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DOI: https://doi.org/10.4012/dmj.2018-361

Posted at the Zurich Open Repository and Archive, University of Zurich
ZORA URL: https://doi.org/10.5167/uzh-181470
Journal Article
Published Version

Originally published at:
Jansen, Jan Ulrich; Lünkmann, Nina; Sener, Beatrice; Stawarczyk, Bogna (2019). Comparison of fracture toughness measurements for zirconia materials using two test methods. Dental Materials Journal, 38(5):806-812.
DOI: https://doi.org/10.4012/dmj.2018-361
Comparison of fracture toughness measurements for zirconia materials using two test methods

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To determine the influence of the fracture toughness (KIC) methods [single-edge-V-notch-beam (SEVNB) and chevron-notch-beam (CNB)] as well as an optional heat treatment on the KIC of three different zirconia generations (1st: ZI, 3rd: FX, 4th: HT). One hundred and twenty specimens each (3x4x45 mm) were fabricated, sintered, notched (n=360) and half of them heat treated before KIC measurements with 4-point-flexural-strength test. SEM images of the notches were recorded. Highest KIC was found for ZI followed by HT and FX. SEVNB resulted in significantly higher KIC than CNB. Heat treatment resulted in decrease for SEVNB and increase for CNB of KIC (except for FX). Groups tested using CNB showed higher reliability of values (Weibull modulus) than tested using SEVNB. SEM images present crack path and fracture surface. Different zirconia materials lead to different KIC values. The test method and a prior heat treatment showed an influence on the KIC values and their reliability.

Keywords: Single-edge-V-notch-beam, Chevron-notch-beam, Zirconia

INTRODUCTION

Zirconia is becoming increasingly popular9 as its outstanding biocompatible5, aesthetic and mechanical propertiesprevail and manifest themselves in clinical success6,8. As a result, the material has been the focus of development in recent years and four successive generations of zirconia have been developed with the aim to improve the optical properties and to maintain the mechanical properties as high as possible8. This is also linked with the increasing interest in using monolithic restorations rather than veneered frameworks, which places higher demands on the flexural strength and can most likely be fulfilled by zirconia due to mechanical resilience and an efficient manufacturing process but only with enhanced translucency at the same time6.

A decisive factor represents the comparatively high fracture toughness (KIC) of zirconia among ceramic materials which describes the local stress concentration at the crack tip at the critical value that leads to material failure7. The high value of the parameter results from phase transformation toughening: Due to tensile stress triggering tetragonal zirconia transforms to the monoclinic phase and inhibits crack growth by volume increase at the crack tip3,5,8,9.

The zirconia development comprises four generations that mainly differ in their chemical composition by the proportion of alumina (Al2O3) and yttria (Y2O3). First developed zirconia (1st generation) is characterized by high flexural strength, low translucency, 3 mol% Y2O3 and a phase composition of 90.1 wt% tetragonal and 9.6 wt% cubic phase10. Although it shows reliable, long-term resilience and an efficient manufacturing process but only with enhanced translucency at the same time6.

By reducing the content of Al2O3, a more translucent material with smaller grain sizes and modified molecular structure is obtained (2nd generation). An increase of the Y2O3 doping to 5 mol% leads to the 3rd generation zirconia with higher translucency as well as a complete stabilization induced by approx. 53% cubic phase10,12. However, since this material has poorer mechanical properties, the need for further, improved zirconia has remained and the proportion of Y2O3 has been reduced to 4 mol% in the 4th generation. This represents a compromise between the 2nd and 3rd generation in terms of translucency and flexural strength.

The single-edge-V-notch-beam (SEVNB) method, which is well-established, widely used and easy to implement, leads to a high scattering of the measured values13 and is considered unsuitable for fine-grained materials with a grain size smaller than 1 μm14, as the so-called “notch root radius effect” and an overestimation of the KIC occurs15-17. With regard to the sharpness of the SEVNB notch root radius, the notches produced with razor blades are significantly limited by the razor blade geometry (approx. 1817 or 10 μm19) and for the Y2O3 stabilized zirconia with grain sizes of 437 nm (3 mol% Y2O3) or 858 nm (5 mol% Y2O3) insufficiently thin17. Instead of an initial crack, the CNB process is based on a triangle cross-section where the crack proceeds from one tip through the material with an increasing geometry factor until it fractures7,14. By this, the notch root radius effect is avoided but the technical implementation of the two-sided notch is complicated15.

Heat treatment protocols influence the stress state and can be used to reduce residual stresses in the material, for instance after mechanical exposure due to grinding or airborne-particle abrasion19,20. By heating zirconia to a temperature of 1,350°C, which is slightly
above the transition temperature from the tetragonal to monoclinic phase (1,170°C), recrystallizing processes are triggered.

The aim of this investigation is to determine the $K_{IC}$ of 1st, 3rd and 4th generation zirconia materials using either the SEVNB or CNB method with and without prior heat treatment. The null-hypotheses assume that neither (I) the different zirconia materials, nor (II) the $K_{IC}$ method (SEVNB, CNB), or (III) the heat treatment has an influence on the results of $K_{IC}$ and Weibull modulus.

MATERIALS AND METHODS

In total, 360 bar specimens (3.0±0.2×4.0±0.2×45.0±0.5 mm) of three different zirconia generations, namely Ceramill Zolid ZI (ZI; 1st generation, $n=120$), Ceramill Zolid FX (FX; 3rd generation, $n=120$) and Ceramill Zolid HT$^+$ (HT; 4th generation, $n=120$) (Table 1), were milled (Ceramill Motion 2, Amann Girrbach, Koblach, Austria), ground (SiC-Paper P1200, Buehler, Lake Bluff, IL, USA) and sintered (Therm 2; Amann Girrbach).

The total amount of specimens was divided according to two $K_{IC}$ methods, specifically SEVNB ($n=60$/zirconia) and chevron-notch-beam (CNB; $n=60$/zirconia). The notch geometries were produced in compliance with DIN EN ISO 6872 for SEVNB and DIN EN14425-3 combined with ISO 24370 for CNB$^{14,21,22}$.

Half of specimens ($n=50$/subgroup) were additionally heat treated (1,350°C for 30 min) before $K_{IC}$ measurements (LHT 02/16, Nabertherm, Lilienthal, Germany) (Fig. 1.4).

Preparation and measurement of CNB specimens

The CNB specimens were inserted individually into a customized mounting for a tabletop cut-off machine (Secotom 50, Struers, Ballerup, Denmark). The mounting (Fig. 1.1b) featured two positions in order to perform two coplanar cuts which resulted in the necessary chevron notch featuring an angle of 26°. After performing the first cut with a 150 µm-saw-blade (MoD08, Struers), a displacement speed of 0.05 mm/min, a blade rotational velocity of 5,000 min$^{-1}$ and a cutting length of 15 mm, the specimen was turned, the position of the specimen in the holder changed (Fig. 1.2b) and the second cut performed. After cleansing with distilled water and ultrasound (Sonorex RK102H, Bandelin electronic, Berlin, Germany), the final geometry was measured microscopically (Zwick/Roell Z 2.5, Zwick, Ulm, Germany) and the compliance with ISO 24370 verified (Fig. 1.3). For $K_{IC}$ measurements, 4-point-flexural-strength was determined with foregoing defined loading in compression with a force of 2–3-times of the fracture load 3-times (1445 Zwick/Roell' Zwick, Fig. 2.left). After turning the specimen, flexural strength was measured with a traverse speed of 0.05 mm/min (displacement controlled) and fracture shutoff at 70% of the maximal

| Zirconia | Abbreviation | $Y_2O_3$ in mol% | $Al_2O_3$ in wt% | Material | LOT      |
|----------|--------------|------------------|------------------|----------|----------|
| 1. Generation | ZI | 3 | ca. 0.25 | Ceramill ZI | 1610000 |
|           |    |    |        |          | 1706000 |
|           |    |    |        |          | 1702000 |
| 3. Generation | FX | 5 | ca. 0.05 | Ceramill Zolid FX | 1512004 |
|           |    |    |        |          | 1512008 |
| 4. Generation | HT | 4 | ca. 0.05 | Ceramill Zolid HT$^+$ | XY406339G |

Product name and LOT (all materials from Amann Girrbach)
force (Figs. 1.5b and 2.right). The validity of the stable crack growth was determined by analyzing the force-displacement-curve according to the normative regulations of DIN EN 14425-3 and ISO 24370. The fracture toughness \(K_{IC,CNB}\) was calculated according to the following equation:

\[
K_{IC,CNB}=\frac{F}{b^{3/2}w^{1/2}} \cdot Y^{*}_{\min} \sqrt{1000}
\]

with \(K_{IC,CNB}\)=fracture toughness [MPa√m], \(F\)=fracture load [N], \(s_{b}\)=bearing range [m], \(s_{i}\)=inner range [m], \(b\)=specimen thickness [m], \(w\)=specimen width [m], \(Y^{*}_{\min}\)=form factor of stress intensity. The geometric relationship was determined by:

\[
0.3874-3.0919(l/w)+4.2017(l/w)^2
\]

with \(Y^{*}_{\min}\)=form factor of stress intensity, \(w\)=specimen width [m], \(l_{c}\)=chevron notch depth [m], \(l_{a}\)=average notch length [m].

**Preparation and measurement of SEVNB specimens**

For SEVNB, five specimens were simultaneously placed on the narrow side in a customized specimen holder and fixated (Futar D Fast, Kettenbach, Eschenburg, Germany). With the help of a tabletop cut-off machine and a diamond cut-off wheel (M1D13, Struers), a pre-notch —with a depth of 600—700 µm and width of approx. 450 µm— was produced in the center of the specimen perpendicular to the longitudinal axis of the specimens (Fig. 1.1a). Subsequently, the specimen holder was transferred into a notching machine (SD Mechatronik, Feldkirchen-Westerham, Germany), which produced a V-notch at the bottom of the notch by actuating a razor blade (Lutz Blades, Emil Lux, Wermelskirchen, Germany) with the usage of diamond polishing suspension (DiaPro Allegro Largo and DiaPro Dac3, Struers, Fig. 1.2a). In order to achieve normed notch geometries with a depth of 0.8—1.2 mm, cycles in a range of 15,000 to 20,000 and a weight of 600—900 g were chosen. After cleansing with detergent (F 100 Super, HWR-Chemie GmbH, Emmering, Germany) and ultrasound, the final geometry was measured microscopically (Fig. 1.3). Four-point-flexural-strength was determined with a traverse speed of 0.5 mm/min (displacement controlled) and a fracture shutoff at 60% of the maximal force (Fig. 1.5a). The fracture toughness \(K_{IC,SEVNB}\) was calculated according to the following equation:

\[
K_{IC,SEVNB}=\frac{F}{b^{3/2}w^{1/2}} \cdot Y^{*}_{\min} \sqrt{1000}
\]

with \(K_{IC,SEVNB}\)=fracture toughness [MPa√m], \(F\)=fracture load [N], \(s_{b}\)=bearing range [m], \(s_{i}\)=inner range [m], \(b\)=specimen thickness [m], \(w\)=specimen width [m], \(Y^{*}\)=form factor of stress intensity. The geometric relationship was determined by:

\[
Y=1.9887−1.326α−3.49−0.68α+1.35α^2/(1+α)^{1.5}
\]

with \(Y\)=form factor of stress intensity, \(α\)=relative depth of the V-notch.

**SEM crack and fracture analysis**

SEM images were recorded to demonstrate that the crack path originated from the inserted notch and to present the fracture surface of CNB and SEVNB specimens (Zeiss Supra 50VP FESEM, Carl Zeiss, Oberkochen, Germany). The images were taken with an acceleration voltage of 0.2—30 kV and a maximum working distance of 11.1 mm. Prior to SEM analysis, specimens were selected, thermal etched and sputtered with tungsten (CCU-010, Safamatic, Bad Ragaz, Switzerland).

**Statistical analysis**

Data were statistically analyzed (IBM SPSS Statistics 23, IBM, Armonk, NY, USA) using Kolmogorov-Smirnov and non-parametric test, such as Kruskal-Wallis and Mann-Whitney-U. To estimate the reliability of \(K_{IC}\) values, two-parametric Weibull analyses, using the maximum likelihood estimation method at 95% confidence level were computed\(^{20}\). The defined level of significance was adjusted by Bonferroni correction (0.05/12=0.004).
RESULTS

The Kolmogorov-Smirnov test indicated a higher rate of violations of the normality assumption (16.67%), therefore non-parametric tests were used.

Significant differences of $K_{IC}$ were found for ZI, FX and HT within SEVNB and CNB ($p<0.001$). Among the groups without heat treatment, SEVNB (ZI: 8.26 MPa$\sqrt{m}$; HT: 6.68 MPa$\sqrt{m}$; FX: 3.53 MPa$\sqrt{m}$) resulted in significantly higher median of $K_{IC}$ than CNB (ZI: 4.42 MPa$\sqrt{m}$; HT: 3.53 MPa$\sqrt{m}$; FX: 2.64 MPa$\sqrt{m}$) ($p<0.001$) (Table 2) and for both methods, ZI had the highest median values followed by HT and FX ($p<0.001$). Heat treatment showed significant impact on $K_{IC}$ in all groups ($p<0.001$), except for FX in CNB ($p=0.128$). After heat treatment, $K_{IC}$ decreased for SEVNB (ZI: 5.11 MPa$\sqrt{m}$; HT: 4.29 MPa$\sqrt{m}$; FX: 3.19 MPa$\sqrt{m}$) but increased for CNB (ZI: 4.47 MPa$\sqrt{m}$; HT: 3.64 MPa$\sqrt{m}$; FX: 2.66 MPa$\sqrt{m}$). Figure 3 shows the linear correlation of median $K_{IC}$ values within one group. Weibull modulus (m) showed higher values for the CNB method. Here, the Weibull modulus decreased after heat treatment and led to significant differences for HT (60.22→18.28) and FX (48.96→31.05) whereby the values for ZI with CNB did not differ significantly (with: 50.55; without: 61.90).

For SEVNB, a slight increase in Weibull modulus was observed after heat treatment (ZI: 6.93→10.40; HT: 4.25→4.84; FX: 6.02→7.51).

Table 2  Fracture toughness, Weibull modulus and characteristic fracture toughness

| Fracture toughness method | Heat treatment | Material | Fracture toughness | Weibull modulus |
|--------------------------|----------------|---------|--------------------|-----------------|
|                          |                |         | Min/Median/Max $K_{IC}$ [MPa$\sqrt{m}$] | M   | 95% CI |
| CNB                      | without        | ZI      | 4.21/4.42/4.58$^{bc}$ | 61.90 | 42.4/90.1 |
|                          |                | FX      | 2.53/2.64/2.81$^{a,b}$ | 48.96 | 33.5/71.3 |
|                          |                | HT      | 3.38/3.53/3.64$^{ab}$ | 60.22 | 41.2/87.7 |
|                          | with           | ZI      | 4.13/4.47/4.71$^{bc}$ | 50.55 | 34.6/73.6 |
|                          |                | FX      | 2.48/2.66/3.57$^{a,b}$ | 31.05 | 21.2/45.2 |
|                          |                | HT      | 3.04/3.64/4.40$^{ab}$ | 18.28 | 12.4/26.7 |
| SEVNB                    | without        | ZI      | 5.03/8.26/10.01$^{bc}$ | 6.93  | 4.6/10.1  |
|                          |                | FX      | 1.99/3.53/4.96$^{ab}$ | 6.02  | 4.0/8.8   |
|                          |                | HT      | 3.65/6.68/9.12$^{ab}$ | 4.25  | 2.8/6.2   |
|                          | with           | ZI      | 4.16/5.11/6.89$^{bc}$ | 10.40 | 7.0/15.2  |
|                          |                | FX      | 2.15/3.19/4.38$^{a,b}$ | 7.51  | 5.0/11.0  |
|                          |                | HT      | 1.29/4.29/5.56$^{ab}$ | 4.84  | 3.2/7.1   |

Capital letters indicate significant differences within method and material choice. Small letters indicate significant differences within method and heat treatment group. Asterisks (*) marks groups deviating from normal distribution.

Fig. 3  Linear correlation of median $K_{IC}$ values within one group.
The SEM images of the fracture surface of CNB and SEVNB specimens present the crack path and show that it originated from the inserted notch (Figs. 4a–d). Figures 4a and c show the overview of the fracture surfaces along the inserted notches of a CNB and SEVNB specimen respectively. Both inserted notches resulted in plane and smooth fractures. For CNB a small area of fracture lines is observed at the lower edge of the chevron notch (Fig. 4b), while the fracture line and crack path along the V-notch was even more prominent (Fig. 4d).

DISCUSSION

The most outstanding results are the higher $K_{IC}$ for the SEVNB than for the CNB, the opposite effect of heat treatment on $K_{IC}$ results, and the differences among the zirconia materials of 1st, 3rd and 4th generation. Further on, differences for the Weibull moduli are visible. Hence, all null-hypotheses are rejected.

Differences of the methods

The first mentioned observation of significantly higher $K_{IC}$ values for SEVNB aggregates to the concerns of overestimated values found in literature$^{13,16}$, which are attributed to the method itself: SEVNB leads to higher $K_{IC}$ when fine-grained $Y_2O_3$ stabilized zirconia is investigated because of the notch root radius sizes, which exceed the magnitude of the microstructure. Several investigations and a round robin have highlighted this limitation$^{10,13,15,17}$. To the current state, even adaptation factors to the theoretical fracture toughness, like 1.5$^{16}$, have been examined. Therefore, the present values and the success in approaching the sharp notch root radius can generally be compared with other SEVNB results in literature, but assuming the $K_{IC}$ values determined by SEVNB as the true fracture toughness should be made with caution.

These difficulties of SEVNB have motivated to use an alternative method of $K_{IC}$ determination, namely the CNB method. At first glance, CNB differs from the SEVNB method in that no cracking geometry is initiated$^{13,24}$. Beneficially, the chevron tip or rather the notch does not need to be sharp$^7$ in comparison to the notch required for SEVNB. Nevertheless, CNB is susceptible to subcritical crack growth, also slow crack growth, and thus to unstable crack growth. According to DIN EN 14225-3 and ISO 24370$^{21}$, this assumes to reject measurements with sudden fracture from the final evaluation as it would falsify the results even when a stable fracture growth is obtained from force-displacement-curve due to slow crack growth. Literature describes an underestimation of the $K_{IC}$ values obtained by CNB, caused by slow crack growth because the standardized measurement speed of maximal 0.05 mm/min$^{21}$ does not prevent slow crack growth and should be increased$^{18}$. Belli et al. referred to a crack stability that occurred for loading rates/measurement speeds of $\leq$0.01 mm/min$^7$. Generally, the measurement speed of
CNB is known to influence the $K_{IC}$, since with increasing measurement speed the $K_{IC}$ increases\textsuperscript{10}, however the present measurements of $K_{IC}$ via CNB method were performed at a measurement speed of 0.05 mm/min with predominantly valid curve shapes as demanded in DIN EN 14425-3 and ISO 24370. In order to obtain stable crack growth, a preloading with 166 N in compression has been applied before determining $K_{IC}$ by CNB. By this, the material might be weakened resulting in an additional underestimate of the $K_{IC}$. Nevertheless, both theories are a setback, since the fracture toughness represents a material property that should not depend on the test method. Apart from that the mean values obtained for FX (3.54±0.70 MPa$\sqrt{m}$) approximately correspond to the investigations of Nassary Zadeh et al. (3.56±0.47 MPa$\sqrt{m}$)\textsuperscript{25}.

**Material characteristics**

The decreasing fracture toughness from 1st, to 3rd and 4th generation zirconia is linked to the decreasing strength and results from the chemical compositions and the associated crystal lattice structures. The higher content of Al$_2$O$_3$, and its phase transformation ability effect in 1st generation zirconia is one plausible reason for the high fracture toughness of the 1st generation material ZI obtained with both methods (CNB and SEVNB). Y$_2$O$_3$ has a stabilizing effect and keeps phases like the tetragonal and cubic stable at room temperature leading to coarser grains with increasing content. This harms the strength and the fracture toughness. In addition, it limits the phase transformation which results in the uncommonly high fracture toughness. Thus, a lower fracture toughness is expected when more Y$_2$O$_3$ is added to zirconia. In addition to the static effects, a higher content of cubic phase influences the aging process of zirconia and prevents hydrothermal aging in which the tetragonal phase is converted to the monoclinic phase due to heat and humidity and the material is weakened. Since both, CNB\textsuperscript{24,26} and SEVNB\textsuperscript{26} are known for their sensitivity to moisture, constant conditions during the measurements were ensured in this study. Due to notching, cutting and cleansing under aqueous environment and capillary action, stress crack corrosion and hydrothermal aging could be accelerated as well as fracture toughness be affected in different manners for the materials and methods. This might be an explanation why no changes in $K_{IC}$ were observed for third generation material (FX) using the CNB approach and confirms current theories that cubic zirconia is more stable to aging and phase transformation\textsuperscript{5,27}. Additional measurements of the crystalline phase content obtained by XRD measurements for example, would be usefully to supplement and discuss the present findings.

**Heat treatment and aging**

Heat treatments, which are known by their stress reducing property and simultaneously influence the moisture content, result in an increase of $K_{IC}$ for CNB. This could mean that the reliability of the materials rises when less tensions are present. At first glance, the results of the SEVNB method contradict to this. This could be due to the fact that the micro cracks of SEVNB are already introduced before the heat treatment and other phenomena play a role. For example, zirconia can be weakened by annealing processes of various temperature ranges from 100 to 400°C and also above 1,000°C\textsuperscript{28,29}. In general, zirconia crystallizes in three different crystal phases depending on the temperature: cubic (2,680–2,360°C), tetragonal (2,360–1,170°C) and monoclinic (1,170°C–room temperature). Through the addition of stabilizing oxides, zirconia is either stabilized in the tetragonal or cubic phase at room temperature depending on the concentration of the stabilizing oxide. When high tensile stresses are applied, the tetragonal phase can transform ($\tau$=$\tau$m transformation) into the monoclinic phase originating from a crack tip. The existing tensile stress reduces the pressure of the matrix on the zirconia grains and causes the transformation from $\tau$=$\tau$m. Since the formation of the monoclinic phase and the increase in volume of the crystals prevents further crack growth, the $\tau$=$\tau$m transformation is also known as martensitic transformation or toughening mechanism. The transformation can theoretically be reversed by a heat treatment using temperatures slightly above 1,170°C as it was performed in the present investigation with 1,350°C.

However, a heat treatment leads to a targeted drying process and thus to a change of moisture content. For SEVNB an increased humidity up to 10% resulted in higher $K_{IC}$\textsuperscript{26}, which contradicts the present outcomes. For CNB, moisture leads to subcritical crack growth and smaller $K_{IC}$ values\textsuperscript{13}, which can be observed by the higher $K_{IC}$ after heat treatment in this study. Moisture and heat are also strongly associated with the term “hydrothermal aging” and a weakening of the zirconia material due to phase transformation from tetragonal to monoclinic phase as mentioned above\textsuperscript{3,9,30}. It remains unclear how the interplay of tension, heat, humidity and aging could have influenced the behavior of the zirconia generations, but differences have clearly occurred due to the heat treatment step.

**Weibull statistics**

Originally used to analyze flexural strength results, Weibull statistics are applied to the fracture toughness of zirconia\textsuperscript{25,31}, providing additional insight in addition to $K_{IC}$ values. Weibull modulus distribution considers method reliability and the distribution of brittle materials such as zirconia\textsuperscript{32}. The lower Weibull modulus for SEVNB demonstrates that the CNB method is the more reliable method which also persuades with a smaller sample size. Previous findings proved a significantly smaller scatter of the results for CNB than for SEVNB\textsuperscript{13}. The higher Weibull modulus for SEVNB with heat treatment than without can be explained by the above-mentioned stress-reducing and reliability increasing effects of heat treatments because stress states are leveled, and effects of moisture are avoided. Differences between the fracture toughness determination methods are obvious due to the different fracture mechanics of
CONCLUSION

Summarizing, the different zirconia materials of 1st, 3rd and 4th generation lead to different $K_C$ values. The test method as well as a heat treatment showed an influence on the $K_C$ values. CNB method seems to provide more reliable $K_C$ values compared to SEVNB.

ACKNOWLEDGMENTS

The authors would like to thank Amann Girrbach for supporting this study with zirconia materials. This research was partially supported by research grant ZF4052004AG (AiF Projekt, Berlin, Germany, ZIM-Kooperationsprojekte, Projekträger des BMWI).

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