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Electrical discharge machinable alumina zirconia niobium carbide composites – influence of stabilizer content

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Abstract

Manufacturing of customized components of hard and tough structural ceramics by conventional technologies suffers from high cost in the final hard machining step. Electric discharge machining can be a cost efficient alternative as the machining process is contact-free and thus material removal becomes independent on mechanical properties. To be ED-machinable, ceramics require a certain electrical conductivity, which can be achieved by addition of an electrically conductive phase. In this study materials containing a micron-size conductive dispersion of 24 vol.% of niobium carbide in a sub-micron-size matrix of zirconia toughened alumina containing 17 vol.% of zirconia of various yttria stabilizer contents were studied. Starting powders were alloyed by a mixing and milling process in 2-propanol and samples were produced by subsequent hot pressing at 1525°C for 2 h at 40 MPa axial pressure in a graphite die. Mechanical properties, microstructure and phase composition were studied. The ED-machinability in a wire cutting process was investigated with respect to machining speed, surface quality and ED-induced subsurface damage. It was found that yttria stabilizer content affects mechanical properties. A maximum fracture resistance was found at a stabilizer content of 1 mol.% while bending strength varied little with stabilizer content. ED-machinability was unaffected by stabilizer content.
Keywords: ZTA, ZrO_2, Al_2O_3, NbC, Electrical discharge machining, Mechanical properties

KOMPOZYTY TLENEK GLINU – TLENEK CYRKONU – WĘGLIK NIOBU DO OBRÓBKI ELEKTROEROZYJNEJ – WPŁYW ZAWARTOŚCI STABILIZATORA

Wysokie koszty finalnej obróbki skrawaniem negatywnie oddziałują na wytwarzanie na zamówienie komponentów z twardej i wytrzymałej ceramiki konstrukcyjnej za pomocą tradycyjnych technologii. Obróbka elektroerozyjna może stanowić kosztowo efektywną alternatywę, ponieważ proces obróbki jest bezkontaktowy i dlatego usuwanie materiału staje się niezależne od właściwości mechanicznych. Aby być obrabialną elektroerozyjnie, ceramika wymaga pewnej przewodności elektrycznej, którą można osiągnąć poprzez dodanie fazy przewodzącej elektrycznie. W niniejszej pracy badano materiały zawierające przewodzącą dyspersję węglika niobu w ilości 24% obj. w submikronowej osnowie tlenku glinu wzmacnianego tlenkiem cyrkonu, która zawierała 17% obj. tego tlenku z różną zawartością stabilizatora. Wyjściowe proszki homogenizowano drogą mieszania i mielenia w 2-propanolu, a próbki wytwarzano poprzez następcze prasowanie na gorąco w 1525°C przez 2 h pod jednoosiowym ciśnieniem 40 MPa w formie grafitowej. Zbadano właściwości mechaniczne, mikrostrukturę i skład fazowy. Obrabialność elektroerozyjną w procesie cięcia włóknem badano w odniesieniu do szybkości obróbki, jakości uzyskanej powierzchni i podpowierzchniowego uszkadzania, wywołanego obróbką elektroerozyjną. Stwierdzono, że zawartość stabilizatora w postaci tlenku itru oddziałuje na właściwości mechaniczne. Maksymaną odporność na pękanie stwierdzono przy udziale stabilizatora wynoszącym 1% mol., podczas gdy wytrzymałość na zginanie zmieniała się nieznacznie wraz z zawartością stabilizatora. Obrabialność elektroerozyjna nie zależała od zawartości stabilizatora.
Słowa kluczowe: ZTA, ZrO_2, Al_2O_3, NbC, obróbka elektroektroerozyjna, właściwości mechaniczne

1. Introduction

Compared to plain alumina, zirconia toughened alumina ceramics (ZTA) offer better strength and fracture resistance at a moderate tradeoff in hardness [1]. The improvement of mechanical properties is attributed to different reinforcement mechanisms such as transformation toughening, microcracking and residual stress fields. While the basic mechanisms are well known for decades their exploitation by a knowledge based approach is difficult as this would require full control over the size and distribution of the zirconia phase in the alumina, a proper choice of stabilizer type and content and a correct adjustment of sintering parameters. Recent studies of ZTA materials containing 10–24 vol.% of 1–2 mol% yttria sintered at 1475°C for 1–3 h have shown that optimum parameter combinations lead to materials of considerable strength (1200 MPa) and fracture resistance (8.5 MPa√m) but they have also revealed that slight deviations from the optimum parameter set lead to a leveling of strength and toughness at 800 MPa / 4 MPa√m [2, 3]. It was shown that transformation toughening is not the main effect governing fracture resistance in these materials. A proper setup of the state of residual stress and possibly transformation-induced microcracking during fracture might be more relevant.

In case of ED-machinable ZTA with a conductive TiC dispersion developed for engineering applications the variation of stabilizer content has much lower effect. Fracture resistance values are commonly much lower than in case of plain ZTA, ranging from 4–5 MPa√m with a tendency to rise with decreasing stabilizer content in the range between 2 mol.% to 0.8 mol.% [4]. This may partly be caused by the low fracture resistance of the TiC dispersion (K_{IC} = 2 MPa√m). ZTA-WC composites containing the tougher tungsten carbide (K_{IC} = 4.4 MPa√m [5] led to much better mechanical properties, however ZTA-WC composites compared to ZTA-TiC had inferior ED-machinability [4, 5]. Niobium carbide having a fracture resistance of 3.5 MPa√m, a cubic lattice structure like TiC and a higher CTE ($7.2 \cdot 10^{-6}$ K^{-1}) than WC ($5.8 \cdot 10^{-6}$ K^{-1}) may be an interesting candidate for a suitable electrically conductive dispersion in ZTA with the aim to maintain the good machinability of ZTA-TiC and improve the moderate fracture resistance.

Literature on composites of oxide ceramics and NbC is relatively rare, some quotes exist on alumina-NbC used for cutting tools [6–9] and Y-TZP-NbC with NbC content of 0–40 wt.% [10, 11]. Apart from a recent study by the authors [12] the system ZTA-NbC is yet nearly unexplored.

2. Experimental Procedure

The powders used for this study are α-alumina (APA0.5, d_{50} = 300 nm, S_{BET} = 8 m²/g, Ceralox, USA), unstabilized zirconia (TZ-0, S_{BET} = 15 m²/g, crystallite size 25 nm, Tosoh, Japan), niobium carbide (d_{50} = 0.8–1.2 µm, ABCR, Germany) and yttria (purity 99.9%, Sigma Aldrich, USA). Batches of 200 g of powder blends were mixed and milled in 250 ml 2-propanol in an attrition mill at 400 rpm for 2 h using Y-TZP balls of 2 mm diameter. Blends contained 24 vol.% of NbC and 17 vol.% of zirconia of various stabilizer contents. The stabilizer contents in the zirconia added were adjusted between 0.75 mol.% and 2 mol.% in 0.25 mol.% increments by addition of yttria (the weight of yttria was counted thus as integral part of the stabilized zirconia). After separation of the milling media, the resulting slurry was dried at 45 °C overnight and screened through a 100 µm mesh to crush agglomerates and separate debris from the polymer milling container. The as-screened material was then ready for hot pressing.

Hot pressing (FCT Anlagenbau, Germany) was carried out in a boron nitride clad graphite die of 45 mm diameter at 1525 °C for 2 h dwell at 40 MPa axial pressure in vacuum, a heating rate was 50 K/min. Two disks of 22 g and one disk of 50 g weight separated by graphite disks were pressed simultaneously to obtain samples for mechanical characterization and ED-machining tests. After completion of the pressing cycle the press was filled with argon and samples were cooled in the hot press with the heater switched off.

The samples were subsequently lapped with 15 µm diamond suspension and polished with 15 µm, 6 µm and 1 µm diamond suspension for 30 min each to achieve a mirror-like finish (Struers Rotopol, Denmark). Density using Archimedes principle, Young's modulus and Poisson's ratio by the acoustic method, Vickers hardness HV10 and indentation toughness by direct crack length measurement were measured on as polished disks. For bending tests and fracture resistance according to the residual strength method (ISB) the two thinner disks were cut into bars of 4 mm width using a diamond wheel (Struers Accutom 50, Denmark). The sides of the bending bars were lapped and polished with 15 µm diamond suspension. The edges were carefully chamfered using a 20 µm diamond disk to remove any cutting induced defects. Bending strength was measured on a minimum of 10 samples in a 4-pt setup with 20/10 mm outer/inner span at a crosshead speed of 0.5 mm/min. For the ISB test, 4 samples were notched by placing a HV10 indent at a distance of 12 mm from the end of the bars with the cracks parallel and perpendicular to the sides. The residual strength was measured immediately after notching with the indent on the tensile side placed within the inner span of the same 4-pt setup at a crosshead speed of 2.5 mm/min to avoid subcritical crack growth. The phase composition of the samples was measured by XRD (Bruker D8, France, CuKα, graphite monochromator) on the surface of polished disks and on the fracture faces of broken bars from the ISB-test. Intensities of the monoclinic (-111) and (111) and the tetragonal (101) reflex in the 2theta range between 27–33° were integrated. The monoclinic contents calculated according to the calibration curve of Toraya [13].

The microstructure of the samples was investigated by SEM (Zeiss Gemini, Germany, secondary electrons, in-lens-technology) on samples which were thermally etched in hydrogen at 1280 °C for 5 min prior to measurement to reveal the grain boundaries.

Table 1. Machining parameters.

Machining Step	Average discharge current I_{avg} [A]	Average discharge voltage U_{avg} [V]	Average discharge duration t_e [µs]
MC	22.2	46.2	1.08
1st trim	21.5	54.0	1.05
2nd trim	8.4	83.4	0.24

Table 2. Relative density of the hot pressed ceramics.

Stabilizer content [mol.%]	0.75	1.0	1.25	1.5	1.75	2.0
Relative density [%]	99.8	99.8	99.6	100	100	100

Electric conductivities of the samples were determined by a 4-pt measurement using bending bars of ~40 mm length, 2 mm thickness and 4 mm width.

Basic EDM tests by wire cutting (Charmills CUT 1000 Oel Tech) were carried out using zinc coated brass wire with a diameter of 0.1 mm [AC-Cat A 900–0.1] and an oil based dielectric fluid (Oelheld, IonoPlus IME-MH). The parameters listed in Table 1 were used for the machining of the ceramics.

The machining results were evaluated by measurements of the surface roughness by white light interferometry (Bruker, Contour GT, USA). The sub-surface structure of ED-machined material was studied by optical microscopy of polished cross sections.

3. Results and Discussion

3.1. Mechanical and electrical properties

All samples showed relative densities higher than 99.8% of theoretical densities. Hot pressing parameters (time and temperature) were appropriately chosen to fully densify the materials. The calculation of the theoretical densities was done taking into account the real contents of monoclinic and tetragonal phase determined in XRD measurements shown in Fig. 2. Up to 1.25 mol.% yttria content, relative densities are slightly below the calculated theoretical densities of the ceramics. At yttria contents higher than 1.25 mol.%, fully dense ceramics were obtained.

In as fired condition, none of the samples showed structural damage, which is a hint that the chosen range of stabilizer content was appropriate.

The Young´s modulus of the sintered ceramics was in the range of 366–374 GPa, which is in line with the rule of mixture and comparable to prior investigations [12]. Considering a measurement accuracy of ± 3 GPa, a clear correlation of stabilizer content and Young´s modulus could not be identified.

Stabilizer content effects both hardness and toughness of the ceramic. Hardness rises with increasing stabilizer content from 1700 HV10 up to 1900 HV10. The maximum hardness was observed at a stabilizer content of 1.5 mol.% yttria. Hardness subsequently remains on this high plateau (Fig. 1). Beside hardness, fracture resistance of the material is influenced by stabilizer content. The fracture resistance, according to ISB measurements, is in the rage of 4.42–4.95 MPa√m (Fig. 1). A maximum of the fracture resistance was observed at 1 mol.% yttria content. Higher stabilizer contents than 1.25 mol.% curtail the fracture resistance of the ceramics.

Excessive stabilization reduces the transformability of the zirconia reinforcement and thereby leads to a decline in fracture resistance. This fact is confirmed by XRD measurements. The maximum value of transformability (24.5%) is reached at 1.0 mol.% stabilizer content. Ceramics with lower stabilizer content are already monoclinic to a large extent in the as fired state. Further increase of stabilizer concentration leads to steep decline in transformability to an asymptotic limit value of ~ 5 vol.% transformability reached at a stabilizer concentration as low as 1.5 mol.% (Fig. 2).

Contrary to hardness and toughness, the 4-pt bending strength and electrical conductivity remain almost unaffected by the stabilizer content. For all investigated stabilizer contents bending strength stays on a high level (> 700 MPa). An intermediate maximum is observed at 1.25 mol.% yttria (774 ± 82 MPa). The lowest bending strength (710 ± 103 MPa) was observed at a stabilizer content of 2 mol.%. The slight trend to increasing strength with lower stabilizer contents is however within the range of the observed standard deviations.

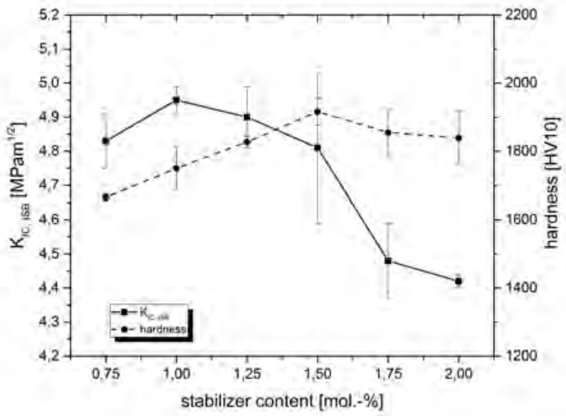


Fig. 1. Toughness and hardness as a function of stabilizer content.

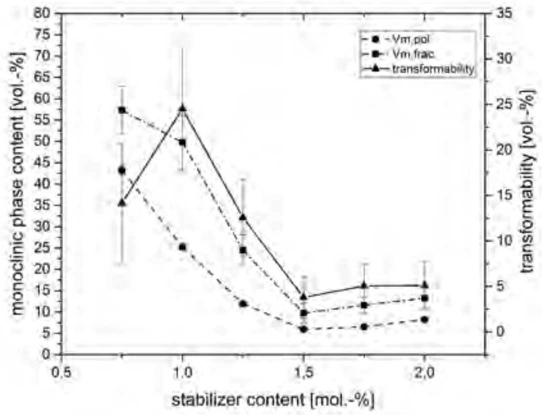


Fig. 2. Transformability and monoclinic phase content of the investigated ceramics.

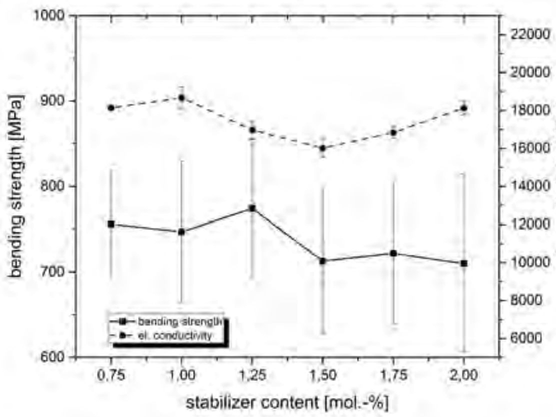


Fig. 3. 4-pt bending strength and el. conductivity as a function of stabilizer content.

Electrical conductivity is in the range of 16000–18600 S/m (Fig. 3). A direct dependence of stabilizer content and electrical conductivity was not observed

3.2. Microstructure

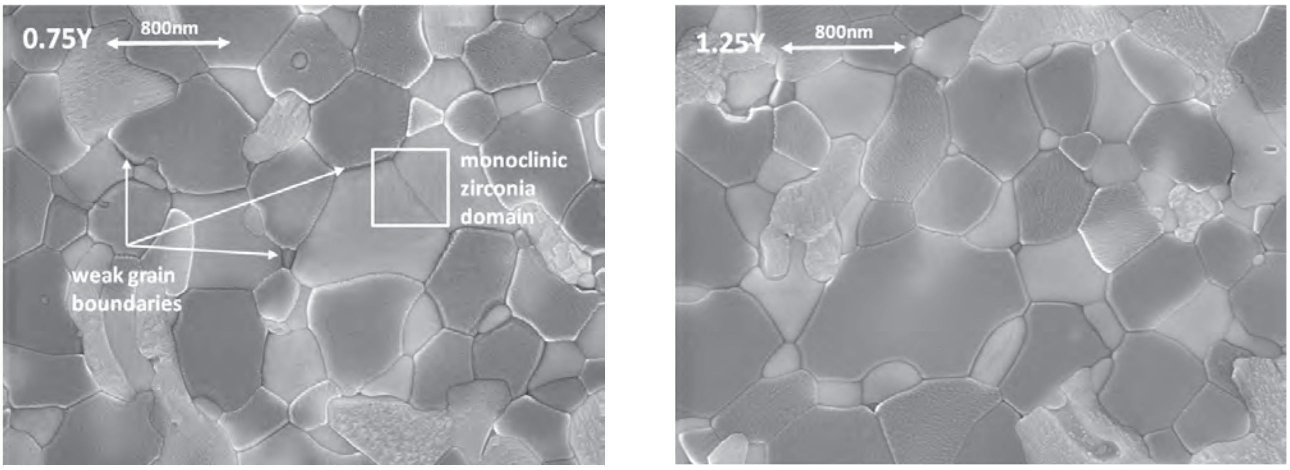
SEM images show homogeneous dispersion of the three phases and sub micron sized grains for all constituents (Fig. 4). Grey grains are alumina, zirconia appears as light grey grains. The electrical conductive phase (NbC) can be easily identified due to "crystal growth planes" with cubic structure resulting from thermal etching. NbC grains show irregular morphology, often consisting of aggregates of smaller crystallites, compared to the starting powder no visible grain growth is observed. The zirconia dispersion appears as grains well below 1 µm with an average grain size of ~500 nm, which is beneficial for the retention of the tetragonal phase after cooling. The grains of the alumina matrix are regular shaped, grain sizes are typically < 1 µm.

As indicated by the XRD results ceramics with < 1.25 mol.% yttria contain noteworthy fractions of monoclinic zirconia (Fig. 2). SEM studies confirm this result. As indicated by square (Fig. 4a) the material with 0.75 mol.% yttria shows the characteristic twin like dovetail structures of monoclinic domains in larger zirconia grains, moreover this partial retransformation leads to weakened grain boundaries in the adjacent grains indicated by arrows. In composites with 1.5 mol.% yttria (Fig. 4b) such features cannot be detected.

3.3. Electrical discharge machining

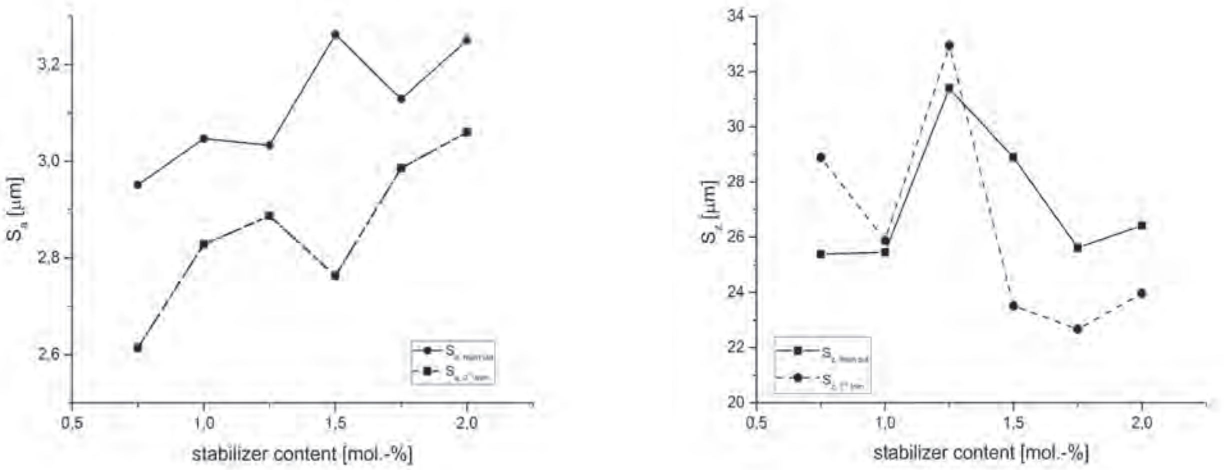
EDM basis tests showed that machining of all composites was possible, but accompanied by several wire breaks. Number of wire breaks was not influenced by the stabilizer content. Higher stabilizer contents lead, with respect to the mean roughness index S_a, to lower surface quality. The surface roughness of the main cut and the second trimming operation rise continuously with increasing stabilizer content (Fig. 5).

Contrary to the mean roughness index, the average depth of roughness shows no direct dependence on the stabilizer content. As shown in Fig. 5 the average depth



a) b)

Fig. 4. Weak grain boundaries and monoclinic zirconia domains due to understabilization of 0.75 Y stabilized ZTA-NbC (a) and 1.25 Y stabilized ZTA-NbC (b).



a) b)

Fig. 5. Mean roughness index S_a (a) and average depth of roughness S_z (b) of the machined surfaces.

of roughness has an intermediate maximum at 1.25 mol.% yttria. Up to this point the subsequent trimming operations do not lead to an improvement of the average depth of roughness. At higher stabilizer contents than 1.25 mol.% yttria the surface quality can be improved by subsequent trimming operations.

SEM graphs prove that the subsequently fulfilled trimming operations help to improve the surface quality. As shown in Fig. 6 a decrease of the roughness and a smoothing of the surface is observed. Pictures of polished cross sections confirm this observation (Fig. 7). Two main effects can be identified: On the one hand the ratio of main roughness of the main cut to the 2nd trim decreases with increasing stabilizer content and on the other hand the mean roughness of the samples rises with increasing stabilizer content. SEM pictures of the machined surfaces show also a higher number of gas inclusions in the surface layer with increasing stabilizer content. It seems that increasing stabilizer contents lead to preferred evaporation of one phase (probably alumina). The surface structure is sponge like and shows a thin glassy layer on the top. A deep influenced zone ("white layer"), as known from the machining of cemented carbides, was not observed. The zone influenced by the machining process has a maximum thickness of around 20 µm. ED machined surfaces showed no percolating crack network; only a few cracks can be observed. The number and size of the cracks are unaffected by the stabilizer content.

As shown in Fig. 7 cracks perpendicular to the surface layer, which would impair the mechanical properties, were not detected.

4. Discussion

The mechanical characterization of the ceramics showed that a stabilization of the zirconia phase by a minimum of 1 mol.% is necessary. This assumption is supported by SEM micrographs and XRD measurements. XRD measurements showed that the monoclinic phase content in as fired samples increases nearly exponentially with reduced stabilizer contents. In the 0.75 mol.% stabilized zirconia dispersion ~ 45% zirconia is already monoclinic prior to fracture so that the material can be considered insufficiently stabilized. SEM images showed weak grain boundaries and monoclinic zirconia domains for this composition. According to thermodynamics [14] the zirconia is fully tetragonal at sintering temperature; these features developed upon retransformation during cooling are responsible for the missing density increment of ~0.2% at low stabilizer contents. Materials showing no microcracks are fully dense. The increase of stabilizer content above 1.5 mol.% however not only reduces the monoclinic content in as fired materials but completely almost eliminates contribution of transformation toughening to total fracture resistance.

The S-shaped decline of toughness between 1–2 mol.% yttria content is probably caused by a superposition of transformation toughness, residual stress and microcracking. At 0.75 mol.% transformation toughening is low, the high monoclinic content leads to tensile stress in the matrix and the toughness is predominantly determined by microcracking. At 1 mol.% monoclinic content in as fired state is ~25% so that the matrix is almost stress neutral [15, 16]. Still a few microcracks exist and transformation toughening

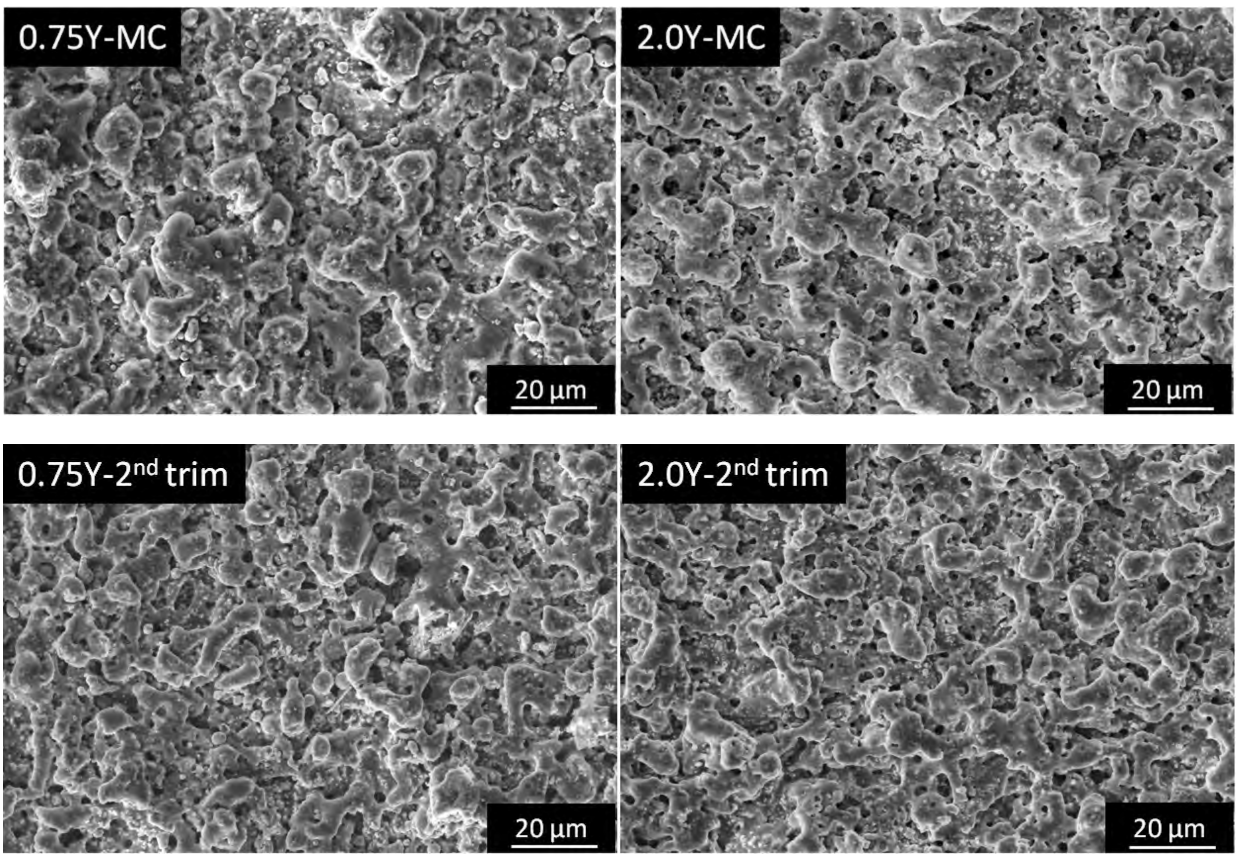


Fig. 6. SEM graphs of the machined surfaces (top: main cut 0.75 and 2.0 mol.% yttria; below: 2nd trim 0.75 and 2.0 mol.% yttria).

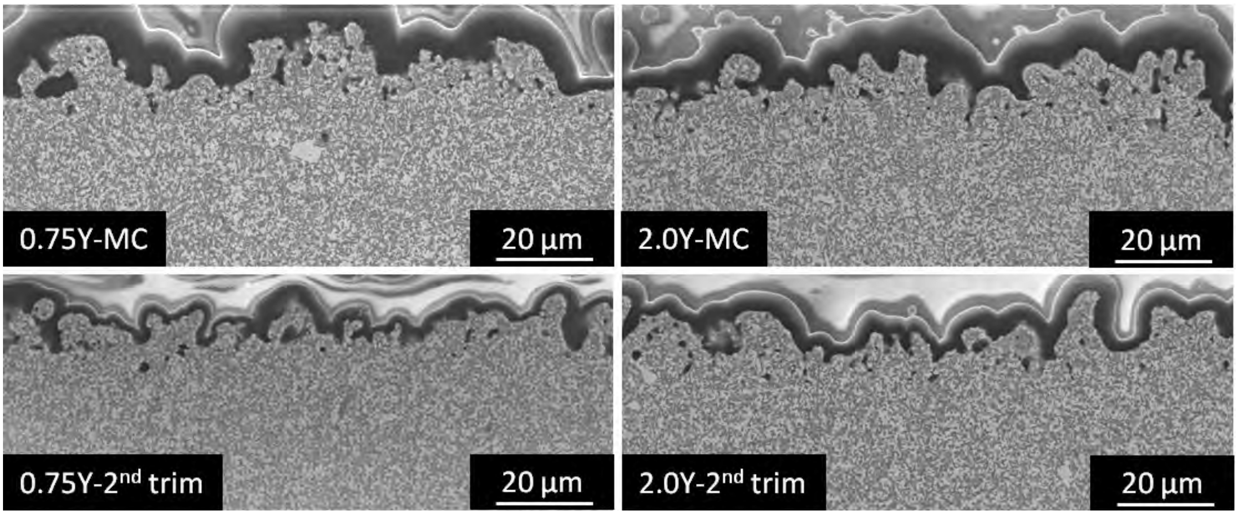


Fig. 7. Polished cross sections of ED machined samples (above: main cut of 0.75 Y and main cut of 2.0 Y stabilized ZTA-NbC; below: 2nd trim of 0.75 Y and main cut of 2.0 Y stabilized ZTA-NbC)

reaches its optimum. At higher yttria contents microcracks are absent; transformation toughening progressively decreases but the matrix is now under compressive stress which provides some additional toughness. In all materials NbC grains having the highest Young's modulus of all constituents may provide a minor amount of additional toughness by crack deflection and bridging, this increment is probably not very high as the grain size of the NbC dispersion is low. Compared to plain ZTA [2, 3, 16] the stabilizer content variation in the ZTA-NbC composites has a lower effect on strength and toughness. This is probably due to the mutual grain growth inhibition of the phases in the ternary composite and to the brittleness of the NbC phase.

According to the classical linear elastic fracture mechanics of Griffith [17] and Irwin [18] we may assume a linear correlation between strength and toughness – provided that the flaw size is constant. This seems to be the case from 1.25 mol.% yttria upwards. At lower yttria contents than 1.25 mol.% strength declines which can be correlated to the rising amount of microcracks which do not only provide toughness but are also structural defects. The absolute change in strength is however almost negligible considering the standard deviation. Due to the constant content and particle size of the three components zirconia, alumina and NbC the electrical conductivity is unaffected. According to percolation theory [19–21], this was expected.

Electrical discharge machining of all the material compositions was possible. However frequent of wire breaks were observed which disrupt the process and lower its performance. Obviously the content of conductive phase, 24 vol.% NbC, is not sufficient to maintain stable machining conditions. In a recent publication [12] it is shown that a stable ED-machining of ZTA-NbC is possible by using a minimum of 28 vol.% NbC as electrical conductive phase. The variation of the stabilizer content has only little influence on the ED machinability in general. Measurements of the surface roughness by white light interferometry showed a strong influence of the stabilizer content on the main roughness index. A closer look at SEM images of the machined surfaces cannot finally prove these measurements. Polished cross sections of the machined specimens show that stabilizer content does not influence the surface quality that strong as it appears from the white light interferometry. Surface quality, can be improved by subsequent trimming operations due to the partially removal of the influenced surface layer and the creation of a new surface layer. This new surface layer is smoothened due to the milder processing conditions of the trimming cuts.

5. Summary and conclusion

In the present study the influence of the stabilizer content on the mechanical properties and the ED-machinability of ZTA-NbC was investigated. It was found that stabilizer content affects fracture toughness, hardness and to certain extent 4pt-bendig strength. Young's modulus and el. conductivity are not influenced by the variation of stabilizer content. Bending strength is on a high lever (> 710 MPa) and fracture resistance (K_{IC} = 4.42 – 4.95 MPa√m) is also on an attractive level for ZTA ceramics with a dispersion of a transition metal carbide. Compared to the ZTA-TiC material system (K_{IC} = 4.22 MPa√m) [22] the fracture toughness can be improved by ~17%. The negligible influence of the stabilizer content on material properties such as el. conductivity and Young´s modulus is good accord with the percolation theory. Wire electrical discharge machining of all the compositions was possible. But, as the wire breaks show, the volume fraction of electrical conductive NbC is not sufficient for the ED machining process. Results concerning machining quality are somewhat contradictory. The mean roughness index decreases, while the average depth of roughness is reduced by increasing the stabilizer content of the material composition.

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