Comparative study on methods for estimating the toughness of brittle materials polished by cerium oxide pellets

H. BENZAAMA[a,*], M. BENTOUMI[b] and A. BENCHIHA[c]
a,c Laboratory of Applied Biomechanics and Biomaterials (LABAB), National Polytechnic School of Oran MA. Algeria
c. *Laboratory of Applied Optics, Institut of Optics and Precision Mechanics, University of Ferhat Abbas, Setif, Algeria*
*Contact: habenza@yahoo.fr, phone +213-41-29-07-73/poste 129

Abstract:

The pellets polishing is used to finish different type of brittles materials (glass and ceramic glass). Improved surfaces are obtained via this technique. In this paper, we present a methodology allowing producing a surface roughness lower than 8 nm. In addition, the toughness of brittle materials is estimated in pellets polishing. For that, the instrumented indentation tests were performed by varying the load. Pop-in are observed from 50 N in the ceramic glass. For the Dense flint glass SF2, these pop-in does not appear only from 100 N when they are not highlighted on the Crown glass K5. Different relationships based on assumptions about the geometry of the cracks, were used to estimate the fracture toughness KIc from the length of formed cracks. The method by optical Profilometer used after the indentation test, is established to characterize the fracture toughness of four glasses studied, this technique based on the measurement of the crack length, minimizes the uncertainties, gives better results and improves precision compared to that of the classic indentation method. The results obtained on these glasses, as well as their dispersions are compared.

Keywords : toughness, polishing, cerium oxide pellets.

1 Introduction

The instrumented indentation is used to estimate the toughness, KIc of a material from the length of the cracks formed. The methods used determine the KIC toughness with an uncertainty of up to 50% [1-2]. The characterization of fragile materials depends on the surface condition obtained by two manufacturing processes, the smoothing and the polishing [3-4-6]. All these methods generally allow the determination of the KIc toughness with an uncertainty of up to 50% [5].

The hardening operation is carried out on a steel or bronze lap [7-8] by free abrasive grains leading to local microcracks of the surface layer of the material with a small chip removal [9-10]. Same as polishing, polishing uses a softer polisher and finer abrasive grains, generating minimal surface roughness with less material removal [11-12].

2.1 Materials

The materials used are Schott glasses based on alkali silicate; Crown glass K5, borosilicate crown BK7, dense flint glass SF2 and ceramic glass Zerodur® by Schott. The samples used are parallelepipedal of 32x22x6 mm³ for SF2, a square shape of (20x20 mm2) for the Crown glass and (44x44x8mm³) for the Zerodur® and in circular shape of 70x12mm² for the BK7.

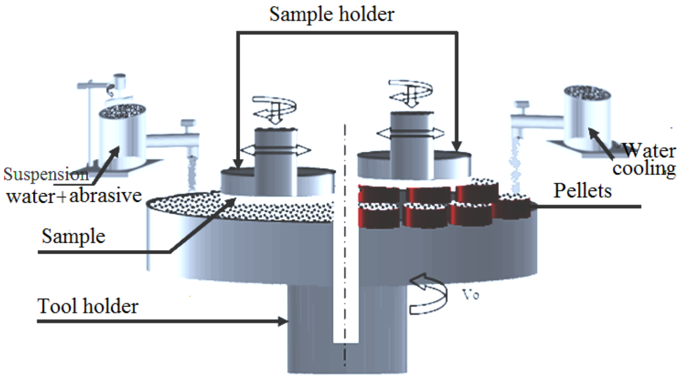


Fig. 1 Principle of glass polishing by free abrasive grains and by pellets

2 Experimental Methods

2.1 Pellets elaboration

The abrasive used for thinning is based on 80% alumina in the form of grains bonded by a 20% resin. The mixture is heated for 5 to 10 minutes to make it homogeneous. The mixture (0.720 g) is compressed to 100 MPa equivalent to 15 KN. The pellets are then heated to a temperature below that of melting, for a period of 45 to 60 minutes. The cooking temperature is 150 ° C. This results in a process of polishing and polishing.

The mixture (0.720 g) is compressed to 100 MPa equivalent to 15 KN. The pellets are then heated to a temperature below that of melting, for a period of 45 to 60 minutes. The cooking temperature is 150 ° C. This results in a process of polishing and polishing.

The chemical and physical compositions of the four grades determined by X-ray diffraction and by measurement of the refractive index (Carl Zeiss Abbe Refractometer) are shown in Table 1:

Table I: Chemical composition of glass (ρ: density in gr / cm3, n: refractory index, v: number abbe and ν: coefficient of fish)

Glasses	Physical properties				Composition %												
	P	n	V	v	SiO_2	Na_2O	CaO	K_2O	MgO	Al_2O_3	P_2O_5	Li_2O	TiO_2	ZrO_2	A_sO_3	B_2O_3	BaO
BK7	2.51	1.517	64.2	0.20	68.4	8.3	0.9	7.3	-	1	-	-	0.4	-	0.9	10,6	2.2
K5	2.27	1.522	69.5	0.21	70.1	10.6	8.2	5.4	3.9	1.3	-	-	0.5	-	-	-	-
SF2	3.86	1.64	33.9	0.25	37.2	-	-	6.1	-	-	56.7	-	-	-	-	-	-
Zerodur®	2.53	1.542	59.6	0.24	55.8	0.5	-	-	0.9	26.,5	7.2	3.7	3.3	1.8	0.3	-	-

2.2 Grinding and polishing

The fine grinding operation was conducted on a conventional machine, comprising a tool holder which rotates at a rotational speed of 124 rev / min. The pellets are glued onto the tool holder, which rotates in the opposite direction to the sample, fixed to the workpiece holder by resins. The latter rotates at a constant speed and oscillates at adjustable amplitudes according to the diameter of the lap. The abrasive grains used are oxides of alumina (Al2O3). The three types of glass: Schott's Ceramic (Zerodur®), Crown K5 and Flint heavy SF2 are successively ground in different sizes of grains: 40, 20, 15, 7 and 3 µm for a period of 60 minutes and then polished by Pellets made of cerium oxide CeO2 with a diameter of 0.5 µm.

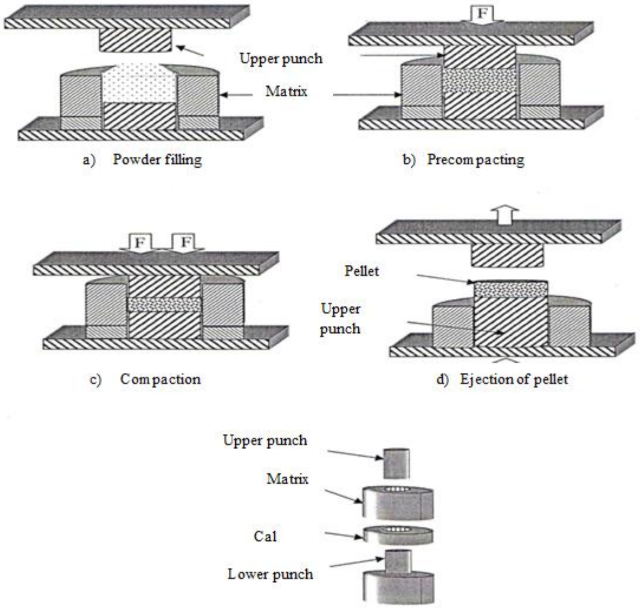


Fig. 2 Principle of pellet formation

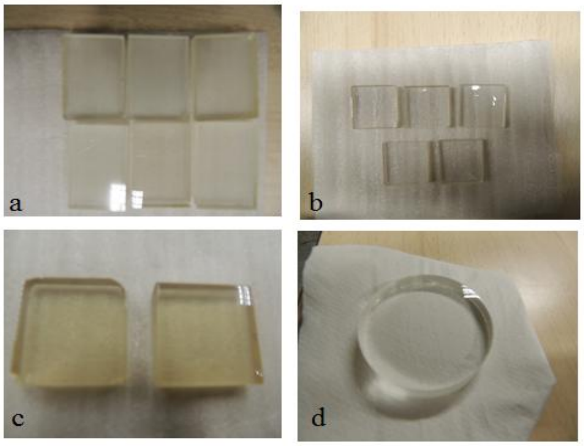


Fig. 3 Polished glasses with pellets: (a) heavy flint SF2, (b) crown K5, (c) Zerodur® and (d) BK7

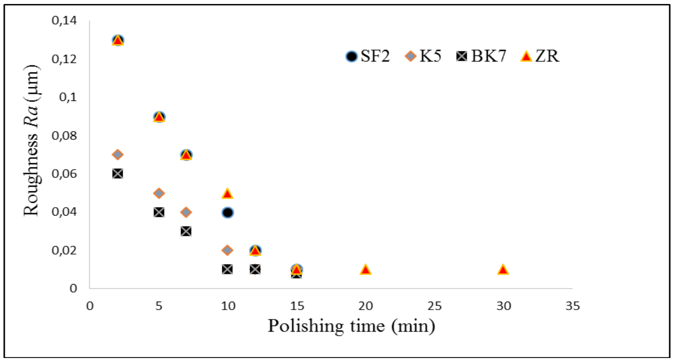


Fig.4 Evolution of roughness *Ra* as a function of polishing time

2.3 Evaluation of the toughness KIc using the different relationships

The indentation tests are carried out using the Zwick ZHU 2.5 instrumented hardness-tester.

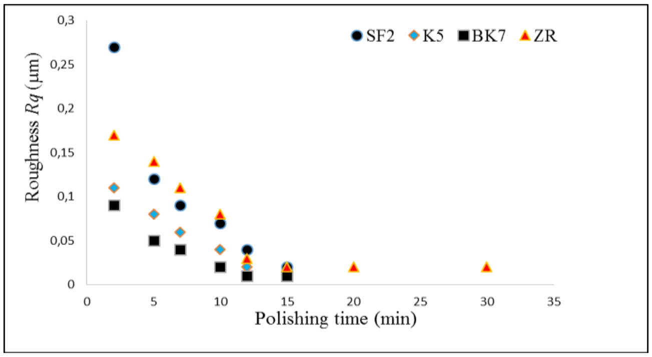


Fig. 5 Evolution of roughness *Rq* as a function of polishing time

The applied loads making it possible to measure the toughness KIc of a fragile material is determined from the length of cracks formed. In our case, the toughness of the glass studied was estimated from the four relations for the four glasses studied are from 5 to 50N. The toughness The applied load are presented in table2:

TableII: Equations used to calculate fracture tenacity for different forms of cracks

Nº	Equations	References
1	$K_{IC}=0.016(E/H)^{1/2}. P.C^{-3/2}$	Anstis [26]
2	$K_{IC}=0.022(E/H)^{2/5}. P.C^{-3/2}$	Tanaka [27]
3	$K_{IC}=0.0089(E/H)^{2/5}. (P/a).C^{-1/2}$	Niihara [28]
4	$K_{IC}=0.0134(E/H)^{1/2}. P.C^{-3/2}$	Evans [29]

The mean values obtained from KIc, E and HV are grouped in Table 3:

TableIII: Fracture tensity, young modulu and average values of hardness Vickers of glasses

Glasses	Young Modulus	Vickers Hardness	Fracture Hardness K_{IC} (MPa.m$^{1/2}$)			
	E(GPa)	H(GPa)	Anstis	Evans	Tanaka	Niihara
BK7	77.90 ± 1.8	5.42 ± 0,3	0.59 ± 0,02	0.49 ± 0.04	0.66 ± 0,05	0.71 ± 0,05
K5	56.21 ± 0.2	6.3 ± 0.1	0.54 ± 0,02	0.45 ± 0,02	0, 63 ± 0,05	0, 65 ± 0,04
SF2	73.37± 0.9	4.58 ± 0,2	0.51 ± 0,03	0.42 ± 0,03	0, 59 ± 0,04	0, 73 ± 0,05

The evolution of the Vickers indentations obtained under different loads ranging from 5 to 90 N for the four materials studied is shown in FIG. 6. We also note a similarity in the appearance of the indentation curves in the four cases. The loading and unloading curves are not linear, show that the responses of the four materials used exhibit elastoplastic behavior.

In FIG. 7, the impression followed by radial cracking is clearly observed from the first applied load of 5 N for each type of glass. Only Zerodur® glass has a radial crack with slight deformation of the impression. From the loads between 10 and 20 N, and during the withdrawal of the penetrator, a slight deformation of the impression due to elastic return is observed for the four materials. This deformation takes the form of a deflection of the faces of the residual impression and the appearance of a number of scales. For loads greater than or equal to 20 N, scales are systematically observed. The imprint and the cracks are clear enough for borosilicate crown glass BK7, dense flint glass SF2 and crown glass K5.

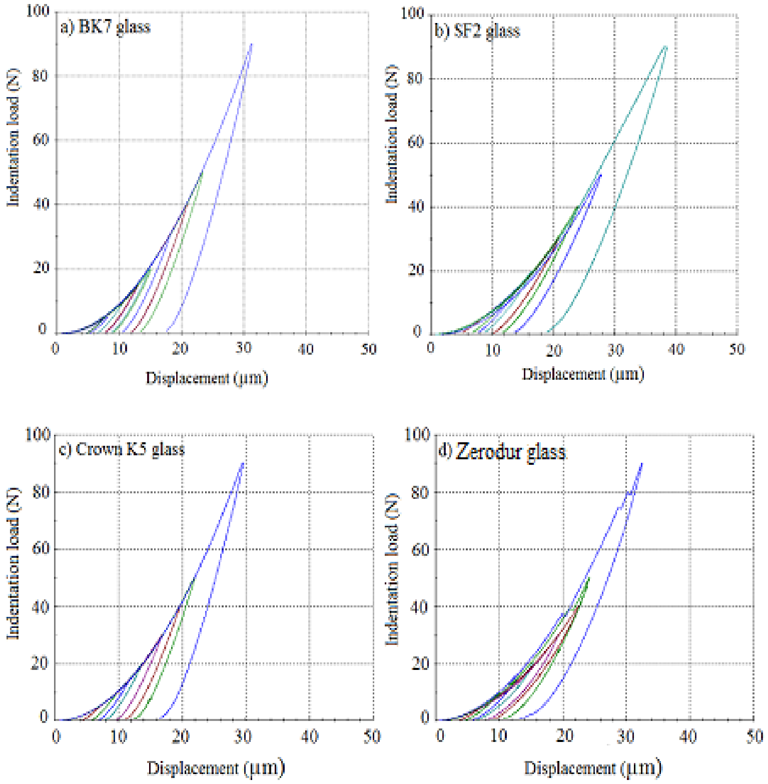
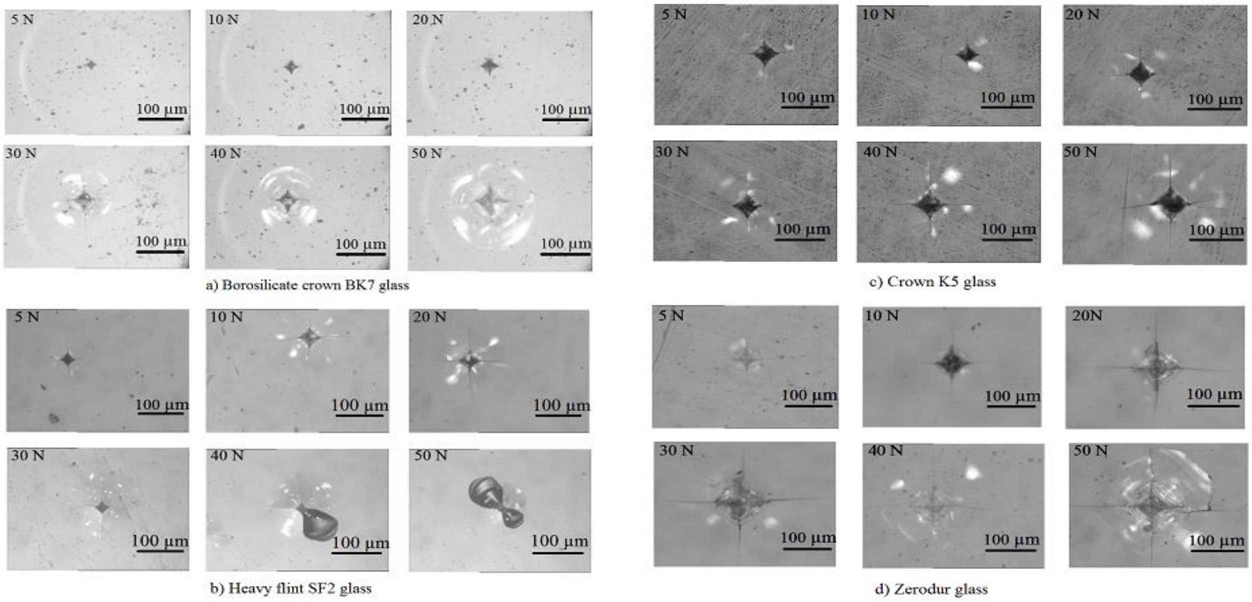


Fig. 6 Glasses load - displacement curves from 5 to 90N



a) Borosilicate crown BK7 glass

b) Heavy flint SF2 glass

c) Crown K5 glass

d) Zerodur glass

Fig. 7 Vickers indentation imprint on the four glasses studied

On the other hand, for Zerodur® glass, they tend to disappear, the size of the defects increases, the lateral and radial cracks widen and form a more or less regular closed contour. Finally, and for loads of 50 N, the radial cracks of the impression widen for the two glasses BK7 and flint dense SF2. For crown glass K5, lateral cracks widen progressively with scaling and for Zerodur® glass, the total impression deformation and flaking gradually develop, and their shape tends to become circular.

The indentation curves shown in Fig. 6 are obtained with progressive loads ranging from 5 to 50 N. The tests show a fairly good reproducibility, although the indented zones are different. By increasing the load from 60 to 90 N the behavior becomes more homogeneous, and there are discontinuities on the force-displacement curve for the Zerodur® glass during loading. These phenomena, called pop-in, correspond to an unstable propagation of the crack. The shape of the force-displacement curves highlights the elastoplastic behavior of the materials. We notice that the plastic deformation of the dense flint glass is superior to other glasses. For a charge of 90 N, for example, the residual penetration of the SF2 flint glass is of the order of 19 µm whereas it is only 13, 16 and 17 µm for Zerodur®, crown glass K5 and glass Crown borosilicate BK7 respectively.

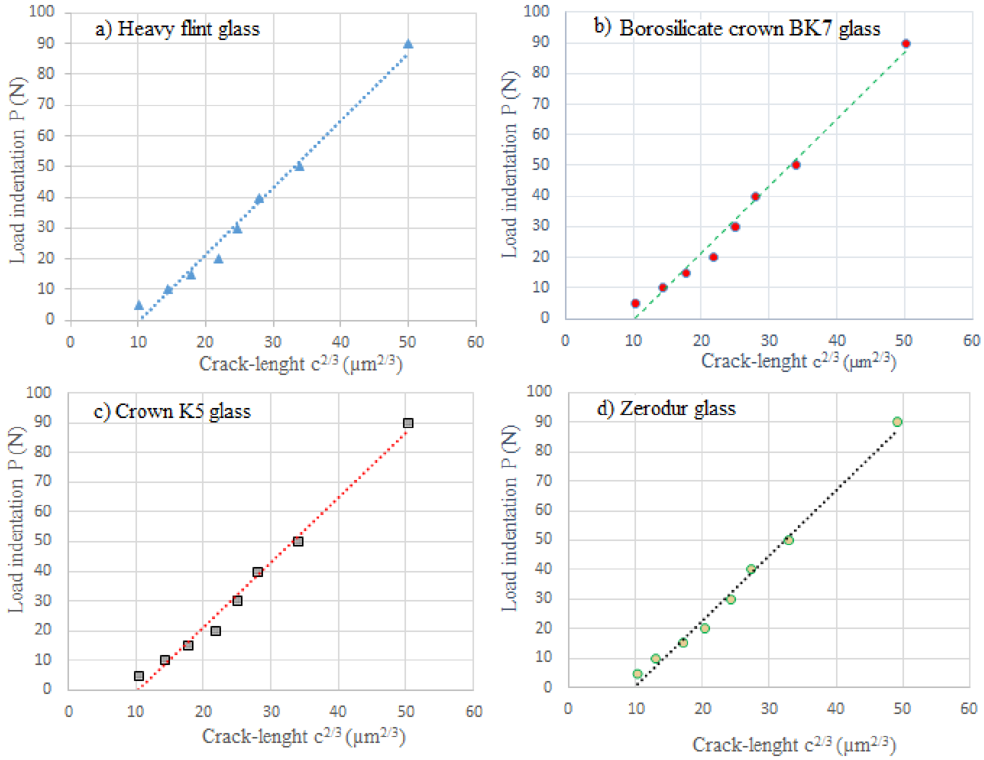


Fig. 8 Evolution of the load P as a function of the length of the crack for the four glasses studied

The first pop-in appears for the Zerodur® glass for a 50 N charge. No pop-in is detected on the charge-discharge curve for the other glasses at this load.

For the samples used, and across the standard deviation, we note that the dispersion rate of the results calculated by the Anstis, (Evans and Tanaka) and Niihara relation does not exceed 3%, 4%, and 5% respectively.

Fig. 8 shows the evolution of the load P as a function of the length of the crack for the four glasses. The applied load is proportional to c-3/2, irrespective of the shape change of the cracks, which corresponds to a constant toughness, indicating that the polishing has not introduced significant residual stresses.

3 Conclusion

The abrasive grains (pellets) in the processes of doubling and polishing the surfaces of the fragile materials. The polishing by pellets shows a better surface quality with respect to the polishing by free abrasive grains and makes it possible to obtain roughness values and the order of 8 to 10 nanometers.

The hardness behavior of the indented glasses was compared using the force-displacement curves obtained during the instrumented test and a comparative study of different equations was made for the calculation of the tenacity of the four glasses used.

Indeed, according to the load-displacement curves, we note that the residual penetration after unloading in the case of the dense flint glass SF2 is greater than the other glasses, it corresponds to 19 µm for the selected loads 90 N and is not (16 µm), (17 µm) and (13 µm) for Crown K5 glasses, borosilicate glass BK7 and Zerodur® respectively.

In addition, the sizes of radial fingerprints and cracks increase with the load. By applying loads ranging from 0 to 90 N, a good reproducibility is observed on the load-displacement curves, although the three curves correspond to the borosilicate crowns BK7, crowns K5 and dense flints SF2, show a homogeneous behavior, The Zerodur glass, presents a discontinuity on the force-displacement curve during loading where the phenomenon of pop-in appears, which corresponds to an unstable propagation of the crack.

KIC toughness was calculated by the Evans and Niihara equation, with ranges of 9 to 19% and 11 to 22%, respectively; Whereas the tenacity values found by the Tanaka and Anstis relationships are between 6 and 9%.

In addition, the crack length is determined from the optical profilometer method to estimate the KIC toughness. This reliable technique makes it possible to obtain better results, therefore may be admissible, since the uncertainty found is minimal, it is of the order of 1 to 2%. Nevertheless, this technique has a disadvantage which lies in the constraint of the time involved and in the mode of operation in which the latter is carried out only if by combining by the conventional indentation method.

The average tenacity values found by the Tanaka and Niihara relation are quite large compared to Anstis and Evans. For example, the estimated toughness of the Zerodur glass is of the order of 1.26, 1.21, 0.95 and 1.14 for Niihara, Tanaka, Evans and Anstis respectively. The disadvantage lies in the uncertainty that is high enough for the Tanaka and Niihara relation, it is of the order of 5% and of the order of 2 to 3% that of Anstis.

Consequently, the Anstis relation can be chosen for the calculation of the KIC toughness of fragile materials (crown borosilicate glass, optical glass or ceramic glass), since the latter presents less dispersion compared to the other relations and is the one that is The most commonly used in the literature, which makes it possible to compare the tenacity of different materials.

References

[1] S. Palmqvist, "Method of determining the toughness of brittle materials, particularly sintered carbides," Jernkontorets Ann, Vol. 141, pp. 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, Vol. 33, pp. 629–663, 1962.

[3] X.Shaobo, L.Guangxi, L.Changchun, "Application of indentation technique in determinating fracture toughness of ceramics," Engineering Fracture mechanics, Vol. 31, pp. 309-313, 1988.

[4] A.Moradkani, H.Bahardvandi, M.Tadjari,H.Latifi,J.Martikhaninen, "Determination of fracture toughness using the area of micro-crack tracks left in brittle materials by Vickers indentation test," Journal of Advanced Ceramics, Vol.2,N°1, pp. 87-102, 2013.

[5] A.Iost, "Détermination de la ténacité de matériaux fragiles ou ductiles à partir de l'essai d'indentation", Rev. Métal., 110, pp.215-233, 2013.

[6] C.J.Evan, E.Paul, D.Dornfeld, D.A.Lucca, G.Byrne, "Material removal mechanisms in lapping and polishing," CIRP Annals-Manufacturing Technology, Vol.52, Issue2, pp.611-633, 2003

[7] H. H. Karow, "Fabrication methods for precision optics". Wiley-interscience ISBN0-471-70379-6 USA, 2004.

[8] K. Phillips, G. M. Crimes, T. R. Wilshaw, "On the mechanism of material removal by free abrasive grinding of glass and fused silica," Wear, Vol. 41, pp.327–350,1977.

[9] G.F.Vander Voort, "Metallography Principles and Practice". Mc Graw Hill Inc., p.100, 1982.

[10] J.C. Lambropoulos, Su Xu, Tong Fang, "Loose abrasive lapping hardness of Optical glasses and its interpretation", Applied Optics, Vol.36, N°7, pp.1501-15, 1997.

[11] M. Bentoumi, D. Bouzid, Isabelle H. and A Iost. "Etude comparative par indentation des caracteristiques mécaniques des verressodo-calciques et vitroceramiques rodés et polis par des grains abrasifs liés. Matériaux & Techniques 103, 611 (2015).