

Title	In silico non-linear dynamic analysis reflecting in vitro physical properties of CAD/CAM resin composite blocks
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1 Research paper 2 In silico non-liner dynamic analysis reflecting in vitro physical properties of CAD/CAM 3 4 resin composite blocks 5 Dr. Oğuzcan Karaer^a, DDS, Dr. Satoshi Yamaguchi^{b*}, Ph.D., Dr. Yutaro Nakase^{b,c}, DDS, Dr. 6 Chunwoo Lee^b, DDS, Prof. Satoshi Imazato^{b, d}, DDS, Ph.D. 7 8 ^a Department of Prosthodontics, Faculty of Dentistry, Ankara University, Ankara 06560, 9 Turkey 10 ^b Department of Biomaterials Science, Osaka University Graduate School of Dentistry, 1-8 11 Yamadaoka, Suita, Osaka 565-0871, Japan 12 ^c Department of Pediatric Dentistry, Osaka University Graduate School of Dentistry, 1-8 13 14 Yamadaoka, Suita, Osaka 565-0871, Japan ^d Department of Advanced Functional Materials Science, Osaka University Graduate School 15 of Dentistry, 1-8 Yamadaoka, Suita, Osaka 565-0871, Japan 16 17

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27 Abbreviations 28 CAD/CAM Computer-aided design/computer-aided manufacturing 29 NTP Notchless triangular prism 30 FEA Finite element analysis 31 SEM Scanning electron microscopy

ABSTRACT

- 35 *Purpose.* The aim of this study was to assess the validity of *in silico* models of three-point
- 36 bending tests to reflect *in vitro* physical properties obtained from three commercially
- 37 available computer-aided design/computer-aided manufacturing (CAD/CAM) resin composite
- 38 blocks and demonstrate notchless triangular prism analysis with those properties.
- 39 *Material and Methods*. Three types of commercially available CAD/CAM resin composite
- 40 blocks were used: Cerasmart 300 (CS300; GC, Tokyo, Japan), Katana Avencia P Block (AP;
- 41 Kuraray Noritake Dental, Tokyo, Japan), and KZR CAD HR3 Gamma Theta (GT; Yamakin,
- 42 Osaka, Japan). In vitro/in silico three-point bending tests were conducted to obtain elastic
- modulus and fracture strain for nonlinear dynamic finite element analysis (n=10/each).
- Fractured surfaces of specimens after *in vitro* NTP tests were observed, and the fracture
- 45 toughness of each CAD/CAM resin composite was obtained by in silico NTP analysis.
- 46 **Results.** Both in vitro and in silico load–displacement curves obtained from three-point
- bending tests were significantly correlated (p < 0.05). The elastic moduli of CS300, AP, and
- 48 GT were 8.0 GPa, 10.0 GPa, and 9.0 GPa, respectively. The fracture toughness values
- obtained from in silico NTP analysis of CS300, AP, and GT were 5.057 MPa·m^{1/2}, 4.193
- 50 MPa·m^{1/2}, and 4.880 MPa·m^{1/2}, respectively. There was no significant difference in the length
- of the stable region among the three CAD/CAM resin composites (p = 0.09).

Conclusions. The in silico approach established in this study showed acceptable reflection of
 in vitro physical properties and will be useful for assessing fracture toughness related to the
 longevity of CAD/CAM resin composites without wastage of materials.
 KEYWORDS: Resin composites, Fracture toughness, Finite element analysis, Computer
 aided design, Computer aided manufacturing

1. INTRODUCTION

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Computer-aided design/computer-aided manufacturing (CAD/CAM) resin composite blocks are esthetic restorative materials and may possibly be used as an alternative to metals or ceramics (Ruse and Sadoun, 2014). CAD/CAM resin composite blocks have greater mechanical properties than conventional filling resin composites because of their highly dense nanofillers (Coldea et al., 2013a, b; Okada et al., 2014) and pre-polymerization procedures (Nguyen et al., 2012, 2013). After approval by Japanese health insurance companies in 2014, clinical treatment with CAD/CAM resin composites in the molar region have steadily increased. However, the longevity of CAD/CAM resin composite molar crowns is still unknown (Yamaguchi et al., 2018a). The fracture toughness of resin composites is one key criterion for evaluating resistance to crack propagation from the existing initiation points related to the fatigue performance of CAD/CAM resin composites. Additionally, a significant weak correlation between wear performance and clinical fractures relating to the fracture toughness has been reported (Heintze et al., 2017). The single-edge notched beam and the chevron notch short rod/beam tests are available to evaluate the fracture toughness of dental materials (Fujishima and Ferracane, 1996; Ilie et al., 2012; Ornaghi et al., 2014; Tantbirojn et al., 2003). However, it is difficult to reproduce the shape of the notch, making the specimen preparation challenging.

1996) as a simpler method to evaluate both the fracture toughness of materials and bonded

Notchless triangular prism (NTP) tests have been proposed by Ruse et al. (Ruse et al.,

79 interfaces between the adhesive and restorative materials (Eldafrawy et al., 2018; Mesmar and

Ruse, 2019). Tensile stress calculated by finite element analysis (FEA) using the NTP test and

the chevron notch short rod test showed no differences in distribution (Ruse et al., 1996).

FEA is well established as a method for assessing stress distribution, and is useful for investigating fracture initiation points in dental materials (Ausiello et al., 2004; Coelho et al., 2008; Dal Piva et al., 2018; Della Bona et al., 2013; Yamaguchi et al., 2018a; Yamanishi et al., 2014). It is difficult to determine which is the best composition of CAD/CAM resin composites to optimize fracture toughness, and *in vitro* fracture toughness tests are still conducted by using actual specimens. *In vitro* experimental results involve complex factors such as shape, size, materials, layout of fillers, and coupling conditions between fillers and the resin matrix (Ferracane, 2011). One of the advantages of FEA is the ability to evaluate the stress distribution focusing on specific factors related to mechanical performance (Yamaguchi et al., 2017). This feature makes FEA a particularly useful tool in materials design.

Non-linear dynamic FEA can represent fracture propagation corresponding to the fracture criteria of materials, as well as the stress distributions (Yamaguchi et al., 2014). Fracture criteria are important factors for obtaining accurate fracture loads reflecting the *in vitro*

results. The maximum principal strain has been reported as the best fracture criterion for evaluating the flexural strength of resin composites (Yamaguchi et al., 2018b). The combination of nonlinear dynamic FEA and the fracture criterion makes it possible to develop *in silico* models of NTP tests to reflect the *in vitro* results.

The aim of this study was to assess the validity of *in silico* models of three-point bending tests to reflect *in vitro* physical properties obtained from three commercially available CAD/CAM resin composite blocks and demonstrate notchless triangular prism analysis with those properties.

2. Materials and methods

2.1 CAD/CAM resin composites blocks

Three types of commercially available CAD/CAM resin composites blocks were used:

Cerasmart 300 (CS300; GC, Tokyo, Japan), Katana Avencia P Block (AP; Kuraray Noritake

Dental, Niigata, Japan), and KZR CAD HR3 Gamma Theta (GT; Yamakin, Osaka, Japan).

Details of the material composition of each block are summarized in Table 1.

Table 1. Composition of CAD/CAM resin composites used in this study

	Code	Manufacturer	Filler and monomer	Filler content (wt%)	
Cerasmart 300	CS300	GC	Barium glass, silica powder	78	
	C3300		UDMA, Bis-MEPP, polyfunctional methacrylate	76	
Katana Avencia P Block	AP	Kuraray Noritake Dental	Mixed filler with silanated barium glass, silanated colloidal silica, Cured resins consisting of methacrylate monomer, UDMA	82	
KZR-CAD HR3 Gamma Theta	GT	YAMAKIN	Inorganic oxide filler, fluoride-releasing filler, spherical nanofiller (SiO ₂) Methacrylate monomer	75	

2.2 *In vitro/in silico* three-point bending tests

In vitro three-point bending tests were conducted using each material $(4.0 \times 1.2 \times 14.0 \text{ mm})$ mm specimen, n = 10) after storage in water for 24 hours using a universal testing machine (EZ-SX, Shimadzu, Kyoto, Japan) with a 1.0 mm/min cross head speed. The obtained load–displacement curve was converted into a stress–strain curve. The flexural modulus was obtained from the initial slope of the stress–strain curve, and the fracture strain was recorded at the end

of the curve. Micro computed tomography (micro-CT; R_mCT2, Rigaku, Tokyo, Japan) analysis for calculating the volume and the weight measurement of each specimen were conducted to calculate densities. Using the data obtained by these *in vitro* tests, the elastic modulus of each material was obtained by *in silico* non-linear dynamic FEA (LS-DYNA, LSTC, Livermore, CA, USA). The flexural modulus and the elastic modulus are not equal. The flexural modulus was used as an initial elastic modulus for three-point bending analysis. After the first analysis, there is some difference between *in vitro* and *in silico* load displacement curve. Until converging those curves, the analysis was repeatedly conducted. Finally, the elastic modulus of each material was determined. The Poisson's ratio of each material was set to 0.38 according to that of dental composites (Greaves et al., 2011).

2.3 Specimen preparation and *in vitro* notchless triangular prism tests

For the notchless triangular prism (NTP) test, $6 \times 6 \times 6 \times 12$ mm specimens were prepared by milling machine (DWX-51D, Roland DG, Hamamatsu, Japan) and polished with #2000 SiC paper. All the specimens were immersed into 37°C water for 24 hours. A fracture initiation point 0.1 mm in depth was opened on the middle edge of the triangular prism specimen using a stainless steel razor blade using custom-made jigs (TRUE SEED, Kyoto, Japan). The edge of the triangular specimens was carefully observed under an optical

microscope (HM-211, Mitsutoyo, Kawasaki, Japan) with 10× magnification. The specimens were wiped and fixed on custom-made jigs for the NTP tests (TRUE SEED). The NTP tests were conducted with a universal testing machine (EZ-SX, Shimadzu, Kyoto, Japan) with a 1.0 mm/min crosshead speed.

The fractured surfaces of the specimens were then gold-coated using Quick Coater (SC-701, Sanyu Electron, Tokyo, Japan) and observed by scanning electron microscopy (SEM; JSM-6390BU, JEOL, Tokyo, Japan) under 30× magnification at 5 kV. The length from the crack initiation point to the boundary between the stable and unstable regions was measured using Image J software (National Institute of Health, Bethesda, MD, USA).

2.4 In silico notchless triangular prism analysis

Using the elastic moduli and fracture strains obtained from *in vitro* three-point bending tests and densities, NTP analysis was conducted by *in silico* non-linear dynamic FEA using the same CAD models designed for the custom-made jigs for the NTP tests (Fig. 1). The fracture toughness (K_{1c}) of each model was calculated using the following equation:

$$K_{1C} = \frac{P}{D\sqrt{W}}Y_{\min}$$

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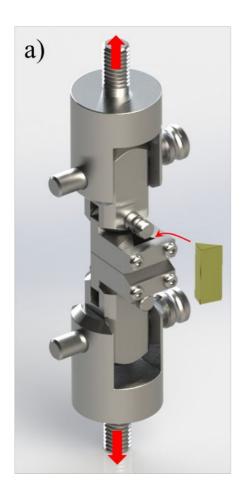
where P is the load at fracture, D (12.0 mm) is the length of the prism, W (10.5 mm) is the

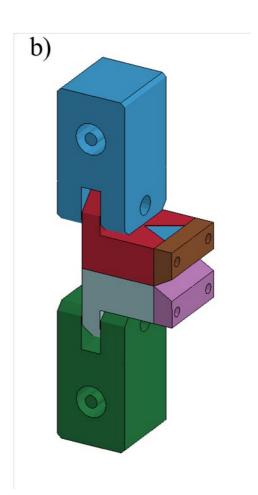
length of the mold, and Y_{min} (28.0) is the stress intensity factor.

The maximum principal strain distribution was obtained once there was 0.1 mm displacement

until fracture.

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Fig. 1 CAD models for NTP tests. a CAD models of in vitro NTP tests for fabricating custom-

made jigs. **b** CAD models for *in silico* NTP analysis.

2.5 Statistical analysis

Loads at each time point obtained from the *in vitro* three-point bending tests were compared statistically with the value determined by *in silico* analysis using Pearson's correlation tests (PASW Statistics 18, IBM, Somers, NY, USA). Additionally, loads at each time point obtained from the *in vitro* NTP tests were compared statistically with the value determined by *in silico* analysis using Spearman's correlation test.

The lengths of stable region obtained from the SEM images were compared statistically using one-way analysis of variance (ANOVA) with Tukey's post hoc test.

P-values of less than 0.05 were considered as statistically significant for all tests.

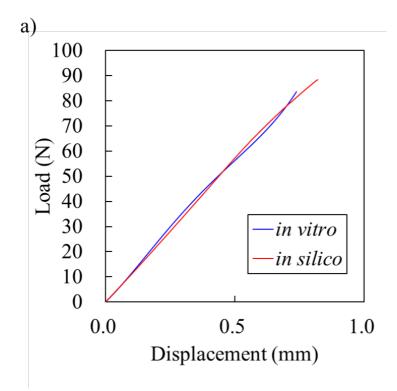
3. RESULTS

3.1 Elastic moduli obtained from *in silico* three-point bending analysis

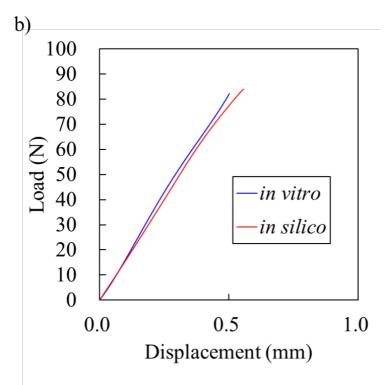
The flexural moduli, densities, and fracture strains of CS300, AP, and GT are summarized in Table 2. *In vitro* / *in silico* load–displacement curves after the three-point bending tests/analysis are shown in Figure 2. The *in vitro* curve was obtained by averaging all loads at each time point. Both *in vitro* and *in silico* load–displacement curves were significantly correlated (CS300: R=0.99, AP: R=1.0, GT: R=0.99, p < 0.05). The elastic moduli of CS300, AP, and GT were 8.0 GPa, 10.0 GPa, and 9.0 GPa, respectively.

Table 2. Physical properties of CAD/CAM resin composites obtained by *in vitro* three-point bending tests, micro-CT analysis, and weight measurement

	Flexural modulus (GPa)	Density (g/cm ³)	Fracture strain (ε)
CS300	6.765 ± 1.042	2.17 ± 0.00682	0.0366 ± 0.00627
AP	9.261 ± 1.774	2.35 ± 0.00994	0.0247 ± 0.00267
GT	7.424 ± 1.053	2.08 ± 0.00364	0.0322 ± 0.00346







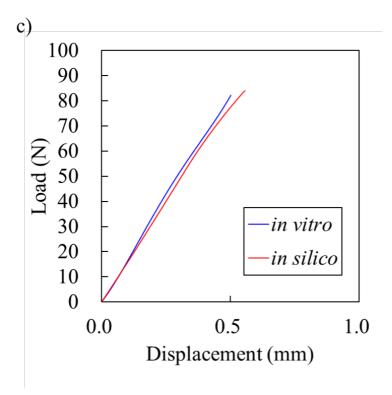


Fig. 2 Load–displacement curve obtained from *in vitro / in silico* three-point bending tests. The *in vitro* curve was obtained by averaging all curves. **a** CS300, **b** AP, **c** GT.

3.2 Fracture toughness after in silico NTP analysis

The fracture toughness values of CS300, AP, and GT obtained in *in silico* NTP analysis were 5.057 MPa·m^{1/2}, 4.193 MPa·m^{1/2}, and 4.880 MPa·m^{1/2}, respectively. *In silico* load—displacement curves of CS300, AP, and GT obtained in *in silico* NTP analysis are shown in Figure 3. The *in vitro* curve was obtained by averaging all loads at each time point. Both *in vitro* and *in silico* load—displacement curves were significantly correlated (CS300: R=1.0, AP: R=1.0, GT: R=1.0, p < 0.05).

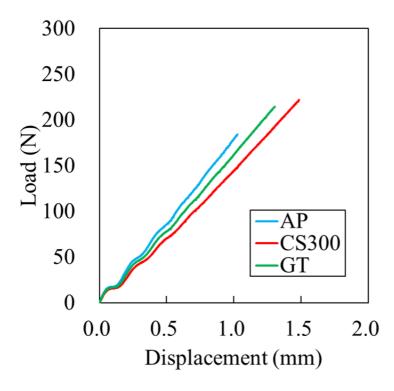
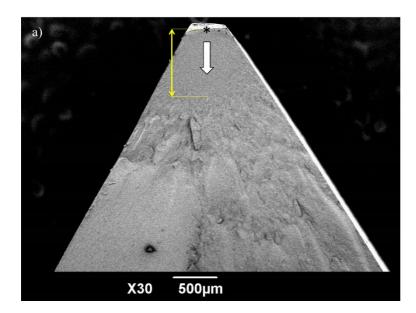


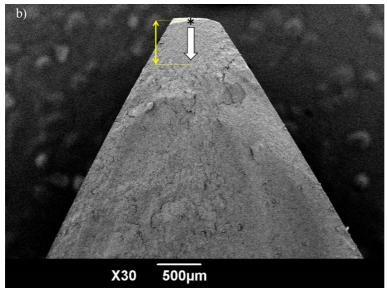
Fig. 3 Load–displacement curves obtained from in silico NTP analysis.

3.3 Fractographic analysis

Figure 4 shows SEM images at one side of the fractured surface from the initiation point after NTP tests for CS300 (Fig. 4a), AP (Fig. 4b), and GT (Fig. 4c). White arrows and bold white arrows indicate crack initiation points and the direction of crack propagation. Stable and unstable areas are indicated by yellow lines and were observed throughout the fracture surface of the specimens. The means and standard deviations of the length of the stable region are

shown in Figure 5. There was no significant difference among the three CAD/CAM resin composites (p = 0.09).





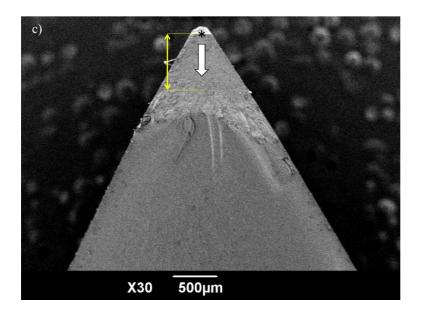


Fig. 4 SEM images of fractured surface of specimens after in vitro NTP tests. The yellow arrows

indicate the length of the stable region. The black asterisks indicate the fracture initiation point.

The white arrows indicate the direction of crack propagation. a CS300, b AP, c GT.



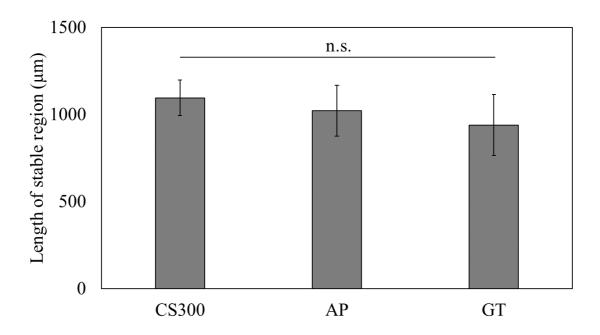
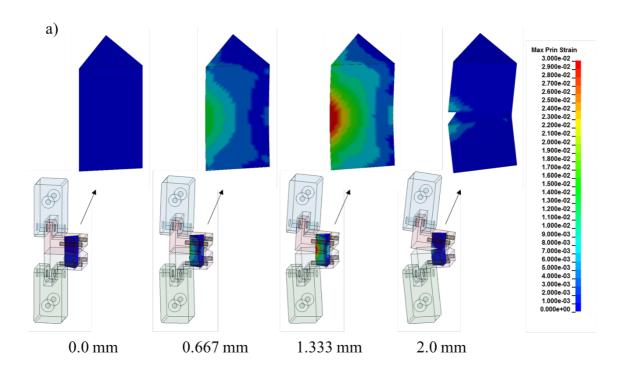


Fig. 5 Length of the stable region on the fracture surface after in vitro NTP tests.

3.4 Maximum principal strain distribution in the specimens

Figure 6 shows the maximum principal strain distribution during deformation and after fracture for CS300 (Fig. 6a), AP (Fig. 6b), and GT (Fig. 6c). The tensile strain (positive value of maximum principal strain) is concentrated on the middle edge of the specimens. Fracture was observed at the stretch after exceeding the fracture strain of each material.



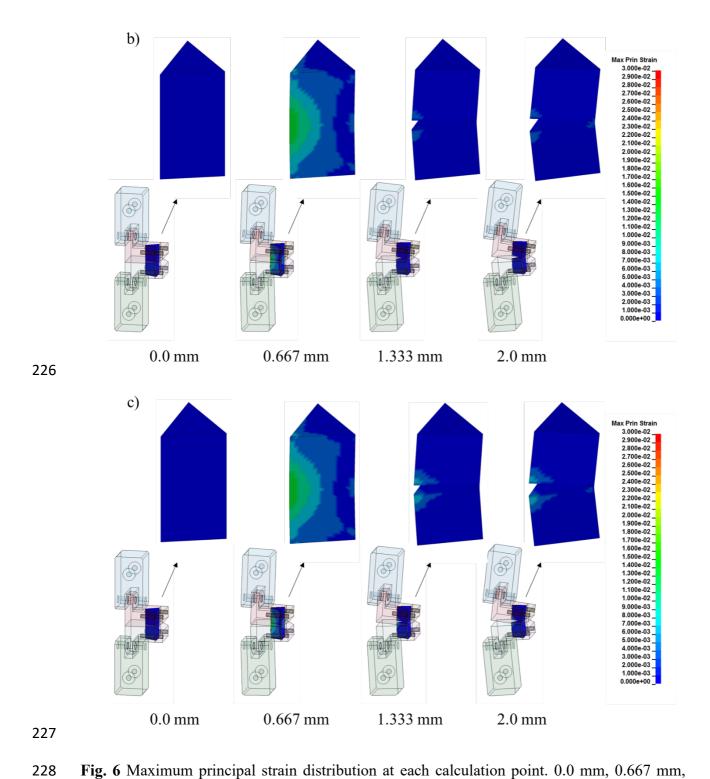


Fig. 6 Maximum principal strain distribution at each calculation point. 0.0 mm, 0.667 mm,

1.333 mm, and 2.0 mm from left to right. a CS300, b AP, c GT.

4. DISCUSSION

An *in silico* three-point bending model was successfully developed and could be used to predict fracture toughness to reflect *in vitro* physical properties obtained from three commercially available CAD/CAM resin composite blocks and demonstrate *in silico* notchless triangular prism (NTP) analysis with those properties. We believe that the *in silico* approach using non-linear dynamic FEA established in this study will become an alternative way to assess fracture toughness related to the longevity of CAD/CAM resin composite crowns without wastage of materials.

The three-point bending tests indicated that the elastic moduli of CS300 and GT were lower than that of AP. From the composition of the materials as shown in Table 1, the inorganic filler content of CS300 and GT was 78 wt% and 75 wt%, respectively, while that of AP was 82 wt%. A lower filler content of resin composites results in a lower flexural modulus (Randolph et al., 2016)/strength (Yamaguchi et al., 2018b) and the volume of the matrix resins must be increased in relation to the amount of fillers. Thus, the lower filler content of CS300 and GT may have resulted in a lower elastic modulus. The elastic modulus is one of the mechanical properties used for evaluating and ranking resin composites (Ilie et al., 2017), and is defined as the initial slope of the stress–strain curve for a given material under loading.

In silico three-point bending analysis using non-linear dynamic FEA successfully reflected the *in vitro* testing results. These results suggest that the elastic modulus, density, and fracture strain of each CAD/CAM resin composite could be useful for predicting the flexural modulus and flexural strength. These parameters may be applied to other testing methods such as biaxial flexural tests, four-point bending tests, and fracture toughness tests such as the NTP tests used in this study.

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Fracture toughness is an intrinsic material property and is a measure of the energy required to propagate a crack from an existing defect. The NTP test for evaluating fracture toughness adopted in this study was originally proposed by Ruse et al., 1996). The applications of NTP tests are now expanding to evaluate the interfacial properties of dental materials (Alkadi and Ruse, 2016; Eldafrawy et al., 2018; Mesmar and Ruse, 2019). The in silico load-displacement curve of GT in the NTP tests was well matched to the in vitro curve. For CS300 and AP, lower fracture loads and displacements were observed in vitro compared with in silico with statistically significant correlations. One of the reasons for these differences was the construction of jigs for the NTP tests. The specimen cannot be fixed to the jigs without fixation jigs with screws. Even if there are slight differences in the specimen's dimensions at the microscale order, static and dynamic friction properties among the jigs and the specimen will be dramatically changed on the fixation force. Thus, the in silico approach could avoid these frictional effects and allow direct comparison by focusing on the different material composition of each CAD/CAM resin composite block.

The SEM images showed that both stable and unstable areas (Alkadi and Ruse, 2016) resulted in mode I fracture (the tensile loading direction is vertical to the fracture surface). The length of the stable area from the crack initiation point to the boundary between the stable and unstable areas was not significantly different among the CAD/CAM resin composites, while a shorter length of stable area has been reported in lithium disilicate glass ceramics (Alkadi and Ruse, 2016; Mesmar and Ruse, 2019). These results suggest that CAD/CAM resin composites inhibit crack propagation to a greater degree than lithium disilicate glass ceramics.

Stable and unstable areas were not observable on the *in silico* models because the mesh size of 0.3 mm was too large to represent the nano/microstructural details of the fractured surface. The calculation cost would need to be increased corresponding to a higher resolution mesh. Multi-scale analysis may be helpful to find the crack initiation point at the nano/microscale level (Takano et al., 2010; Takano et al., 2003; Yamaguchi et al., 2017). *In silico* nanoscale models of CAD/CAM resin composites blocks have been developed using electron microscopy with cryo-stage and focused ion beam (Lee et al., 2019). The combination of non-linear dynamic FEA and multi-scale analysis using nanoscale models may accelerate

elucidation of the fracture mechanism and the development of new materials with greater longevity.

While of CAD/CAM resin composites exhibit greater fracture toughness, hydrolysis of the silane coupling and matrix resins in the oral environment (Drummond, 2008; Kusuma Yulianto et al., 2019) should be considered in future studies. Nanoscale models containing a silane coupling layer are under development for investigating the influence on the mechanical properties of CAD/CAM resin composites.

5. CONCLUSIONS

The *in silico* approach established in this study acceptably reflected the *in vitro* physical properties and will be useful for assessing fracture toughness related to the longevity of CAD/CAM resin composites without wastage of materials.

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