**REPETITIVE NANO-IMPACT TESTS AS A NEW TOOL TO MEASURE FRACTURE TOUGHNESS IN BRITTLE MATERIALS**

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*Keywords*: Nano-indentation; Repetitive-nano-impact test; High strain rate test; Dynamic hardness; Fracture toughness.

***Abstract***

Along this work, the feasibility of using repetitive-impact tests with a cube-corner tip and low loads for obtaining quantitative fracture toughness values will be shown. It will be displayed that the impacts are able to produce a cracking similar to the pattern developed for the classical fracture toughness tests in structural ceramics. Moreover, it will be shown how it is possible to identify the crack geometry evolution from Palmqvist crack to half-penny crack with each new impact being able to study the proper evolution of fracture toughness in terms of different indentation models and as a function of the strain rate, . Fracture toughness values of Al2O3 descend from ~6.10 (), to ~3.52 (). These values correspond to those found in the literature for α-Al2O3 demonstrating that the use of repetitive-nano-impact tests provides good reproducibility, high accuracy for reliable fracture toughness testing.

***1. Introduction***

Instrumented indentation technique is one of few available to investigate the mechanical properties along any kind of length range, i.e. from small volume, for instance: coating or thin layers, to bulk material. The resulting load-depth curve of indentation provides abundant and varied information that be used to determine several mechanical properties such as: hardness, Young’s modulus, yield strength, viscoelastic properties, etc [[[1]](#endnote-1)]. In addition, evaluation of the wear-resistance by hardness and/or scratch testing, has become routine for a multitude of materials [[[2]](#endnote-2),[[3]](#endnote-3),[[4]](#endnote-4)]. Nevertheless, the results are not always accurate, particularly when the surfaces are subjected to erosive wear during service and fail by a fatigue process. For the purpose of providing a solution, cyclic impact technique have been developed to extend the capability of depth-sensing indentation/scratch instrumentation to perform fatigue testing on a wide variety of surfaces, such as DLC and amorphous carbon [[[5]](#endnote-5),[[6]](#endnote-6),[[7]](#endnote-7)], plasma electrolytic oxidation surface [[[8]](#endnote-8)], polymers [[[9]](#endnote-9)] and numerous coatings for cutting tools [[[10]](#endnote-10),[[11]](#endnote-11),[[12]](#endnote-12),[[13]](#endnote-13)]. The cyclic impact technique produces a repetitive impact under a high stress and high strain rates simulating the fatigue conditions under repetitive contact conditions. Depending on the choice of parameters such as indenter geometry, acceleration distance, load, number of cycles and test frequency, different types of damages may be induced. Thereby, for a fixed acceleration distance, depending on the nature of the material and the load magnitude used to produce the impacts, different events can be observed during the test.

For ductile materials, at low impact acceleration loads, depth initially increases and then decreases (to negative values) with increasing number of impacts. This feature indicates that some thermal expansion, or a blistering phenomenon, is causing the sample to swell outwards from the indenter [5]. At mid-range impact loads, the depth asymptotically increases with successive impacts towards a deformation depth limit, i.e. fatigue limit, for that impact load. At high impact loads, depth swiftly increases during the first few impacts prior to reaching a plateau. As depth is then almost constant, each impact is effectively subjecting the material to a high strain fatigue rate. After a number of these fatigue impacts, the material is observed to fracture and fail.

In contrast, for brittle materials, if the load magnitude is not high enough to overcome the yield strength, depth will be constant along the impacts and it will display an abrupt jump when the fatigue limit is reached. However, if the load magnitude is high enough to achieve the yield strength, indentation depth will progressively increase with each new impact, as a consequence of the material fracture, until reaching a plateau where the material is submitted once again to fatigue conditions. Therefore, the resulting depth-number of cycles curve could reveal abrupt jumps with physical effects depending on the elasto-plastic properties of the material.

Although cyclic impact testing was not originally designed for measuring fracture toughness, depending on the material ductility and the load magnitude fracture of the surface may be achieved. Therefore, the question of whether these tests are suitable for calculating fracture toughness values, KC, which is considered as one of the most important properties for structural materials, is totally open. Its calculation would be of particular interest for coatings or thin layers, whose dimensions are between a few microns and hundreds of nanometers, and for which conventional methods such as single edge notched beam (SENB) [[[14]](#endnote-14)], chevron notched beam (CVNB) [[[15]](#endnote-15)] and double cantilever beam (DCB) [[[16]](#endnote-16)] are not applicable.

One possible way to determine fracture toughness makes use of the indentation models (IM), which have been developed for measuring the fracture toughness in mode I, KIC, with sharp indenters in bulk materials [[[17]](#endnote-17),[[18]](#endnote-18),[[19]](#endnote-19),[[20]](#endnote-20),[[21]](#endnote-21),[[22]](#endnote-22),[[23]](#endnote-23),[[24]](#endnote-24),[[25]](#endnote-25),[[26]](#endnote-26)]. In the majority of these IM models, KIC is proportional to two ratios: and , where E is the Young’s modulus, H is the hardness, P is the load and C is the crack length. Moreover, depending on the IM models, the coefficients *n* and *m* adopt different values. Nevertheless, the use of these IM models is, in general, limited to brittle materials because they are only applicable when assumptions of linear elastic fracture mechanics are met. In other words, the load-deflection response of cracked samples must show a linear elastic behaviour up to the point where unstable fracture occurs. Thereby, the plastic region localized at the origin of the crack tip has to be very small for the purpose of not affecting the overall load deflection behaviour. This limitation becomes more important in case of coatings and thin layers, since the thicknesses are reduced.

The first point to consider when assessing the applicability of the IM models (for the fracture toughness calculation from repetitive impact test) is to select the appropriate indenter, since it will determine the minimum load value for generating cracks. A Berkovich indenter showed a load threshold at which it is not possible to induce half-penny crack, therefore this indenter must not be used to estimate the fracture toughness for low loads. For small range of loads, Jang et al. [[[27]](#endnote-27),[[28]](#endnote-28),[[29]](#endnote-29)] recommended the use of cube-corner tips with an angle of 35.3º, which is sharper than the Berkovich and Vickers tips with values 65.3º and 68º, respectively. Thereby, the cracking threshold is reduced by 1-2 orders of magnitude due to its much sharper angle, which induced a higher stress level beneath the indenter [[[30]](#endnote-30),[[31]](#endnote-31)]. Therefore, it is possible to produce high plastic strain (200 %) in a small volume of material underneath the cube-corner tip for a small applied load, increasing the probability of crack nucleation and its propagation parallel to the loading direction. The second point is related to the crack geometry, since until nowadays this has been developed from a Vickers indenter (four-sided pyramidal) and, therefore, the use of these IM models for three-sided pyramidal is questionable.

Palmqvist [[[32]](#endnote-32)] suggested that the crack length, *c*, produced with Vickers indenter, emanating from the corners of its footprint (Pyramid-square) and they are connected underneath the indenter tip in the majority brittle materials. However, Niihara et al. [[[33]](#endnote-33)] distinguished between Palmqvist cracks, which are developed at low indentations loads and/or in materials with high toughness, from median or half-penny cracks by using different ratios: *c/a* or *l/a*, where *a* is the half diagonal length of the indenter and *l* is length from the corner of the indenter to the end of the crack [[[34]](#endnote-34)]. Both found that at higher values of crack-to-indent ratio (*c/a* ≥ 3.5) the crack profile corresponded with a half-penny type, and that at lower ratio values (*l/a* ≤ 2.5 or *c/a* ≤ 3.5) the crack profile is of Palmqvist type. Thereby, the main issue to be addressed is the veracity (or lack thereof) of these ratios as well as which values are more appropriate for the constant presents in the Anstins and Laugier indentation models ( and , respectively) [17,[[35]](#endnote-35)], in the case of a cube-corner geometry.

This work studies the feasibility of using repetitive impact tests with a cube-corner tip and low loads for obtaining quantitative fracture toughness values. For this purpose, it will be assumed that the impacts are able to produce a cracking in the coating, similar to the pattern developed for the classical fracture toughness tests in bulk materials, and therefore, from the crack developed in the repetitive impacts will be evaluated the suitability of the classical indentation models for measuring fracture toughness. For this purpose, fracture toughness will be analysed in a brittle bulk -Al2O3 material, since its mechanical properties are well known, and therefore theses can be compared with the reported values in the literature in order to validate the accuracy of this tool to be used in thin layers and/or coatings, which dimensions not allow use of traditional methods for fracture toughness determination.

***2. Experimental procedure***

*2.1 Materials*

Discs of pure Al2O3 (>99.98), denominated as HIP VitoxTM and prepared by Morgan Matroc Ltd, have been used. Dimensions of these discs are 2 mm thickness and 25 mm diameter. Surface roughness (Ra) is about 56 nm.

*2.3 Microstructural characterization*

Microstructural characterization was performed by conventional metallographic techniques and scanning electron microscopy (SEM) with a field emission gun (FEG) coupled with an energy dispersive X-ray (EDX) system for chemical analysis. Grain size pattern was revealed from discs broken at room temperature after immersion for several minutes in liquid nitrogen.

*2.3 Mechanical characterization*

Mechanical properties were determined by nanoindentation experiments by using a Nanotest Vantage equipment from Micro Materials (Wrexham, UK). Nanoindentation was performed on the top surface by using a Berkovich tip with a load ranging between 1– 5 mN. Loading and unloading time were fixed at 20 and 5 seconds in order to fix the strain rate at 0.05 and 0.2 s-1, respectively. In all cases, the holding time was fixed at 15 seconds. Berkovich hardness, HB and Young’s reduced modulus, ER, were evaluated from the load-depth indentation curves using the Oliver and Pharr method [1], by using the following equations:

(1)

(2)

In Eq. (1), Pmax and AC represent the maximum load and the projected contact area between the indenter and specimen at maximum load, respectively. In Eq. (2), andi, and Ef and Ei denote the Poisson’s ratio and the Young’s modulus for the film and the indenter, respectively. ER refers to the reduced Young’s modulus of the specimen determined according to Oliver and Pharr procedure. Young’s modulus (Ei) and Poisson’s coefficient (i) of the diamond Berkovich tip are 1141 GPa and 0.07, respectively. Both hardness and Young’s modulus results for a given load correspond to average values of at least 10 experiments.

The nano-impact pendulum impulse configuration of the Nanotest-Advantage was used to make repetitive impacts at regular intervals at the same location with precisely controlled force [6]. A solenoid connected to a timed relay was used to produce these repetitive impacts directly on the surface. Impacts for three different energy levels have been studied. To that end, a cube-corner diamond tip was accelerated, from a distance of 5000 nm, towards to the surface with three forces (15, 20 and 25 mN). The experiments were computer-controlled so that repetitive impact occurred at the same position every 5 seconds over a period of 600 seconds (120 impacts in total). The penetration depth was registered after every impact. Average values were obtained from at least 10 experiments performed in randomly selected areas on the surface of the coating.

***3. Principle of cyclic impact technique***

A repetitive impact test takes place as a solenoid is use to pull off the indenter (which can be of different geometries such as spherical, Berkovich, cube-corner, etc) from the surface and re-accelerated towards it from a small and fixed distance, producing consecutive impacts on the same spot [[[36]](#endnote-36)]. The increase in the depth reached along the repetitive impacts is related to plastic deformation and/or fracture below the surface impact point. Depending on the load, P, and the distance between the indenter tip and the surface, S, different energies transmitted, εt, to the surface can be selected. This energy is defined as:

, (3)

where hi (0 ≤ i ≤ n-1, n = impacts number) is the impact depth accumulated inside of the surface along the successive impacts. The larger S and/or P, the greater the initial εt with which the indenter impacts against the surface. Thereby, depending on the magnitude of the initial εt and the material ductility, the fracture of the surface may be induced [10,13,[[37]](#endnote-37),[[38]](#endnote-38)].

It is important to note that the energy transmitted is not constant along all impacts because of the distance between the indenter tip and surface changes with each new impact. The energy transferred during the impact is absorbed by the material similarly to that which occurs in a Charpy test where the energy absorbed corresponds to the difference between the initial and final potential energy, which is related to the difference between initial and final depth reached during the test. This energy, εa, can be defined as:

, (4)

where P is the load used during the repetitive impacts, hi is the impact depth reached inside of the surface from the second impact until the end of test and is the projected area after first impact and for new impact, which can be defined as:

, (5)

The reason the first impact depth, h1, is subtracted from the rest depths is because the first impact is actually a normal indentation made at high strain rate, , whose purpose is to produce a notch with similar shape to the indenter used. Therefore, hardness values for a cube-corner indenter, HCC, ought to be obtained from usual hardness definition:

, (6)

where *P* is the applied load during repetitive impact test and *AP* is the projected contact area in the first impact. However, this hardness can be only understood from the point of view of dynamic hardness, Hd, which is related to the decrease of dynamic flow stress with increasing stress beyond a certain strain rate value. After the first impact, depending on the material strength and the initial εt used, a different strain rate, , may be shown along the accumulative impact depth versus impact number. It is a consequence that is proportional to the ratio , and therefore the higher the growth rate of the depth, , and the lower the impact depth, h, the greater the value of . Thereby, from the repetitive impact test is possible to measure the dynamic hardness, Hd, evolution for each impact as a function of εa. For this it is necessary to subtract the initial impact depth, h1, from the rest of the impacts depths, because the first impact is actually an indentation with a high strain rate albeit lower in comparison with the rest impacts with strain rate comprised between 102 and 103 s-1. Thereby, the dynamic hardness may be defined as:

(7)

***4. Results***

*4.1 Microstructural characterization*

Cross sectional examination by SEM of the fractured samples, Fig. 1, reveals that Al2O3 discs are compact and dense, with average grain size of about 2 µm.

*4.2 Mechanical characterization*

*4.2.1 Nano-indentation testing*

Figure 2 shows the variations of the Young's reduced modulus, ER, and the Berkovich hardness, HB, as a function of the maximum indentation depth, h. It can be seen that the Berkovich hardness and Young's reduced modulus do not show any gradient with the indentation depth, which is indicative of a high microstructural homogeneity. The HB and ER values are 22.8±1.7 and 313±4 GPa, respectively. Knowing the value of Poisson's ratio (ν= 0.22 for Al2O3), it is possible to calculate the true Young's modulus E from Eq. 2, whose value is reported together with HB and ER values in table 1.

*4.2.2 Cyclic impact testing*

In the particular case of Al2O3, depending on the magnitude of the initial εt, its yield strength, and therefore its maximum strength may be reached at the first impact. Thereby, if the initial εt valueis sufficiently high to overcome it, the nucleation of a single crack and its propagation from the apex of the footprint may take place. This would be reflected in big decrease in the initial growth rate of the depth, , along the curve of the accumulative impact depth, h, as a function of the impacts number (Fig. 3). It is important to remark that all h values showed in this figure have been obtained by subtracting the depth reached at the first impact, h1, since it is actually a normal indentation. After a certain number of impacts, which could be attributed as a continuous opening of the surface around the cube-corner indenter, initially high values approach zero, reaching a plateau where there is not a change in the impact depth, h. The impacts along the plateau produce the nucleation of new small cracks underneath the tip, whose natural tendency is to coalesce to form a big crack. The aperture of this new crack is reflected as an abrupt jump in the impact depth, rapidly increasing the value again until a maximum impact depth value is reached. This event indicates the production of crack coalescence, again resulting in the opening of the surface. Nevertheless, depending on the magnitude of the initial εt value, more than one plateau may be reached, as can be seen in Figure 3. For an εt value of 75 nJ there is only one plateau, whereas for an εt value of 125 nJ there are three.

As has been mentioned above, the first event is actually an indentation, which aims to produce a notch with similar shape to a cube-corner tip. For the three initial values of εt studied: 75, 100 and 125 nJ, the h1 values achieved are 163±8, 184±9 and 217±6 nm, respectively. As expected, the higher the initial εt, the greater the h1 and, therefore, the larger the initial size of the notch is. From these h1 values it is possible to calculate the projected area, Ap, associated with the initial notch, and finally their hypothetical cube-corner hardness, HCC, (Table 2). It is important to note that no correction related to size of the cube corner tip has been used in HCC calculus. This HCC value is almost independent of the initial εt values, as can be seen in table 2. Nevertheless, it is possible to see how the higher the initial energy, the greater the notch is, and therefore the higher the stress level concentrated below the vertex of the cube corner footprint. If the comparison between both hardness values (HCC and HB) is done, it is clear to see as the ~216 GPa, corresponding to cube corner hardness value, obtained for εt =75 nJ, is too high in comparison to ~23 GPa, corresponding to the Berkovich hardness value, HB. This discrepancy is related to the fact that this indentation is done a high strain rate, , and therefore its value can be only understood from the point of view of dynamic hardness.

The dynamic hardness tests are typically performed with a spherical indenter, which is accelerated against a surface. Along the successive impacts, a large fraction of the impact energy is elastically accommodated due to the low impact depths compared to the indenter radius. This elastic energy fraction causes a high surface hardening, which will be higher for greater loads. However, in our case, the indenter has a cube-corner tip, which significantly differs from spherical. Thereby, along the first successive impacts, the indenter progresses inwards, opening the Al2O3 surface around the indenter, without causing plastic deformation because of the experimented high strain rates. Therefore, the hardness values are obtained from an elastic process, being similar to the dynamic hardness, Hd, whose values are dependent on the strain rate magnitude, which is not constant. As the number of impacts increases, progressively decreases because, as decreases and h increases with each new impact, εt does not remain constant throughout all impacts. Thereby, it is possible to study the dynamic hardness evolution, as the impacts number increases, from the Eq. 7, since for each new impact the decreases until achieving a plateau in its value.

Figure 4 shows the evolution in Hd for the lowest and highest initial εt values (75 and 125 nJ). In both cases, the initial dynamic hardness values show a strong decrease till reaching the initial plateau, whose value is coincident with the HB value. The impact number required to achieve the HB value for the Al2O3 depends on the initial εt value. The higher the εt value, the greater the depths reached in each impact and the lower the number of impacts necessary to achieve it. Thus, for an initial energy of 75 nJ the HB value is reached after 14 impacts, whereas for an energy of 125 nJ, 8 impacts are needed. It is evident that the energy absorbed in each impact, εa, by the Al2O3 is directly related to the evolution in εt. This absorbed energy is per unit volume, i. e. the greater the impact depth, h, the higher the energy consumed in the increasing indented volume. However, the amount of energy transmitted, with each new impact, gets smaller because of the increase of h. Thereby, it is possible to define an energy absorbed, εa, unique for each impact. This εa is transformed into an increase in the notch size as the surface is opening around the cube-corner tip. From Eq. 4 it is possible to calculate its evolution as a function of the impacts number for the three initials values of εt studied. The absorbed energy evolves similarly to the dynamic hardness. The higher the initial εt value, the lower the number of impacts are required to reach the first plateau, due to increasing εa with each new impact. In fact, in brittle materials like Al2O3, the initial εa values can be considered similar to the impact strength, *IS*, because their plastic strain is negligible. This *IS* represents the ability to absorb mechanical energy in the process of deformation (elastic or plastic) and fracture under impact loading, i. e. the amount of energy absorbed before fracture. If there is only elastic deformation, this impact strength is similar to a resilience, which is defined as:

, (8)

where *σI* represents the material impact stress and *E* its Young’s modulus.

Thereby, it is possible to see how this impact stress changes with each new impact, since is not constant along the test. To calculate this *σI* evolution is necessary equalize the absorbed energy with the impact strength in each new impact and therefore it is possible to see its evolution. The higher the impact depth, *h*, the lower the impact stress, *σI*, is. On the other hand, the impact stress reaches a plateau when εt is not great enough to induce an opening in the surface around the indenter. i.e. when the energy absorb in each impact is not enough to propagate the crack generate below the apex of the footprint, increasing its size. Along the plateau, the impact process can be considered as quasi-static and therefore, the *σI* value is equal to the yield strength of the Al2O3. Therefore, the yield strength of the alumina can be calculated with each new impact, as shown in Fig. 5. For the lower initial energy transmitted, εt = 75 nJ, the *σI* value achieve along the plateau is ~ 275 MPa, whereas for the highest energy transmitted, εt = 125 nJ, this value is ~ 265 MPa. These values, close to each other, show how they are similar to the typical values showed by α-Al2O3. Thereby, it is possible to study the evolution of the impact stress from high strain rates value ( ~103 s-1) to quasi-static strain rates value ( ~10-3 s-1).

Proof of similar behaviour has been showed for several authors. Saikat Acharya et. al. have shown as alumina ceramics, beyond the critical strain rate (dε/dt), is sensitive to variations in strain rate [[[39]](#endnote-39)]. At (dε/dt) ≤ 102 s-1 the subcritical growth of axial crack causes the strain rate sensitivity of compressive strength to occur. However, for (dε/dt) ≥ 103 s-1 the crack inertia controls the strength. Thereby, their results indicates that at high strain rates the magnitudes of compressive strength of alumina ceramics are sensitive to grain size, processing methodology and experimental conditions.

On the other hand, once this first plateau is achieved, the ability to prevent the coalescence of small cracks (before abrupt fracture of the surface and its subsequent growth by fatigue) will depend of the initial value of εt. After of the abrupt fracture showed in figure 5, i.e., after the coalescence of small cracks as a consequence of fatigue process along the plateau, the impact depth is too high and, therefore, the energy transmitted is not enough to produce the coalescence of new cracks and therefore, its growth by fatigue. The impact depth tends to reach a maximum value, which will be higher for a greater initial εt.

*4.2.3. Fracture toughness*

As stated previously, depending on the indenter geometry, the initial εt, and the nature of the material, the nucleation and propagation of a crack can be induced along a nano-impact test. From the first impact, which is actually a high strain rate indentation, is possible to produce a notch with the same shape as the cube-corner indenter, whose purpose is to concentrate the maximum possible stress around the impact point just underneath of the apex of this notch. Therefore, if initial εt is high enough, to match the material yield strength, and the capacity of the material for accumulates plastic deformation is negligible; the hypothetical crack (nucleated in the first impact) will be propagated from the apex of the notch along the next successive impacts and its length corresponds to the accumulative impact depth, h, reached with each new impact. From the graphical point of view, this crack propagation is equivalent to the opening of the material surface around the indenter because the successive increase in the impact depth (accumulated with each new impact) is similar to the length travelled by the crack. Thereby, once the notch is made, the fracture toughness of brittle materials, might be obtained, in theory, from a single nano-impact test.

At this point, it is important to remember that the impacts are done at high strain rate, ~103 s-1, and therefore, the fracture toughness value will be higher than that obtained from quasi-static indentation where strain rate value is typically ~10-3 s-1. Indeed under quasi-static compressive strain rate conditions and in the absence of lateral restrictions, the normal failure mechanism is via nucleation, growth and coalescence of a multitude of axial micro-cracks. For values up to 102 s-1, however, there is a transition into a regime of rapid strain rate strengthening, whereby the cracks are nucleated and propagated at such a rate that the strength of the material is proportional to the strain rate, even for brittle materials, for which an increase in the fracture strength [[[40]](#endnote-40),[[41]](#endnote-41),[[42]](#endnote-42)] and fracture toughness [[[43]](#endnote-43),[[44]](#endnote-44)] values is produced. However, the initial εt value does not remain constant, therefore decreasing the amount of energy transferred with each new impact. This feature causes the fall in strain rate, , because the grow rate of the depth, , decreases and the impact depth, h, increases with each new impact. Since εt is high enough for opening the surface around the indenter, the crack propagation will take place and will be able to measure the fracture toughness evolution as a function of the strain rate.

*4.2.3.1 Notation, assumptions and approaches*

Given that the impacts are similar to high strain rate indentations, it would be possible to use the same models that have been developed to analyse the fracture toughness in mode I, since the forces generated by the cube-corner tip have the same two components. The first one acts normal to the surface and drives the cube-corner indenter deeper with each new impact. The second one acts perpendicular to the impact direction, separating the surface under the tip. This second force produces tensile stresses, which are normal to the impact depth reached, i.e. perpendicular to the crack propagation from the apex of the tip. However, the accurate KIC measurements require a proper knowledge of the crack morphology in order to select the most appropriate expression for evaluating KC. Two of the most commonly used fracture toughness formulations in mode I, KIC, have been proposed by Anstis et al. [17] and by Laugier [23]. Both equations are based on the Lawn et al. extension of the Evans and Charles analysis [[[45]](#endnote-45)]. Lawn et al., by using Hill’s expanding cavity solution for an elastic-plastic solid and assuming a half-penny crack configuration, suggested the following expression to calculate KIC [[[46]](#endnote-46)]:

(9)

where *P* is the indentation load, *c*, is the crack length, i.e., the accumulated value of h, *E*, is the Young’s reduced modulus, *H*, is the hardness and is the calibration coefficient, which depends on the tip and crack geometries. For half-penny cracks, Anstis et al. proposed for the calibration coefficient a value of 0.016±0.004. This value is well established when half-penny cracks are generated with Vickers indenters in the range of macro and microindentation. However, the non-axisymmetric nature of Berkovich or cube-corner indenters does not always allows half-penny cracks to join below the footprint and, therefore, the applicability of Eq. 9 is questionable. For this reason, other expressions have been developed for the calculation of fracture toughness from superficial cracks, such as Palmqvist crack [17]. Laugier adapted the Lawn half-penny formalism to take into account the actual crack morphology [21]:

(10)

where *a* and *l* are the lengths related to the half of the diagonal and the crack length from the apex of the footprint, respectively, while is the constant similar to , which is around 0.016 for Vickers indenters. However, the use of a cube-corner indenter is more appropriate because its threshold load to induce cracks in a normal indentation is several orders of magnitude lower than for a Berkovich indenter. Therefore, the values for the and parameters must be recalculated for a cube-corner indenter. If it is assumed that both parameters are related only to the tip angle, the expected value for a cube-corner indenter is 0.033. However, depending on the material toughness range, different values as 0.0319 [[[47]](#endnote-47)], 0.036 [28], 0.040 [[[48]](#endnote-48)] or 0.057 [[[49]](#endnote-49)] have been reported. The main reason for these discrepancies is related to the equation chosen to fit the experimental data, which is determined by the crack geometry.

*4.2.3.2 Fracture toughness calculation from Anstins indentation model*

A priori, the selection of the Anstins IM seems to be the right one, since the surface is opening more and more with each new impact and the material slide around the lateral surface until the initial high achieve quasi-static conditions. This way of breaking the surface is similar to half-penny crack morphology; since the hypothetical crack origin is located in the apex of the footprint, i.e., in the vertex of the initial notch, which acts as a stress concentrator. Crack growth, from this point, will produce morphology similar to a half-penny-crack. Thereby, from Anstins formulation and knowing the true Young modulus, E, obtained from conventional nano-indentation tests, the dynamic hardness values, Hd, and the crack length, h, for each new impact it is possible to calculate the fracture toughness, Kc. Figure 6 shows the KIC evolution obtained from Hd values and the crack length for the loads of 15 and 25 mN and with = 0.040. As happened with the Hd and the IS values, KIC exhibits similar behaviour, decreasing until reaching a constant value. In both cases, KIC values for the first impact, , and the value achieve on the initial plateau, , are virtually identical: 5.33 MPa, for 15mN, and 5.64 MPa, for 25mN, and 3.48 MPa, for 15mN, and 3.56 MPa, for 25mN, respectively. The equality of KIC values for different loads, demonstrates the validity of the Anstis formulation (Eq. 9) for calculating fracture toughness, since the proportionality between the ratio and KIC, regardless of the load value, is retained. Nevertheless, the difference between using a higher or lower load, lies in the greater or smaller impacts number needed to reach the initial plate where the condition of quasi-static strain rate are achieved. For a given distance S, between the indenter tip and the surface and the greater the load, P, few impacts are required to achieve the quasi-static strain rate. Because for impacts performed with a higher initial εt, the impact depth, h, reached with each new impact is greater, and therefore the decrease in the strain rate is more marked. Similarly, in the table 3 are collected the values, whom have been also calculated for the rest of values. As it can be seen, fracture toughness values obtained for value lower than 0.040 are too low if these are compared with the recently values reported in the literature for Al2O3 (~5.2 MPa) with similar hardness (~19 GPa) and density (~98 %) [[[50]](#endnote-50),[[51]](#endnote-51)]. However, for =0.057, value is ~5 MPa, quite similar to the one reported in the literature referred to above. Thereby, =0.057 may be the most appropriate value to calculate the fracture toughness from Anstins IM.

On the other hand, whereas in the Anstis formulation (Eq. 9) in which the ratio between the hardness and Young's modulus is implicit, another possibility for the calculation of KIC makes use of the only one Berkovich hardness value, HB (22.8 GPa), instead of the Hd values. For comparison, figure 6 also shows the evolution in fracture toughness values, replacing in Eq. 9 the Hd values for the constant HB value. In this figure it can be seen that the values, obtained using HB, are extremely high and do not correspond with real ones. As in the previous case, fracture toughness values evolve until reaching the same plateau. Initial value is shown in the table 3 for the two higher values (0.040 and 0.057). Therefore, the use of the Berkovich hardness values is not recommended because of it is a constant value not reflecting the evolution of hardness, which is a consequence of the strain rate evolution. Nevertheless, these fracture toughness values show also as the strain rate drops from a value ~103 s-1 to achieve the quasi-static conditions (10-3 s-1); this being point at which both hardness values, dynamic and Berkovich, are coincident.

***5. Discussion***

Despite the number of works addressing the evaluation of fracture toughness by nanoindentation tests, KIC is often evaluated using Eqs. 9 and 10, without proper consideration of crack morphology. This morphology depends on the indentation load, tip geometry and material toughness. For cracks with half-penny morphology, the condition that must be met is that the c/a ratio has to be larger than 3.5. In contrast, for Palmqvist crack morphology, the l/a ratio has to take a smaller value between 1.1 and 2.5. Thereby, the main issue to be addressed is the veracity (or lack thereof) of these ratios and therefore, which values are more appropriate for the constant: and , in the case of Cube-corner geometry.

N. Cuadrado et. al. [46] have suggested different values for these constants depending on the crack morphology. These authors have demonstrated that the crack morphology has a semi-elliptical shape and therefore, Eq. 10 should be used to calculate the fracture toughness. Thereby, the use of the equation proposed by Astins (Eq. 9) is fully justified while the condition c/a > 3.5 is maintained. Therefore, it is necessary to calculate the evolution of a, which is the length from the centre of the projected area until the corner, and l, which is the length from the corner of the indenter until the end of the crack, in order to identify the evolution of h/a and l/a parameters with each new impact. For this we have to calculate different lengths related to cube-corner geometry:

, (11)

where h1 is the impact depth achieved by the indentation done at high strain rate and θ is the semi angle, whose value for a cube-corner indenter is 35.26°;

o, (12)

where d is the length from the point f, which is located at the middle of a lateral, belonging to the projected area, to the opposite corner;

o, (13)

where t is the length from the point f to the centre of the projected area. Once we know the values of the parameters d and t, it is possible to calculate the parameter a, i.e., length from the centre of the projected area to the corner, since this value corresponds to the subtraction of the previous parameters (a = t - d). Thereby, from the initial value of length a, it is possible to calculate the ratio h/a, where varies from 2 to n (n = number of impacts). The calculation of the other characteristic length, l, is easier than previous one because l is equal to the subtraction of known parameters: .

Figure 7 shows the evolution in fracture toughness for the lowest εt, using the value of 0.040 for Anstins indentation model and 0.057 for Laugier indentation model. The blue colour data shows the KIC evolution calculated from Eq. 9 (Anstins IM), whilst yellow data shows the KIC evolution calculated from Eq. 10 (Laugier IM). For the first impact h/a and l/a values are 1.63 and 0.64, respectively. Thereby, the conditions for any formulations are not achieved and fracture toughness values are too high (Table 4). However, from the second to the eleventh impact Laugier IM for Palmqvist type crack morphology condition is met (1.1≤ l/a ≤ 2.5) and fracture toughness values descend from 6.01 to 3.65 . Then, for higher number of impacts, Laugier condition is not met because there is a change of crack morphology from Palmqvist type to half-penny type. As a consequence, Anstins IM can be used to obtain fracture toughness values, since from this impact h/a is higher than 3.5. From the eleventh to fifteenth impact, the fracture toughness evolved from 3.62 to 3.48 where both dynamic and Berkovich hardness values are coincident. Thus, using the appropriate indentation model for each impact is possible to see as fracture toughness values decrease from ~ 6 to ~ 3.50 , as a consequence of the strain rate, along the repetitive impacts, decrease from high values to quasi-static conditions. This evolution in fracture toughness values ​​highlights the importance of the impact depth, h, value reached in each new impact, since in some way it is equivalent to the thickness in which the crack propagates. As it is well known, for relatively thin specimens, the KC value depends on the thickness. This dependence is manifested as a decrease in the values ​​of KC by increasing the thickness, exactly equal occur in the present study with the crack propagation through thickness each new impact up to the plateau. In this plateau, strain rate is quasi-static and KC values ​​are independent of the impact depth, h, or equivalently to the thickness in the case of thin specimens. Therefore, the performance shown by the fracture toughness values, obtained from repetitive-nano-impact test, reflect changes in measurement conditions from plane stress to plane strain measurement conditions from which is possible to obtain the typicalKIc values.

Along the plateau, where the quasi-static condition is achieved, crack propagation is interrupted because the energy transmitted in the successive impacts is not enough to propagate the crack. Thereafter, the surface is subjected to a fatigue process where a multitude of cracks are nucleated until a new propagation process takes place. In any case, the choice of the value 0.040 for Anstins IM and 0.057 for Laugier IM appears to be adequate, since along the plateau both formulations show the same value, which is typical for this ceramic material in quasi-static indentation conditions. Robustness of our approach is supported by identical fracture toughness values found in the literature for α-Al2O3 such as: 3.50 in the work of Bocanegra-Bernal et al. [47], 3.20 in the work of Bartolome et al. [[[52]](#endnote-52)], or 3.90 in the work of Anstins et al. [17].

***6. Conclusion***

In this work we considered repetitive nano-impact tests as a tool to obtain fracture toughness of brittle materials. Two distinct types of tests can be performed depending on the value of the initial impact energy, εt, i.e, depending on the value of the product between the load, P, and the distance travelled by the tip of the indenter, S, to the surface. The surface of the material is subjected to a fatigue test if the depth reached at the successive impacts is kept constant, since the value of the initial energy is low. If, however, the value of this energy is higher, the depth reached by tip, h, increases with each impact.

In the particular case of brittle material, which ductility is negligible, using sharp indenter geometry like cube-corner and an appropriate initial εt value, the nucleation and propagation of a crack is induced. This crack propagates from the apex of the notch, which is produced in the first impact, until it reaches a stationary value as a consequence of the decrease in the initial εt value. Therefore, the crack length can be measured, since it is similar to the opening of the material surface around the indenter due to successive increasing of the impact depth accumulated with each new impact. However, increase of the accumulative impact depth with each new impact leads to drop in an initial strain rate, , and initial impact energy, εt. During the impact test the strain rate decreases from high strain rate conditions (103 s-1)to quasi-static conditions (10-3 s-1). Thus, it is possible to study the evolution experienced by different mechanical properties such as hardness, yield strength and fracture toughness as a function of successive impacts.

On the other hand, repetitive nano-impact test, carried out with cube-corner geometry, have proven to be highly efficient in selecting the correct indentation model for calculating fracture toughness, since it allows studying the evolution of c/a or l/a ratios as a function of the impacts. Thereby, it is possible to identify for each impact the crack geometry, which evolves from Palmqvist crack (Laugier IM) to half-penny crack (Anstins IM). Consequently, we are able to study the proper evolution of the different values of fracture toughness in terms of both indentation models (Laugier and Anstins) and as a function of the strain rate decrease. Fracture toughness values for bulk discs of Al2O3 decreased from ~6.10 , for high value, to ~3.52, for quasi-static value. These values are to identical to those reported for α-Al2O3, which demonstrates that proposed method could capture well not only qualitative information related to fracture resistance of brittle materials but as well quantitative aspect, i.e. value of fracture toughness.

**Acknowledgements**

The authors wish to express their thanks for the financial support of the Czech Science Foundation through the project 14-32801P. Nanoindentation was carried out on equipment funded by EPSRC through EP/K005103/1 project.

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