Ceramics-Silikáty 68 (4), 516-526 (2024)
www.ceramics-silikaty.cz doi: 10.13168/cs.2024.0050

MECHANICAL CHARACTERISATION OF CAD/CAM DENTAL MATERIAL AND NATURAL TEETH

#MÁRIA KOLÁŘOVÁ, ALEXANDRA KLOUŽKOVÁ, JANA KRÁLÍKOVÁ

Department of Glass and Ceramics, University of Chemistry and Technology Prague, Technická 5, 166 28 Prague

#E-mail: maria.kolarova@vscht.cz

Submitted May 15, 2024; accepted July 11, 2024

Keywords: Fracture toughness, Dental ceramics, Vickers indentation fracture test, Mechanical testing, Leucite

The aim of this study was to investigate the hardness and the fracture toughness of dental ceramic materials by means of the Vickers indentation technique. One type of most common commercial restorative dental ceramic materials fabricated using CAD/CAM system leucite-based glass-ceramics was selected for study and comparison with laboratory synthetized leucite composites and natural teeth. Obtained results showed that the measurement of hardness and fracture toughness depends on the used conditions (the indenter geometry, the indentation load and the duration of maximal force), morphology of polished surface, type of crack system developed or measured indentations and of course on the empirical equation (Casellas, Shetty et al., Niihara et al., Lankford, Niihara, Laugier, Nakahira) used for calculation of fracture toughness. The results proved that the values of Vickers hardness of our laboratory prepared leucite composites (7.76 – 8.92 GPa) are very close to the values of commercial IPS Empress blocks. The Vickers indentation test was verified as a suitable method for evaluation of fracture toughness of small sized samples of leucite-based dental composites when using low indentation load of 9.807, 19.61, 29.42 and 39.23 N and indication of Palmqvist cracks.

INTRODUCTION

Several materials, i.e. restorative materials, dental cements and bonding agents are very promising materials in the dental market. Contemporary and effective dental materials processed by computer-aided-design and computer-aided-manufacturing (CAD/CAM) technology are one of the most commonly used restorative materials in dental implantology together with materials used for 3D printing or tissue engineering. The variety of modern CAD/CAM silicate ceramics that have been expanding for several decades are commonly divided on the basis of differences in microstructure, which means variations in amount or type of crystalline and amorphous phases [1-6]. Currently, among the most widely used all-ceramic types in dentistry are glass-ceramics and zirconia oxide ceramics [8-10]. Primarily silica-based glass matrix of glass-ceramics is strengthened by a crystalline phase, mainly lithium disilicate or leucite. Dental glass-ceramics vary in composition but always contain a glassy phase and one or more embedded crystalline phases, the amount of which ranges from 30 to 70 % (the proportion of the crystalline phase can range from 0.5 to 99.5 %). Reinforcement of the glass matrix with crystals provides excellent aesthetic quality, allows for etching and cementation, and ultimately enhances resistance to crack propagation [2, 9-13]. Clinical indications of glass-ceramics include inlay, onlays, overlays, veneers, single crowns and fixed partial dentures to restore teeth, replace previous faulty fillings or cover discolorations or defects [5, 7-9, 14, 15]. The properties of dental materials are determined according to ISO standards [16]. The optical, chemical, and mechanical properties should be comparable to those of natural teeth, which is a crucial requirement of all standards. The degree of difference in individual parameters and properties depends on the type of application but above all on the character of the required property. For example, chemical durability should be higher for restorative materials compared to natural teeth. On the contrary, mechanical properties such as hardness (H), fracture strength (σ), fracture toughness (K_{IC}), and wear resistance should be within certain limits so that no unwanted wear of the enamel of the opposite teeth will occur.

The aim of this study was to investigate and correlate mechanical properties, specifically hardness and fracture toughness, of dental leucite-based glass-ceramic systems and laboratory prepared leucite composites. Partially the study was focused on determination of appropriate measurement conditions (indenter geometry, indentation load and maximum force duration), selection of suitable mathematical model for fracture toughness calculation. An additional objective was to determine the hardness of the natural tooth enamel and dentin, considering variations due to damage and age.

REVIEW

Mechanical properties in addition to biocompatibility are key properties that significantly affect their long-term durability and ensure the functionality of biomaterials. Fracture toughness and hardness are some of the most important mechanical properties of biomaterials that should be carefully studied and evaluated before implantation [17]. The clinical potential of all--ceramic restoratives confides in mechanical properties such as flexural strength, hardness and fracture toughness, which show the response to loading forces and crack propagation [18-24].

The hardness (H), which is defined as the resistance to localised plastic deformation, is one of the most frequently measured mechanical properties of brittle materials like ceramics or glass. The main advantage of conventional micro- or nano-hardness measurements using Vickers or Knoop diamond indenter is that this method does not require a preparation of a large number of samples of precisely defined size and shape, and could be used for measurement of dental ceramics. The indenter with a certain force is applied for a specified period. Tests of mechanical properties such as strength and hardness have yielded favourable results across a wide range of applications but have significant limitations in predicting clinical performance. These limitations can be overcome by measuring fracture toughness [25, 26].

Therefore, the most suitable property for characterisation of dental all-ceramic materials is the fracture toughness [27, 28]. Fracture toughness is the value of fracture resistance and is defined as the critical value of stress intensity level in "mode I" or the opening mode which refers to the existence of unstable crack propagation, fracture initiation or failure [18, 19, 21, 29-31]. The fracture toughness (K_{IC}) of dental restorative glass ceramics vary in values that rarely exceed 5 $MPa \cdot m^{1/2}$. Fracture toughness measurement methods could be divided into conventional fracture mechanism measurement techniques, which require specific sample preparation and sophisticated high precision equipment, and techniques based on a defined indentation fracture test [29-37]. The conventional techniques require special samples with artificially created cracks, the so-called precracks, which are introduced by a diamond saw and sharpened by e.g. a razor-blade, bridge-anvil or thermal shock. According to ISO 6872, pre-cracked samples are loaded in a three- or four-point bending test arrangement. The most common and frequently used methods for measurement of brittle ceramics are SENB (Single Edge Notch Beam), SEVNB (Single Edge V Notch Beam), SEPB (Single Edge Precracked Beam), CNB (Chevron Notched Beam) or SCF (the surface crack in flexure), which use Knoop indenter to make a small pre-crack [16, 20, 33, 38-45].

Recently, increased attention has been given to indentation techniques involving samples with small indentations created by a hardness tester indenter. The value of stress intensity factor (K_{Ia}), corresponding to the interruption of the propagating crack, is applied in the indentation fracture (IF) methods [21, 22, 29]. IF are methods based on the measurement of the length of cracks on the polished surface of the sample. The equality $K_{Ia} = K_{IC}$ is applied for the determination of the fracture toughness of ceramics. Vickers indenter and the so-called Vickers indentation fracture toughness test (VIF) are the most commonly used method, which is based on the formation of radial cracks in the diagonals of the indentation under a sufficiently high load. For the mechanical characterisation of metals, glass, polymers, composites, coating, biomaterials, hard bio-logical tissues, etc. is the VIF technique increasingly applied [36, 37, 46-48]. The KIC values could be calculated using the model formulas as a function of crack length, Young´s modulus of the material tested and Vickers hardness calculated. Palmqvist first developed the concept of obtaining the fracture toughness value of brittle materials by means of the indentation method. A Vickers diamond indenter was used to describe the relationship between the fracture process and other parameters (e.g. hardness) [49]. The Palmqvist theory was after a few decades followed by other studies, who established several relations. Then, the VIF method was implemented as a fracture toughness testing method. Evans and Charles suggested several equations and proved that Vickers indentation could be applied to characterising the fracture of different materials with properties of hardness, toughness, and Poisson´s ratio that range at least between: 1 – 70 GPa, 0.9 – 16 $MPa \cdot m^{1/2}$ and 0.2 – 0.3 [50]. Their measurement claimed that fracture toughness data obtained by indentation achieved high precision with knowledge of Young´s modulus. The equations generalised by Evans and Charles are valid for Palmqvist or median (half-penny) cracks. Figure 1 shows the difference between two types of crack system, when at lower loads Palmqvist cracks are usually formed at lower loads and half-penny or radial-median cracks predominate at higher applied loads. Several authors followed up and developed alternative relations between K_{IC}, the nature of the crack, the load values, the lengths of the diagonal indentation, the Vickers hardness value of the material, the elastic modulus and the crack length from the centre of the indentation. So, it was not possible to establish a single formula for the calculation of K_{IC} of various materials because of the difference in brittle behaviour of the materials and different types of cracks. The profile of individual cracks could be distinguished according to the ratio of the crack length (c) and the indenter radius (a). The commonly used equations for Palmqvist cracks and median cracks are listed in Table 1.

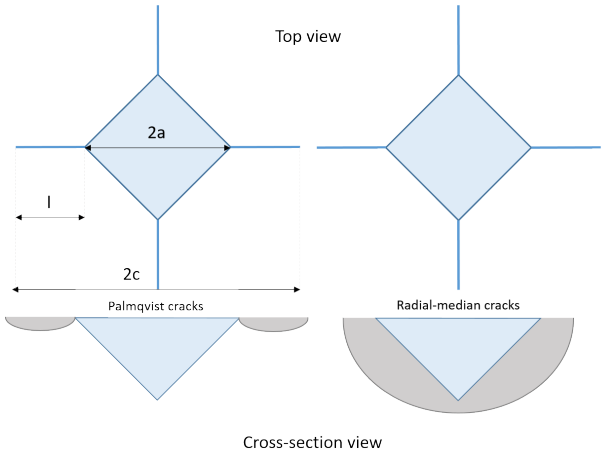


Figure 1. Schematic Vickers indentation radial crack system morphologies.

EXPERIMENTAL

Materials

Chemical composition of CAD/CAM blocks, purity of input powders for synthesis and glass matrix was identified using X-ray fluorescence sequential WD-XRF spectrometer ARL Performix (Thermo ARL, Switzerland). The acquired data were evaluated by means of UniQuant software package implemented in Oxsas software.

Phase composition and crystallinities of samples were identified and calculated using X-ray diffractometer PANalytical X´Pert^3 Powder (Malvern Panalytical, Netherlands) by means of parafocusing Bragg-Brentano geometry and CuKα radiation over the angular range of 5 – 80° 2Theta. Data evaluation was performed by Panalytical High Score Plus 4.1 software package and semi-quantitative analysis was calculated using Reference Intensity Ration (RIR) values form reference database PDF4$^+$.

The microscopic observations of samples microstructure and indentation cracks were performed using

Table 1. Empirical equations for evaluation of fracture toughness.

Author	No.	Equation	Type of crack
Casellas [51]	1	$K_{IC} = 0{,}024\frac{F}{c^{1{,}5}}\left(\frac{E}{HV}\right)^{0{,}5}$	Palmqvist
Palmqvist [50]	2	$K_{IC} = 0{,}0028HV^{0{,}5}\left(\frac{F}{T}\right)^{0{,}5}$	Palmqvist
Shetty et al. [52]	3	$K_{IC} = 0{,}0319\frac{F}{al^{0{,}5}}$	Palmqvist
Niihara et al. [19]	4	$K_{IC} = 0{,}0089\frac{F}{al^{0{,}5}}\left(\frac{E}{HV}\right)^{\frac{2}{5}}$	Palmqvist
Niihara [53]	5	$K_{IC} = 0{,}0122\frac{F}{al^{0{,}5}}\left(\frac{E}{HV}\right)^{\frac{2}{5}}$	Palmqvist
Laugier [54]	6	$K_{IC} = 0{,}0143\left(\frac{E}{HV}\right)^{\frac{2}{3}}\left(\frac{a}{l}\right)^{0{,}5}\frac{F}{c^{1{,}5}}$	Palmqvist
Nakahira [47]	7	$K_{IC} = 0{,}035\left(\frac{l}{a}\right)^{-0{,}5}\left(\frac{Ek}{HV}\right)^{\frac{2}{5}}\frac{HVa^{0{,}5}}{k}$	Palmqvist
Anstis [32]	8	$K_{IC} = 0{,}016\frac{F}{c^{1{,}5}}\left(\frac{E}{HV}\right)^{0{,}5}$	Radial-median
Laugier [55]	9	$K_{IC} = 0{,}0095\left(\frac{E}{HV}\right)^{\frac{2}{3}}\frac{F}{c^{1{,}5}}$	Radial-median
Evans and Charles [50]	10	$K_{IC} = 0{,}0752\frac{F}{c^{1{,}5}}$	Radial-median
Tanaka [56]	11	$K_{IC} = 0{,}0725\frac{F}{c^{1{,}5}}$	Radial-median
Niihara, Morena a Hasselman [57]	12	$K_{IC} = 0{,}0309\left(\frac{E}{HV}\right)^{\frac{2}{5}}\frac{F}{c^{1{,}5}}$	Radial-median
Blendell [58]	13	$K_{IC} = 0{,}0303(HVa^{0{,}5})\left(\frac{E}{HV}\right)^{\frac{2}{5}}log\left(8{,}4\frac{c}{a}\right)$	Both types
Lankford [47]	14	$K_{IC} = 0{,}0782(HVa^{0{,}5})\left(\frac{E}{HV}\right)^{\frac{2}{5}}\left(\frac{c}{a}\right)^{-1{,}56}$	Both types

Note: F is applied load (N), HV is Vickers hardness (GPa), E is Young´s modulus (GPa), c is the crack length from centre of the indentation (m), a is a half of the indentation diagonal length (m), l is crack length measured from vertices of the indentation (m), k is a material constant and T is the total crack length (m).

The aim of this study was focused on characterisation of mechanical properties of commercial and laboratory leucite-based restorative materials. Specifically, the study sought to determine optimal measurement conditions and identify the most accurate empirical equations for use with the VIF method.

the Olympus BX51 optical microscope (Olympus, Japan), scanning electron microscope Vega III (Tescan, Czech Republic) and laser confocal microscope LEXT OLS5100 (Olympus, Japan).

Young´s modulus of laboratory composites was measured using RDFA 23 system (IMCE, Belgium) that includes automated impulse excitation measurements at room temperature. Cylindrical rods were prepared by pouring into the mould and firing them in a vacuum furnace. The resonant frequencies of the samples were used for the calculation of Young´s modulus.

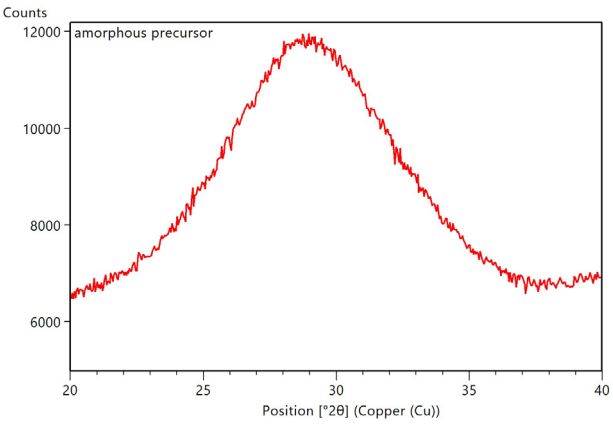
Sample preparation

The samples were divided into two groups that were evaluated and compared with each other. The first part of the experiments was performed on commercial CAD/CAM ceramic blocks IPS Empress (Ivoclar Vivadent). The IPS Empress CAD ingots contain leucite crystals (diameter 1 – 5 μm) which are homogeneously distributed in a glass matrix (35 – 45 vol. %) [13]. The detailed specification of the IPS blocks investigated listed in the manufacturer´s scientific documentation is in Table 2 together with the XRF results. IPS Empress CAD prism-shaped samples in the translucency level HT were cut from prefabricated ingots into 5 mm thick slices using cutting and grinding device Geoform 102 (Metkon Instruments Inc., Turkey). The samples were ultrasonically cleaned and dried. In order to measure the mechanical properties, it was necessary to impregnate them and subsequently pour by epoxy resin. After curing, the samples were ground and polished using a Forcipol-TS polisher equipped with a Forcimat-TS head unit (Metkon Instruments Inc., Turkey) using diamond suspensions to achieve a perfectly polished surface of plane-parallel samples.

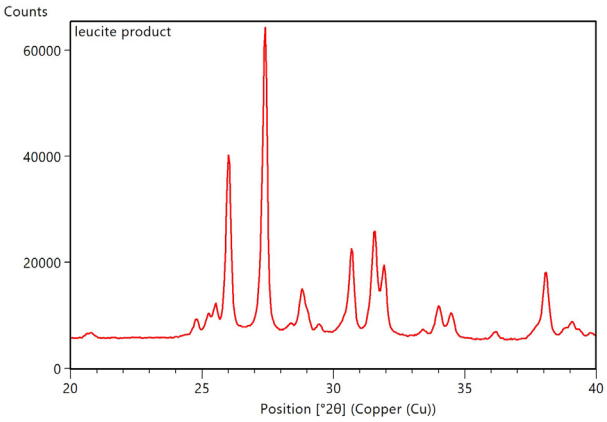
Table 2. Composition of commercial IPS blocks in wt. %.

Component	Technical data	XRF analysis
SiO_2	60 – 65	64.46 ± 0.40
Al_2O_3	16 – 20	16.92 ± 0.20
K_2O	10 – 14	9.39 ± 0.2
Na_2O	3.5 – 6.5	5.94 ± 0.1
Other oxides	0.5 – 7	2.74 ± 0.3
Pigments	0.2 – 1	0.25 ± 0.14

The second group of samples consisted of laboratory-prepared leucite composites, which contained tetragonal leucite (40 wt. %) and a glassy matrix (60 wt. %). Crystalline phase leucite was synthesised by a hydrothermal method from input powders (silicon oxide POCH Gliwice, aluminium powder Aipra and potassium hydroxide Lach-Ner s.r.o.) and the precursor was subsequently calcined in a laboratory furnace [59, 60]. The precursor powders were prepared from sols by mixing the silicate and the aluminium solution, which was made by stirring of the aluminium powder in the KOH solution with molarity 3 ml·L^{-1}. Calcined product (24 hours at 1100 °C) contained a tetragonal leucite as is presented in Figure 2.



a) amorphous precursor



b) leucite product

Figure 2. XRD pattern of amorphous precursor and calcined product.

An amorphous glass matrix with the composition specified in Table 3 was melted in a platinum crucible for 2 hours at 1500 °C in a laboratory furnace. Fused glass frit was crushed and ground using a planetary mono mill Pulverisette 6 (Fritch, Germany) and an average particle size of ca. 26 μm was obtained. The pre-prepared components (t-leucite with kalsilite and glass matrix) were homogenised with a small amount of water (5 wt. %) in the Reax 2 overhead shaker (Heidolph, Germany) with corundum balls for 1 hour. The mixture was press into the bars of 12.7 mm by uniaxial hydraulic press Silfradent using the pressure of 250 MPa. The bars were fired in a laboratory vacuum furnace Classic with a regime determined from SciGlass 6.7 software. The heating rate was 30 °C·min^{-1} up to the final temperature of 1000 °C with a dwell time of 1 – 3 min, the optimal dwell time was checked using an optical microscope to obtain optimal sintering of composite samples.

Table 3. Composition of glass frit prepared in laboratory conditions determined by XRF analysis.

Oxide	SiO_2	Al_2O_3	K_2O	Na_2O	CaO	B_2O_3	ZrO_2
wt. %	65.85	15.42	7.94	5.38	2.54	1.80	1.03
Est. Err.	0.53	0.18	0.14	0.11	0.12	-	0.11

The additional set of samples was prepared from natural teeth of various location in the mouth, especially molars and incisors, and age i.e. moderate wear. Mechanical properties of teeth different age were examined for both dentin and enamel of natural teeth samples.

The samples of composites and natural teeth were prepared for the hardness and fracture toughness test in a similar way to commercial IPS Empress materials according to petrographic process standards.

Hardness and fracture toughness testing

High-quality polishing of the sample surface is required for measurement of Vickers hardness and determination of fracture toughness by Vickers indentation fracture test (VIF). The plane parallelism and smoothness of the surface of samples for measurement must be of high quality to gain results of high assurance. Hardness testing was performed using the Falcon 611 (Innovatest, The Netherlands) hardness tester with Vickers and Knoop diamond indenters, and data were evaluated using Impression software with four optical objectives. The indentations of the commercial (IPS Empress) and laboratory samples (t-leucite in glassy matrix) were measured using a Vickers square-based sharp pyramidal diamond indenter at a maximum load of 4.903 N (HV0.5) for 10 s. Natural enamel have to be measured at maximum load of 2.942 N (HV0.3). For each sample, 10 or 15 measurements were performed. Three different samples of each type of samples were measured so that the maximum of 30 – 45 measurements were averaged.

The determination of the fracture toughness was carried out using the Vickers indentation fracture method (VIF) on the same set of samples with preciously polished surfaces, which were used for the hardness measurements. High indentation loads ranging from 9.807 (HV1) to 39.23 N (HV4) were applied for 10 s. Figure 3 shows typical cracks developed from the vertices of the Vickers indentation. Higher loads, leading to distortion of the indentation zone, initiated Palmqvist cracks. The results obtained from different indentation loads were compared - mainly indentation lengths and crack lengths were correlated. Indentations were formed in laboratory conditions using the same hardness tester and Vickers indenter as in hardness evaluation. The diagonals and cracks were measured using integrated optical instruments using Impression software. Each sample was tested ten times per load and the measured length was used for the determination

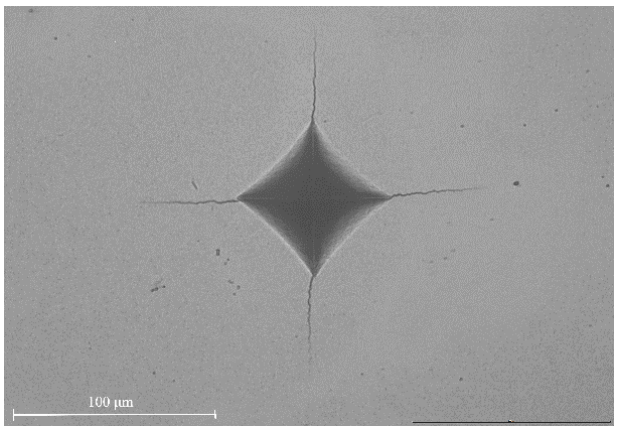


Figure 3. Micrograph of Vickers indentation with developed under load of 19.61 N (HV2).

of fracture toughness using selected equation models (Equation no. 1, 3, 4, 5, 6, 7 and 14 from Table 1). Due to the difficulties in preparing samples of specific dimensions to measure Young's modulus using impulse excitation technique (IET) or from the linear elastic region of the uniaxial stress-strain curve obtained by the standard 3- or 4-point bending test, Young's moduli were defined from scientific documentation of commercial ISP Empress [13]. Young´s modulus of laboratory prepared composites was measured on cylindrical rods using IET method.

RESULTS AND DISCUSSION

Composite characterisation

Laboratory prepared leucite composite was characterised using XRD analysis to identify crystalline phase. The XRD patterns proved that only tetragonal leucite is included in samples after firing in a vacuum furnace. The two most intensive peaks of t-leucite at positions 25.97 and 27.32° 2Theta are shown on the XRD patterns in Figure 4.

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Vickers hardness test

Results of Vickers hardness (HV) obtained from at least 30 measurements per each sample type were statistically evaluated and the values of the commercial and laboratory samples were compared.

The typical indentation at 4.903 N load for 10 s is presented in Figure 5 and the results of the microhardness test are shown in Figure 6. Laboratory

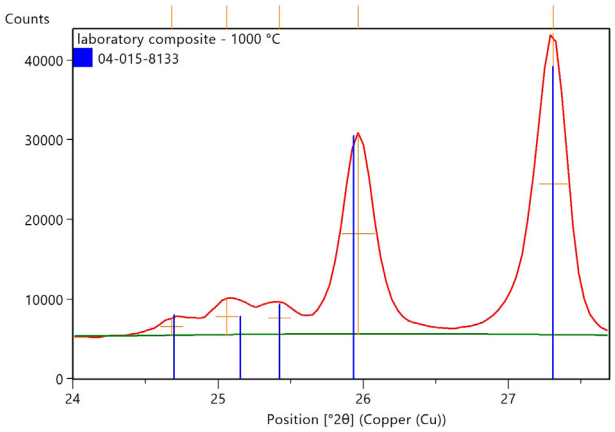


Figure 4. XRD patterns of composite samples prepared from calcined product with blue lines which are stick pattern no. 04-015-8133.

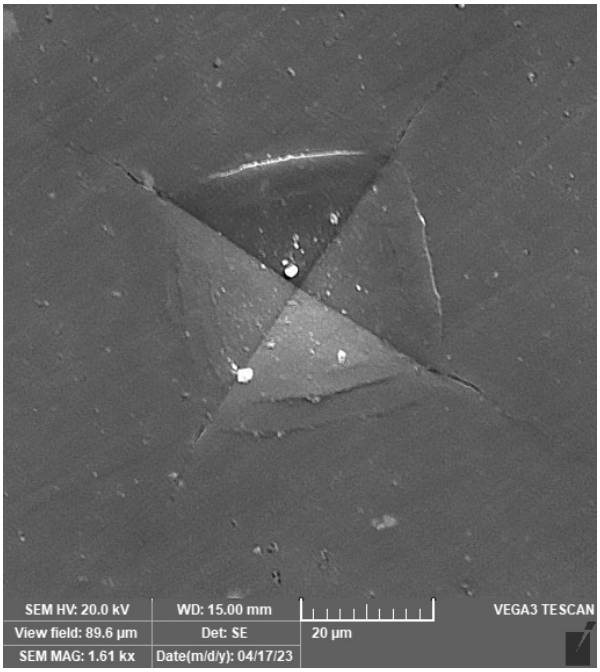
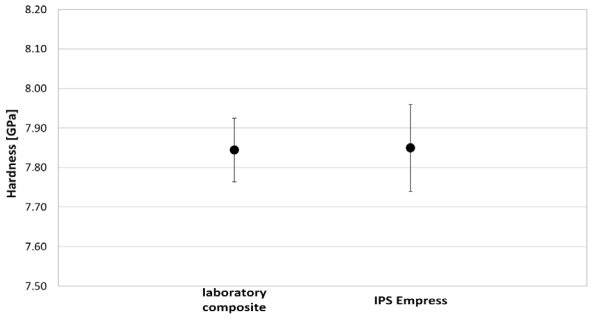


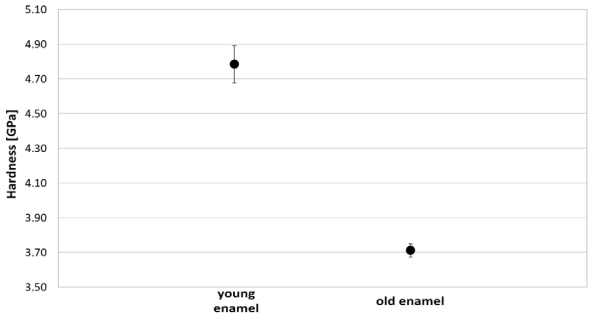
Figure 5. SEM image of Vickers indentation in IPS Empress.

composites made from calcined products exhibit hardness values comparable to the commercial IPS Empress sample, while also exhibiting reduced variation among individual data points.

Finally, both types of all-ceramic restorative samples were compared with the values obtained for dentin and enamel of natural teeth. Figure 6 shows that the values of natural enamels reach almost half the values of the both types of all-ceramic restorations. The lower values of HV of natural teeth are caused by lower load of the measurement. The main point of this comparison is that confidence interval of 95 % evaluated by Weibull statistics has a decreasing trend in a row IPS Empress, laboratory composite, young

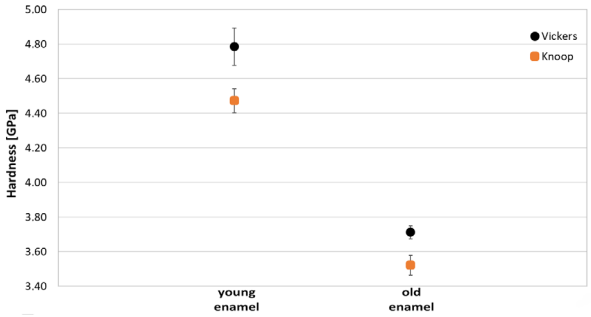


a) laboratory composites and IPS Empress

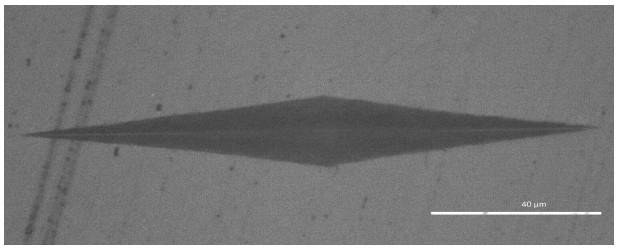


b) young and old natural teeth enamel

Figure 6. Comparison of Vickers hardness.



a) hardness values



b) micrograph of Knoop indentation

Figure 7. Vickers and Knoop hardness of natural teeth.

enamel and old enamel. So the higher value of hardness does not mean the higher reliability, which is not consistent with the value of the hardness.

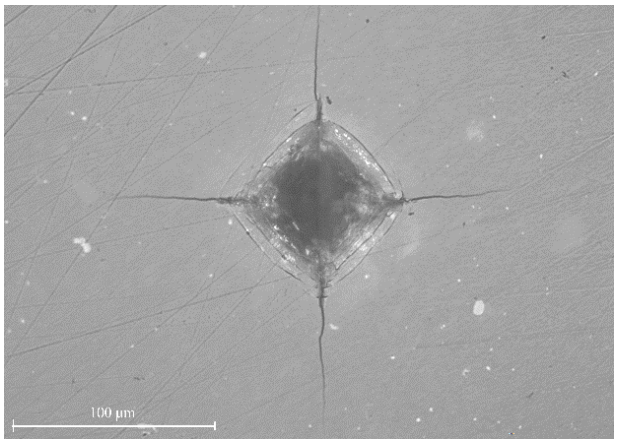
An additional goal of evaluation of natural enamel and dentin and the impact of ageing on hardness was performed by comparison of Vickers and Knoop hardness. Figure 7 shows the correlation of Vickers

and Knoop hardness of young and old enamel. The data revealed a clear trend of decreasing hardness with increasing age, likely due to demineralisation and other aging factors. The overall average Vickers hardness for the young and old enamels was 4.78 ± 0.22 and 3.71 ± 0.08 GPa, respectively. The Vickers hardness test was also used to determine the hardness of natural dentin, which is significantly lower at 1.39 ± 0.04 GPa and in correlation with other studies [61]. However, its value is not crucial for the choice of dental restorative material. According to the data obtained, the use of the Knoop hardness measurement is more appropriate for evaluation of the hardness of natural teeth and the choice of suitable dental restoration materials. Knoop hardness values are more accurate when measuring anisotropic materials with a higher deviation in hardness due to the elongated shape of the indentation (Figure 7b). Knoop hardness measurements provide more precise data due to better determination of well-defined indentations at lower loads and at microscopic scale. Knoop hardness values for both groups were 5 – 6 % lower than Vickers hardness and reach 4.47 ± 0.12 and 3.42 ± 0.05 GPa. The main intention of using the Knoop hardness test for the identification of the hardness of natural teeth was to minimise the effect of layering.

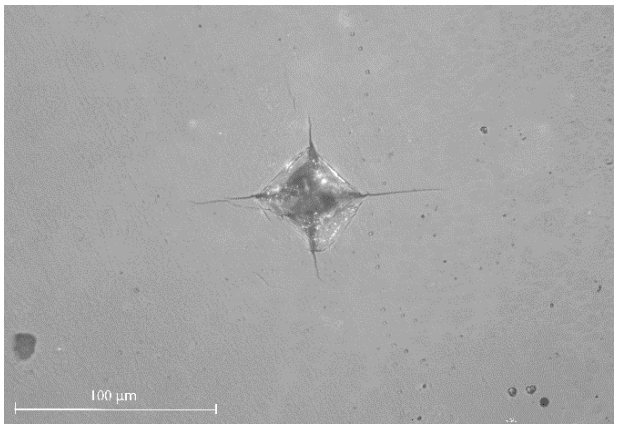
Fracture toughness determination by VIF test

The selected model equations commonly used for the calculation of K_{IC} listed in Table 1 were applied to determine the toughness of the indentation fracture of leucite ceramic composite samples. The test conditions were adapted to induce Palmqvist cracks using lower indentation loads of 9.807 N (HV1) for laboratory prepared samples and loads of 19.61 N (HV2), 29.42 N (HV3) and 39.23 N (HV4) for commercial samples. The Palmqvist type crack morphology is developed at the edges of the plastic indentation and elongates on the surface from the vertices of the indentation radially, as is shown in schematic in Figure 1 and in micrograph in Figure 8. In cross section view has an elongated semi-elliptical shape.

Graphical verification of the occurrence of a Palmqvist crack was performed by plotting the length of the crack as a function of the applied loads for both types of samples. The relationship is linear, points lie exactly on the line and the value of the correlation coefficient is positive. The appearance of radial-median crack type can be proven by sudden change in linear slope at higher loads, which was not examined. The type of induced crack is also affected by the value of the c/a ratio, which has been verified for all indentation loads used and did not exceed 2.5 for the Palmqvist crack system.



a) IPS Empress sample under 39.23 N load



b) laboratory prepared composite under 9.807 N load

Figure 8. Micrograph of Palmqvist crack propagation.

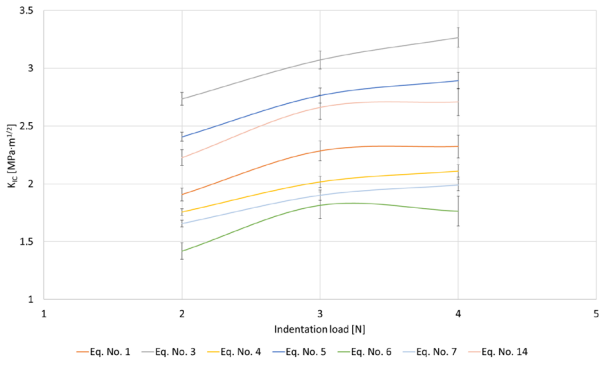


Figure 9. Fracture toughness dependence on applied indentation load of IPS Empress composite.

The dependence of the calculated fracture toughness on the applied load was used to evaluate a suitable empirical model. The results in Figure 9 show that the assessment of fracture toughness using the VIF technique strongly depends on the choice of the empirical model and, as already mentioned, the length of the crack and the precision of its measurement are related to this. Equations no. 4 and 7 were decided as the most relevant equations for determining

the fracture toughness of the IPS Empress composite with the constant linear dependence on the indentation load and the expansion of the indentation crack. Evident diversity was proved from the K_{IC} values calculated by the different equations using the same crack system. Equations no. 5 and 3 for determining the K_{IC} of leucite dental composites may also be considered applicable if necessary. However, these equations (no. 5 and 3) provide higher absolute values, which must be considered in the evaluation. The advantage of Niihara and Shetty et al.'s equations lies in their simplicity, allowing for fracture toughness calculations with knowledge of fewer material properties. However, for more accurate results, it is preferable to use more complex equations, such as equations no. 4 and 7, which incorporate additional factors and provide a more comprehensive analysis.

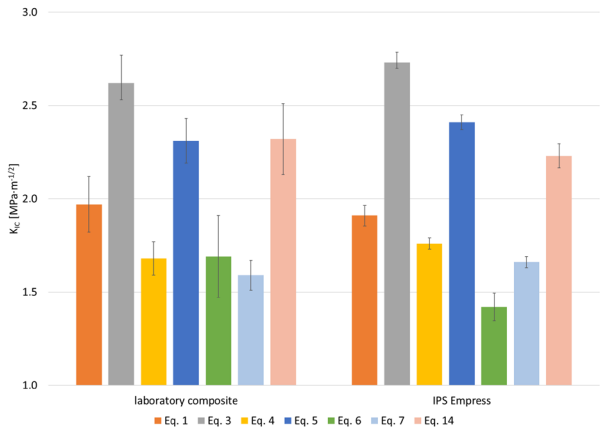


Figure 10. Comparison of KIC values of laboratory and commercial leucite composites.

Table 4. Selected empirical models suitable for leucite ceramics.

Composite type	K_{IC} [MPa·m$^{1/2}$]		
	Eq. no. 4	Eq. no. 5	Eq. no. 7
Laboratory composite	1.68 ± 0.18	2.31 ± 0.25	1.59 ± 0.17
IPS Empress	1.76 ± 0.06	2.41 ± 0.08	1.66 ± 0.05

Equations no. 4, 5 and 7 were selected as the most suitable for the evaluation of the fracture toughness of dental leucite composites, as the K_{IC} values of the commercial IPS Empress and laboratory prepared composite samples are most consistent and in the highest accordance. The K_{IC} value defined by the scientific documentation [14] for the commercial material IPS Empress is 1.3 MPa·m$^{1/2}$. However, these values given by the documentation were measured by the SEVNB method, in this work the Vickers indentation test (VIF) was used. Our analysis indicates that equation no. 7 (type of crack - Palmqvist, author of equation - Nakahira) for VIF calculation yields results that closely align with the manufacturer-provided SEVNB data.

CONCLUSIONS

Polished samples of dental leucite composites and natural teeth were prepared. Based on the tests performed under various conditions, we identified a suitable combination of indenters, indentation loads, and maximum force durations.

Following the results of investigated mechanical properties, it could be concluded that:

- The Vickers microhardness values of commercially and laboratory-prepared all-ceramic restorative samples can be compared to the hardness of natural teeth of different ages. A comparison of the measured data revealed that the reliability interval of the applied statistical test decreases in the following order: IPS Empress, laboratory composite, young enamel, and old enamel. The Vickers hardness of the all-ceramic restorative material ranges from 7.74 – 7.96 GPa, while that of dentin is 1.39 ± 0.04 GPa and enamel 4.78 ± 0.22 GPa.
- The evaluated Vickers and Knoop hardness of natural enamel and dentin of different age and state of mineralisation showed that hardness is one of the key properties of the material to choose a suitable restorative material. The Knoop hardness values for natural teeth were more accurate due to the elongated shape of the indentation. Knoop hardness values (4.47 ± 0.12 and 3.42 ± 0.05 GPa) were approximately 5 % lower compared to Vickers hardness values.
- The fracture toughness measurement proved that the VIF test is a useful method of evaluation of small volume samples and the estimated values definitely depend on the choice of empirical equation, accurate sample preparation, use of indentation load and type of crack system developed. The most appropriate equations for the evaluation of the fracture toughness of leucite dental composites were defined by Niihara et al. and Nakahira. The estimated fracture toughness using these model equations were calculated in the range of 1.50 – 1.84 and 1.42 – 1.71 MPa·m$^{1/2}$ respectively.

The study demonstrated that the selection of appropriate measurement conditions, which were defined within the study, is crucial for obtaining accurate, comparable, and reliable data.

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