueous environments or ambient humidity at low temperature (RT to 400 °C) [11, 12], a phenomenon commonly known as low temperature degradation (LTD). This is expressed as a spontaneous transformation of the metastable tetragonal ZrO<sub>2</sub> phase into the monoclinic phase, accompanied with a volume expansion, microcrack formation and complete material degradation. Double yttria + neodymia stabilized ZrO<sub>2</sub> however has a higher hydrothermal stability than yttria stabilized ZrO<sub>2</sub> with the same overall amount of stabilizer [13].

In this work, the hydrothermal stability of 2 mol% yttria stabilized ZrO<sub>2</sub>-WC (60/40) is compared with that of 1 mol% yttria + 1 mol% neodymia co-stabilized composites. Furthermore, a range of Y<sub>2</sub>O<sub>3</sub> + Nd<sub>2</sub>O<sub>3</sub> co-stabilized ZrO<sub>2</sub>-WC (60/40) ceramic composites were processed using pulsed electric current sintering (PECS) at 1350 and 1450 °C. The Y<sub>2</sub>O<sub>3</sub> stabilizer content was fixed at 1 mol% while the Nd<sub>2</sub>O<sub>3</sub> stabilizer content was varied between 0.75 and 1.5 mol%. The Vickers hardness, fracture toughness,

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bending strength, electrical resistivity as well as the microstructural properties were investigated. The neodymia stabilizer content and PECS temperature were optimized to obtain the best combination of mechanical properties.

### Experimental procedure

Commercial ZrO<sub>2</sub> (TZ-0, Tosoh, Japan), mechanomade WC (grade J300, MBN, Italy) and Al<sub>2</sub>O<sub>3</sub> (SM8, Baikowski, France) starting powders were used for the composite preparation. As shown in Fig. 1a, the WC powder is highly agglomerated with an agglomerate size range of 0.1–10 μm, although the primary crystal size is claimed to be about 20 nm (Supplier information), whereas the ZrO<sub>2</sub> powder is nanometric with a particle size of about 50 nm (Fig. 1b). The Al<sub>2</sub>O<sub>3</sub> powder has an average grain size of 0.4 μm.

The neodymia and yttria were applied by a stabilizer coating technique on the ZrO<sub>2</sub> starting powder prior to mixing with the WC powder. For this purpose, the proper amount of neodymia (99.9%, Chempur, Germany) and yttria (99.9%, ACROS, Belgium) was dissolved in nitric acid (65%, Sigma-Aldrich, Germany) at 100 °C on a magnetic stirrer. The resulting nitrate solution was applied to the monoclinic zirconia powder via a suspension coating process [14]. The nitrate-coated powder was calcined for 1 h at 800 °C. The yttria stabilizer content was fixed at 1 mol% whereas the neodymia content was varied between 0.75 to 1.5 mol%. Al<sub>2</sub>O<sub>3</sub> (0.8 wt.%) was added as a ZrO<sub>2</sub> grain growth inhibitor and sintering aid. Moreover, the addition of 0.8 wt.% Al<sub>2</sub>O<sub>3</sub> was claimed to improve the hardness [8].

Fifty grams of ZrO<sub>2</sub>-WC with 40 vol% of WC was mixed on a multidirectional Turbula mixer (type T2A, Basel, Switzerland) in ethanol in a polyethylene container of 250 mL during 48 h at 60 rpm. To the container, 250 g of cemented carbide milling balls (Ø = 4–5 mm, Ceratizit grade MG15) were added to the container to break the agglomerates in the starting powder and to enhance powder mixing. The ethanol was removed after mixing using a

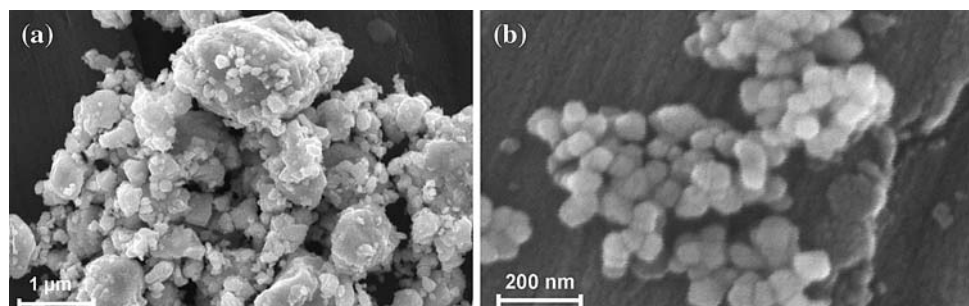
rotating evaporator. The dry powder mixture was sieved (315 mesh) to remove the agglomerates, prior to densification.

The powder was densified into 30 mm diameter discs by means of pulsed electric current sintering (Type HP D 25/1, FCT Systeme, Rauenstein, Germany) at 1350 or 1450 °C in a graphite tool set-up. Details on the equipment, die/punch set-up and temperature control are given elsewhere [15]. A heating rate of 400 °C/min was applied up to 1050 °C with a dwell time of 1 min and subsequent heating at 200 °C/min up to the sintering temperature. The initial pressure was increased from 7 to 30 MPa during the 1-min dwell time at 1050 °C and from 30 to 62 MPa during the first minute at the sintering temperature. The total dwell time at maximum temperature was 3 min.

Microstructural investigation was performed by scanning electron microscopy (SEM, XL-30FEG, FEI, Eindhoven, The Netherlands). X-ray diffraction (Seifert 3003 T/T, Ahrensburg, Germany) was used for phase identification and calculation of the relative monoclinic and tetragonal ZrO<sub>2</sub> phase content. The m-ZrO<sub>2</sub> volume fraction is calculated according to the method of Toraya et al. [16]. The transformability of a ceramic is defined as the difference in m-ZrO<sub>2</sub> content of a fractured and polished surface, whereas the transformability during hydrothermal aging is the % m-ZrO<sub>2</sub> difference between the steam-treated and pristine-polished surface. The average grain size of the phases in the densified composites was measured according to the linear intercept method. The reported values are the average of about 200 grains measured by means of Image Pro software on SEM micrographs. The electrical resistivity of the composites was measured according to the 4-point contact method using a Resistomat (TYP 2302 Burster, Gernsbach, Germany).

The density of the samples was measured in ethanol, according to the Archimedes method (BP210S balance, Sartorius AG, Germany). The Vickers hardness (Model FV-700, Future-Tech Corp., Tokyo, Japan), HV<sub>10</sub>, was measured with an indentation load of 98 N with a dwell time of 10 s. The indentation toughness, K<sub>IC</sub>, was calculated according to the formula of Anstis et al. [17] based on crack length measurements of the median crack pattern

**Fig. 1** Scanning electron micrograph of WC (a) and ZrO<sub>2</sub> (b) starting powders



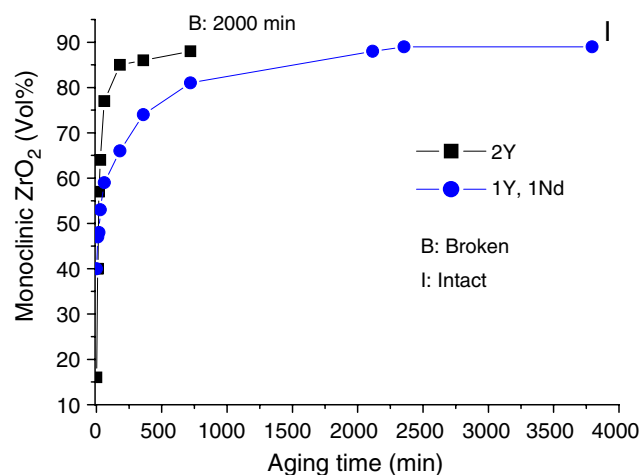
produced by Vickers  $HV_{10}$  indentations. The elastic modulus of the ceramic specimens was measured using the resonance frequency method [18], measured by the impulse excitation technique (Model Grindo-Sonic, J.W. Lemmens N V., Leuven, Belgium). The test was done on  $25.0 \times 5.4 \times 2$  mm samples. The reported values are the mean and standard deviation of 10 measurements. The sample surfaces for E-modulus and strength testing were ground with a diamond grinding wheel (type MD4075B55, Wendt Boart, Brussels, Belgium) on a Jung grinding machine (JF415DS, Göppingen, Germany).

Low temperature degradation (LTD) tests were performed at 200 °C under a saturated  $H_2O$  pressure of 1.55 MPa. For this purpose, rectangular bars ( $20 \times 5 \times 2$  mm) were polished and inserted in a stainless steel autoclave. The autoclave was inserted in a molten salt bath to establish an internal temperature of 200 °C as monitored by a thermocouple in the autoclave.

## Results and discussions

### Hydrothermal stability

The results of the hydrothermal stability testing, performed on 2 mol% yttria (2Y) and 1 mol% yttria + 1 mol% neodymia (1Y,1Nd) stabilized  $ZrO_2$ -WC (60/40) composites PECS at 1450 °C, are summarized in Fig. 2. The 2 mol% yttria stabilized composite completely degraded after 2000 min in 1.5 MPa saturated water vapour at 200 °C, while the 1Y-1Nd stabilized  $ZrO_2$ -WC composite sample remained intact even after 4000 min. In general, the mechanisms which increase the hydrothermal stability of  $ZrO_2$  ceramics, such as a reduction in  $ZrO_2$  grain size or an increased



**Fig. 2** Hydrothermal stability of 2 mol%  $Y_2O_3$  and 1 mol%  $Y_2O_3$  + 1 mol%  $Nd_2O_3$  stabilized  $ZrO_2$ -WC (60/40) composites

stabilizer content, etc. [1, 11], decrease the t- $ZrO_2$  phase transformability and concomitant fracture toughness. Beside the higher hydrothermal stability, the (1Y,1Nd) stabilized  $ZrO_2$ -WC composite also has a higher fracture toughness than the 2Y stabilized  $ZrO_2$ -WC (see Table 1). The reason for the superior hydrothermal stability of double stabilized yttria + neodymia  $ZrO_2$  over yttria stabilized  $ZrO_2$  was reported to be the larger lattice parameter of double stabilized  $ZrO_2$  as a result of the incorporation of  $Nd^{3+}$  ions and the absence of a thermally activated martensitic transformation above room temperature [13]. The same mechanism applies to the  $ZrO_2$ -based composite systems.

### Mechanical properties

The microstructure of a polished cross-section and fracture surface of the (1Y,1.25Nd) stabilized composite, PECS at 1450 °C, is shown in Fig. 3. The fracture surface shows that the  $ZrO_2$  grain size is in the of 100–150 nm range, while the WC particle size ranges from 20 nm up to 2  $\mu$ m. Three phases can be differentiated on the atomic number contrast image of the polished cross-section, i.e.  $ZrO_2$  (dark), WC (white) and  $Al_2O_3$  (black). Assuming a density of 6.0, 15.8 and 3.9  $g/cm^3$  for (1Y,1–1.5Nd) stabilized  $ZrO_2$  [13], WC and  $Al_2O_3$ , respectively, a theoretical density of 9.80  $g/cm^3$  is calculated for  $ZrO_2$ -WC (60/40) composites. The relative density of the ceramic grades is about 99% of the theoretical density and hardly changes with sintering temperature.

The strength of the ceramics PECS at 1450 °C is substantially higher than for those sintered at 1350 °C, although the density is comparable (see Table 1).  $ZrO_2$  and WC have a very good high temperature chemical stability below 1500 °C [19]. Hence, the  $ZrO_2$ -WC grain-boundaries should be comparable for the ceramics PECS at 1350 and 1450 °C. The higher PECS temperature however could result in a stronger  $ZrO_2$ -WC interphase strength explaining the higher strength of the composites PECS at 1450 °C. The bending strength of the 2 mol% yttria stabilized composite is comparable with that of the  $Nd_2O_3$  co-stabilized ceramic (see Table 1).

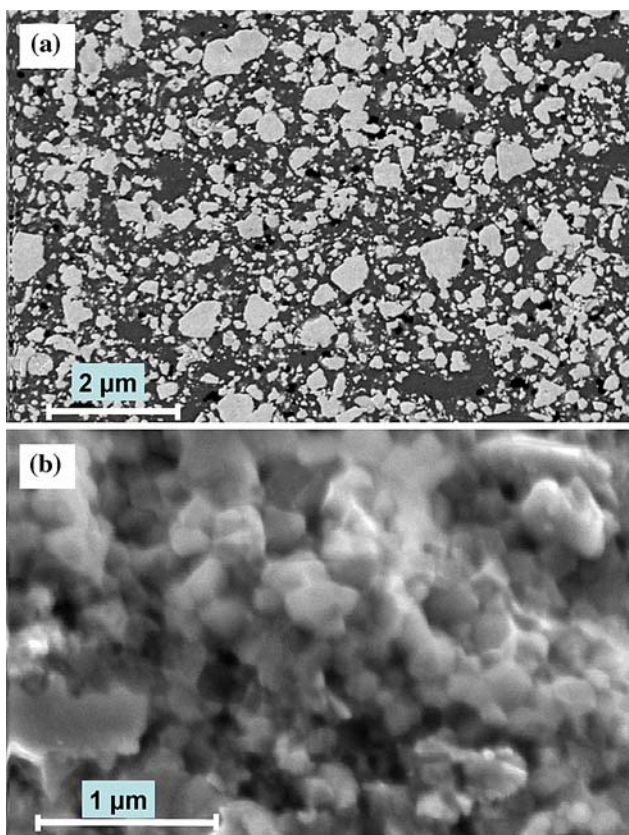
Only a minor amount of monoclinic  $ZrO_2$  (m- $ZrO_2$ ) could be detected by means of X-ray diffraction on the polished surfaces of the ceramic composites, as shown in Fig. 4. A small amount of m- $ZrO_2$  could be present in the bulk of the lowest stabilizer content grade, whereas the small amount of m- $ZrO_2$  on the 1 and 1.25 mol%  $Nd_2O_3$  co-stabilized grades could also have been generated during polishing, due to the higher transformability of the latter grades.

Different toughening mechanisms are active in  $ZrO_2$ -based composites [1, 10, 20–22]. The main toughening

**Table 1** Mechanical and electrical properties of 1 mol%  $Y_2O_3$  + 0.75–1.5 mol%  $Nd_2O_3$  and 2 mol% yttria stabilized  $ZrO_2$ -WC (60/40) composites, PECS at 1350 or 1450 °C

Temperature (°C)	$Nd_2O_3$ (mol%)	Transformability (%)	Density ( $g/cm^3$ )	E (GPa)	HV <sub>10</sub> (GPa)	$K_{IC-10}$ ( $MPa\ m^{0.5}$ )	Strength (MPa)	Resistivity ( $\Omega\ m$ )
1350	0.75	30	9.79	356	15.24 ± 0.19	9.2 ± 0.4	1513 ± 64	1.3E-05
	1	45	9.76	350	15.38 ± 0.29	10.1 ± 0.2	1486 ± 164	1.0E-05
	1.25	52	9.68	349	15.62 ± 0.17	10.0 ± 0.5	1561 ± 165	1.6E-05
	1.5	29	9.81	344	15.78 ± 0.16	9.0 ± 0.4	1497 ± 200	1.2E-05
1450	0.75	31	9.77	359	15.52 ± 0.23	9.6 ± 0.4	2045 ± 80	6.9E-06
	1	44	9.75	356	15.48 ± 0.26	9.7 ± 0.4	1808 ± 139	1.5E-05
	1.25	41	9.78	356	15.92 ± 0.23	9.9 ± 0.5	1849 ± 101	1.2E-05
	1.5	26	9.76	362	15.79 ± 0.27	9.1 ± 0.4	1863 ± 4	9.6E-06
	0 <sup>a</sup> [9]	44	9.78	372	16.20 ± 0.15	7.3 ± 0.1	1873 ± 90	–

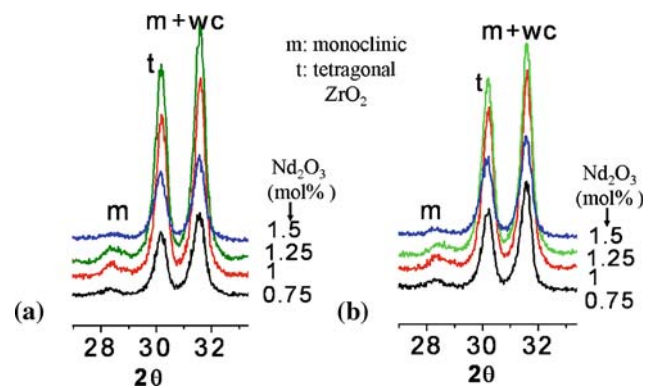
<sup>a</sup> 2 mol% yttria stabilized  $ZrO_2$ -WC (60/40)



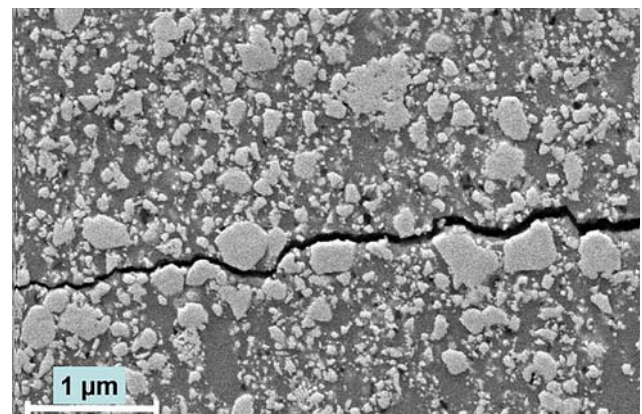
**Fig. 3** SEM micrograph of polished and fracture surface of (1Y,1.25Nd) stabilized  $ZrO_2$ -WC (60/40), PECS for 2 min at 1450 °C and 60 MPa.  $ZrO_2$  (dark), WC (white) and  $Al_2O_3$  (black)

mechanism is  $ZrO_2$  transformation toughening [4, 10], whereas crack deflection is clearly operational as shown in Fig. 5.

All  $ZrO_2$ -WC (60/40) composites stabilized with 1 mol%  $Y_2O_3$  + 0.75–1.5 mol%  $Nd_2O_3$ , have a toughness of 9–10  $MPa\cdot m^{0.5}$ , despite a substantial difference in  $ZrO_2$  phase transformability varying from 30 to 52% (Table 1).



**Fig. 4** XRD pattern of (1Y,1-1.5Nd) stabilized  $ZrO_2$ -WC (60/40) PECS at 1350 (a) and 1450 °C (b)



**Fig. 5** Crack path initiated from the corner of a Vickers indentation on a (1Y-0.75Nd) stabilized  $ZrO_2$ -WC (60/40) composite PECS at 1350 °C

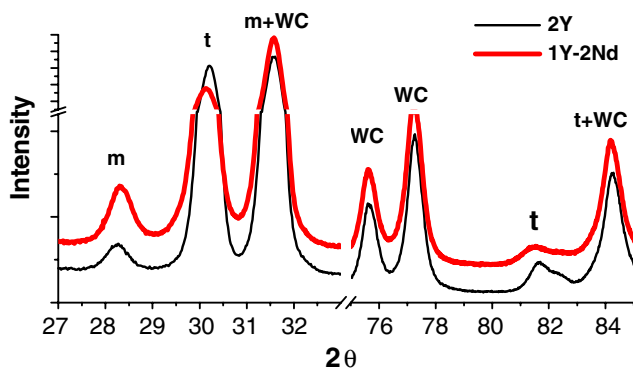
The toughness of the 1.5 mol%  $Nd_2O_3$  co-stabilized composite is statistically slightly lower, whereas the toughness of the other grades is the same, which could be attributed to the higher stabilizer content in agreement with a lower

phase transformability. Although the transformability of the 0.75 mol%  $\text{Nd}_2\text{O}_3$  co-stabilized composite is also lower and comparable to that of the 1.5 mol%  $\text{Nd}_2\text{O}_3$  grade, the toughness is as high as for the other grades. The loss in  $\text{ZrO}_2$  transformation toughening in the lower stabilizer content grade might be compensated by a higher tendency for the formation of microcracks resulting in crack tip shielding by microcracking. Although no clear microcracks could be observed by means of SEM, the lower stabilizer content  $\text{ZrO}_2$  grades should be more prone to microcracking.

The transformability is hardly influenced by the PECS temperature. That can be attributed to the high heating rate of 200 °C/min and short dwell time of 3 min, which does not allow to establish a substantial difference in  $\text{ZrO}_2$  grain size that would strongly influence the  $\text{ZrO}_2$  phase transformability. The comparable grain size is also reflected in a comparable hardness and stiffness.

The toughness and hydrothermal stability of the (1Y,1-1.5Nd) stabilized  $\text{ZrO}_2$ -WC (60/40) composites, PECS at 1450 °C is higher than for the 2 mol% yttria stabilized equivalent (see Table 1 and Fig. 2), whereas the strength and hardness are comparable.

The toughness of the 1 and 1.25 mol%  $\text{Nd}_2\text{O}_3$  co-stabilized composites is higher than for the 2 mol% yttria stabilized equivalent, although the  $\text{ZrO}_2$  phase transformability is comparable. Since the  $\text{Nd}^{3+}$  ionic radius (0.110 nm) is larger than for  $\text{Y}^{3+}$  (0.102 nm) [23, 24], the co-stabilized t- $\text{ZrO}_2$  phase has a larger lattice parameter [13]. This is confirmed by the peak shift of the t- $\text{ZrO}_2$  phase towards a lower  $2\theta$  angle at low and higher  $2\theta$  values, whereas the m- $\text{ZrO}_2$  and WC phase shows no peak shift, as shown in Fig. 6. At the same t- $\text{ZrO}_2$  phase transformability, the volume expansion resulting from the t- $\text{ZrO}_2$  phase transformation in double stabilized  $\text{ZrO}_2$  composites will therefore be larger than in the yttria stabilized equivalent. This results in a higher residual compressive stress on the propagating crack tip and concomitantly higher fracture toughness in double stabilized  $\text{ZrO}_2$  composites.



**Fig. 6** XRD patterns of 2Y and (1Y,2Nd) stabilized  $\text{ZrO}_2$ -WC (60/40) composites, PECS at 1450 °C. (m = m- $\text{ZrO}_2$ , t = t- $\text{ZrO}_2$ )

## Conclusions

$\text{ZrO}_2$ -based composites with 40 vol% WC and stabilized with 1 mol% yttria and 0.75–1.5 mol% neodymia were pulsed electric current sintered (PECS) to full density at 1350 and 1450 °C. The (1Y,0.75Nd) co-stabilized composite densified for 2 min at 1450 °C under a pressure of 62 MPa combines a hardness of  $15.5 \pm 0.2$  GPa, an excellent toughness of  $9.4 \pm 0.4$   $\text{MPa}\cdot\text{m}^{0.5}$  and impressive 3-point bending strength of  $2.04 \pm 0.08$  GPa.

A very high fracture toughness of about 9–10  $\text{MPa}\cdot\text{m}^{0.5}$  was measured for all the composites irrespective of the neodymia stabilizer and PECS temperature. The toughness was hardly influenced by the PECS temperature and a  $\text{Nd}_2\text{O}_3$  co-stabilizer content ranging from 0.75 to 1.25 mol%. The excellent bending strength of about ~1900 MPa for the double stabilized composites PECS at 1450 °C was substantially higher than for the grades PECS at 1350 °C (~1500 MPa).

The 1 mol% yttria + 1 mol% neodymia co-stabilized  $\text{ZrO}_2$ -WC (60/40) composites have a higher fracture toughness, hardness as well as hydrothermal stability in steam at 200 °C than the equivalent 2 mol% yttria stabilized composites, whereas the bending strength and stiffness is comparable.

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