

On the feasibility of the Chevron Notch Beam method to measure fracture toughness of fine-grained zirconia ceramics

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Abstract

Objective

The fracture toughness determination of fine-grained zirconia ceramics using the chevron notched beam method (CNB) was investigated to assess the feasibility of this method for quality assurance and material characterization.

Methods

CNB tests were performed using four different yttria-stabilized zirconia ceramics under various testing modes and conditions, including displacement-controlled and load-rate-controlled four point bending to assess the influence of slow crack growth and identify most suitable test parameters. For comparison, tests using single-edge V-notch beams (SEVNB) were conducted.

Results

It was observed that the CNB method yields well-reproducible results. However, slow crack growth effects significantly affect the measured K_{IC} values, especially when slow loading rates are used. To suppress slow crack growth, the application of high loading rates is recommended.

Significance

Despite a certain effort needed for setting up a sample preparation routine, the CNB method is considered to be very useful for measuring and controlling the fracture toughness of zirconia ceramics.

Introduction

Due to their excellent mechanical properties and inertness, zirconia-based ceramics have been developed successfully and are broadly used for dental restoration [1-3] and orthopaedics [4]. In particular, very high fracture toughness and mechanical strength values can be realised by tailoring the microstructure and composition of zirconia. However, the effect of transformation toughening mechanism that enable excellent strength properties may also be weakened in presence of permanent exposure to corrosive environment and mechanical stresses [1, 5-8]. Zirconia-based ceramics with enhanced resistance against stress crack corrosion and low-temperature degradation have been developed [9].

The fracture toughness K_{IC} is one of the most important properties of ceramic materials exposed to cyclic loading, like it is the case in the field of e.g. dental applications. Therefore, the K_{IC} is strongly recommended to be evaluated wherever mechanical stability of materials and components are of interest. Moreover, the fracture toughness has become a key property to evaluate and compare material quality.

To measure fracture toughness, the Single-Edge V-Notch Beam (SEVNB) method has become the most widely used standard [10]. Here a V-notched beam is used which is prepared by a sharp blade in combination with a diamond suspension. This method was proved to be practical, reliable and accurate enough to yield reproducible results [11,12]. However, it has been doubted that this standard is applicable for very fine-grained zirconia ceramics, due to the fact that even by using very sharp blades, the notch tip diameter would certainly be much larger than the average grain size, and small differences of the blade quality and type of diamond suspension might have a strong influence on the measured toughness value causing systematic deviations [13,14]. A particularly critical drawback of the SEVNB method is that the lower the notch quality the higher will be the obtained K_{IC} value, since lower notch qualities generally have increased notch tip diameters. It has been supposed

that in case of low-quality notches the SEVNB technique may overestimate the K_{IC} value of modern fine-grained zirconia ceramics by a factor of 1.5 [13].

As an alternative testing method, the Chevron Notch Beam (CNB) test has been identified, which is also a standardised method to evaluate the fracture toughness of ceramic materials [15, 16] and was previously reported to yield well-reproducible results with low standard deviation also in glass [17]. By accurately following the prescribed preparation steps of this standard, a pre-crack should be produced that will at first grow stably upon bending. The corresponding Stress-Intensity Factors (SIF) as a function of crack length are known [18], and from the maximum load measured during the bending test, the K_{IC} value can be easily determined. The main advantage of this method is that it is independent of the notch tip diameter, since due to stable crack growth, the K_{IC} of a real (natural) crack is measured.

Despite the expected benefits for measuring the K_{IC} of fine-grained zirconia ceramics, there have been some drawbacks of the chevron notch tests: First, the stadium of stable crack growth is not reached in every test. Therefore, there is always a considerable number of tests that do not yield a valid result. This difficulty has been counteracted by two ways:

1. Inducing crack formation in compression as recommended in DIN EN 14425-3 [15]
2. slow loading rates

However, while an appropriate pre-cracking step significantly enhances the rate of valid test results, the use of slow load rates leads to a significant influence of subcritical crack growth effects, which result in lower measured K_{IC} values [19-21].

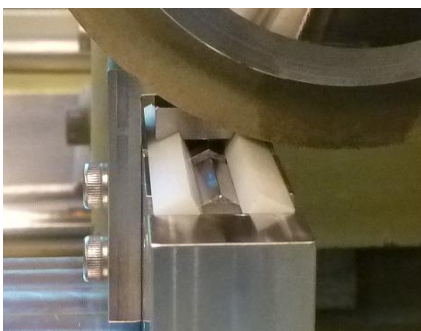
Aim of the present work was to assess the feasibility of CNB test method for the measurement of the K_{IC} of zirconia ceramics. In particular, it was surveyed if this test may be a preferred method for the quality control of zirconia ceramics manufacture. Four zirconia materials were selected and tested using different test parameters to identify suitable conditions and parameters for the K_{IC} determination.

Experimental

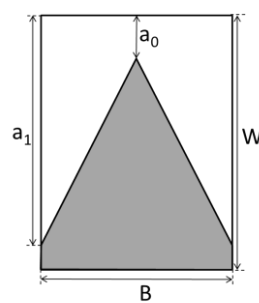
Sample preparation

The specimens were prepared out of pre-sintered dental milling blanks via high pressure waterjet-cutting followed by grinding to the desired size on a surface grinding machine. The sintering was performed according to supplier recommendations. The final specimen size according EN14425-3 was about $3.0 \times 4.0 \times 45.0 \text{ mm}^3$.

Two samples were mounted in a special sample holder (fig. 1) with antidromic angulation of $26^\circ 30'$. The chevron notch was then cut with a precision diamond saw (Buehler ISOMET 4000) and a metal bound wafering blade with low diamond content (Buehler series 15LC; $\varnothing 76 \text{ mm} \times 0.20 \text{ mm}$). Revolution and cutting speed were 5000 rpm and 4 mm/min respectively. After the first cut, the position of the two samples was exchanged, ensuring a final chevron notch geometry in compliance with EN14425-3. Since small variations of the notch geometry – even within the allowed geometry range of the EN14425-3 – have a significant influence on the obtained K_{IC} values, the geometry parameters of the Chevron notches (fig. 1b) should be close to the upper limits but avoid overcuts (a_0 : $0,80 \pm 0,08$ and a_1 : min. 3,80 to max. W).



(a)



(b)

Fig. 1: (a) Samples preparation setup and (b) sketch of the notch geometry, described by the dimensions a_0 , a_1 , W and B

After cutting, samples were cleaned for 5 minutes with acetone in an ultrasonic bath (Bandelin electronic RK 510) and afterwards dried for 30 Minutes at 110 °C in a drying cabinet (Heraeus UT 6060).

The CNB tests were conducted according to DIN EN 14425-3, sect. 7. After measuring the dimensions of the samples and the notches, the samples were precisely inserted into the 4-point-test assembly. The tests were conducted at room temperature in a lab environment (max. 50% humidity). According to sect. 7.2 (comment 2), the rate of valid test results can be increased by loading the notch in compression: This was done by turning the notched bend bar upside-down and loading it three times with a load of 200 to 220 N (which was three times the maximum fracture load of the notched samples) (fig. 2). This preloading step in compression induces the formation of a small pre-crack in the notch.



Fig. 2: Load configuration for the pre-crack formation under compression: The Chevron notch is on the upper (compression) side.

Directly after the preloading step, the CNB test was performed in the usual 4 point bending configuration (fig 3). The DIN standard allows a free selection of the cross-head speed lower than 0.05 mm/min, while the fracture should occur within 2 to 5 minutes. As will be shown later, the speed limit of 0.05 mm/min will not yield plausible results. It was therefore decided to perform further tests with constant loading rates between 1 and 10 N/s, which resulted in test durations between only 10 and 20 seconds. Some additional tests were conducted in an inert atmosphere

using silicone oil to prevent subcritical crack growth to adversely affect the test results. These tests were performed at a loading rate of 1 N/s and a displacement rate of 0.05 mm/min.

After the test the notch geometry was measured. From the valid tests, the load maxima were determined, and the resulting K_{Ic} values were calculated according to the following formula (acc. to DIN EN 14425-3) [15]:

$$K_{I,CNB} = Y_{min}^* \left[\frac{P_{\max}(S_o - S_i)}{BW^{3/2}} \right] \quad (1)$$

with
$$Y_{min}^* = (3.08 + 5.00\alpha_0 + 8.33\alpha_0^2) \left(1 + 0.007 \left(\frac{S_o S_i}{W^2} \right)^{1/2} \right) \left(\frac{\alpha_1 - \alpha_0}{1 - \alpha_0} \right) \quad (2)$$

$$\alpha_0 = a_0/W \text{ and } \alpha_1 = a_1/W. \quad (3)$$

The other variables are given in Figs 1 and 3.

It should be taken into account that ISO 24370 [16] has a deviant calculation approach for Y^* leading to a slightly deviant K_{Ic} -value.

The tests at varying loading rates were conducted at the two test labs available by the authors using electromechanical testing machines (Zwick/Roell Z020 with a 500 N load-cell type Xforce P, Instron 1380). In both labs 4 point bend test setups are in accordance with ISO 14704 [22].

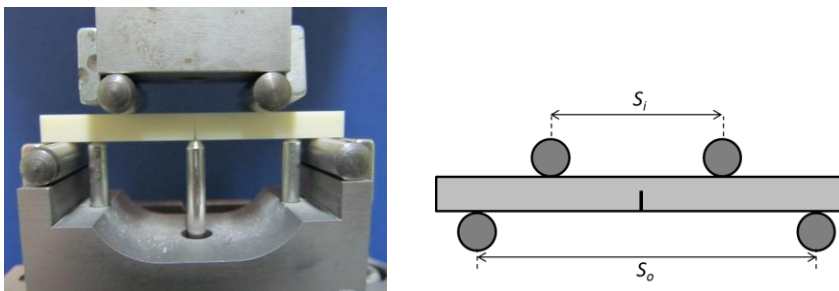


Fig. 3: Test configuration for the CNB test

For comparison with the CNB results, K_{IC} values were measured using the SEVNB method according to ISO 23146. It was made sure, that the achieved notch tip diameter was below 5 μm , which should lead to acceptable fracture toughness values.

Materials

An overview of the tested materials is given in Table 1. Though all four materials have the same composition (3 mole% Y_2O_3 , 97 mole% ZrO_2), certain differences in microstructure (e.g. grain size and Yttria distribution) are expected to have an influence on their fracture toughness.

Table 1: Overview of tested 3Y-TZP materials.

Materials	Supplier
Lava TM Plus High Translucency Zirconia	3M Deutschland GmbH
ZY	OxiMaTec GmbH
Z-CAD [®] HTL	Metoxit AG
ZirDent TM	C5 medicalwerks

Results

Results of displacement-controlled tests

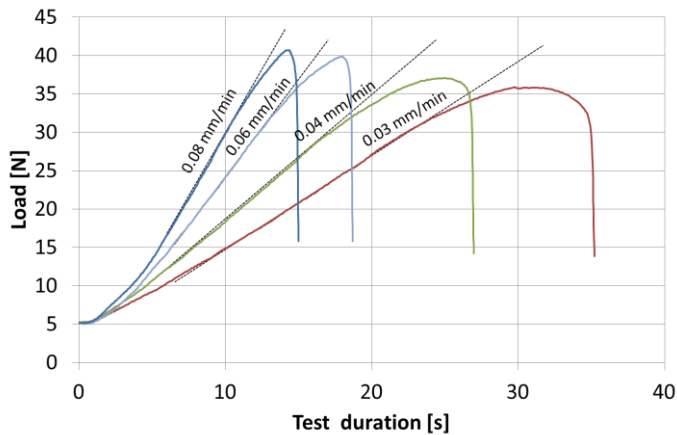


Fig. 4: Typical results of load over time of CNB tests at crosshead speeds in the range of 0.03 to 0.08 mm/s (Material: Lava™ Plus)

First tests were conducted at varying cross-head speeds between 0.03 and 0.08 mm/min. Typical data of the load as a function of time are plotted in fig. 4. It can be observed that directly after the start of the tests at a pre-load of 5 N, there is an initial non-linear load increase, which within 5 seconds transforms into a linear mode. When approximately 30 to 35 N are reached, the slope of the load curve decreases again, which is assigned to the onset of stable crack growth leading to increased compliance of the samples [23,24]. However, it is difficult to accurately determine the load, at which the slope starts to decrease. It should be noted that the period of stable crack growth is dependent on the cross-head speed and lies within a range of app. 2 to 10 seconds.

The most important point is however that the load maximum systematically decreases with the cross-head speed from 41 N at a speed of 0.08 mm/s to 36 N at 0.03 mm/s. This shows that the measured K_{IC} value is load-rate dependent and may systematically decrease with the loading rate.

The resulting K_{IC} values of the CNB tests at different cross-head speeds are plotted in Fig 5. It is obvious that the higher loading rates lead to higher values. The loading-rate dependence of the K_{IC} values diminishes at higher speeds.

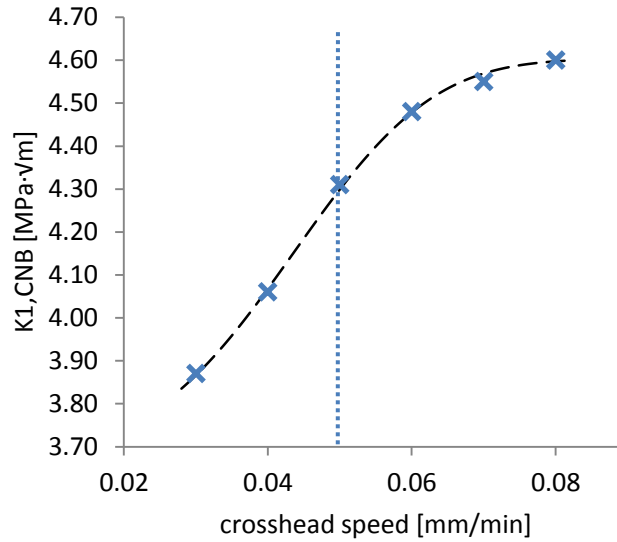


Fig. 5: Results for displacement-controlled CNB tests. The dotted vertical line marks the crosshead speed limit as given by DIN EN 14425-3 (Material: Lava™ Plus)

The dashed line in fig. 5 is a fit using the Johnson-Mehl-Avrami-Kolmogorow-Equation [25-29] :

$$K_{IC}(v) = K_{IC_{min}} + (K_{IC_{max}} - K_{IC_{min}}) \cdot (1 - e^{-(bv)^n}) \quad (4)$$

where $K_{IC}(v)$ is the fracture toughness for a given crosshead speed v , the maximum and the minimum K_{IC} values were taken as $K_{IC,max}$ and $K_{IC,min}$ respectively. The Avrami-exponent n and constant b were then determined by plotting $\ln(v)$ vs $\ln\ln((K_{IC,max} - K_{IC,min}) / (K_{IC,max} - K_{IC}))$ (fig. 6). This yields a straight line with slope n and intercept $\ln(b)$.

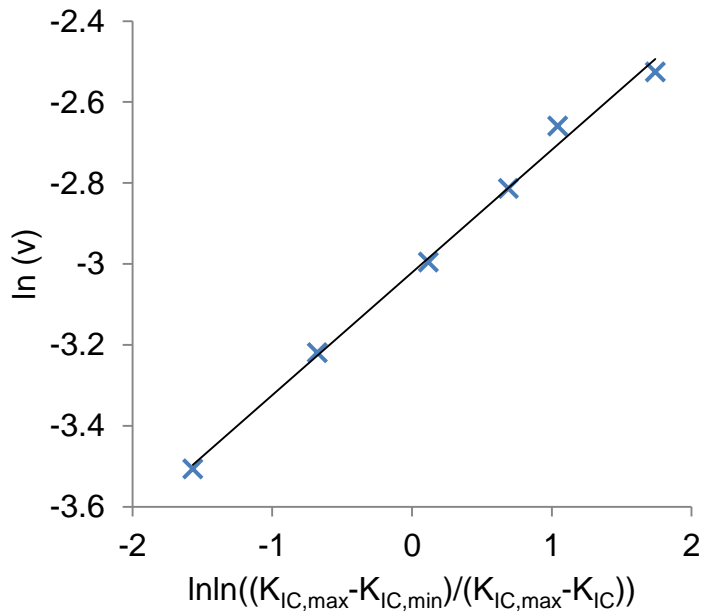


Fig. 6: Plot of $\ln \ln \left(\frac{K_{IC,max} - K_{IC,min}}{K_{IC,max} - K_{IC}} \right)$ versus $\ln(v)$ to determine the Avrami exponent n and the constant b of the Johnson-Mehl-Avrami-Kolmogorow-Equation (Material: Lava™ Plus)

To reduce the period of stable crack growth during the tests as much as possible, the testing mode was changed from displacement-controlled to load controlled. Typical data, obtained from these tests are illustrated in figs. 7 and 8. Fig. 7 shows that depending on the loading rate, the test duration may be very short: at high rates, it takes only few seconds to reach the maximum load. It is remarkable that initially, the displacement increases linearly with increasing load. At loads between 40 and 50 N, the displacement-rate gradually increases, i.e. the crosshead speed increases in order to keep the loading rate constant. This event can be correlated with the onset of stable crack-growth, which increases the compliance of the sample [23]. The additional crosshead acceleration shortens the period of stable crack growth. Fig. 8 shows that the load increase is a linear function of the displacement until the onset of stable crack growth at around 40 N. The maximum load, from which the K_{IC} value is determined, was reached at a slightly smaller displacement, and the maximum load is higher at higher loading rate.

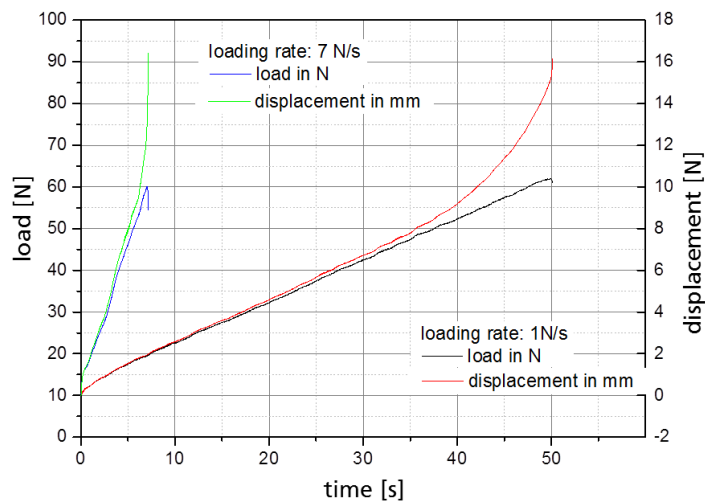


Fig. 7: Typical data of load-controlled CNB tests: Load and displacement as a function of testing time (Material: ZY).

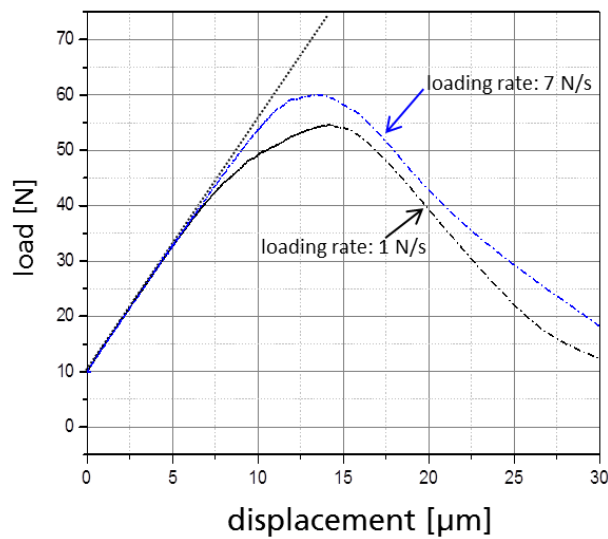


Fig. 8: Representative load-displacement curves obtained at two different loading rates (Material: ZY).

An overview of the K_{IC} results obtained by CNB tests at different loading rates is given in the following chart (fig. 9). The tests were conducted at two different labs using electromechanical testing machines (Instron, lab1 - Fraunhofer IWM, Freiburg; Zwick, lab2 - Institut Straumann AG, Basel). This overview shows that there is a systematic difference between the values obtained at low loading

rates (1 N/s) and higher loading rates (above 6 N/s): In accordance with the results of the displacement-controlled tests (fig. 5) the values are significantly higher, when high loading-rates are used.

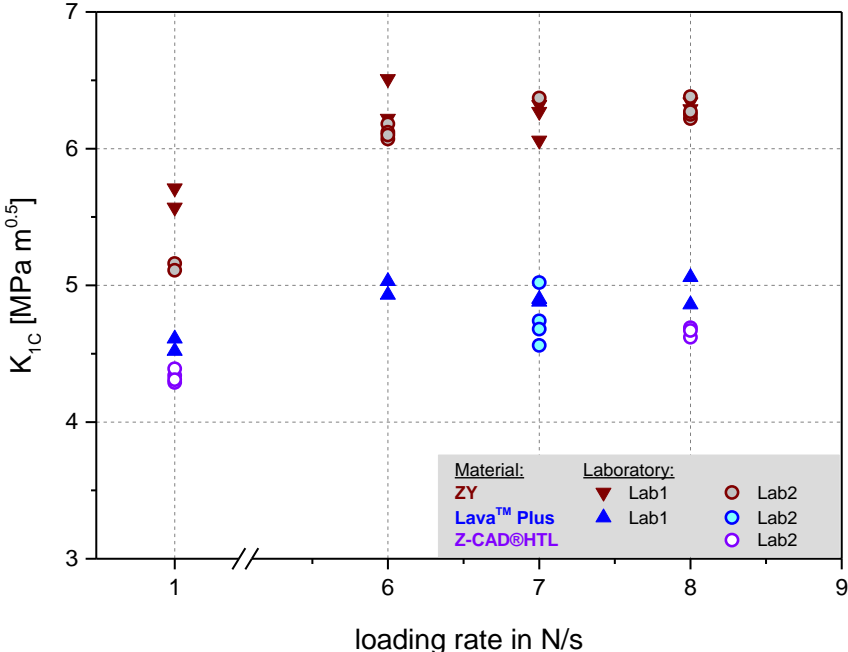


Fig. 9: K_{IC} values of three selected materials obtained at different loading rates in two different labs.

In table 2 and fig. 10 the results of CNB tests of four different materials are compared with the corresponding K_{IC} values obtained by SEVNB. I was observed that there are some differences between these values and that the standard deviation of the SEVNB values are considerably higher than those of the CNB values.

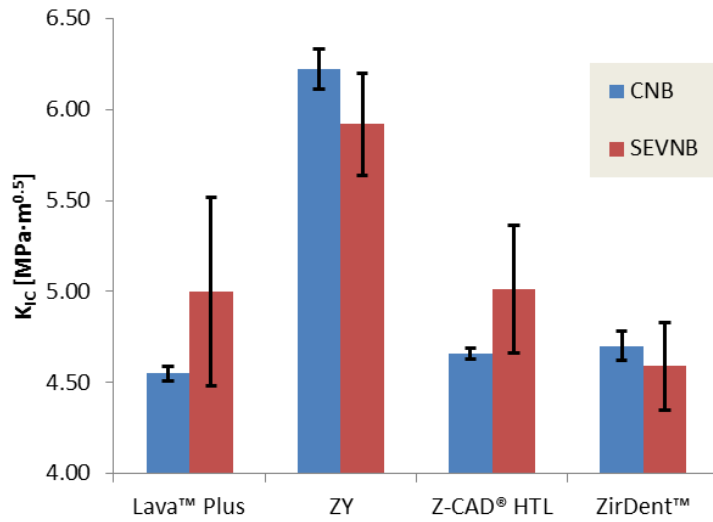


Fig. 10: Comparison chart of K_{IC} values of different materials determined by CNB (for loading rates ≥ 6 N/s) vs. SEVNB.

For comparison, some CNB tests of Z-CAD® HTL were conducted at displacement rates of 0.05 mm/min using silicone oil to prevent stress crack corrosion during the tests (table 3). The resulting K_{IC} of 4.63 ± 0.12 MPa·m^{0.5} is in the same range as obtained at high loading rates without silicone oil (4.66 ± 0.03 MPa·m^{0.5}). However, with the use of silicone oil at tests with loading rates of 1 N/s the obtained K_{IC} values (4.76 ± 0.05 MPa·m^{0.5}) are slightly higher.

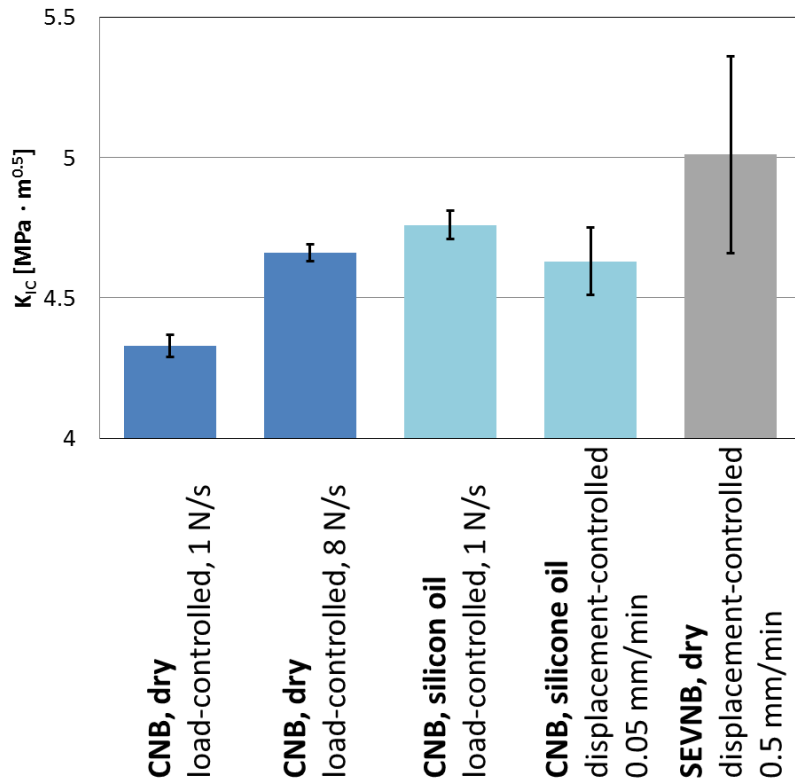


Fig. 11: Comparison chart of K_{IC} values of Z-CAD[®] HTL determined via CNB with different loading modes and with and without silicone oil.

Table 2: Average Values and Standard Deviations of K_{IC} values measured by CNB (for loading rates ≥ 6 N/s) and SEVNB.

Material	$K_{IC, CNB}$ [MPa·m ^{0.5}]	$K_{IC, SEVNB}$ [MPa·m ^{0.5}]
Lava [™] Plus	4.55 ± 0.04	5.00 ± 0.52
ZY	6.22 ± 0.11	5.92 ± 0.28 *
Z-CAD [®] HTL	4.66 ± 0.03	5.01 ± 0.35
ZirDent [™]	4.70 ± 0.08	4.59 ± 0.24

* notch width < 6 μ m

Table 3: K_{IC} values of Z-CAD® HTL obtained at different test conditions.

Condition (Test method, environment, loading type and rate)	$K_{IC, CNB}$ [MPa·m ^{0.5}]
CNB, dry, load-controlled, 8 N/s	4.66 ± 0.03
CNB, dry, load-controlled, 1 N/s	4.33 ± 0.04
CNB, silicone oil, load-controlled, 1 N/s	4.76 ± 0.05
CNB, silicone oil, displacement-controlled, 0.05 mm/min	4.63 ± 0.12
SEVNB, dry, displacement-controlled, 0.5 mm/min	5.01 ± 0.35

Discussion

The first results of the CNB tests (see figs. 4 to 8) revealed that stable crack growth effects significantly affect the measured K_{IC} values. Since it is known [5-8] that most Zirconia materials are prone to subcritical crack growth caused by stress crack corrosion and microstructural degradation at the crack tip, it might be difficult to get realistic K_{IC} values by CNB tests. The dotted line in fig. 5 marks the upper cross head speed limit as given by DIN EN 14425-3, which still is in the range of systematically varying K_{IC} values. In consequence, the measurement of plausible K_{IC} values within this range of the recommended crosshead speeds is not possible. The corresponding SEVNB value for this material is 5.01 MPa·√m. Therefore the test parameters were modified to shorten the period of stable crack growth as much as possible. It was shown in fig. 9 for three different materials that in load-controlled tests with loading rates beyond 6 N/s the obtained K_{IC} values were on a constant level.

To obtain more realistic K_{IC} data it was decided to significantly increase the loading rate and to change the testing mode from displacement-controlled (constant crosshead speed) to load-controlled (constant load increase), because the load controlled mode offers the opportunity to increase the loading rate under a more controlled way. Moreover, in the moment of crack growth that causes a higher compliance of the bend bars, there is an instant increase of the crosshead speed to keep the loading rate constant (see fig. 7). Therefore this test mode causes significantly faster crack growth and thus reduces the influence of subcritical crack growth.

As mentioned in the introduction, the K_{IC} is strongly affecting the lifetime-performance of dental implants and restorations under biodynamic loading. The knowledge about a realistic K_{IC} is therefore of crucial importance in the development of new materials and designs. Especially if considering minimal invasive approaches.

The comparison of the corresponding K_{IC} values obtained by SEVNB and CNB (s. table 2 and fig. 10) shows that the CNB values generally follow the values gathered by SEVNB method. However, no systematic deviation trend can be detected. Due to the fact that the notch tip diameter has a significant influence on the measured K_{IC} value in SEVNB tests [30], there is a remaining uncertainty on the reliability of these data. It is important to note that the standard deviation of the results of the CNB tests is clearly smaller than in the case of SEVNB tests, which was seen as a clear advantage of the CNB method. Further comparison of test results obtained at different test conditions (see fig. 11 and table 3) showed that load-controlled test using a high loading rate (e.g. 8 N/s) yield realistic and reliable values that are even in the range of K_{IC} values measured with the use of silicone oil.

Conclusions: Benefits and drawbacks of CNB method versus SEVNB

The feasibility of CNB tests to measure fracture toughness of Zirconia-based ceramics was investigated. It was observed that there is a strong influence of subcritical crack growth, which leads to lower K_{IC} results at low loading rates. Increasing the loading rate and changing from displacement-controlled tests to load-controlled tests, the obtained results were well reproducible and the statistical scatter of the results was small. Providing an inert testing atmosphere additionally enhances the values.

The benefits of this test method in comparison to SEVNB testing are

- + There is a good comparability of materials and results obtained using different testing machines.
- + The scatter of the results for CNB is significantly smaller than for SEVNB.

+ The influence of sample preparation is small, because in this test there is a period of stable crack growth, during which the K_{IC} value is determined.

However,

- stable crack growth is influenced by subcritical crack growth occurring in Zirconia ceramics. Thus, high loading rates or/and inert testing atmosphere must be used.
- there were few tests, at which the stable crack-growth was not reached. In this case the load increases to higher values and the test ends with a sudden fracture of the sample. However, this kind of failure is easy to discern.
- CNB tests are not yet standardized appropriately. Besides, there are significant differences between the test procedures prescribed in DIN EN 14425-3 [6] and ISO 24370 [7]. The rate limits given in the DIN standard are not suitable for zirconia ceramics. Instead of claiming an upper limit of the load rates, we recommend to use a lower loading rate limit for zirconia-based ceramics or to use silicone oil as described in the ISO 24370.

The chevron notch cut requires accurate preparation equipment. But once the preparation methodology has been established, the effort for sample preparation will be low.

The obtained results of the CNB tests slightly differ from the results of SEVNB tests, but so far we have not observed a systematic deviation. SEVNB is the broadly used standard method. However, the benefits of this test method, as described above, show that CNB tests yield well-reproducible results with low scatter, despite its faster and easier preparation procedure. Therefore, once there will be a sufficient database to compare the K_{IC} results of Zirconia that are obtained by CNB, this method will be favorable and very useful for both giving reference values for K_{IC} on data sheets as well as for quality control.

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