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SPIE.

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Contribution of interferometry to Vickers indentation toughness determination of glass and ceramic glass

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Abstract. Cracking resistance or fracture toughness of brittle materials, such as optical glasses, can be determined by Vickers indentation, which allows generating cracks along the diagonals of the indent whose length depends of the loading rate. Subsequently, a relationship between the crack length and the corresponding indentation load is applied to calculate the indentation fracture toughness. As a result, the precision of the fracture toughness will depend on the accuracy of the crack length measurement. To minimize the uncertainties of this parameter, we propose its measurement more precisely by a profilometric method. On the other hand, as the glass surface roughness is recognized to have an important influence on the crack growth, different surface preparations are studied to minimize this roughness effect. In this work, four types of glasses are studied, namely Classical Crown K5, Borosilicate Crown BK7, Dense Flint SF2, and Ceramic Zerodur[®] glasses. The surface polishing is performed by using CeO₂ pellets, which allow obtaining a roughness value for R_a to be <10 nm. Vickers instrumented indentation tests, allowing the plot of the applied load as a function of the indenter displacement, are performed with various maximum applied loads of up to 90 N. Different relationships based on assumptions on the crack shape (half-penny/median or Palmqvist cracks) are then used to calculate the indentation fracture toughness. Additionally, to accurately measure the crack length, we present an original approach based on the analysis of the interferometry map obtained with an optical profilometer using a monochromatic wavelength. © 2019 Society of Photo-Optical Instrumentation Engineers (SPIE) [DOI: 10.1117/1.OE.58.3.034102]

Keywords: optical glass; indentation fracture toughness; Vickers indentation; interferometric measurement; polishing method.

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1 Introduction

The classical indentation technique has been first developed for determining the hardness of the materials. But for brittle materials, when using a Vickers indenter, cracks are often generated along the diagonals of the indent depending on the applied load. By connecting the crack length to the corresponding applied load, Palmqvist^{1,2} was one of the first authors in the 1960s to propose a relationship to characterize the cracking resistance based on the work required to produce a crack. Since then, a great number of formulas derived from the indentation test have been proposed to calculate the fracture toughness, K_{IC} , taking into account the crack shape that develops under the indent (half-penny/median or Palmqvist cracks). Ponton and Rawlings^{3,4} have realized an important review by collecting the relationships used to estimate the fracture toughness divided in two groups accordingly with the crack shape. Conclusively, for brittle materials, indentation technique is probably the most used technique for determining the fracture toughness⁵⁻¹⁶ for different reasons, namely, the indentation test is easy to perform, it requires no specific shape for the specimen, and it allows the characterization of local and bulk fractures. Unfortunately, this technique gives highly dispersive results, with errors reaching values >50%.¹⁷ As an example when considering the pioneer's work in Anstis et al.,¹⁸ the

proposed formula is established according to the hypothesis that median-type cracks is formed during the discharge, below the indenter, due to the action of the tension residual stresses which maintain the cracks in equilibrium at the value of force critical crack extension. Then the constant which appears in this relationship is calculated by comparing indentation and classical tests in fracture mechanics as double cantilever beam test. In this condition, the coefficient is given as $\pm 25\%$ of incertitude. Several reasons can be advanced to explain this dispersion: (i) the cracks are well developed and the crack length is higher than two times the half-diagonal of the indent, (ii) no chipping may occur, (iii) the original relation has been proposed not only for median cracks but also for materials presenting Palmqvist cracks as carbide-cobalt alloys, and (iv) a part of this uncertainty (5%) can be attributed to Young's modulus and hardness standard deviations, which can significantly increase when the material exhibits indentation size effect (hardness-load dependence). On the other hand, the main difference between the experimental fracture mechanics and the indentation test is connected to the size of the specimen. Indeed, indentation is a local test and the results may be lower due to the lower probability of crack encountering a defect compared in a massive specimen. Contrarily, the influence of surface defects such as roughness or residual stresses are more important for the indentation test. Therefore, the one

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solution proposed by Hervas et al.¹⁹ and Iost²⁰ is the use of different complementary techniques to calculate the indentation fracture toughness.

Another reason which can explain the discrepancy in the fracture toughness evaluation is connected to the accuracy of the indentation crack length measurement. For example, Miyazaki and Yoshizawa²¹ have performed a round robin analysis using two alumina ceramics involving 12 laboratories. As a main result, they show that the error in reading the crack length was reduced by using a powerful optical microscopy with an objective lens of 40× or 50× and jointly a traveling stage. Additionally, Miyazaki and Yoshizawa,²² in another paper, discussed the improved accuracy in relation to the mechanism of the high contrast image of the crack tips. This was achieved by improving the visibility of crack tip by applying a transparent solution to the surface.

In this paper, we suggest the use of an optical profilometer jointly to the indentation experiments to estimate the crack length, to determine the fracture indentation toughness of four optical glasses (Classical Crown K5, Borosilicate Crown BK7, Dense Flint SF2, and Ceramic Zerodur® glasses). Unfortunately, this method presents disadvantages of time consuming and dislocation of workpiece from indentation testing area to optical microscopy measuring area. However, this is more accurate in determining the crack length. On the other hand, the quality of the surface of optical materials, i.e., roughness and presence of superficial defects, can greatly affect the measurement, and therefore specific attention is paid to the lapping and polishing processes to prepare the specimen.²³ As an example, the lapping process of the surfaces by means of bonded abrasives or pellets allows the use of metal tools in planar or spherical form, where the effective area contains abrasive grains. Note that the use of water allows the tool to cool down, removes debris, and facilitates the flow of particles between the sample and the tool.^{24–27} By applying this methodology, we have obtained high-precision optical components of a dioptic glass. As a result, convex and concave spherical surfaces were made with a radius of curvature measuring 5 mm and the root-mean-square roughness (R_q) is found to be <20 nm.^{28–34} The polishing principle is similar to the lapping process, except that the polishing agent is softer and the abrasive grains are finer. In fact, polishing surface of glass with finer abrasive grains reduces surface roughness to <1 nm. Indeed, a nanodimensional shape can be obtained

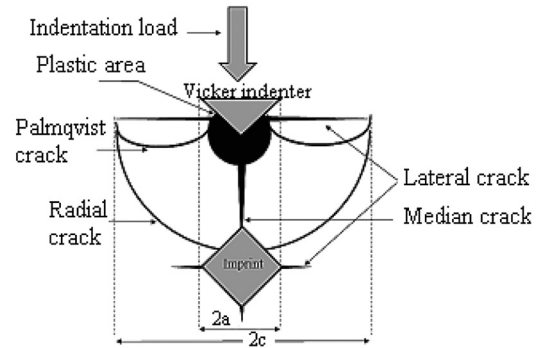


Fig. 2 Schematic representation of the crack systems for a Vickers indentation.³⁸

by polishing the spherical surfaces.³⁵ The ceramic glass Zerodur of Schott can even reach a roughness R_q of about 0.2 nm.³⁶ Figure 1 shows the principle of polishing optical glass with free abrasive grains and pellets.³⁷

The determination of the indentation fracture toughness requires polished and flat surface material and suitable experimental equipment for measuring the dimensions of the cracks. In addition, the shape can take different forms, i.e., Palmqvist (P) or half-penny/median (M), as schematically shown in Fig. 2.

2 Materials and Experiments

2.1 Material

The materials under study are alkali silicate-based glasses of Schott. The Crown glass K5 is a classic optical glass, with low refractive index and dispersive power and its Abbe number is >50. It is particularly adapted for the production of optical systems, such as lenses, prisms, and mirrors. The Borosilicate Crown glass BK7 is used in optics for the manufacture of optical components. It has a low refractive index of about 1.52 and a high Abbe number. This glass is recognized for its high homogeneity, low porosity, and machining facility, thereby conferring it to be a good material for optical transmission for transmission ranges between 380 and 2100 nm. The Dense Flint glass SF2 contains mainly lead oxide (56.7% PbO). It has a high refractive index and it is more dispersive than the Crown glass K5. Because of its brightness, the dense flint glass is used in many fields,

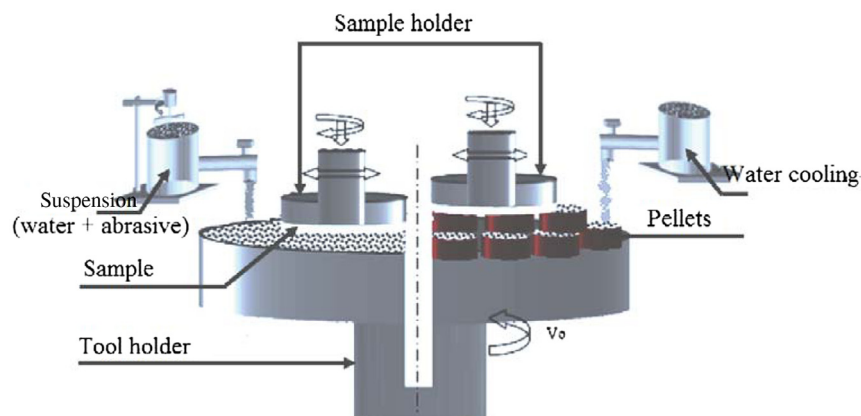


Fig. 1 Glass polishing principle by free abrasive grains and by pellets.³⁷

such as art glass, screens, and protection in the field of optical glasses. Indeed, the optical design of crown glasses is associated with flint glasses in an optical pair in order to correct chromatic or spherical aberrations. The ceramic glass Zerodur of Schott consists of microcrystals dispersed in a glass phase. This sort of glass is relatively transparent because the refractive indices of the two glass matrices and crystalline phase are close. However, the dispersion created by the presence of nanocrystals prevents its use in passive optics, that is, for transmitting light.³⁹⁻⁴¹ The ceramic glass Zerodur has a low coefficient of expansion and it presents an exceptionally high resistance to thermal shock, $>600^{\circ}\text{C}$, and it is used in the hot plates and kitchens for terrestrial and space-telescope mirrors and laser gyro. The tested samples present different shapes and dimensions:

- Crown glass K5, a square shape ($20 \times 20 \text{ mm}^2$),
- Borosilicate Crown glass BK7, a circular shape ($70 \times 12 \text{ mm}^2$),
- Dense Flint glass SF2, a rectangular shape ($32 \times 22 \text{ mm}^2$),
- Ceramic glass Zerodur, a square shape ($44 \times 44 \text{ mm}^2$).

The chemical compositions and some physical properties of the four studied glasses are collected in Table 1. The

Table 1 Some physical properties (ρ , density; n , refractive index; V , Abbe number; and ν , Poisson's coefficient) and the chemical compositions of the studied glasses.

Optical materials		Crown K5	Borosilicate Crown BK7	Dense Flint SF2	Ceramic glass Zerodur
Physical property	ρ (g/cm ³)	2.51	2.27	3.86	2.53
	n	1.51	1.52	1.64	1.54
	V	64.2	69.5	33.9	59.6
	ν	0.20	0.21	0.25	0.24
Chemical composition (in w%)	SiO ₂	68.4	70.1	37.2	55.8
	Na ₂ O	8.3	10.6	—	0.5
	CaO	0.9	8.2	—	—
	K ₂ O	7.3	5.4	6.1	—
	MgO	—	3.9	—	0.9
	Al ₂ O ₃	1	1.3	—	26.5
	PbO	—	—	56.7	7.2
	Li ₂ O	—	—	—	3.7
	TiO ₂	0.4	0.5	—	3.3
	ZrO	—	—	—	1.8
	A _s O ₃	0.9	—	—	0.3
B ₂ O ₃	10.6	—	—	—	
BaO	2.2	—	—	—	

chemical compositions have been determined by means of the ZSX Primus IV Sequential wavelength dispersive x-ray fluorescence, and the Abbe number as well as the refractive index, is determined by means of the Abbe refractometer Model 98.490 of Novex-Holland.

2.2 Pellets Preparation

The abrasive used for lapping is mainly composed of alumina Al₂O₃ corundum type in the form of grains bound by an organic binder or vitrifying. The preparation involves a mixture of 80% alumina and 20% resin in weight percentage as a binder during 5 to 10 min to homogenize the mixture. The contents of abrasive grains and binder within the mixture were determined by preliminary experiments. Formatting has been conducted at the institute of optics and precision mechanics of the University of Setif (Algeria). The principle was to submit a given quantity of mixture (0.720 g) deposited in a closed container to a compression stress of 100 MPa corresponding to a force of 15 KN. The pellets are then heated at a temperature below the melting point during a period ranging from 45 to 60 min. The heating temperature is about 150°C. Figure 3 schematically illustrates the pellets' preparation.

2.3 Lapping and Polishing Processes

The controlled lapping operation was conducted by means of a conventional machine, including a tool holder which rotates at a rotational speed of 124 rpm. The pellets which have been elaborated with different particle sizes are used to eliminate subsurface damage from the sample preparation process such as grinding.⁴² The pellets are pasted in the tool holder, which rotates in the opposite direction of the sample piece attached to the door by resins. The latter rotates at a constant speed and oscillates at adjustable amplitudes according to the diameter of the lap. The abrasive grains contain alumina oxide (Al₂O₃) (Abralex, quality E produced by Pieplow Brandt GmbH Company). The four types of glasses: Borosilicate Crown BK7, Zerodur, Crown K5, and Dense Flint SF2 of Schott are lapped successively by different grain sizes of 40, 20, 15, 7, and 3 μm , for a period of 60 min and then are polished by pellets cerium oxide CeO₂ of 0.5 μm in diameter. Polishing by pellets allows getting a transparent and better quality surface (Fig. 4). The polishing time for the crown and flint glasses is <15 min, whereas it is up to 30 min for the ceramic glass Zerodur, which is obviously more difficult to polish.

2.4 Vickers Indentation Tests

The Vickers indentation tests have been conducted using the instrumented indenter Zwick ZHU 2.5. The applied loads are ranged between 5 and 90 N. Six tests were performed for each maximum indentation load, to study the reproducibility of the indentation tests. The fracture indentation toughness of brittle materials is then determined from the length of the cracks formed along the diagonal of the indent in relation to the corresponding indentation load. In this work, fracture indentation toughness of the studied glasses was computed from four relationships presented in Table 2. The two relations proposed by Anstis et al.¹⁸ and Lawn et al.⁴³ are related to the median-cracking mode and those of Laugier⁴⁴ and Niihara et al.⁴⁵ to the Palmqvist-cracking mode. In this

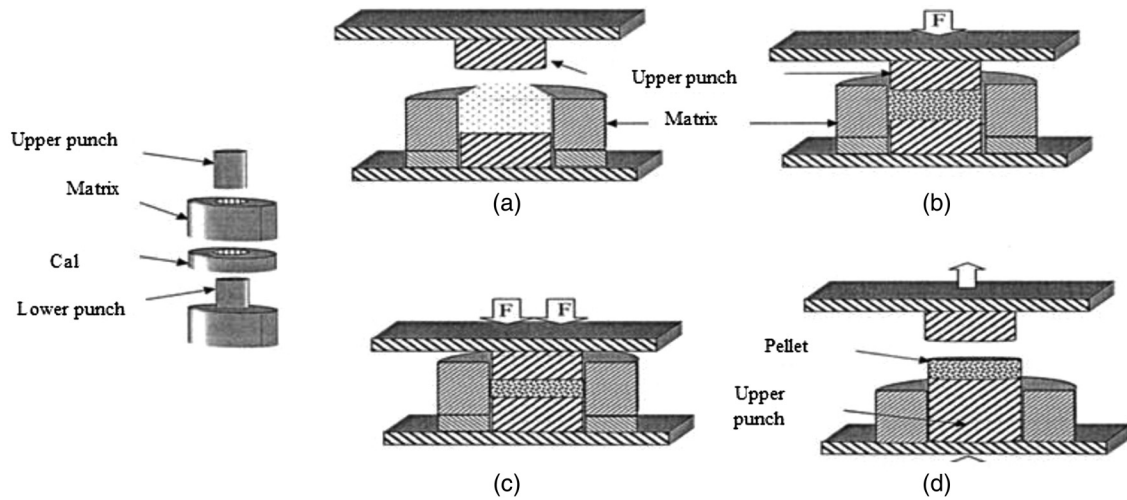


Fig. 3 Formatting principle of pellets preparation (a) powder filling, (b) precompaction, (c) compaction, and (d) ejection of pellet.

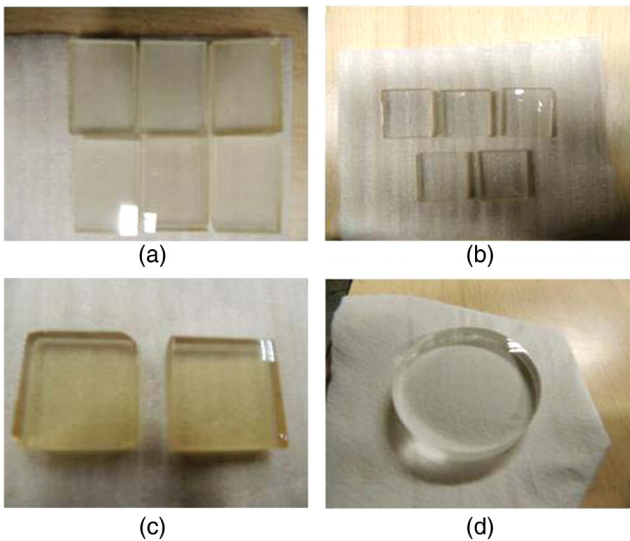


Fig. 4 Polished glasses with pellets for (a) Dense Flint SF2, (b) Crown K5, (c) Zerodur, and (d) Borosilicate Crown BK7.

table, P corresponds to the load, c corresponds to the crack length measured from the center of the indent, l denotes the crack length measured from the corner of the indent, and a denotes the half diagonal of the indent. Here, E is the Young's modulus and HV the hardness. Note that IIT allows the determination of the mechanical properties E and HV from the load-depth curve resulting in the same experimental indentation test as the cracking analysis.

2.5 Crack Length Measurement

An optical profilometer Veeco NT9300 with monochromatic light centered on a wavelength $\lambda = 550$ nm was used. Such an instrument is often used to characterize a sample surface to access its roughness. In this work, it was also used to determine the crack lengths performed on the different samples after indentation. Different objective sizes are mounted on a turret with Michelson and Mirau optical systems. The objective used is the Michelson 5 \times with a working distance of 6.7 mm. The system works in two different modes, i.e., phase-shifting interferometry (PSI) mode and vertical scanning interferometry mode. In the first mode, a piezoelectric transducer moves linearly over the reference surface at a smaller distance to produce a phase shift between the test and reference beams. The system records the intensity and then converts the intensity to wavefront data by integrating the intensity data. This technique is used when the difference between the adjacent measurement points is <25% of λ . Otherwise, a higher error could be made. Thus, this technique is only used for fairly smooth and continuous surfaces. For the second mode, the interferometric objective moves vertically to scan the surface at varying heights. As the system scans downward, an interference signal for each point on the surface is recorded. The vertical position corresponding to the peak of the interference signal is extracted for each surface point. Finally, this instrument is used to not only detect the crack extremity but also get a scratch profile on the glass to determine the fracture toughness with a scratch tester.

From a practical point of view, the length of cracks around a Vickers indent was measured through an optical

Table 2 Relationships used in calculating the fracture indentation toughness for different crack geometries.

No.	Equations
1	$K_{IC} = 0.016(E/HV)^{1/2} \cdot P \cdot c^{-3/2a}$
2	$K_{IC} = 0.0134(E/HV)^{1/2} \cdot P \cdot c^{-3/2b}$
3	$K_{IC} = 0.0134(E/HV)^{1/2} \cdot P \cdot c^{-3/2c}$
4	$K_{IC} = 0.0089(E/HV)^{2/5} \cdot P / (a \cdot l^{1/2})$ for $\approx 0.25 \leq (l/a) \leq \approx 2.5^d$ to 3

^aAnstis et al.,¹⁸
^bLawn et al.,⁴³
^cLaugier,⁴⁴ and
^dNiihara et al.⁴⁵

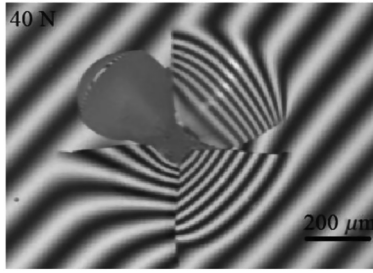


Fig. 5 Example of an observation of a Vickers indent obtained with an indentation load of 40 N applied to the Dense Flint glass SF2 and the corresponding interference fringes located around the indenter.

profilometer. Usually, the imprints performed in glass are unstable. The size and shape of cracks performed by Vickers indentation change for each applied load, and the crack propagation are different. Sometimes they can be accompanied by spalling. Therefore, it is difficult to accurately and correctly measure the value of the crack lengths. To obtain the crack length by means of an optical profiler, the intensity image that was taken previously by the optical microscope of the Vickers indentation system was used. As a crack corresponds to a local discontinuity of the surface, it is possible to

detect interference fringe discontinuities (Fig. 5). The fringe period is similar to the altitude variation, so the fringe phase variation allows the crack extremity detection with greater sensitivity than classical methods.

The Veeco Wyko NT9300 is an optical profilometer that is used for measuring the three-dimensional (3-D) surface profile without any contact at a nanometric resolution. This instrument is used to observe the surface of the samples and to measure the crack lengths ($2c$) and ($2c'$), generated by Vickers indentation, through the interferometry fringes. This technique is based on a white-light reflected by a reference mirror combined with the light reflected by the sample which then produces interferential fringes. As the white-light has a short coherence length, the high-contrast fringes are present at a greater depth, thus increasing the accuracy of the measurements. On the other hand, the method based on the analysis of the interferometry fringes requires that the surface of the sample is flat, correctly polished, and presenting no consequent surface defects. When moving the objective toward the sample, the fringes converge and stabilize themselves over the entire sample surface. Afterward, a rapid scan is performed in a few seconds to obtain a clear image where the cracks can be observed along the edge of the indent to be measured by means of a cursor with a relatively good accuracy.

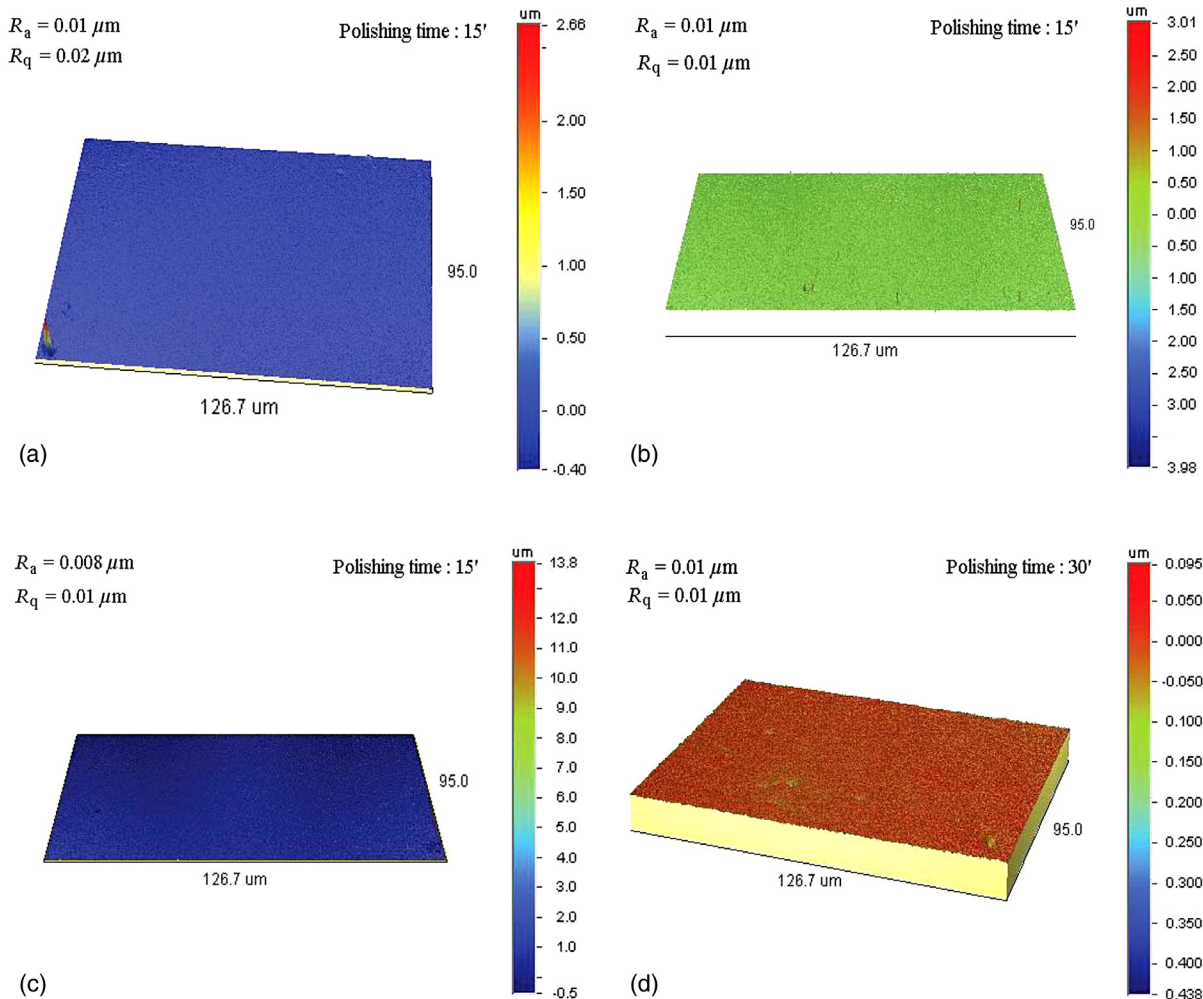


Fig. 6 Three-dimensional pictures of roughness obtained after polishing by pellet for (a) Dense Flint SF2, (b) Crown K5, (c) Borosilicate Crown BK7, and (d) Zerodur glasses.

3 Results and Discussion

3.1 Surface Characterization

The roughness has been studied by means of the optical Profilometer Veeco Wyko NT9300. As a main result, the polishing technique using the pellets gives a good surface quality for the four glasses studied. Indeed, the roughness values R_a are very similar and close to 10 nm for the Dense Flint SF2, Crown K5, and Zerodur glasses and slightly lower for Borosilicate Crown BK7 glass, i.e., the average roughness, $R_a = 8$ nm. Concerning the roughness parameter R_q , it has been found equal to 10 nm for the Crown K5, Borosilicate Crown BK7, and Zerodur® glasses and 20 nm for the Dense Flint SF2 glass (Fig. 6). This result can be explained by the different grain sizes used, the working conditions, the nature of the glass, and the difficulty of polishing the glasses by pellets.

To obtain reliable and mean values, six experimental tests have been performed. Table 3 collects the values obtained for R_a and R_q expressed in nanometer, the standard deviation in nanometer, and the uncertainty in percentage have been added to this table. Note that an uncertainty located between 2% and 4% has been found for the roughness parameter R_a , whereas the uncertainty reached values ranging between 4% and 8%.

Figure 7 represents the variation of the roughness parameters, R_a and R_q , as a function of the polishing time. The variations of the two roughness parameters follow the

Table 3 Mean values for the two parameter toughness R_a and R_q associated with their standard deviations as well as their uncertainties obtained for the four studied optical glasses.

Optical materials		Crown K5	Borosilicate Crown BK7	Dense Flint SF2	Ceramic glass Zerodur
R_a	Value (nm)	9.80	8.10	10	9.90
	SD (nm)	0.31	0.19	0.5	0.53
	Unc. %	±3.1	±2.3	±5.0	±5.4
R_q	Value (nm)	10.50	9.93	20	10.30
	SD (nm)	0.86	0.37	0.15	0.68
	Unc. %	±8.2	±3.7	±5.8	±6.6

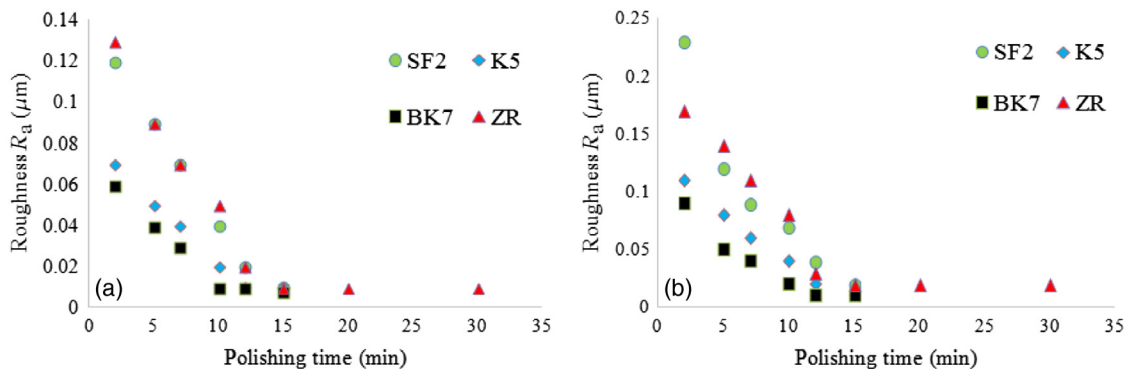


Fig. 7 Variation of (a) R_a and (b) R_q parameters as a function of polishing time.

same tendency for the different glasses; they rapidly decrease during the first 10 to 15 min to reach the minimum and constant values.

3.2 Vickers Indentation Results

The Vickers instrumented indentation test (IIT) determines the mechanical properties, i.e., the Vickers hardness (HV) and the Young's modulus (E) which have been introduced in the relationships to calculate the indentation fracture toughness (K_{IC}). As the determination of the cracking mode (median- or Palmqvist-cracking modes) is experimentally difficult to achieve, we suggest the computation of the indentation fracture toughness by applying the different toughness expressions in Table 2, without previous knowledge of the cracking mode. As a result, Table 4 collects the Young's modulus and hardness values, both expressed in gigapascal, and the indentation toughness expressed in megapascal.m^{1/2}.

3.3 Interferometry Analysis

The optical profilometer Veeco was used after the indentation test. For the fracture toughness calculation, the values of the mechanical properties, E and HV, are those obtained with IIT. Figure 8(a) shows the principle of measurement of the crack lengths applied as an example to the Dense Flint glass SF2.

To thoroughly analyze the surface of the studied materials, we used the optical profilometer by analyzing the surface in three-dimensions (x , y , and z) without any contact, where x and y define the observation plan and z represents the distance between the sensor and the plan $x - y$. The PSI mode is preferred to improve the study of smooth or quasismooth surface of optical glasses. This methodology is based on the integration of the variation of the light intensity as a function of the height z . A series of lenses focuses the white-light beam on an optical blade that emits a part of the light beam on the sensor and the remaining on the lens of the microscope. Afterward, the light beam through the lens of the microscope crosses a Mirau interferometer before it gets reflected on the test surface. Thus, the two light beams will be collected by the sensor with a shift leading to interference fringes having a given appropriate height z . These fringes are necessary to adequately analyze the sample. They indicate that the instrument is well calibrated and that the uncertainty will be even lower. Obviously, a good calibration will lead to a better focus and consequently

Table 4 Young's modulus (E in GPa), Vickers hardness (HV in GPa), and indentation toughness (K_{IC} in $\text{MPa}\cdot\text{m}^{1/2}$) obtained for all the studied glasses.

Optical materials		Crown K5	Borosilicate Crown BK7	Dense Flint SF2	Ceramic glass Zerodur
HV (GPa)		6.3 ± 0.1	5.4 ± 0.3	4.6 ± 0.2	7.8 ± 0.1
E (GPa)		56.2 ± 0.2	77.9 ± 1.8	73.4 ± 0.9	90.6 ± 0.3
Fracture toughness, K_{IC} in $\text{MPa}\cdot\text{m}^{1/2}$	K_{IC}^a	0.54 ± 0.03	0.60 ± 0.03	0.50 ± 0.03	1.14 ± 0.04
	K_{IC}^b	0.45 ± 0.04	0.49 ± 0.04	0.40 ± 0.04	0.99 ± 0.05
	K_{IC}^c	0.60 ± 0.05	0.69 ± 0.05	0.59 ± 0.04	1.21 ± 0.06
	K_{IC}^d	0.65 ± 0.04	0.71 ± 0.05	0.73 ± 0.05	1.30 ± 0.06

^aAnstis et al.,¹⁸
^bLawn et al.,⁴³
^cLaugier,⁴⁴ and
^dNiihara et al.⁴⁵

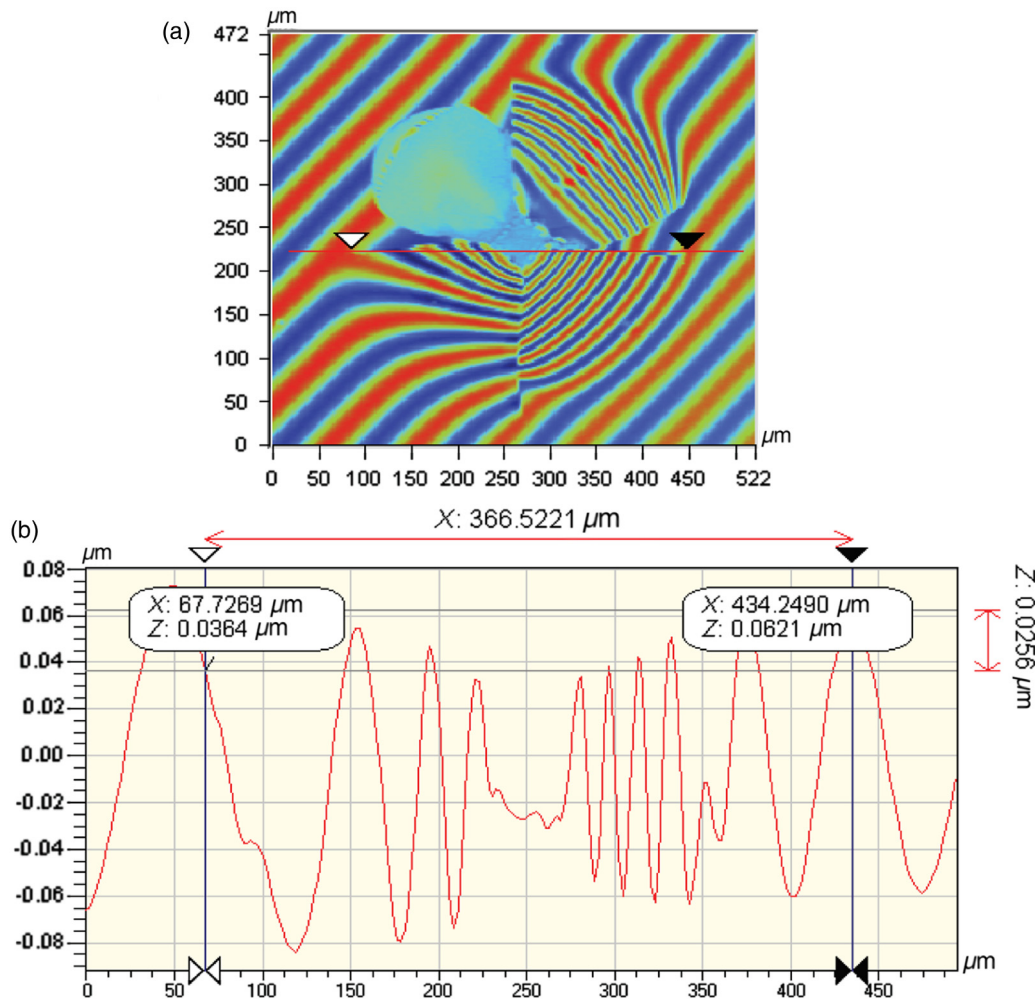


Fig. 8 (a) Intensity cartography of a print ($P = 40 \text{ N}$) with the measurement cursors placed and (b) intensity variation of the picture cartography between the two measurement cursors.

well-defined fringes will be obtained. As an example, Fig. 9 shows the result around a Vickers indent.

The measurement of the two crack lengths ($2c$) and ($2c'$) by using the optical profilometer method (OPM)

was used to calculate the indentation fracture toughness. The average values of the indentation fracture toughness associated with their standard deviations obtained by the OPM were collected in Table 5 and compared with

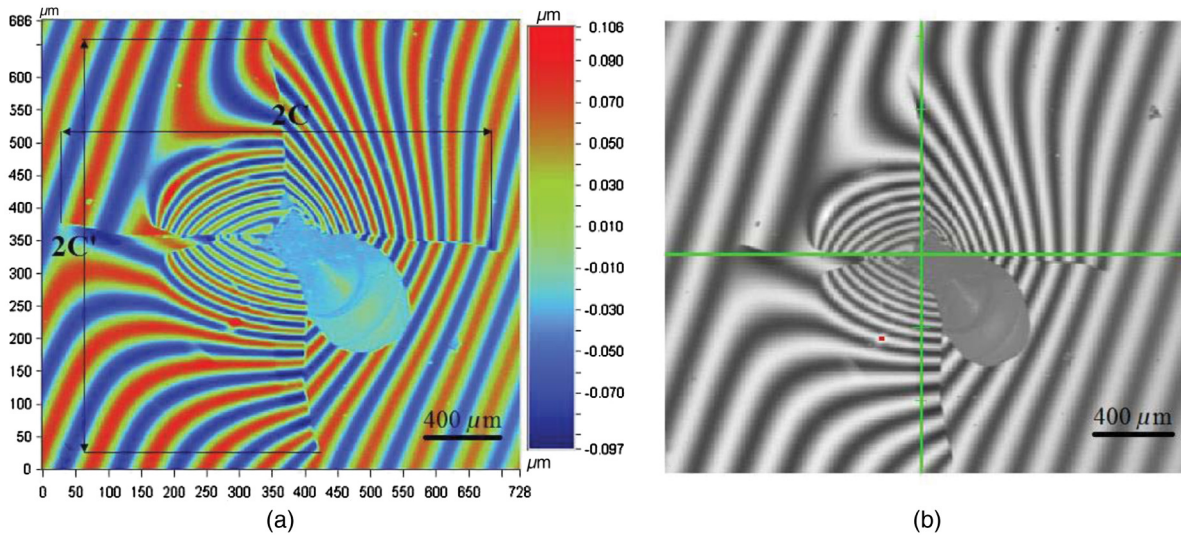


Fig. 9 Vickers indent resulting from the application of 40 N indentation load and observed by optical profilometer (a) intensity cartography and (b) derived picture.

those obtained by indentation test for the four studied glasses.

By comparing the results obtained by means of the OPM and of the optical microscope of the indenter, the values for the indentation fracture toughness are located in the same range of magnitude. On the other hand, the standard deviation is found to be lower. To compare the uncertainties of these two methods, we suggest the calculation of the standard deviation (SD) to the indentation fracture toughness (K_{IC}) ratio. When considering all the studied glasses and the methodology (or relationship) applied^{18,43–45} for calculating the SD/K_{IC} , we obtained $6.5\% \pm 1.7\%$ when using only the indentation test for measuring the crack length, and this value reduced to $3.2\% \pm 1.0\%$ when the crack length measurement from the profilometer was used. This result confirms that the crack length measurement by OPM leads to more accurate results with a lower standard deviation than the results obtained by measuring the crack lengths with

classical optical device. Finally, this relatively low dispersion allows us to assess the accuracy and reproducibility of the OPM to estimate the crack length.

Figure 10 represents the variation of the indentation fracture toughness K_{IC} as a function of the load for the Crown K5 [Fig. 10(a)], the Dense Flint SK2 [Fig. 10(b)], the Borosilicate Crown BK7 [Fig. 10(c)], and the ceramic Zerodur [Fig. 10(d)] glasses.

To compare the cracking fracture behaviors of the four glasses studied in this work and owing to the fact that the cracking mode, i.e., median- or Palmqvist-cracking modes, has not been defined, we suggest the calculation of a mean value of the indentation fracture toughness by considering all the relationships because they lead to similar values and the two methods employed to measure the crack length. In these conditions, we obtained the following values:

- Crown glass K5: $K_{IC} = 0.55 \pm 0.08 \text{ MPa}\cdot\text{m}^{1/2}$.

Table 5 Indentation fracture toughness K_{IC} determined by OPM and IIT measurements for the four studied glasses.

Optical materials		Crown K5	Borosilicate Crown BK7	Dense Flint SF2	Ceramic glass Zerodur
K_{IC}^a	IIT	0.54 ± 0.03	0.60 ± 0.03	0.50 ± 0.03	1.14 ± 0.04
	OPM	0.51 ± 0.02	0.59 ± 0.02	0.49 ± 0.01	0.99 ± 0.02
K_{IC}^b	IIT	0.45 ± 0.04	0.49 ± 0.04	0.40 ± 0.04	0.99 ± 0.05
	OPM	0.43 ± 0.02	0.48 ± 0.02	0.39 ± 0.02	0.97 ± 0.02
K_{IC}^c	IIT	0.60 ± 0.05	0.69 ± 0.05	0.59 ± 0.04	1.21 ± 0.06
	OPM	0.59 ± 0.02	0.64 ± 0.02	0.58 ± 0.02	0.98 ± 0.02
K_{IC}^d	IIT	0.65 ± 0.04	0.71 ± 0.05	0.73 ± 0.05	1.30 ± 0.06
	OPM	0.63 ± 0.02	0.68 ± 0.02	0.72 ± 0.02	1.24 ± 0.03

^aAnstis et al.,¹⁸

^bLawn et al.,⁴³

^cLaugier,⁴⁴ and

^dNiihara et al.⁴⁵

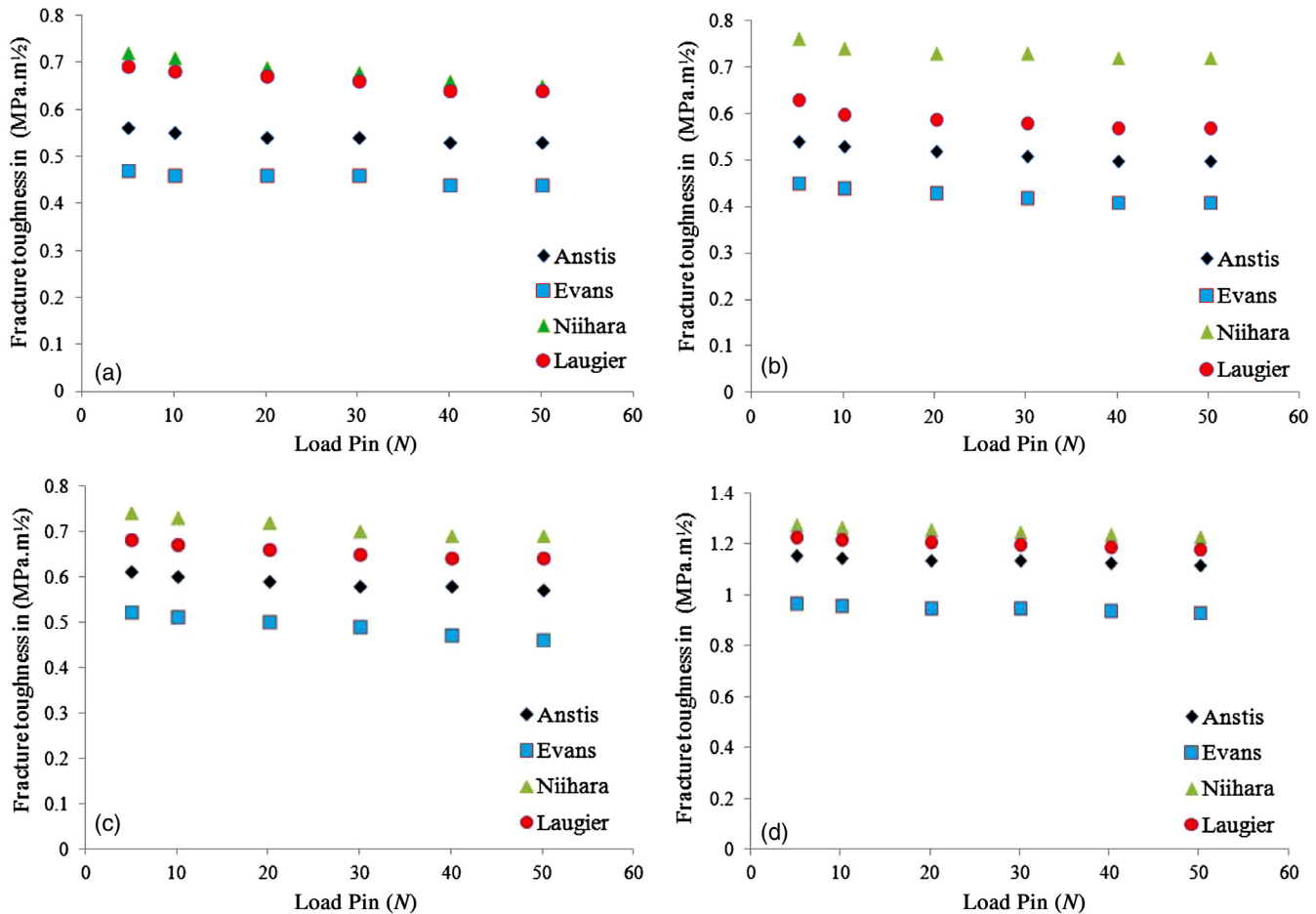


Fig. 10 Fracture toughness K_{IC} variation as a function of the applied load P for (a) Crown K5, (b) Dense Flint SF2, (c) Borosilicate Crown BK7, and (d) ceramic Zerodur.

- Borosilicate Crown glass BK7: $K_{IC} = 0.61 \pm 0.09 \text{ MPa}\cdot\text{m}^{1/2}$.
- Dense Flint glass SF2: $K_{IC} = 0.55 \pm 0.13 \text{ MPa}\cdot\text{m}^{1/2}$.
- Ceramic glass Zerodur: $K_{IC} = 1.10 \pm 0.14 \text{ MPa}\cdot\text{m}^{1/2}$.

As a main result, the ceramic glass Zerodur presents the highest fracture toughness resistance, whereas the three others glasses have more or less the same lower fracture resistance. However, even if the ceramic Zerodur presents the best cracking resistance, the load-displacement curves obtained for this material reveal the presence of pop-ins which appear during the indentation loading, contrarily to the three others materials (Fig. 11).

In addition, Fig. 11 confirms the good reproducibility of the IIT for the loading part of the load-displacement curves except for the ceramic glass Zerodur which presents disparateness over all the loading part, indicating that the cracking behavior is not regular. This result is also due to the presence of pop-ins (sudden bursts of displacements at specific indentation loads) in the case of the ceramic glass Zerodur at a load higher than 50 N but not regularly since successive pop-ins is observed.

It is also noticeable that the plastic deformation of the dense flint glass is superior to the other glasses. This can be estimated through the area under the unloading curve which can be assimilated to the indentation work.

To simplify this calculation, we suggest the use of only the residual penetration depth, which can be used as an indicator of the plastic deformation. As an example for the maximum indentation load of 90 N, we can observe that the residual penetration depth of the Dense Flint glass SF2 is $\sim 19 \mu\text{m}$, whereas it is equal only to 13, 16, and $17 \mu\text{m}$ for the Zerodur, Crown glass K5, and Borosilicate Crown glass BK7, respectively.

4 Conclusions

The cracking resistance of a variety of optical glasses has been studied by Vickers indentation, allowing for the computation of the fracture indentation toughness. To obtain precise values, the optical profilometer has been used complementarily to the optical device associated with the IIT for obtaining an accurate value of the crack length. Independently of the nature of the tested ceramic glasses, the standard deviation to the fracture indentation toughness ratio value is found to be two times less when measuring the crack length by means of the profilometer method. This result clearly indicates that the profilometer is helpful in obtaining the consistent fracture indentation toughness values. As a result, the mean fracture indentation toughness values found by applying the relationship of Anstis et al. and Lawn et al. are slightly lower compared to that obtained when using the relationships of Laugier and Niihara et al.

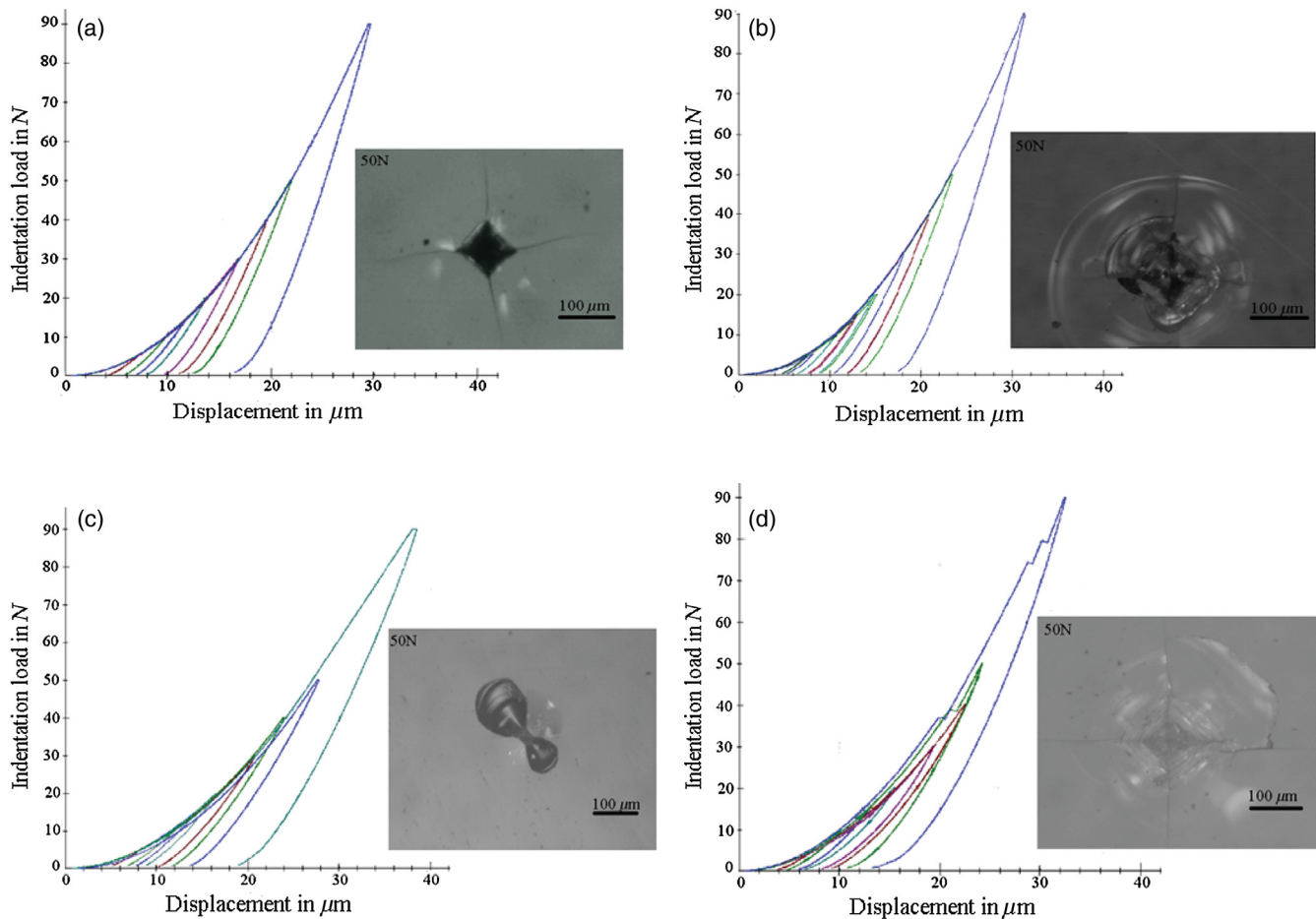


Fig. 11 Load-displacement curves obtained by IIT with a maximum load ranging between 5 and 90 N for (a) Crown glass K5, (b) Borosilicate Crown glass BK7, (c) Dense Flint glass SF2, and (d) Ceramic glass Zerodur.

The observed discrepancy was expected as the two groups of relations are related to different cracking modes, i.e., median- and Palmqvist-cracking modes, respectively. Independent of this observation, it is possible to calculate a mean fracture indentation toughness parameter which allows the classification of the mechanical behavior of the tested optical glasses according to their cracking resistance. From the higher to the lower one, the glasses are classified as follows: ceramic glass Zerodur, Borosilicate Crown glass BK7, Dense Flint glass SF2, and crown glass K5.

Finally, it is important to note that IIT is very interesting for fracture studies. Indeed, in addition to the crack formation, this mechanical test allows the determination of the mechanical properties (i.e., the hardness and the Young's modulus in the same test), which are required for computing the toughness, irrespective of the relationship applied. Moreover, compared to the classical indentation test where only the test can be analyzed postmortem, the load-displacement curves obtained by IIT give additional information such as the reproducibility of the indentation experiment, the presence of pop-ins indicating cracking and/or chipping, and the plastic deformation rate.

References

1. S. Palmqvist, "Method of determining the toughness of brittle materials, particularly sintered carbides," *Jernkontorets Ann.* **141**, 300–307 (1957).
2. S. Palmqvist, "The work for the formation of a crack during Vickers indentation as a measure of the toughness of hard metals," *Arch Eisenhuettenwes* **33**, 629–663 (1962).
3. C. B. Ponton and R. D. Rawlings, "Vickers indentation fracture toughness test part 1: review of literature and formulation of standardized indentation toughness equations," *J. Mater. Sci. Technol.* **5**, 865–872 (1989).
4. C. B. Ponton and R. D. Rawlings, "Vickers indentation fracture toughness test part 2: application and critical evaluation of standardized indentation toughness equations," *J. Mater. Sci. Technol.* **5**, 961–976 (1989).
5. B. R. Lawn and R. F. Cook, "Probing material properties with sharp indenters: a retrospective," *J. Mater. Sci.* **47**, 1–22 (2012).
6. G. D. Quinn and R. C. Bradt, "On the Vickers indentation fracture toughness test," *J. Am. Ceram. Soc.* **90**, 673–680 (2007).
7. J. H. Lee et al., "Cohesive interface simulations of indentation cracking as a fracture toughness measurement method for brittle materials," *Acta Mater.* **60**, 5448–5467 (2012).
8. T. Rouxel et al., "Indentation deformation mechanisms in glass: densification versus shear flow," *J. Appl. Phys.* **107**, 094903 (2010).
9. A. Moradkani et al., "Determination of fracture toughness using the area of micro-crack tracks left in brittle materials by Vickers indentation test," *J. Adv. Ceram.* **2**(1), 87–102 (2013).
10. B. R. Lawn and D. B. Marshall, "Indentation fracture and strength degradation in ceramics," in *Flaws and Testing. Fracture Mechanics of Ceramics*, R. C. Bradt, D. P. H. Hasselman, and F. F. Lange, Eds., Vol. **3**, pp. 205–229, Springer, Boston, Massachusetts (1978).
11. N. Cuadrado et al., "Evaluation of fracture toughness of small volumes by means of cube-corner nanoindentation," *Scripta Mater.* **66**, 670–673 (2012).
12. D. S. Harding, W. C. Oliver, and G. M. Pharr, "Cracking during nano-indentation and its use in the measurement of fracture toughness," in *Thin Films: Stress. and Mech. Properties V, Mater. Res. Soc. Symp. Proc.*, p. 663, Materials Research Society, Pittsburgh, Pennsylvania (1995).
13. G. M. Pharr, "Measurement of mechanical properties by ultra-low load indentation," *Mater. Sci. Eng. A* **253**, 151–159 (1998).

14. T. Scholz et al., "Fracture toughness from submicron derived indentation cracks," *Appl. Phys. Lett.* **84**(16), 3055–3057 (2004).
15. J. Gong, "Indentation toughness of ceramics: a statistical analysis," *Ceram. Int.* **28**, 767–772 (2002).
16. G. D. Quinn and J. J. Swab, "Fracture toughness of glasses as measured by the SCF and SEPB methods," *J. Eur. Ceram. Soc.* **37**, 4243–4257 (2017).
17. J. J. Kruzic et al., "Indentation techniques for evaluating the fracture toughness of biomaterials and hard tissues," *J. Mech. Behav. Biomed. Mater.* **2**(4) 384–395, (2009).
18. G. R. Anstis, P. Chantikul, and B. R. Lawn, "A critical evaluation of indentation techniques for measuring fracture toughness: I, Direct crack measurements," *J. Am. Ceram. Soc.* **64**, 533–538 (1981).
19. I. Hervas et al., "Fracture toughness of glasses and hydroxyapatite: a comparative study of 7 methods by using Vickers indenter," *Ceram. Int.* **42**(11), 12740–12750, (2016).
20. A. Iost, "Détermination de la ténacité de matériaux fragiles ou ductiles à partir de l'essai d'indentation," *Revue de Métallurgie* **110**(3), 215–233 (2013).
21. H. Miyazaki and Y. I. Yoshizawa, "Refined measurements of indentation fracture resistance of alumina using powerful optical microscopy," *Ceram. Int.* **40**(2), 2777–2783, (2014).
22. H. Miyazaki and Y. I. Yoshizawa, "Novel measurement technique of crack length for indentation fracture (IF) method using high contrast image of crack tips through thin film coating," *J. Eur. Ceram. Soc.* **35**(10), 2943–2948 (2015).
23. C. J. Evan et al., "Material removal mechanisms in lapping and polishing," *CIRP Ann. Manuf. Technol.* **52**(2), 611–633 (2003).
24. H. H. Karow and H. Hank, "Fabrication methods for precision optics," in *Fabrication Methods for Precision Optics*, H. H. Karow, Ed., Wiley-Interscience, Hoboken, New Jersey (2004).
25. D. S. Glancy and M. J. Damgaard, "A study in the dynamics and wear of rigid disc systems," in *24th Ann. Convention Int. Metall. Soc. Monterey*, California, USA, p. 3 (1991).
26. K. Phillips, G. M. Crimes, and T. R. Wilshaw, "On the mechanism of material removal by free abrasive grinding of glass and fused silica," *Wear* **41**, 327–350 (1977).
27. S. D. Jacobs et al., "MRF: computer-controlled optics manufacturing," *Am. Ceram. Soc. Bull.* **78**, 42–48 (1999).
28. W. I. Rupp, "Loose abrasive grinding of optical surface," *Appl. Opt.* **11**(12), 2797–2810 (1972).
29. G. F. Vander Voort, *Metallography: Principles and Practice*, p. 752, McGraw-Hill Book Co., ASM International 1999 (1984).
30. A. A. Tesar and B. A. Fuchs, "Removal rates of fused silica with cerium oxide and pitch polishing," *Proc. SPIE* **1531**, 80 (1991).
31. D. Golini, J. C. Lamropoulos, and P. D. Funenbus "Surface micro roughness of optical glasses under deterministic micro grinding," *Appl. Opt.* **35**(22), 4448–4462 (1996).
32. H. H. Pollicov, "Computer aided optics manufacturing," *Opt. Photonics News* **5**(6), 15–19 (1994).
33. J. Liedes, "Optic SM update," *J. Opt. Soc.* **1**, 1917–1983 (1996).
34. J. C. Lambropoulos, S. Xu, and T. Fang, "Loose abrasive lapping hardness of Optical glasses and its interpretation," *Appl. Opt.* **36**(7), 1501–1516 (1997).
35. J. P. Marioge, *Surface optique: Méthodes de fabrication et de contrôle, recherches*, pp. 26–33, EDP Sciences, France (2000).
36. R. Komanduri, D. A. Luca, and Y. Tani, "Technological advances in fine abrasive processes," *CIRP Ann.* **46**(2), 545–596 (1997).
37. N. Belkhir, T. Aliouane, and D. Bouzid, "Correlation between contact surface and friction during the optical glass polishing," *Appl. Surf. Sci.* **288**, 208–214 (2014).
38. R. L. K. Matsumoto, "Evaluation of fracture toughness determination methods as applied to ceria-stabilized tetragonal zirconia polycrystal," *J. Am. Ceram. Soc.* **70**(12), 366–368 (1982).
39. J. Petzoldt, "Transparent metastable glass ceramics containing quartz solid solutions," *Glastechnische Berichte* **43**(4), 127–134 (1970).
40. W. Pannhorst, "Recent advances in LAS-glass-ceramics," *Am. Ceram. Soc. Bull.* **82**(9), 9301–9306 (2003).
41. G. Mueller, "Glass-ceramic materials. Schott information," **2**, 1–7 (1972).
42. F. Lakhdari et al., "Surface and subsurface damage in Zerodur glass during ultrasonic assisted grinding," *Int. J. Adv. Manuf. Technol.* **90**(5–8), 1993–2000 (2017).
43. B. R. Lawn, A. G. Evans, and D. B. Marshall, "Elastic plastic indentation damage in ceramics: the median/radial crack system," *J. Am. Ceram. Soc.* **63**(9–10), 574–580 (1980).
44. M. T. Laugier, "New formula for indentation in ceramic," *J. Mater. Sci. Lett.* **6**, 355–356 (1987).
45. K. Niihara, R. Morena, and D. P. H. Hasselman, "Evaluation of K_{IC} of brittle solids by the indentation method with low crack-to-indent ratios," *J. Mater. Sci. Lett.* **1**, 13–16 (1982).

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