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Loading rate effect on the mechanical behavior of zirconia in nanoindentation

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ABSTRACT

This paper reports the loading rate effect on the mechanical behavior of zirconia using nanoindentation and in situ scanning probe imaging techniques. Nanoindentation tests were performed at a peak load of 10 mN and 0.1–2 mN/s loading rates. The results show that the contact hardness increased by 31% with the loading rate while the Young's modulus was loading rate independent (ANOVA, $p > 0.05$). A strain rate sensitivity model was applied to determine the strain rate sensitivity and the intrinsic contact hardness. A pressure-sensitive idealized yield criterion model was applied to analyze the pressure hardening coefficient and the intrinsic compressive yield stress. Extensive discontinuities and largest maximum and contact depths were also observed on the force–displacement curves at the lowest loading rate. These phenomena corresponded to nanoindentation-induced strain softening. The in situ scanning probe images of indentation imprints showed plastic deformation without fracture at all loading rates and dislocation-induced pileups around indentation imprints at the low loading rate. The amount of pileups decreased with increase in loading rate. Finally, these results provide scientific insight into the submicron material removal mechanisms for zirconia during sharp abrasive machining.

1. Introduction

Zirconia has high strength, high fracture toughness, low thermal conductivity, high ionic conductivity and biocompatibility. It is widely used in load-bearing and wear-resistant structures in engineering and medicine/dentistry as thermal barrier coatings, fuel cell electrolytes, pump components, valve seals, bushings/bearings, body implants, dental restorations, etc. [1–6]. The high fracture toughness of zirconia is associated with the tetragonal-to-monoclinic phase transformation [7–9] or ferroelastic domain switching [10,11], resulting in a high damage tolerance [12]. Zirconia structures are usually shaped by abrasive grinding and polishing. In dentistry, sandblasting with alumina abrasives is also used to treat zirconia cementation surfaces for improved adhesion [2,13,14]. These abrasive processes can trigger the tetragonal-to-monoclinic phase transformation in zirconia which can enhance its strength [15,16]. However, the tensile stresses generated by abrasive machining can also induce surface and subsurface damage in zirconia that change its surface properties and deteriorate its structural integrity and material reliability [17,18]. To improve zirconia's mechanical performance, machining-induced damage in the material must be minimized by applying ductile mode grinding conditions [19,20].

Understanding the mechanical behavior of zirconia can provide scientific insights into ductile regime machining mechanisms for the material, which can be simulated by indentation mechanics [21,22]. The mechanical contact conditions in indentation are geometrically similar to the conditions in abrasive machining. Machining forces, cutting speed and abrasive geometries can be simplified by indentation loads, loading rates, and indenter geometries, respectively. Thus, indentation features reflect the essential abrasive machining mechanics.

Indentation techniques have been used to characterize the mechanical behavior of zirconia. In particular, Hertzian indentation was performed to study the deformation and fracture of zirconia [23–25], which is ideal to measure the elastic properties and the elastic–plastic transitions [26]. In general, sharper indenters, such as Vickers, can induce larger stresses and strains sufficient to displace large material volumes and impose large amounts of shear stresses allowing easy plastic deformation [26,27]. Thus, Vickers indentations were used to study the plastic behavior of zirconia [28–31]. Those microscale indentation studies also demonstrated fracture mechanisms for zirconia in which critical load thresholds for median/radial crack formation were exceeded.

As the scale of deformation becomes small in the sub-threshold region, materials can be removed from zirconia by plastic flow

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leaving a crack-free surface [32]. Nanoindentation can be used to characterize such scale deformation events, including probing the contact hardness, H_c, and Young's modulus, E, based on the Oliver and Pharr method [33,34]. Combining with in situ scanning probe imaging, nanoindentation can also discern various material deformation behaviors underneath the indenter [35]. Several nanomechanical studies were conducted on the zirconia behavior influenced by grain size [36,37], ageing [38–40], indentation size effect [41] and crystallographic orientation [42]. However, those studies were conducted at constant loading rates which assumed equilibrium deformation conditions. In fact, loading rates can affect the material hardness [27], which is an important material design consideration where hardness is taken as a basis for predicting strength, machinability, wear and erosion characteristics.

At the microscale, loading rate effects on the mechanical behavior of alumina, alumina oxynitride, silicon nitride, silicon carbide, zirconia and Pyrex glass have been investigated in post-threshold indentations in which fractures occurred [29,30,43]. It was also found that loading rates influenced the behavior and properties of bulk metallic glasses in nanoindentation [44–48]. However, the loading rate effect on the mechanical behavior of zirconia is not studied at the submicron scale, which is vital in the understanding of removal mechanisms in ductile regime machining for the material.

This paper aimed to study the loading rate effect on the mechanical behavior of zirconia using nanoindentation and in situ scanning probe imaging. The contact hardness, H_c, and Young's modulus, E, were measured as a function of loading rate in the range 0.1–2 mN/s at the peak load of 10 mN. Nanoindentation-induced discontinuities in zirconia were markedly manifested in loading–unloading curves at the lowest loading rate whereas they tended to be imperceptible at the highest loading rate. These loading rate-dependent mechanical responses of zirconia to different phenomena were analyzed. Finally, the linkage between the nanoindentation responses and the modes of material removal of zirconia was established.

2. Experimental procedures

2.1. Materials

Cylindrical blocks of pre-sintered zirconia (IPS e.max ZirCAD, Ivoclar Vivadent) were selected in this study, which are commonly processed in chair-side dental CAD/CAM systems for crowns and bridges. This material consisted of 87–95 wt% ZrO_2, 4–6 wt% Y_2O_3, 1–5 wt% HfO_2, 0.1–1 wt% Al_2O_3, 97% tetragonal and 3% monoclinic phases [4,49,50]. Indentation samples with dimension of 15 mm × 15 mm × 2 mm were obtained using the procedures described by Alao and Yin [49]. Briefly, they were metallographically ground and polished using successively finer diamond paste to obtain mirror surfaces. The root-mean-squared surface roughness, R_q, was approximately 72.3 nm from the scan area of 50 µm × 50 µm [49] using scanning probe imaging (NT-MDT NTEGRA, Hysitron, USA). After polishing, those samples were sintered in a furnace (MTI GSL1500X) to 1300 °C for 2 h at 10 °C/min heating rate and then naturally cooled to room temperature. These sintering conditions agree with the specifications for clinical zirconia restorations [51].

Sintered zirconia samples were rescanned using the scanning probe imaging. Fig. 1(a) shows a scanned sintered zirconia surface on which the root-mean-squared surface roughness, R_q, was 102 nm from the same scan area of 50 µm × 50 µm for pre-sintered samples. This indicates that sintered zirconia surfaces became rough due to sintering-induced grain coarsening, shrinkage, residual stresses and monoclinic to tetragonal phase transformation. To meet the sample roughness requirement for nanoindentation, sintered samples were repolished using the same metallographic process for pre-sintered zirconia. After repolishing, zirconia surfaces were scanned using the scanning probe imaging. Fig. 1(b) shows a scanned repolished sintered zirconia surface, obtaining the root-mean-squared surface roughness, R_q, of approximately 7.7 nm in a scan area of 50 µm × 50 µm and an average zirconia grain size of 500 nm.

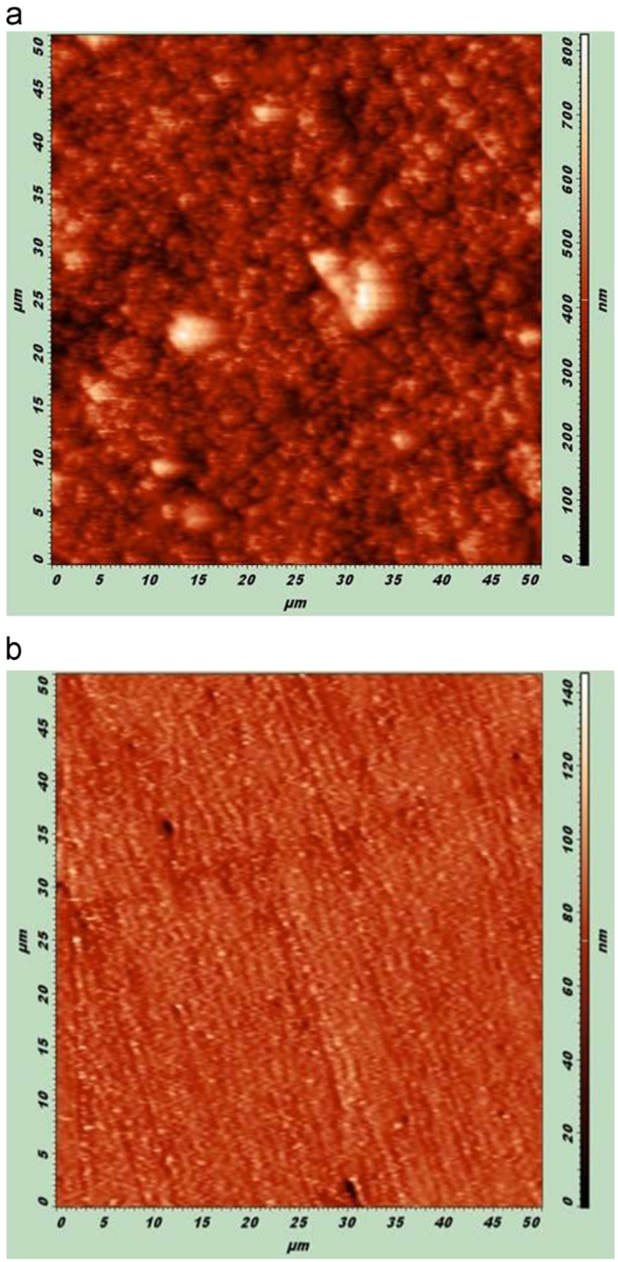

Fig. 1. 2D scanning probe images of zirconia surfaces (a) after sintering and (b) after repolishing.

2.2. Nanoindentation tests

Nanoindentation tests were conducted using a Hysitron Triboscope (Hysitron, USA), which has load and depth sensing resolutions of 1 nN and 0.0002 nm respectively. A Berkovich diamond indenter of approximately 150 nm tip radius was applied. Prior to