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Repetitive nano-impact tests as a new tool to measure fracture toughness in brittle materials

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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, $\dot{\varepsilon}$. Fracture toughness values of Al_2O_3 descend from $\sim 6.10\, MPa\sqrt{m}$ ($\dot{\varepsilon} = 10^3 s^{-1}$), to $\sim 3.52\, MPa\sqrt{m}$ ($\dot{\varepsilon} = 10^{-3} s^{-1}$). These values correspond to those found in the literature for α-Al_2O_3 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]. In addition, evaluation of the wear-resistance by hardness and/or scratch testing, has become routine for a multitude of materials [2–4]. 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 [5–7], plasma electrolytic oxidation surface [8], polymers [9] and numerous coatings for cutting tools [10–13]. 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

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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, K_C, 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], chevron notched beam (CVNB) [15] and double cantilever beam (DCB) [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, K_{IC}, with sharp indenters in bulk materials [17–26]. In the majority of these IM models, K_{IC} is proportional to two ratios: $\left(E/H\right)^n$ and P/C^m, 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. [27–29] recommended the use of cube-corner tips with an angle of 35.3°, which is sharper than the Berkovich and Vickers tips with values 65.3° and 68°, respectively. Thereby, the cracking threshold is reduced by 1 or 2 orders of magnitude due to its much sharper angle, which induced a higher stress level beneath the indenter [30,31]. 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] 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] 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]. Both found that at higher values of crack-to-indent ratio ($c/a \geq 3.5$) the crack profile corresponded with a half-penny type, and that at lower ratio values ($l/a \leq 2.5$ or $c/a \leq 3.5$) 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 (χ_ν and ξ_ν^R, respectively) [17,35], 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 α-Al_2O_3 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 Al_2O_3 (>99.98), denominated as HIP Vitox™ 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.

2.2. 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 s 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 s. Berkovich hardness, H_B and Young's reduced modulus, E_R, were evaluated from the load-depth indentation curves using the Oliver and Pharr method [1], by using the following equations:

$$H = \frac{P_{max}}{A_C} \quad (1)$$

$$\frac{1}{E_R} = \frac{1-\nu^2}{E_f} + \frac{1-\nu_i^2}{E_i} \quad (2)$$

In Eq. (1), P_{max} and A_C represent the maximum load and the projected contact area between the indenter and specimen at maximum load, respectively. In Eq. (2), ν and ν_i, and E_f and E_i denote the Poisson's ratio and the Young's modulus for the film and the indenter, respectively. E_R refers to the reduced Young's modulus of the specimen determined according to Oliver and Pharr procedure. Young's modulus (E_i) and Poisson's coefficient (ν_i) of the diamond Berkovich tip are 1141 GPa and 0.07, respectively. Both hardness