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SEM observation and wettability of variously processed and fractured surface of dental zirconia

Naoyoshi Tarumi [a,b,*], Motohiro Uo [c], Eiji Yamaga [a], Fumio Watari [b]

[a] Sapporo Dental Laboratory, Kita 24 jo Nishi 2 chome, Kita-ku, Sapporo, Hokkaido, 011-0024, Japan
[b] Graduate School of Dental Medicine, Hokkaido University, Japan
[c] Tokyo Medical and Dental University, Bunkyo-ku, Tokyo, 113-8549, Japan

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ABSTRACT

Current dental zirconia has several problems in clinical application such as chipping, fracture and detachment. To reduce these problems the surface after various treatments was analyzed by SEM observation, contact angle measurement and surface roughness measurement, and compared. The surface after mirror polishing was smooth. Porcelain layering was smooth except large formed grooves by bubbles. After sandblast and tribochemical treatments, the surfaces showed several micron-sized caving with micron to submicron-level irregularities. Sandblast and tribochemical treatments with the lager roughness had the smaller water contact angle than silicone wheel polishing. Clinically fractured surface of zirconia showed a more complex structure than manually fractured surface, which may be due to the various mode of stress to be imposed repetitively to various direction.

1. Introduction

Recently, all ceramic crowns and bridges using zirconia fabricated by CAD/CAM for core frame have been used more and more for wider cases in dental clinics. They are especially effective in the cases of metallic allergy and esthetic prostheses. However there are some problems such as fracture of frame in certain cases.

Various surface treatments were done onto zirconia in the fabrication process of all-ceramic crowns. Silicone wheel was used to polish the frame surface. Sandblast treatment was done to clean and to make the surface sufficiently rough for adhesion of resin cement and bonding of porcelain [1]. Tribochemical treatment was used as a pretreatment for the silane coupling treatment processing. Acid treatments were done to dissolve porcelain layer on the surface of frame at the rebuilding of used zirconia prostheses.

Chipping or fracture and detachment of zirconia frame are major problems in clinical practice. Wettability of surface has a large influence on the adhesion effect [2]. In this study, the surface after various treatments was analyzed by SEM observation, contact angle measurement and surface roughness measurement, and compared to reduce the problems in clinical application such as chipping, fracture and detachment. The clinically derived and experimentally attained fracture surfaces of zirconia were also observed by SEM and compared.

2. Materials and methods

2.1. Surface processing

Pre-sintered zirconia of cylinder block of 20 mm in diameter (Cercon Base Basic, DeguDent, Germany) was cut into 10 mm thick disks. Disk surface was polished by waterproof paper (#240 & #800). After sintered at 1350 °C in accordance to the sintering program recommended by product company, all samples were polished by (1) SW: silicone wheel (CoreMaster fine, Shofu, Kyoto, Japan), then these surfaces were variously treated. These surface treatments are (2) SB: sandblast with alumina (Cobra 2 μm at 2.5 atm: Renfert, Germany), (3) TC: tribochemical (Rocatec 30 μm at 2.5 atm: 3 M ESPE, Minnesota, United States), (4) MP: mirror polishing using the buff with diamond paste (Gradia Diapolisher: GC, Tokyo, Japan). (5) AC: 24-h acid with the solution of 13% HF + 16% H_2SO_4 (Uniclean: Shofu, Kyoto, Japan), and (6) PL: porcelain layering treatments (Cerabien CZR: Noritake, Aichi, Japan).

2.2. Fracture surface

The block of 3 mm high, 50 mm long with the upper surface of 2 mm width and the lower surface of 1.8 mm width was cut from zirconia cylinder and sintered at 1350 °C as recommended by Product Company. The size of cross-section of zirconia block was

* Corresponding author at: Graduate School of Dental Medicine, Hokkaido University, Japan. Tel.: +81 11 747 3336; fax: +81 11 736 3336.
E-mail address: n-tarumi@y9.dion.ne.jp (N. Tarumi).

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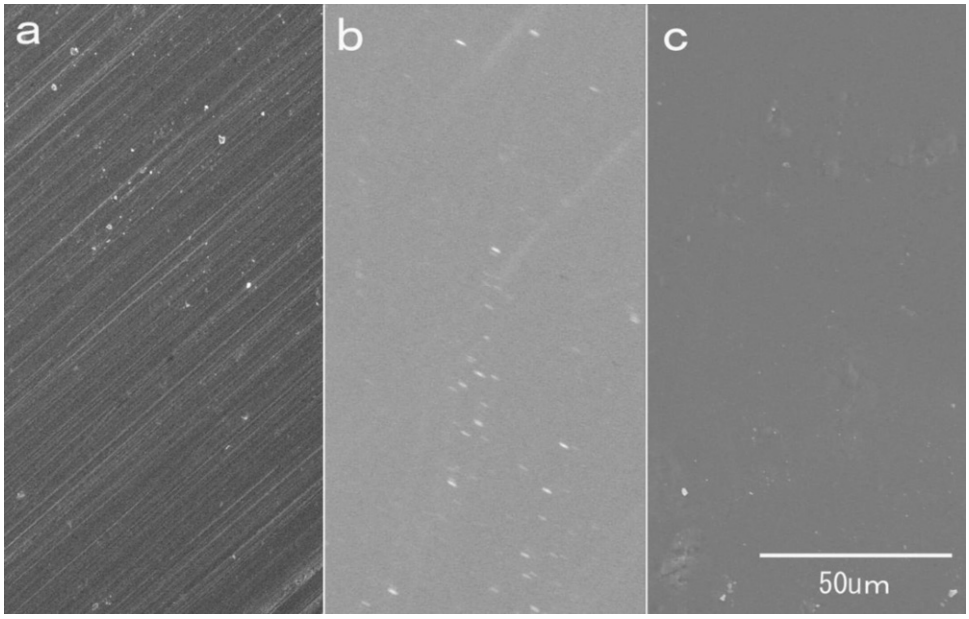

Fig. 1. SEM observation of smoothed zirconia surface. (a) Silicone wheel polishing treatment, (b) mirror polishing, (c) porcelain layering.

selected to fit to the clinical fracture case. It was fractured manually through bending by loading from upper to lower surface without any treatment on the sintered surface. Clinically derived fracture zirconia was also used as sample.

2.3. Observation

Variously treated surfaces and fractured cross-sections were observed by FE-SEM (S4000: Hitachi, Tokyo, Japan).

2.4. Contact angle measurement

The contact angle with water were measured for the surface of zirconia after silicone wheel, sandblast and tribochemical treatments with DMs-200 (Drop Master S series: Kyowa Interface Science Co.Ltd, Saitama, Japan) Measurements were performed 10 times for each. All experimental results were evaluated by Non-repeated Measures ANOVA ($n = 10$) ($p < 0.001$).

2.5. Surface roughness measurement

The surface roughness was measured using SURFCOM 1400A (Tokyo Seimitu, Japan) after the treatments with silicone wheel, sandblast, tribochemical treatments, mirror polishing and porcelain layering.

3. Results

Fig. 1 shows SEM observation of smoothed zirconia surface. Fig. 1a is silicone wheel polishing, Fig. 1b is mirror polishing, and

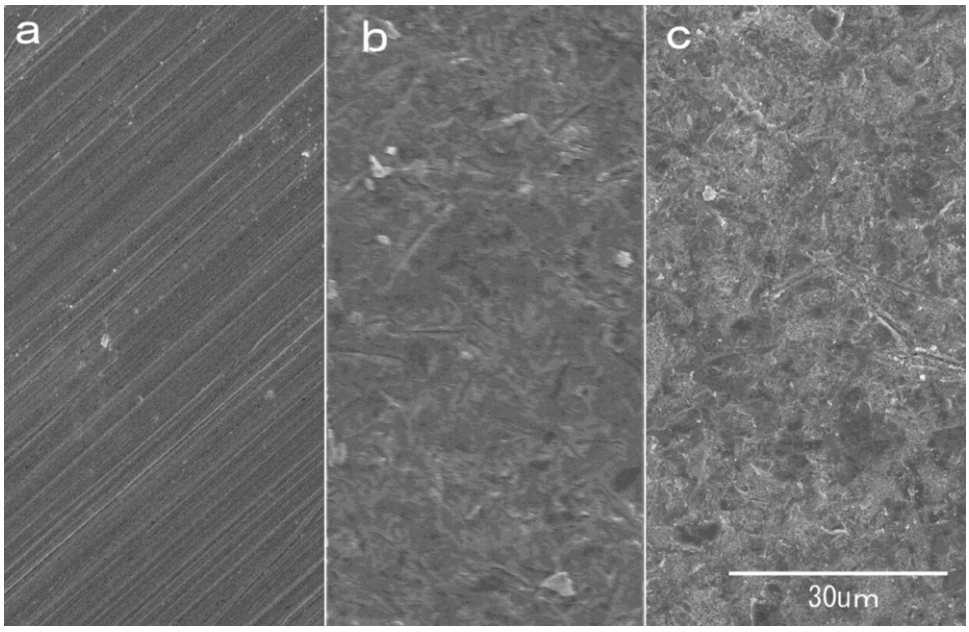

Fig. 2. SEM observation of roughened zirconia surface. (a) Silicone wheel polishing treatment, (b) sandblast treatment, (c) tribochemical treatment.