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Fracture, roughness and phase transformation in CAD/CAM milling and subsequent surface treatments of lithium metasilicate/disilicate glass-ceramics

Abdur-Rasheed Alao[a], Richard Stoll[b], Xiao-Fei Song[c], John R. Abbott[d], Yu Zhang[e], Jaafar Abduo[f], Ling Yin[a,*]

[a] Mechanical Engineering, College of Science and Engineering, James Cook University, Townsville QLD 4811, Australia
[b] Restorative Dentistry, College of Medicine and Dentistry, James Cook University, Cairns QLD 4870, Australia
[c] Key Laboratory of Advanced Ceramics and Machining Technology of Ministry of Education, School of Mechanical Engineering, Tianjin University, Tianjin 300072, China
[d] JCU Dental, James Cook University, Cairns QLD 4870, Australia
[e] Department of Biomaterials and Biomimetics, New York University College of Dentistry, New York, NY 10010, USA
[f] Melbourne Dental School, Melbourne University, 720 Swanston Street, Melbourne, Victoria 3010, Australia

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ABSTRACT

This paper studied surface fracture, roughness and morphology, phase transformations, and material removal mechanisms of lithium metasilicate/disilicate glass ceramics (LMGC/LDGC) in CAD/CAM-milling and subsequent surface treatments. LMGC (IPS e.max CAD) blocks were milled using a chairside dental CAD/CAM milling unit and then treated in sintering, polishing and glazing processes. X-ray diffraction was performed on all processed surfaces. Scanning electron microscopy (SEM) was applied to analyse surface fracture and morphology. Surface roughness was quantitatively characterized by the arithmetic average surface roughness R_a and the maximum roughness R_z using desktop SEM-assisted morphology analytical software. The CAD/CAM milling induced extensive brittle cracks and crystal pulverization on LMGC surfaces, which indicate that the dominant removal mechanism was the fracture mode. Polishing and sintering of the milled LMGC lowered the surface roughness (ANOVA, $p < 0.05$), respectively, while sintering also fully transformed the weak LMGC to the strong LDGC. However, polishing and glazing of LDGC did not significantly improve the roughness (ANOVA, $p > 0.05$). In comparison of all applied fabrication process routes, it is found that CAD/CAM milling followed by polishing and sintering produced the smoothest surface with $R_a = 0.12 \pm 0.08$ µm and $R_z = 0.89 \pm 0.26$ µm. Thus, it is proposed as the optimized process route for LMGC/LDGC in dental restorations. This route enables to manufacture LMGC/LDGC restorations with cost effectiveness, time efficiency, and improved surface quality for better occlusal functions and reduced bacterial plaque accumulation.

1. Introduction

Monolithic ceramic crowns and bridges are proven to be more durable than veneered core restorations where brittle fractures frequently occur in the weak porcelain veneers and the veneer-core interfaces (Beuer et al., 2009; Guess et al., 2010; Swain, 2009; Zhang et al., 2009; 2013a). Ideal ceramic restorations should be made from durable and highly aesthetic materials, such as lithium disilicate ($Li_2Si_2O_5$) glass ceramics (LDGC) (Reich et al., 2014). The high strength and toughness of LDGC arise from ~70 vol% of interlocking needle-like lithium disilicate crystals, which have different thermal coefficients and elastic moduli from their glassy matrix (Apel et al., 2008; Denry, 2013; Denry and Holloway, 2010; Höland et al., 2006a; Kelly, 2008). These differences result in compressive stresses in LDGC, which can deflect advancing cracks (Apel et al., 2008; Denry, 2013; Denry and Holloway, 2010; Serbena and Zanotto, 2012).

Due to the high strength of LDGC and the brittleness of its glassy phase, it is very difficult to machine using chairside or laboratory CAD/CAM milling systems. Alternatively, LDGC restorations are made from low-strength lithium metasilicate (Li_2SiO_3) glass ceramic (LMGC) blocks, which can be easily CAD/CAM-milled to form basic full-contour crowns and bridges (Höland et al., 2000). Meanwhile, milled LMGC blocks require sintering to transform lithium metasilicate to lithium disilicate for formation of strong LDGC. However, the milling process

* Corresponding author.
E-mail address: ling.yin@jcu.edu.au (L. Yin).

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induces surface and subsurface flaws in LMGC, which are difficult to diminish by the subsequent heat treatment, and may compromise the strength of LDGC restorations and shorten their lifespans (Denry, 2013; Rekow et al., 2011; Rekow and Thompson, 2005). Thus, minimization of milling-induced flaws in LMGC is necessary for quality assurance for LDGC restorations.

Further, polishing and glazing are also applied to finalize surface texture, reduce roughness and enhance light reflection (Boaventura et al., 2013; Höland et al., 2006b). In fact, at many dental laboratories or clinics, sintering, polishing and glazing of CAD/CAM-milled LMGC contours are arbitrary procedures (Lin et al., 2012), which can result in variable surface quality. Currently, there is lack of optimized fabrication process selection and sequence to ensure the reliability of LDGC restorations.

Surface quality, such as phase transformation, surface roughness and fracture morphology, plays a critical role in determining the wear and fatigue performance of dental restorations (Alao et al., 2017; Currana et al., 2017; Denry, 2013; Peng et al., 2016; Rekow and Thompson, 2005; Rekow et al., 2011; Ulutan and Ozel, 2011; Zhang et al. 2013b). Clinical studies have shown that CAD/CAM-processed single LDGC restorations achieved 100% cumulative survival rate up to two years (Fasbinder et al., 2010) and 96.3% after four years according to the Kaplan-Meier survival analysis (Reich and Schierz, 2013). The survival rate for three-unit LDGC partial fixed dentures was 93% up to four years (Reich et al., 2014) and 87.9% for up to ten years (Kern et al., 2012). A five-year clinical study indicates that nearly 100% survival rate for LDGC crowns but 70% for fixed partial LDGC dentures (Marquardt and Strub, 2006). The longest clinical observation of LDGC posterior crowns after 15 years reveals 81.9% survival rate (van den Breemer et al., 2017). Clinical analyses of failed LDGC restorations have found that fracture and chipping were the root cause of failure, which originated from surface damage and flaws (Della Bona and Kelly, 2008; Mores et al., 2017; Valenti and Valenti, 2009; van den Breemer et al., 2017; Zhang et al., 2013b). Therefore, the diminishment of surface flaws in LDGC restorations is essential to prolong their lifespans. In addition, surface quality also critically affects cell adhesion, proliferation and protein adsorption (Brunot-Gohin et al., 2013).

In clinical practice, external surfaces of restorations must be finished to a high surface luster to reduce fracture risk, bacterial plaque accumulation, tooth stains, and wear on antagonist/adjacent teeth (De Jager et al., 2000; Jefferies, 2007; Kou et al., 2006; Steiner et al., 2015; Whitehead et al., 1995). Intaglio surfaces of restorations are often roughened to improve bonding to adhesives (Brunot-Gohin et al., 2013). The surface quality of ground LDGC using dental handpieces and diamond burs (Song et al., 2016), and glazed, polished and ground LDGC (Boaventura et al., 2013; Kou et al., 2006; Tholt et al., 2006) have been individually studied. However, it is unclear how the process selection and sequence will influence the surface quality of LMGC/LDGC in CAD/CAM milling, and subsequent sintering, glazing and polishing.

This paper, therefore, aimed to investigate the process-quality relation to determine the optimized processing protocol for LMGC/LDGC. X-ray diffraction was used to analyse crystalline phases and phase transformations. Surface roughness was measured in terms of the arithmetic average roughness R_a and the maximum roughness R_z using a desk-top SEM-assisted morphology analytical software. Scanning electron microscopy (SEM) was applied to analyse the material removal mechanisms, surface fracture and morphology. Finally, an optimal fabrication process for LDGC restorations was proposed to achieve the improved surface integrity.

2. Experimental procedure

2.1. Materials

LMGC blocks of 14.5 mm × 12.4 mm × 18 mm (IPS e.max CAD, Ivoclar Vivadent, Liechtenstein) were selected. The material is fabricated by the manufacturer via melting a base glass consisting of 69.3 wt% SiO_2, 15.4 wt% Li_2O, 6.05 wt% K_2O, 4 wt% ZnO_2, 3.38 wt% Al_2O_3, and 3.84 wt% P_2O_5 at 1450 °C (El-Meliegy and van Noort, 2012; Höland et al., 2006a). This was followed by subsequent annealing at 480 °C for 1 h to precipitate lithium metasilicate crystals (El-Meliegy and van Noort, 2012). After cooling to room temperature, the glass ceramic contains approximately 40 vol%, 0.5–1 μm lithium metasilicate crystals (Bühler-Zemp and Völkel, 2005; El-Meliegy and van Noort, 2012). It has the biaxial strength of 130 ± 30 MPa, fracture toughness of 1 ± 0.1 MPa $m^{1/2}$ and Vickers hardness of 5.4 ± 0.1 GPa (Bühler-Zemp and Völkel, 2005).

2.2. Chairside CAD/CAM milling

LMGC blocks were milled using a chairside CAD/CAM milling unit (CEREC MC XL, Sirona, Germany) with a step bur 12S (Ref 6240167, Sirona, Germany) and a cylindrical pointed bur 12S (Ref 6240159, Sirona, Germany), both of which have the same composition and properties. The step bur consists of three cutting faces with lengths of 3 mm, 3 mm, and 6 mm, and diameters of 2.1 mm, 1.7 mm and 1.3 mm, respectively. The cylindrical pointed bur comprises of two cutting faces with lengths of 4 mm and 8 mm and diameters of 2.1 mm and 1.8 mm, respectively. Both burs are electro-plated with diamond abrasives, and are used to generate flat surfaces as schematically shown in Fig. 1. Wet milling was conducted following the program recommended by the manufacturer, which simulates surface milling of crowns, the most challenging step in the CAD/CAM process (Luthardt et al., 2004). A new step bur was gold-coated and observed using scanning electron microscopy (SEM) (Jeol JSM5410V, Japan). Fig. 2(a) shows the SEM micrograph of the step diamond bur morphology. Fig. 2(b) reveals the diamond cutting edges with an average grit size of approximately 50–60 μm.

2.3. Surface process protocols

After milling of LMGC blocks, the samples were cleaned in acetone and treated by sintering, polishing and glazing to simulate various clinical fabrication processes. These process routes are schematically shown in Fig. 3 and designated as CAD/CAM (i.e., CAD/CAM milling), CAD/CAM-polish, CAD/CAM-sinter, CAD/CAM-polish-sinter, CAD/CAM-sinter-polish, CAD/CAM-sinter-glaze, and CAD/CAM-polish-sinter-glaze processes.

Sintering of milled LMGC samples was carried out in a programed dental furnace (P300, Ivoclar Vivadent, Liechtenstein) at a stand-by temperature of 403 °C. Then, the samples were heated to 770 °C at a heating rate of 60 °C/min and held at the temperature for 10 min. After that, they were heated again to 850 °C at a heating rate of 30 °C/min and held for another 10 min before cooling to 700 °C. Finally, they were

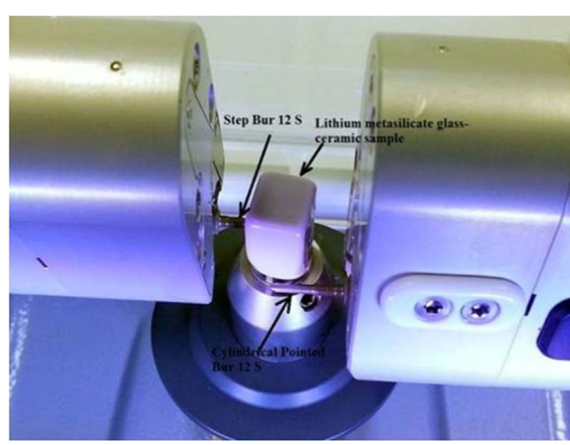

Fig. 1. Chairside CAD/CAM milling of a LMGC block using two diamond burs.