Ductile-Brittle Transition Behaviour of Lithium Metasilicate/Disilicate Glass-Ceramics in Diamond Grinding

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Abstract. Lithium metasilicate/disilicate glass-ceramics (LMGC/LDGC) are attractive composite materials for all-ceramic dental restorations due to their promising strength and biocompatibility. However, their strong mechanical properties make it hard to machine with current dental chairside CAD/CAM-milling. The root cause is the unclarity of their mechanical behaviour under dynamic abrasive machining. This paper conducted single-diamond scratching experiments on LMGC and LDGC to extract the actual diamond grain-material contact in grinding to understand their ductile-brittle transition behaviour. Scanning electronic microscopy (SEM) observation was performed to identify the fully-ductile, ductile-brittle and fully-brittle regimes of the two studied materials. 3D laser confocal microscopy (LCM) was used to measure the critical cutting depths for these regimes. The critical cutting depths for fully-ductile and fully-brittle regimes are 89 nm and 133 nm for LMGC, and 209 nm and 428 nm for LDGC, respectively. These results provide an in-depth understanding of ductile-brittle transition behaviour of LMGC/LDGC under abrasive machining and a guidance for dental technicians to choose proper machining conditions for high-quality LDGC restorations.

1. Introduction
Recent decades have witnessed the growing need for high-quality all-ceramic dental restorations with promising durability, excellent biocompatibility and superior aesthetics [1]. Owing to the high strength and improved translucency, restorations made from lithium disilicate glass-ceramic (LDGC) have attracted increasing attention from patients and clinicians [2, 3]. However, a study for three-unit-fixed LDGC dentures indicates 87.9% survival rate after wearing for ten years [4]. The primary factor for the failure of LDGC restorations was uncovered to be fractures and chippings generated during machining [5]. Therefore, eliminating these machining-induced damages is necessary for the production of LDGC restorations.

LDGC is generated from the SiO2-Li2O-K2O-ZnO-Al2O3-La2O3-P2O5 system in dental laboratory by precise control of nucleation and crystallization [1]. It consists of approximately 70 vol% of fine lithium disilicate (Li2Si2O5) crystals, embedded in glassy matrix [1, 6]. The two different thermal expansion coefficients and elastic moduli of the phase combination bring about tangential compressive stresses and proneness to deflect crack propagation under loads [6]. Therefore, LDGC possesses a notable strength of 303–440 MPa [3, 7]. However, the high strength and brittleness make it difficult to machine directly with the existing chairside CAD/CAM system [8, 9]. Currently, LDGC restorations can only be
produced from lower-strength lithium metasilicate (Li$_2$SiO$_3$) glass-ceramic (LMGC) by CAD/CAM-milling, sintering, polishing, etc. [9, 10]. Nonetheless, the subsequent heat treatment is unable to heal cracks or guarantee dimensional accuracy for final products [6, 9]. Knowledges to producing high-quality surfaces on LMGC and LDGC for dental purposes are far less than sufficiency.

Chairside CAD/CAM-milling of LMGC showed 3D average and maximum roughness of $S_a = 1.125\pm0.224$ μm and $S_z = 10.537\pm1.150$ μm [11]. Simulated diamond grinding of LDGC showed these indices of $S_a = 5–7$ μm and $S_z = 29–42$ μm [8]. These results were far beyond the $Ra = 200$ nm threshold for dental restorations to prevent bacterial colonization [9]. SEM morphologies for these ground surfaces demonstrated the dominant removal mechanism included penetration-induced fractures and shear-induced plastic deformation. Besides, nanoindentations for both materials have also evidenced the presence of fully-ductile removal below 100 nm depth and 10 mN load [12, 13]. However, the actual grain-material interaction in grinding is a dynamic process rather than a quasi-static one in indentation. Thus, understanding to the responses of LMGC and LDGC to diamond machinating are pressingly needed.

This paper aims to understand the mechanical behaviour of LMGC and LDGC in an actual dynamic grinding process. A single-diamond scratching test was conducted using a grinding machine mounted with a single-diamond wheel. The produced scratches were observed in SEM to reveal the material mechanical behaviours and identify the fully-ductile, ductile-brittle and fully-brittle regimes, and measured with 3D LCM to determine the critical cutting depths for each regime.

2. Methodology

2.1. Materials

Lithium metasilicate glass-ceramic (LMGC) blocks of 12.6 mm × 10.5 mm × 15.0 mm (IPS e.max CAD, HT A1/I 12, Ivoclar Vivadent AG, Liechtenstein) and lithium disilicate glass-ceramic (LDGC) blocks with diameter of 12.6 mm and thickness of 9.6 mm (IPS e.max Press, LT A1, Ivoclar Vivadent, Liechtenstein) were selected. Table 1 lists the reported mechanical properties of these two materials.

| Material | $E$ (GPa) | $H$ (GPa) | $K_{IC}$ (MPa m$^{1/2}$) | $\sigma$ (MPa) |
|----------|-----------|-----------|--------------------------|---------------|
| LMGC     | 87–113 $^a$ | 5.2–5.6 $^b$ | 0.9–1.25 $^b$ | 100–160 $^b$ |
| LDGC     | 124–131 $^c$ | 5.3–5.7 $^d$ | 1.15–3.32 $^d$ | 303–440 $^e$ |

$a$ Data from [12]; $^b$ Data from [10]; $^c$ Data from [13]; $^d$ Data from [2, 3]; $^e$ Data from [3, 7]

2.2. Single-diamond scratching

Fig. 1. (a) Scheme and (b) scene of single-diamond scratching experiments. Fig. 1a demonstrates the diamond grain-material contact in the single-diamond scratching experiment. A wheel with a diameter of $d_s$ rotating at a rotational speed $N$ moves along a declining direction of $l$ in length and $d_m$ in maximum depth at a feed rate $v_w$. Single $g_x$ diamond grain is fixed on the wheel circumferential surface. Fig. 1b presents the scene of single-diamond scratching experiments. A sample was mounted on a fixture with its upper surface parallel to the workbench plane. The movement of the wheel was controlled by the grinder controller. Machining conditions are listed in Table 2.

Prior to the scratching experiments, sample surfaces were all polished with consecutive 320#, 600#, 1,000#, 2,000# and 4,000# polishing compounds to guarantee their flatness and flawlessness.
Table 2. Machining conditions for single-diamond scratching experiments

| Grinder         | SMART-B818                     |
|-----------------|--------------------------------|
| Diamond wheel   | \( d_s = 150 \text{ mm in radius, } g_s = 600 \mu\text{m (i.e. 26\#)} \) |
| Rotational speed \( N \) | 1000 rpm                        |
| Feed rate \( v_w \)     | 50 mm/min                       |
| Length of scratch \( l \) | 10 mm                          |
| Final depth of scratch \( d_m \) | 5 \( \mu \text{m} \)          |

2.3. Characterization methods

Produced scratches were measured with a 3D laser confocal microscopy (LCM, UP-24, Rtec Instruments, USA) at 20\( \times \) magnification. Built-in mapping function was used to cover the whole scratch from cut-in to large scale fracture area. Each scratch was then gold-coated and observed in a scanning electronic microscopy (SEM, Merlin platform, Carl Zeiss AG, Germany). Magnifications for LMGC were 5,000\( \times \) for whole scratch and 20,000\( \times \) for details, while for LDGC they were 2,000\( \times \) and 10,000\( \times \), respectively.

SEM images were manually mapped together. Based on SEM observation, the scratches were identified and divided into fully-ductile, ductile-brittle and fully brittle regimes. The corresponding cross sections of the critical transition positions were extracted from 3D LCM images to reveal scratch groove profiles and measure their depths.

3. Results

![Fig. 2. (a) SEM and (b) LCM morphologies of scratch on LMGC; (i) and (iv) are cross sections at critical transition positions; (ii) and (iii) are magnified images showing microcrack, smeared fracture and ductile flow in ductile-brittle regime, and ductile flow in fully-ductile regime, respectively.](image)

Figs. 2a and 2b show SEM and LCM morphologies of scratch on LMGC, respectively. When diamond grain first cut into the material at a shallow cutting depth, ductile flow with no damages was found in the scratch (Fig. 2iii). As the diamond grain cut deeper, microcracks and smeared fractures were observed (Fig. 2ii). Then, extensive brittle burrs were formed distributing alongside the scratch until the material finally broke at large scale (Fig. 2a) with the deepest fracture of 8.6 \( \mu \text{m} \) in depth (Fig. 2b). Based on the removal mechanism, deformation mode could be divided into three types (i.e. fully-ductile, ductile-brittle and fully brittle regimes in Fig. 2a). Correspondingly, cross sections at the two critical transition positions were extracted in Figs. 2iv and 2i, which showed the scratch grooves of ca. 0.169 \( \mu \text{m} \) and 0.337 \( \mu \text{m} \) depths, respectively, while pile-up was revealed in Fig. 2iv.
Fig. 3. (a) SEM and (b) LCM morphologies of scratch on LDGC; (i) and (iv) are cross sections at critical transition positions; (ii) and (iii) are magnified images showing microcrack and ductile flow in ductile-brittle regime, and ductile flow in fully-ductile regime, respectively.

Figs. 3a and 3b show SEM and LCM morphologies of scratch on LDGC, respectively. Similar to LMGC, purely ductile flow (Fig. 3iii) was observed in fully-ductile regime. Microcracks and ductile flow clearly co-existed when the cutting depth became deeper (Fig. 3ii). Finally, large-scale fractures were formed with 5.9 μm at deepest (Fig. 3b). During scratching, minor burrs were visible in Fig. 3a. Cross sections at the two critical transition positions were displayed in Figs. 3iv and 3i, revealing ca. 0.761 μm and 2.968 μm depth scratch grooves, respectively, with pile-up at both positions.

4. Discussion

This work studied the mechanical behaviours of LMGC/LDGC towards dynamic abrasive machining with a single-diamond scratching, with respect to deformation modes, removal mechanisms and scratch groove profiles.

Evidenced in Figs. 2a and 3a, scratches on both LMGC and LDGC revealed ductile flow with no damages at shallow cutting depths. This indicates that fully-ductile removal can be realized for both materials (Figs. 2iii and 3iii), although they are composite ceramics containing crystals embedded with glassy matrix rather than isotropic ones like glass. At deeper cutting depths over 0.169 μm and 0.761 μm in Figs. 2iv and 3iv, microcracks took place surrounded by localized ductile area, revealing ductile-brittle coexisted deformation in this regime (Figs. 2ii and 3ii). When cutting depths reached 0.337 μm and 2.968 μm in Figs. 2i and 3i, the removal mechanism was characterized as fully-brittle.

Both cutting depths for LMGC are shallower than for LDGC, uncovering a more brittle nature of LMGC [14], which is also confirmed by the deepest fractures with depths of 8.6 μm on LMGC and lower 5.9 μm on LDGC, the absence of pile-up prior to the fully-brittle regime on LMGC (Fig. 2i), and the obviously more brittle burrs generated alongside the scratch on LMGC (Fig. 2a). The higher $K_{IC}$ and $\sigma$ contribute LDGC with a stronger ability to deflect crack propagation [6].

Undeformed chip thickness is a dominant parameter controlled by grinding conditions, determining material removal mechanism in a grinding process. The undeformed chip thickness is calculated as [15]

$$ h_m = 2 \left( \frac{v_w}{N} \right) \left( \frac{a_p}{d_s} \right)^{1/2} \left( 1 - \frac{a_p}{d_s} \right)^{1/2} - \left( \frac{v_w}{v_c} \right)^2 / d_s $$

where $v_w$ is the feed rate, $N$ the wheel rotational speed, $a_p$ the cutting depth (corresponded to the measured depth of each scratch groove), $d_s$ the wheel diameter and $v_c$ the linear speed. Based on the conditions in Table 2, the undeformed chip depths $h_m$ at the two critical transition positions in Figs. 2iv and 3i, and Figs. 2iv and 3i for LMGC and LDGC were respectively estimated as

$$ h_{m_{LMGC-D}} = 89 \text{ nm}, \quad h_{m_{LMGC-B}} = 133 \text{ nm} $$
$$ h_{m_{LDGC-D}} = 209 \text{ nm}, \quad h_{m_{LDGC-B}} = 428 \text{ nm} $$

where show $h_m$ for LDGC are approximately 2–3 times those for LMGC.

These results mean that when $h_m$ in a grinding process is lower than 89 nm for LMGC and 209 nm for LDGC, fully-ductile removal can be achieved. Ductile-brittle coexisted removal mechanism happens
at \( h_m \) of 89–133 nm for LMGC and 209–428 nm for LDGC, while the materials are deformed at fully-ductile level at deeper \( h_m \). \( h_m_{LMGC-D} \) and \( h_m_{LMGC-B} \) are approximately 12.2%–50.4% the \( h_m \) of 264–729 nm in a simulated grinding of LDGC [14]. The ratio for \( h_m_{LDGC-D} \) is 28.7%–79.2% while \( h_m_{LMGC-B} \) falls in the range. Thus, CAD/CAM-milled surface on LMGC showed mainly fractures, smears and pulverization with minor milling traces [9], revealing a brittle-dominant removal. CAD/CAM-milled LDGC surface also showed fractures, smears and delamination, while ductile flows were still clear [14], demonstrating the ductile-brittle coexisted removal.

Indentation tests can provide extensive valuable information on the mechanical behaviours of a material [12, 13, 16]. They are conducted under strictly vibrationless and temperature/humidity-stable environments [16]. The moving speed of the indenter is precisely controlled by the loading and/or feeding rate of the indentation system, mostly within millimetres/micrometres per second [12, 13]. However, in actual abrasive machining, linear speeds are mostly at metre per second scale [17]. The high linear speed may bring about large scale specific machining energy [17], leading to serious shock and vibration problems to both workpiece and grinding machine, and consequently deteriorating produced surface quality [18]. Therefore, the single-diamond scratching experiment in this study, extracting every diamond grain-material contact from numerous simultaneous indentations and scratches in real abrasive machining [18, 19], is able to reflect the true mechanical behaviour of the researched materials when being diamond ground [19].

Contact stiffness between workpiece and tool, resulting in final surface quality, is jointly controlled by mechanical properties the working material and stiffness of the mechanical system [18]. Theoretically, under the same machining conditions, produced surface quality on the material with a higher \( h_m \) may be finer, due to a larger portion ductile removal taking place during the machining process [19]. Nonetheless, 3D average and maximum roughness of LDGC surfaces produced in simulated grinding were \( \overline{Sa} = 5–7 \) \( \mu \)m and \( \overline{Sz} = 29–42 \) \( \mu \)m [8], significantly coarser than \( \overline{Sa} = 1.125\pm0.224 \) \( \mu \)m and \( \overline{Sz} = 10.537\pm1.150 \) \( \mu \)m of CAD/CAM-milled LMGC surfaces [11]. This might be attributed to the strong mechanical properties of LDGC and lack of stiffness of the used grinder. Higher \( H \) and \( K_{IC} \) contributed to a higher machinability index of LDGC, leading to higher grinding forces in a grinding process [20] and subsequently severer vibration and rougher surfaces [14]. Thus, a better surface quality on a stronger material also requires a stiffer mechanical system [18].

Besides, it should be noted that single-diamond scratching still cannot represent an actual diamond grinding. The larger \( h_m \) of LDGC produces more plastic debris which adheres to and accumulates on the wheel bond easier than brittle debris from LMGC [20], diminishing the wheel cutting ability [9]. In addition, the stronger mechanical properties of LDGC may result in severer diamond wear [20], reducing active grain number and making the grains sharper to penetrate into the machining surface [9, 14].

5. Conclusions
The nature of material removal mechanisms is determined by grinding conditions and material properties. This study extracted every diamond grain-material contact from numerous simultaneous diamond cutting by conducting single-diamond scratching experiments to reveal the true ductile-brittle transition behaviour of LMGC and LDGC under dynamic abrasive machining. Our results showed that removal mechanism for both materials could be divided into three modes, i.e. fully-ductile, ductile-brittle and fully brittle. The critical transition cutting depths were measured as 89 nm and 133 nm for LMGC, and 209 nm and 428 nm for LDGC. These results provide a guidance for dental technicians to select proper machining conditions to produce high-quality damage-free surfaces on LDGC dental restorations following the given calculations and analyses.

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