Mechanical Properties and Ageing Resistance of Slip Cast and Pressurelessly sintered ZTA - the Influence of Composition and Heat Treatment Conditions

Nicole Yildirim¹, Frank Kern¹*)

¹Institut fuer Fertigungstechnologie keramischer Bauteile IFKB, Universität Stuttgart, D-70569 Stuttgart, Germany

Abstract:
Zirconia toughened alumina (ZTA) ceramics are used in a broad range of mechanical engineering and biomedical applications due to their excellent mechanical and tribological properties. In this study it was tried how the change in stabilizer composition and sintering conditions can be exploited to selectively modify the mechanical properties and low temperature degradation resistance of ZTA materials with a typical biomedical grade composition. It was found that the materials react very sensitively to such changes. Fully dense materials combining favorable mechanical properties such as high strength > 800 MPa, fracture toughness > 5.5 MPa√m and LTD resistance can only be produced in a narrow stabilizer concentration range between 1.1-1.2 %. Beyond this range materials are either insufficiently stabilized which results in poor strength and high tendency to LTD, or over stabilized and no longer susceptible to stress induced phase transformation and therefore too brittle. Changes in reinforcement mechanisms can be attributed to changes in microstructure and phase composition caused by variation of stabilizer content and sintering conditions.

Keywords: Zirconia toughened alumina; Microstructure; Mechanical properties; Low temperature degradation.

1. Introduction

Zirconia toughened alumina (ZTA) ceramics combine high hardness and abrasion resistance of alumina with enhanced strength and toughness introduced by the zirconia dispersion [1, 2]. ZTA materials are therefore frequently applied in mechanical engineering applications such as cutting tools for grey cast iron or biomedical applications such as total hip (THR) or total knee replacement (THR) [3, 4]. The three relevant toughening mechanisms in ZTA ceramics are microcracking, residual stress, and transformation toughening. Each effect as such is well described in literature [5-7].

Occurrence of transformation induced microcracking depends on the size, shape and location of zirconia grains. Heuer showed that non-stabilized intergranular zirconia grains which are tetragonal at sintering temperature tend to retransform spontaneously to monoclinic during cooling above a threshold size of ~ 0.4 μm [8]. This size can be shifted to higher values by addition of yttria as a stabilizer for zirconia. Isolated grains tend to be more stable.

*) Corresponding author: frank.kern@ifkb.uni-stuttgart.de (Prof. Dr. Frank Kern)
than connected zirconia grains. Intragranular grains are less prone to transform than intergranular ones [9].

Under the influence of applied tensile stress metastable tetragonal grains in the wake of a proceeding crack may transform to stable monoclinic phase associated with volume expansion and shear [10]. The compressive stress exerted by volume expansion sets the crack under compression and retards or stops crack propagation. Residual stress is created by thermal mismatch of alumina (lower CTE) and zirconia (higher CTE). Assuming the material is stress-free at sintering conditions the low CTE phase (the alumina matrix) is set under hydrostatic compression, the dispersion (zirconia) is set under hydrostatic tension during cooling. The CTE-based stress can be offset by the stress derived from volume change due to t-m transformation of zirconia [11].

In “real” ZTA materials, depending on composition (fraction of zirconia, stabilizer concentration and starting powder grain sizes) processing and sintering conditions a combination of these reinforcement effects can be observed [2].

The compositional range and requirements for biomedical grade ZTA are listed in the standard ISO 6474-2 [12]. In biomedical applications low temperature degradation (LTD) has to be considered. Exposed to polar body fluids the stabilizer in yttria stabilized zirconia is partially hydrolyzed resulting in spontaneous transformation and volume expansion. Starting from the surface transformed grains put the surrounding grain boundaries under stress and result in a transformed layer proceeding into the bulk of the material, a roughening of the surface and eventually even component failure [13]. The concept in ZTA materials for biomedical applications is to “dilute” with alumina until a concentration is reached where the individual zirconia grains are no longer in contact [14]. Depending on the quality of dispersion and the size of zirconia and alumina grains this percolation threshold is somewhere between 15-17 vol.%. Further dilution would be safer concerning LTD resistance, concerning the mechanical properties this would be unfavorable as the transformation toughening effect and the mechanical strength and toughness scale with the amount of zirconia [9, 14]. For this reason the standard biomedical ZTA material typically contain zirconia fractions of 13-17 vol.% [15]. In this study ZTA materials with 17 vol.% zirconia were prepared from high quality submicron size starting powders, the sintering conditions and the stabilizer fraction of the zirconia added were varied to obtain a systematic overview on the influence of these parameters on mechanical properties and LTD resistance. The chosen manufacturing route by mixing and milling of powders dispersed in water, shaping by subsequent slip casting and firing in air comes much closer to the industrial production cycle than the previous studies where hot pressing technology associated with reducing conditions was applied [16-18].

2. Materials and Experimental Procedures

The starting powders for the study were submicron size alumina (APA0.5, D50 = 300 nm, S_BET = 8 m²/g, Ceralox, USA) and nanoscale unstabilized zirconia (TZ0, S_BET = 17 m²/g, Tosoh, Japan. The zirconia consists of aggregated primary crystallites of 35-50 nm size [17]. The stabilization of the zirconia in the range between 0.9-1.4 mol% Y2O3 was achieved by addition of yttria (99.9 %, Chempur, Germany). The surface chemistry of the alumina and zirconia powders was tested by zetapotential measurements (Malvern, Germany). The solid content was adjusted to 66 wt% according to viscosity measurements (Anton Paar, Germany). A commercial ammonium citrate based surfactant (CE64, Z&S, Germany, 1 ml/100 g powder) was added, the final pH value of the dispersions was 8.5. Powder batches of 300 g containing 17 vol.% stabilized zirconia in the powder fraction were milled in 200 ml with 600 g Y-TZP milling balls (d= 2 mm) at 400 rpm for 2 h. The dispersion quality was measured by laser granulometry (Malvern, Germany). Acrylate based commercial binder (0.5 g/100 g powder, AC95, Z&S Germany) and 1 drop of defoaming agent (Contraspum,
Z&S, Germany) were added 10 min before the end of the milling process to ensure good homogenization and release of the entrapped air.

Five different batches with stabilizer contents of 0.9 mol%, 1 mol%, 1.1 mol%, 1.2 mol% and 1.4 mol% were prepared. The nomenclature for the samples in the text is e.g. 17ZTA1Y-1550 for ZTA containing 17 vol% zirconia stabilized with mol% yttria sintered at 1550 °C. After separation of the milling media and passing the dispersion through a 50 µm screen to remove milling debris the dispersion was de-aired by stirring for 4 h at reduced pressure (200 mbar). Samples of 45x35 mm area were subsequently cast on a pre-dried plaster plate using silicone molds as lateral boundaries. After green body formation the samples were removed from the plate and carefully dried over several days to avoid cracking. Debindering was carried out in air (60 K/h to 800 °C, 1 h dwell). The samples were sintered in air at temperatures between 1500-1550 °C for 3 h (Thermconcept, Gernay, heating rate 2 K/min, free cooling). Sintered samples show a linear shrinkage of ~ 20%. They were manually pre-ground then glued on sample holders and subsequently automatically lapped and polished with diamond suspension (15, 6, 3, and 1 µm) until a mirror-like surface was achieved (Struers Rotopol, Denmark). Density ρ (Archimede’s principle) Vickers hardness HV10 (Bareiss, Germany) and Young’s modulus E (impulse excitation, IMCE, Belgium) were determined on entire plates. The plates were subsequently cut into bending bars of ~ 28 x 2 x 4 mm³ (L x H x W). The sides of the bending bars were machined by lapping to remove cutting defects, the edges were beveled by grinding with a diamond disk (20 µm). Four point bending strength σ_{4pt} and fracture resistance K_{ISB} by indentation strength in bending (ISB) were tested in a four point setup with 20 mm outer and 10 mm inner span. The crosshead speed was 0.5 mm/min for measurement of σ_{4pt} and 2.5 mm/min for K_{ISB}. Samples for the ISB-test were notched with a HV10 indent in the middle of the tensile side with the cracks parallel and perpendicular to the sides and tested immediately. The fracture resistance K_{ISB} was calculated from the residual strength according to Chantikul [19]. The resistance to subcritical crack growth was determined for selected samples sintered at 1525 °C/3 h by stable indentation crack growth in bending (SIGB) method [20]. Bars were indented on the tensile side with for HV10 indents placed along the middle axis at a distance of 2 mm in the space between the inner span of the tensile side of the bending bar. The indented bars were stored for 2 weeks to let the cracks grow to a stable extension. Samples were loaded stepwise with a high crosshead speed of 5 mm/min to avoid subcritical crack growth. The initial stress was ~1/3 of the ISB strength. In the subsequent loading cycles the stress was increased by increments of ~1/10 of ISB strength until failure occurred. After each loading step the crack length of the perpendicular cracks was measured with a microscope. The evaluation of the test was done according to Dransmann et al. [21]. In a plot of ψ ∙ σ ∙ c^{-0.5} (a stress intensity) vs. P ∙ c^{-1.5} (with P = indentation load, c = crack length, σ = applied bending stress and ψ = 1.1 geometry factor) a graph with two branches is obtained. During indentation the indenter applies a certain crack opening stress intensity. Therefore after release of the indenter negative stress intensity is stored in the sample. Crack growth is, therefore, observed if the applied stress intensity K_{app} exceeds this stress intensity K_{app,0}. From now on crack growth is observed until the sample fails. The extrapolated rising branch of the plot intercepts the ordinate at a value of K_{IC}. The difference between K_{IC} and K_{app,0} is the threshold toughness K_{th}, i.e. the stress intensity which can be constantly applied without crack growth. K_{app,0} represents the R-curve dependent part of the toughness, i.e. the contribution of reinforcement mechanisms. The fatigue strength σ_f of the material which is relevant for constantly or repeatedly loaded components such as biomedical implants can be calculated as σ_f = σ_{4pt} · (K_{IO}/K_{IC}) [22]. The quantitative phase composition in polished surfaces and fractured surfaces of zirconia was determined by XRD (Panalytical, NL, CuKα1, Bragg Brentano setup, accelerator detector) by integration of the monoclinic (-111) and (111) as well as the tetragonal (101) peak in the fingerprint range (27-33° 2θ) and applying the calibration curve of Toraya [23]. Based on the phase composition data the transformation zone sizes h can be estimated according to Kosmac.
Knowing transformation zone sizes, transformability and Young’s modulus allows for calculation of the transformation toughness increments $\Delta K_{IC}^T$ according to McMeeking and Evans [6].

$$\Delta K_{IC}^T = \frac{X}{(1-\nu)}V_Z V_f E \varepsilon \sqrt{h} \quad \text{(here } X \text{ = transformation efficiency, } V_Z \text{ is the zirconia content } = 0.17, \varepsilon \text{ = 0.054 Volume change due to transformation, } \nu \text{ = Poisson’s ratio } = 0.25).$$

Here the assumption of a proper value for transformation efficiency $X$ is essential. For pure dilatation $X = 0.22$, for dilatation and shear $X = 0.48$. For yttria stabilized zirconia $X = 0.27$ [10], for a best case scenario (ZTA with isolated zirconia grains) the maximum value of $X = 0.48$ may be assumed [10].

Polished thermally etched surfaces (1350 °C/10 min air) were investigated by SEM (10 kV, in lens secondary electrons, Zeiss Gemini, Germany) in order to quantify the grain sizes and evaluate the homogeneity of the structure.

For a quick assay of ageing resistance polished samples were exposed to saturated water vapor in an autoclave at 134 °C, 2 bar vapor pressure. The dwell was between 10-300 h. It is assumed that 1 h accelerated autoclave ageing corresponds to approximately 3 years in vivo [13].

3. Results and Discussion

3.1. Preliminary experiments to optimize dispersion composition

The zetapotential measurements of stabilized dispersions of the powders (1 ml CE64/100 g powder) are shown in Fig. 1. Zirconia TZ-0 has its isoelectric point at $pH = 2.8 \pm 0.2$, at higher $pH$-values the zetapotential becomes negative and reaches a plateau value of $<-50 \text{ mV}$ at $pH > 7$. The alumina has its isoelectric point at $pH = 5.5$ and reaches a lower maximum stability level of $<-40 \text{ mV}$ at $pH 8.5$, the $pH$ value that was finally chosen for the suspension. Suspensions are considered stable at zetapotential values $> |30\text{mV}|$ [25].

![Fig. 1. Zetapotential of alumina and zirconia suspension in the pH-range 2-11.](image)

Fig. 2 shows the viscosity of 17ZTA1Y dispersions with 75 and 66 wt% solid content measured at different shear rates. Evidently the dispersion with higher solid content has a higher viscosity and a stronger shear thinning behavior. Dispersions with 66 vol% have an almost Newtonian flow behavior while the viscosity between high and low shear rates differs by four orders of magnitude in the dispersion with 75 % solid content. Both suspensions were basically processible by slip casting. Casting trials showed however that the suspension with
high solid loading showed a too slow green body formation rate. Samples were still wet and soft after de-watering overnight, so that the solid content was adjusted to 66%.

Fig. 2. Viscosity vs. shear rate for 17ZTA1Y suspensions with 66 wt% and 75 wt% solid content.

3.2. Density and mechanical properties

Density (not shown here) was higher than 99.6% of theoretical in all cases indicating sufficient sintering time and temperature. Fig. 3 shows the Vickers hardness of the materials containing different amounts of stabilizers sintered in the range between 1500 °C and 1550 °C. A clear trend of rising hardness with increasing stabilizer content is visible. The steepest hardness increase can be observed between 0.9 and 1.0 mol% yttria. Here the hardness rises from 1735-1755 HV10 to 1775-1800 HV10. The subsequent increase in hardness with stabilizer content is less pronounced. It hardly exceeds the standard deviation of measurements. The maximum hardness of 1835 ± 25 HV10 is obtained for 17ZTA1.2Y-1525 and 17ZTA1.4Y-1500. Concerning sintering temperature no clear general trend is visible.

Fig. 3. Vickers hardness HV10 of 17ZTA depending on yttria content and sintering temperature.
The bending strength $\sigma_{bp}$ is plotted in Fig. 4. Evidently the bending strength rises significantly with stabilizer content between 0.9-1.1 mol% yttria. At 1.1 mol% here the maximum bending strength of 820 MPa is reached, then the strength levels off within the standard deviation. The variation of sintering temperature seems insignificant. One exception was 17ZTA1Y-1550 which exhibited low strength; here a closer examination showed that the samples contained many defects so that value has to consider an outlier. The best materials therefore fulfil the requirements of EN ISO 6474-2 ($\sigma_{bp} > 800$ MPa).

![Fig. 4. Bending strength $\sigma_{bp}$ of 17ZTA depending on yttria content and sintering temperature.](image)

The fracture resistance determined by ISB test is shown in Fig. 5. Fracture resistance forms either a distinct maximum (1550 °C) or a small plateau (1500 °C and 1525 °C). The maximum is shifted to higher yttria contents with increasing sintering temperature. Evidently the lowest stabilizer content is only sufficient for the lowest sintering temperature. The maximum toughness of 5.7 MPa√m is reached for 17ZTA1.1Y-1550. For the lower sintering temperatures a maximum toughness between 5.2 MPa√m (17ZTA1Y-1500) and 5.5 MPa√m (17ZTA1-1.1Y-1525) is observed. Stabilizer contents above 1.1 mol% leads to a significant reduction in toughness, to ~5 MPa√m. All samples of >1 mol% yttria fulfill the requirements of EN ISO 6474-2 ($K_{ISB} > 4$ MPa√m).

![Fig. 5. Fracture resistance $K_{ISB}$ of 17ZTA depending on yttria content and sintering temperature.](image)
The Young’s modulus of the samples varied little between 342 ± 7 MPa for all samples which is slightly less than the expected value of 355 MPa by rule of mixture. Fig. 6 shows an example for the determination of the resistance to subcritical crack growth by SIGB method.

![SIGB plot for 17ZTA1Y-1525](image1)

**Fig. 6.** SIGB plot for 17ZTA1Y-1525.

Evidently the applied stress intensity $\psi \sigma \cdot c^{-0.5}$ increases without crack growth up to a level of $K_{\text{app}} = 1.3 \text{ MPa}\cdot\sqrt{m}$. Here a kink in the curve indicates the value for $K_{\text{app},0}$. The extrapolated rising branch intercepts the ordinate at $K_{\text{IC}} = 5.47 \text{ MPa}\cdot\sqrt{m}$. The difference $K_{\text{IC}} - K_{\text{app},0}$ is the threshold toughness $K_{I0}$. The results of the measurements for the other yttria stabilizer contents is summarized in Fig. 7. The materials show a very favorable ratio between $K_{I0}/K_{IC}$, the fatigue strength amounts to >75 % of the bending strength.

![Threshold toughness values](image2)

**Fig. 7.** Threshold toughness values $K_{I0}$, R-curve dependent increments of toughness $K_{\text{app},0}$ and calculated fatigue strength $\sigma_F$ for 17ZTA containing 1.0-1.4 mol% stabilizer sintered at 1525 °C.
3.3. Microstructure

SEM images of the microstructure of 17ZTA with different stabilizer contents sintered at 1500 °C and 1550 °C are shown in Fig. 8. Zirconia (bright grains) is evenly distributed in the alumina matrix (darker grains). Evidently the alumina matrix becomes coarser with increasing sintering temperature at constant yttria content. The coarsening of the matrix also affects the distribution of the zirconia phase, which tends to be displaced by alumina grain growth. Zirconia becomes more aggregated at higher sintering temperature due to this migration of grains. A quantitative evaluation of the grain sizes revealed that - as expected - the grain size of alumina grows by 25% between 1500 °C and 1550 °C while the zirconia grain sizes grow by 30% in the same temperature interval. Surprisingly both alumina and zirconia show the largest grain sizes at an intermediate yttria content of 1.1 mol% (alumina: 1.3 μm at 1500 °C and 1.65 μm at 1550 °C; zirconia: 270 nm at 1500 °C and 350 nm at 1550 °C). Average zirconia grain size is finer than the threshold (0.4 μm) for spontaneous transformation defined by Heuer [8].

![Fig. 8. Microstructure of 17ZTA of different stabilizer contents sintered at 1500 °C and 1550 °C.](image)

The fraction of grains that exceed the critical size for spontaneous transformation during cooling from sintering temperature evidently increases with increasing sintering temperature (and grain size) and decreasing fraction of stabilizer added. At first sight variation of the stabilizer content seems to have no effect on microstructure, a closer look shows, however, that in samples containing small amounts of stabilizer, transformed (monoclinic) zirconia grains and weak grain boundaries can be located.
3.4. Phase composition

The phase composition of zirconia in the polished surface (which represents the bulk composition) shows a decline of monoclinic fraction with increasing stabilizer content and decreasing sintering temperature (Fig. 10).

In detail at 0.9 mol% yttria the stabilizer addition seems absolutely insufficient so that the better part of the zirconia (~60%) is already transformed no matter which sintering temperature. At stabilizer contents ≥ 1.2 mol% the stabilizer content is high enough to keep the material almost entirely tetragonal. The range between 1-1.1 mol% yttria shows strong differences in monoclinic content; this is the range where the compositional and sintering parameters can be exploited to tailor the transformation characteristics. The transformability $V_f$ of zirconia is the difference between monoclinic content in fractured and polished surfaces $V_f = V_{m,fractured} - V_{m,polished}$. 

Fig. 9. Weak grain boundaries (left), small tetragonal and larger monoclinic grains (right) in 17ZTA1Y.

Fig. 10. Monoclinic content $V_{m,polished}$ in polished surfaces of 17 17ZTA depending on yttria content and sintering temperature.
Fig. 11. transformability $V_f$ of tetragonal phase in 1717ZTA depending on yttria content and sintering temperature.

Fig. 11 shows that the transformability always forms a maximum at intermediate stabilizer contents. Evidently at low stabilizer contents most of the zirconia is already transformed to monoclinic and no longer available for stress induced transformation. At high stabilizer contents the zirconia grains are too stable to be transformed induced by stress. The maximum transformability is in the range of 30 %. The maximum of transformability shifts from lower to higher yttria contents with increasing sintering temperature. At 1550 °C the optimum stabilizer concentration range is very narrow while the lower sintering temperatures lead to a broader transformability plateau region between 1-1.2 mol % yttria.

3.5. Transformation toughness

According to the calculation model of Kosmac the maximum transformation zone depth $h$ is up to 3 µm at intermediate yttria stabilizer contents (not shown) [24]. For high and low stabilizer contents and less pronounced transformability this value reduces to 1 µm. The resulting transformation toughness increments $\Delta K_{IC}^T$ calculated with the McMeeking formula [6] and assuming maximum transformation efficiency $X= 0.48$ amounts to approximately 1 MPa$\sqrt{m}$ (Fig. 12). The plot shows the same basic shape as the transformability plot in Fig. 11. Comparing the values for transformation toughness with the R-curve toughness increments $K_{app,0}$ shown in Fig. 7 shows that in the optimum compositional range between 1-1.2 mol% yttria the R-curve related toughness, i.e. the toughness contributed by reinforcement mechanisms can be entirely attributed to transformation toughening. The comparatively high R-curve toughness at 1.4 mol% yttria cannot be explained by transformation toughness alone. An additional toughness increment of ~0.5 MPa$\sqrt{m}$ is due to compressive residual stress in the alumina matrix. At 0.9 mol% yttria the contribution of transformation toughness becomes negligible. Here, due to the high monoclinic content, the alumina matrix is under hydrostatic tension [11]. The toughness contribution by residual stress would therefore become negative, so that the still acceptable toughness can only be explained by the influence of microcracking which can be assumed to provide a toughness increment of ~ 0.5-1 MPa$\sqrt{m}$ [7]. As can be seen from Fig.s 3 and 4 the microcracking-derived toughness is always related to a structural weakening resulting in reduced hardness and strength.
3.6. Low temperature degradation resistance

The low temperature degradation resistance of samples with 1-1.4 mol% yttria stabilizer content sintered at 1525 °C are shown in Fig. 13. The starting value at t= 0 h can be taken from Fig. 10. Stabilizer content of ZTA has an influence on ageing behavior. 17ZTA1Y-1525 shows an increase of monoclinic content from 30% to 50% during the first 30 h, further exposition leads to a maximum monoclinic content of 60% which coincides with the maximum monoclinic content in fracture surfaces (Fig.s 10 and 11). This monoclinic content seems to be the upper limit. 17ZTA1.1Y-1525 shows an initial increase from 24 to 32% during the first 10 h of ageing then the monoclinic content stagnates until 100 h aging time. The initially almost entirely tetragonal materials with 1.2 and 1.4 mol% yttria are quite stable, their final monoclinic content does not exceed 20% at the longest exposition time, they can therefore be considered absolutely safe for biomedical applications and exposition times in vivo of several decades.

![Fig. 12. Transformation toughness increments ΔK_{IC} of 17ZTA depending on yttria content and sintering temperature.](image)

![Fig. 13. Monoclinic fraction of 17ZTA exposed to water vapor (134°C/2bar) at different exposition times.](image)
4. Conclusion

Results have shown that the chosen manufacturing procedure is capable to manufacture ZTA materials for biomedical applications. Materials with 1.4 mol% yttria are strong but brittle; their LTD behavior is excellent. Materials with 1 mol% yttria or less are insufficiently stabilized especially if sintered at high temperature. They show insufficient strength and LTD resistance. Materials in the stabilizer concentration range between 1.1 and 1.2 mol% would fulfill the ISO standard. Slip casting is a suitable model technology to evaluate a broad variety of ZTA recipes due to small batch sizes and a limited experimental effort.

The current study leads to similar results as previous investigations of hot pressed materials [16-18]. The optimum range for a combination of good strength, fracture resistance and sufficient LTD resistance is very narrow. Within this narrow range the characteristics of the material can be adjusted according to requirements and preferences. The changes in toughness and the contribution of toughening mechanisms can be sufficiently explained by the analysis of microstructure and phase composition. With increasing yttria stabilizer content the dominant reinforcement mechanisms shift from microcracking to transformation toughening and finally to an increasing contribution of residual stress by CTE mismatch.

The bending strength of the pressurelessly sintered ZTA just reached the required value of 800 MPa, however, materials were not processed under clean room conditions, especially the milling in a plastic jar may have introduced some debris (plastic flakes) which results in voids reducing the strength.

The strength of hot pressed material produced on the same starting powder basis reached over 900 MPa as previously reported, ZTA materials of the market leader Ceramtec can achieve > 1200 MPa [26]. It has to be considered that the subsequent hot isostatic pressing procedure which is always carried out for biomedical grade ZTA is capable to increase the strength considerably.

5. References

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Синтетовања могу утицати на механичка својства и отпорност на нискотемпературску деградацију цирконијумске керамике ојачане алюмином, типичног биомедицинског састава. Утврђено је да су материјали јако осетљиви на овакве промене. Густи материјали са комбинованим својствима, као што су висока чврстоћа > 800 МПа, жилавост лома > 5.5 МПа/м и нискотемпературска деградација, могу се добити са концентрацијом стабилизатора између 1.1-1.2 %. Изван овог ранга, материјали су или недовољно стабилисани што резултује слабом снагом и великом тенденцијом за нискотемпературску разградњу, или превише стабилисани, и крти. Промене у очвршћавању могу бити приписане променама у микроструктури и фазном саставу услед варијација у садржају стабилизатора и услова синтетовања.

Кључне речи: цирконијум оксид, микроструктура, механичка својства, нискотемпературска деградација.

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