

| Title | Fatigue behavior and crack initiation of CAD/CAM resin composite molar crowns |
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| Author(s) | Yamaguchi, Satoshi; Kani, Renshirou; Kawakami, Kazuma et al. |
| Citation | Dental Materials. 2018, 34(10), p. 1578-1584 |
| Version Type | АМ |
| URL | https://hdl.handle.net/11094/93082 |
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Original paper

Fatigue behavior and crack initiation of CAD/CAM resin composite molar crowns

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Declaration of interest statement

Funding

This research was supported by a Grant-in-Aid for Scientific Research (No. JP15K11195)

from the Japan Society for the Promotion of Science (JSPS).

Abbreviations

CAD/CAM computer-aided design/computer-aided manufacturing

SSALT Step-stress accelerated life test

FEA Finite element analysis

MPS Maximum principal strain

RCB Resin composite block

PVC Polyvinyl chloride

XRD X-ray diffraction

SEM Scanning electron microscopy

ANOVA Analysis of variance

HSD honest significant difference

ABSTRACT

Objective: The aim of this study was to evaluate long-term fatigue behavior using an *in vitro* step-stress accelerated life test (SSALT), and to determine the crack initiation point using *in silico* finite element analysis for computer-aided designed and manufactured (CAD/CAM) molar crowns fabricated from three commercial CAD/CAM resin composite blocks: Cerasmart (CS; GC, Tokyo, Japan), Katana Avencia Block (KA; Kuraray Noritake Dental, Niigata, Japan), and Shofu Block HC (HC; Shofu, Kyoto, Japan).

Methods: Fifty-one mandibular first molar crowns luted on a resin core die were embedded in acrylic resin and covered with a polyvinyl chloride tube. Single compressive tests were performed for five crowns. SSALT was conducted for 36 crowns using three profiles and reliabilities at 120,000 cycles, and a Weibull analysis was conducted. The maximum principal strain of each CAD/CAM resin composite crown model was analyzed by three-dimensional finite element analysis.

Results: Fracture loads of CS and KA ($3784 \pm 144 \,\mathrm{N}$ and $3915 \pm 313 \,\mathrm{N}$) were significantly greater than that of HC ($2767 \pm 227 \,\mathrm{N}$) (p < 0.05). Fracture probabilities at 120,000 cycles were 24.6% (CS), 13.7% (KA), and 14.0% (HC). Maximum principal strain was observed around the mesiolingual cusps of CS and KA and the distobuccal

cusp of HC.

Significance: CAD/CAM resin composite molar crowns containing nano-fillers with a higher fraction of resin matrix exhibited higher fracture loads and greater longevity, suggesting that these crowns could be used as an alternative to ceramic crowns in terms of fatigue behavior.

KEYWORDS

CAD/CAM resin composite molar crown, CAD/CAM resin composite, fatigue resistance, step-stress accelerated life testing, three-dimensional finite element analysis

1 INTRODUCTION

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Computer-aided design / computer-aided manufacturing (CAD/CAM) resin 2 composite blocks (RCBs) containing a high density of nano-filler particles are available 3 4 for use in posterior restorations [1]. Because of the preliminary polymerized resin matrix, the nano-filler particles are homogeneously dispersed in the resin matrix, 5 6 providing stable and excellent mechanical properties such as flexural strength and fracture toughness when compared with those of conventional resin composites used in 7 fillings [2, 3]. As an alternative material to metals or ceramics, CAD/CAM RCBs are 8 attracting much attention because their esthetics are more favorable than metals and 9 their cost is lower than ceramics [4]. CAD/CAM RCBs have excellent fatigue resistance 10 with no catastrophic failures when compared with ceramics [5]. The resin matrix of 11 12CAD/CAM RCBs prevents crack propagation during cyclic loading and leads to greater flexural strength and a lower flexural modulus [6]. 13 The long-term fatigue behavior of bar-shaped CAD/CAM RCB specimens after 14 fatigue treatment has been measured using a three-point bending test, and was found to 15 be comparable to lithium disilicate glass-ceramic [7]. However, the fatigue behavior of 16 17 crown-shaped specimens is still unknown. A frequency of 15 Hz with a load of 10-40 N for 1.2×10^6 cycles (~22 hours) is required to prepare each specimen for the 18

1 three-point bending test.

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movement in the mouth has been used to investigate the longevity of dental implants [8] and all-ceramic crowns [9, 10]. Fracture patterns of bar-shaped specimens after fatigue tests with or without step-stress profiles were similar; that is, SSALT has been validated and could be more time-efficient than fatigue testing with a constant load [11]. The fatigue behavior of CAD/CAM resin composite crowns has been evaluated and was found to be comparable to leucite reinforced glass-ceramic crowns, with no catastrophic failures occurring in the CAD/CAM resin composite crowns [5]. The fracture pattern of crown-shaped specimens after catastrophic failure is important to ascertain the crack initiation point and to improve the composition and physical properties of CAD/CAM resin composite. Fractographic analysis has been used to evaluate the fractured surface after SSALT for dental implants [12, 13], ceramics [14], and polymer infiltrated ceramic network materials [15]. However, it is difficult to determine the specific crack initiation point because the fracture has already occurred. Finite element analysis (FEA) is a powerful tool for calculating stress and strain distribution in dental implants [16, 17] and CAD/CAM RCBs [18]. FEA can be used to

predict the crack initiation point by using maximum principal strain as an effective

A step-stress accelerated life test (SSALT) that mimics the sliding contact

- 1 failure criteria [19].
- 2 The aim of this study was to evaluate long-term fatigue behavior using in vitro
- 3 SSALT, and to determine the crack initiation point using in silico FEA for CAD/CAM
- 4 resin composite molar crowns fabricated from three commercial CAD/CAM RCBs:
- 5 Cerasmart (CS; GC, Tokyo, Japan), Katana Avencia Block (KA; Kuraray Noritake
- 6 Dental, Niigata, Japan), and Shofu Block HC (HC; Shofu, Kyoto, Japan).

2 MATERIALS AND METHODS

2 2.1. CAD/CAM RCBs

- 3 Three types of commercially available CAD/CAM RCBs were used: Cerasmart (CS;
- 4 GC, Tokyo, Japan), Katana Avencia Block (KA; Kuraray Noritake Dental, Niigata,
- 5 Japan), and Shofu Block HC (HC; Shofu, Kyoto, Japan). Details of the composition of
- 6 each block is shown in Table 1.

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2.2. Specimen preparation

- 9 An impression was taken of a mandibular first molar abutment tooth model (A55A-461,
- Nissin, Kyoto, Japan) using silicone impression material. A total of 51 abutment teeth
- were fabricated by incremental build-up and photopolymerization of core resin (Clearfil
- 12 DC Core Automix, Kuraray Noritake Dental) in the impression cavity. The mandibular
- right first molar model (A5A-500, Nissin) and the abutment tooth were scanned (SC-5,
- 14 Kuraray Noritake Dental) and designed (DentalDesigner, Kuraray Noritake Dental).
- Both models were fabricated using a milling machine (DWX-50, Kuraray Noritake
- Dental). To strengthen the adhesion of the crowns [20], the inner surface of the crowns
- were sand blasted with an air pressure of 0.2 MPa using Al₂O₃ particles 30–50 μm in
- diameter. After ultrasonic cleaning for 2 minutes and air-drying, the inner surface of the

- 1 crowns was etched with phosphoric acid gel (K-etchant gel, Kuraray Noritake Dental).
- 2 After 5 seconds, ceramic primer (Clearfil Ceramic Primer, Kuraray Noritake Dental)
- 3 was applied after rinsing with water and air-drying. The crowns were cemented on the
- 4 abutment tooth and exposed to light for 5 seconds, followed by removal of excess
- 5 cement.

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2.3. Single compressive testing

- 8 All specimens were vertically embedded in a 25-mm diameter polyvinyl chloride
- 9 (PVC) tube of acrylic resin (Unifast Lab type F, GC, Tokyo, Japan) with the buccal
- margin of the crown positioned 2 mm higher than the top surface of the acrylic resin.
- 11 After storage in distilled water at 37 °C for 24 hours, a single compressive test
- 12 (AGS-500D, Shimadzu, Kyoto, Japan) was performed at a crosshead speed of
- 0.5 mm/min (n = 5), and mean fracture load and standard deviation were calculated.

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2.4. Step-stress accelerating life testing

- Based on the mean load of the static compression test, three fatigue loading profiles
- were designed for the 12 specimens for SSALT [8]. The designed profiles were
- designated as mild (n = 6), moderate (n = 4), and aggressive (n = 2), following the ratio

- of 3:2:1 (Fig. 1). The crowns were immersed in distilled water (Fig. 2) during SSALT.
- 2 SSALT was performed at a frequency of 5 Hz. Weibull analysis and a reliability
- 3 assessment were conducted. Fractured specimens were observed with stereomicroscopy
- 4 (SMZ-745T, Nikon, Tokyo, Japan) using high dynamic range software (NIS-Elements
- 5 Ver.4.0, Nikon) and scanning electron microscopy (SEM; JSM-6390BU, JEOL, Tokyo,
- 6 Japan) with 20× magnification at 5 kV.

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2.5. Three-dimensional finite element analysis

- 9 FEA models of the indenter, crown, cement, die, acrylic resin, and PVC tube were
- designed from scanned stereolithography data (Fig. 3) for in vitro tests and analyzed
- using voxel-based FEA software (VOXELCON2015, Quint, Tokyo, Japan). Fracture
- loads after single compression tests were applied to the crown, and the location of the
- maximum principal strain on the crown was analyzed.

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2.6. Statistical analysis

- Mean fracture loads obtained from the single compression tests were analyzed by
- analysis of variance (ANOVA) and Tukey's honest significant difference (HSD) test
- 18 (PASW Statistics 18, IBM, Somers, NY, USA). P-values of less than 0.05 were

1 considered as statistically significant.

3. RESULTS

3.1. Fracture loads after single compression test

- Fracture loads of CS $(3784 \pm 144 \text{ N})$ and KA $(3915 \pm 313 \text{ N})$ were significantly
- 4 higher than that of HC (2767 \pm 227 N) (p < 0.05) as shown in Figure 4. An initial load
- of three profiles for SSALT was determined from ~6% of the mean fracture load of
- 6 3489 N (i.e. 200 N).

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3.2. Fracture reliability after SSALT

- 9 Weibull curves obtained from SSALT are presented in Figure 5. The shape
- parameter m (two-sided at 90% confidence bounds) for CS, KA, and HC were 3.8
- 11 (1.6–5.5), 3.1 (1.3–4.4), and 4.8 (2.0–6.9), respectively; and the scale parameter η
- 12 (two-sided at 90% confidence bounds) for CS, KA, and HC were 2844.1
- 13 (2227.8–3695.7), 3672.0 (2706.5–5093.1), and 2774.8 (2279.6–3426.1), respectively.
- Greater reliability at 2000 N, 2500 N, and 3000 N was recorded for KA (85.7%, 73.6%,
- and 58.5%) than HC (81.1%, 54.5%, and 23.4%) and CS (77.2%, 54.4%, and 29.3%).
- Fracture probabilities at 120,000 cycles were 24.6% (CS), 13.7% (KA), and 14.0%
- 17 (HC).

3.3. Fractographic analysis

Figure 6 shows stereoscopic images from the occlusal surface after SSALT with an aggressive profile for CS, KA, and HC. Most of the specimens fractured along the central sulcus of each crown. Figure 7 shows SEM images from the lingual side after SSALT with an aggressive profile for CS, KA, and HC. Large white arrows indicate crack initiation points. Arrested lines indicated by small white arrows were observed

throughout the fracture surface of the crowns. Hackle patterns following the direction of

8 crack propagation are indicated by asterisks.

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3.4. Maximum principal strain (MPS)

The MPS was observed around the mesiolingual cusp for CS (Fig. 8a) and KA

12 (Fig. 8b), and around the distobuccal cusp for HC (Fig. 8c). Large white arrows indicate

the location of the MPS.

DISCUSSION

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CAD/CAM resin composite crowns fabricated from CAD/CAM RCBs have been 2 used as restorations to replace lost tooth structure. After approval by Japanese health 3 4 insurance companies in 2014, the restoration of premolars and molars with CAD/CAM resin composite crowns has grown as an alternative to metal and ceramic restorations. In 5 this study, the long-term fatigue behavior of three commercial CAD/CAM resin 6 composite molar crowns was elucidated in vitro and the crack initiation points at the 7 crowns were determined in silico. 8 The X-ray diffraction (XRD) patterns of all CAD/CAM resin composite powders 9 showed a typical amorphous phase and an increasing order of broad peaks that have 10 been reported by Yoshihara et al. [21]. The broad peak of 22.0° [22] for KA and HC 11 12 were derived from SiO₂ particles, and the other peak of 26.5° [23] for CS was derived from BaO fillers. HC contained spherical filler particles ranging between 1.0 and 13 10.0 µm [21], which is one of the reasons it exhibited a higher peak in the XRD patterns 14 than the other two CAD/CAM resin composites. 15 CS and KA contained nano-fillers of 20-nm and 40-nm SiO₂ particles, respectively, 16 17 and exhibited higher fracture loads after the single compressive test. We have clarified that the compressive strength of CAD/CAM RCBs can be enhanced by loading with 18

- silica nano-filler particles of smaller diameter [18]. Fracture loads after the single
- 2 compressive test using crown-shaped specimens were also consistent with our previous
- 3 findings.
- 4 In the Weibull analysis, the lower shape parameter m of KA indicated greater reliability in strength when compared with CS and HC. Additionally, the higher scale 5 6 parameter η for KA indicated that the load in 63.2% of specimens that failed was higher than those of CS and HC. Interestingly, the order of increase in fracture load after single 7 compressive tests (HC < CS < KA) did not match the order of increase in reliability 8 after SSALT (CS < HC < KA). The filler fractions of KA and HC were 62 wt% and 9 61 wt%, while that of CS was 71 wt%; thus, the resin matrix fraction of KA and HC 10 11 should be higher than that of CS. Therefore, the resin matrix of KA and HC could 12prevent crack propagation during SSALT and might show greater fatigue behavior than CS. The higher fraction of resin matrix might increase the bonding ability between the 13 inner surface of CAD/CAM resin composite crowns and the luting cement, while 14 presenting a risk of an increased amount of water absorption affected by the structure of 15 the polymers. Compared with the leucite reinforced glass ceramics (IPS Empress CAD) 16 17 used by Shembish et al. [5], all the CAD/CAM resin composite crowns used in this study recorded a higher fracture load and greater reliability. The reliability of the IPS 18

- 1 Empress CAD molar crowns used in Shembish et al.'s study was 90% at 1,250,000
- 2 cycles and a clinically relevant load of 200 N, which simulates ~5 years of in vivo wear
- 3 [5]. Sandblasting before bonding could roughen the surface and increase the surface
- 4 area for bonding [20, 24]. The results of the fatigue behavior suggest that all the
- 5 CAD/CAM composite crowns in our study have the potential to last more than 5 years
- 6 in a clinical situation.
- 7 The total time of SSALT using three profiles at 5 Hz was 852,000 s (~10 days) for
- 8 12 crown-shaped specimens, while normal fatigue tests use 1,250,000 cycles at 2 Hz for
- 9 7,500,000 s (~87 days), indicating that SSALT could save 8.8 times as much time as
- 10 normal fatigue tests.
- MPS is applicable as a failure criterion and is effective in predicting the flexural
- strength of composite resins [19]. The crack initiation point of HC was different from
- other CAD/CAM resin composite molar crowns because of the significantly lower
- fracture load of HC compared with those of KA and CS. By increasing the load on HC
- for the FEA, the crack initiation point of HC must migrate from the distal cusp to the
- mesiolingual cusp as in KA and CS. As shown in Figure 8a, the crack initiation point
- was observed around the distal cusp, but the catastrophic failure occurred around the
- mesiolingual cusp. By fractographic analysis, two crack initiation points for all the

- 1 CAD/CAM resin composite molar crowns were considered as candidates in the SEM
- 2 images. The FEA results could help to determine that the actual crack initiation occurred
- 3 around the mesiolingual cusp. The crack initiation point was similar to that of
- 4 zirconia-supported all-ceramic crowns [25].
- 5 The investigation of the pure resin matrix is ongoing to clarify how water
- 6 absorption alters the mechanical properties of bulk CAD/CAM RCBs. An in silico
- 7 nano-scale model of CAD/CAM RCBs [18] investigating the effect of silane coupling is
- 8 under development to evaluate the influence of hydrolysis on the mechanical properties
- 9 [26]. Further studies will accelerate improvements in the long-term fatigue behavior of
- 10 CAD/CAM resin composite molar crowns.

CONCLUSIONS

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- 2 CAD/CAM resin composite molar crowns containing nano-fillers with a higher fraction
- 3 of resin matrix demonstrated higher fracture loads and greater longevity, suggesting that these
- 4 crowns could be used as an alternative to ceramic crowns in terms of fatigue behavior.

5 ACKNOWLEDGEMENTS

- This research was supported by a Grant-in-Aid for Scientific Research (No.
- 7 JP15K11195) from the Japan Society for the Promotion of Science (JSPS).
- 8 We thank Helen Jeays, BDSc AE, from Edanz Group
- 9 (www.edanzediting.com/ac) for editing a draft of this manuscript.

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5

1 TABLES

2 Table 1. Composition of the CAD/CAM resin composite blocks.

| | | | Composition | | | | |
|----------------------|------|--|---------------------|--|--------------|--|--|
| Trade name | Code | Manufacturer | Monomer | Filler | Filler (wt%) | | |
| Cerasmart | CS (| GC, Tokyo, Japan | Bis-MEPP, UDMA, DMA | SiO ₂ (20 nm), Ba glass (300 nm) | 71 | | |
| Katana Avencia Block | | Kuraray Noritake Dental, Niigata, Japan | UDMA | SiO ₂ (40 nm), Al ₂ O ₃ (20 nm) | 62 | | |
| Shofu Block HC | HC S | Shofu, Kyoto, Japan | UDMA, TEGDMA | SiO ₂ , microfumed SiO ₂ , zirconium silicate | 61 | | |

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5 Table 2. Properties of the CAD/CAM resin composite crowns used for finite element

6 analysis.

| | | Crown | | Indenter | Cement | PVC tube | Die and resin |
|-----------------------|-----|-------|------|-----------------|---------|--------------------|---------------|
| Material | CS | KA | HC | Stainless steel | Panavia | Polyvinyl chloride | Acrylic resin |
| Elastic modulus (GPa) | 8.4 | 8.5 | 12.2 | 210 | 12 | 1.5 | 2.979 |
| Poisson's ratio | 0.3 | 0.34 | 0.3 | 0.3 | 0.33 | 0.35 | 0.4 |

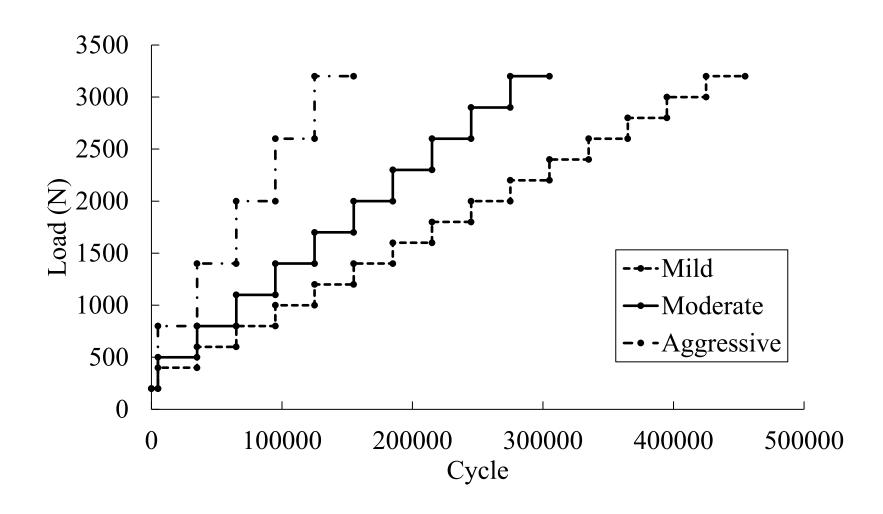
1 FIGURE LEGENDS 2Figure 1. SSALT profiles showing the cycle-to-load assignment for specimens 3 undergoing mild, moderate, or aggressive fatigue profiles. 4 5 Figure 2. Experimental setup of in vitro SSALT. The assembled CAD/CAM resin 6 composite crown embedded in acrylic resin and covered with PVC tube was set onto a 7 custom-made jig. Specimens were stored in water during SSALT. 8 9 Figure 3. CAD models of the indenter, crown, cement, die, acrylic resin, and PVC tube 10 for three-dimensional finite element analysis. 11 12 Figure 4. Mean fracture loads after single compressive testing. Error bar indicates 13 standard deviations. Asterisks indicate significant differences by one-way ANOVA and 14 Tukey's honest significant difference test (p < 0.05). 15 16 17 Figure 5. Weibull curves from SSALT using CAD/CAM resin composite crowns for HC,

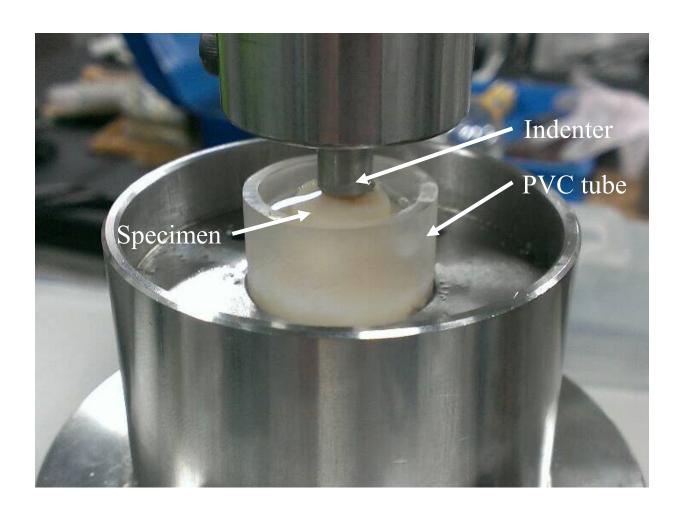
CS, and KA.

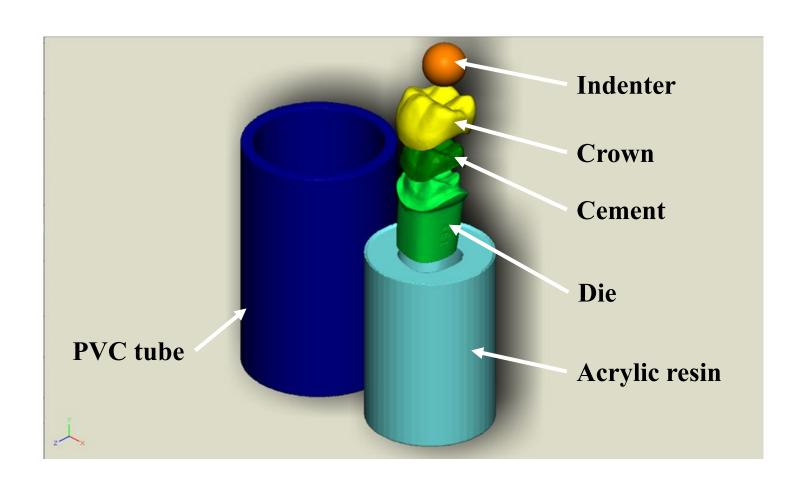
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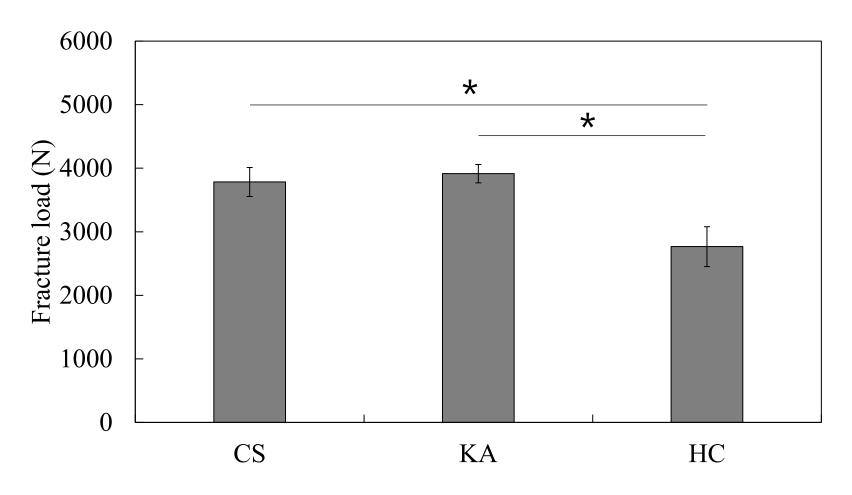
- 2 Figure 6. Stereomicroscopic images processed using high dynamic range software after
- 3 SSALT with an aggressive profile. (a) HC, (b) CS, and (c) KA.
- 5 Figure 7. SEM images of the fractured surface after SSALT with an aggressive profile
- 6 for (a) HC, (b) CS, and (c) KA. Large white arrows indicate crack initiation points.
- 7 Arrested lines are indicated by small white arrows. Hackle patterns following the
- 8 direction of crack propagation are indicated by asterisks.
- Figure 8. Maximum principal strain distribution at the crown of (a) HC, (b) CS, and (c)
- 11 KA.

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One-way ANOVA, Tukey's HSD test (*p < 0.05)

