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In vitro/in silico investigation of failure criteria to predict flexural strength of composite resins

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The aim of this study was to investigate a failure criterion to predict flexural strengths of composite resins (CR) by three-dimensional finite element analysis (3D-FEA). Models of flexural strength for test specimens of CR and rods comprising a three-point loading were designed. Calculation of Young's moduli and Poisson's ratios of CR were conducted using a modified McGee-McCullough model. Using the experimental CR, flexural strengths were measured by three-point bending tests with crosshead speed 1.0 mm/min and compared with the values determined by *in silico* analysis. The flexural strengths of experimental CR calculated using the maximum principal strain significantly correlated with those obtained *in silico* amongst the four types of failure criteria applied. The *in silico* analytical model established in this study was found to be effective to predict the flexural strengths of CR incorporating various silica filler contents by maximum principal strain.

Keywords: *In silico* analysis, Flexural strength, Composite resins, Failure criterion, Finite element analysis

INTRODUCTION

The composition of composite resins (CR) has been rapidly evolving from macro-fill (10–50 μm)[1] to nano-fill (5–100 nm)[2,3] approaches for 50 years[4]. Consequently, mechanical strengths such as flexural strength have continuously improved and recent use of CR in posterior restorations has significantly increased because of good survival rates[5-9]. The main causes of failure in CR are secondary caries and restorative fracture[6,8]. However, the flexural strength and a fracture toughness, which correlate with clinical wear performance[10], have not yet equaled or exceeded that of metal restorations. For further improvement of the mechanical properties, optimization of multi-factorial factors such as weight/volume content, size, type, shape, or geometric layout of fillers[11] are expected to aid progress towards metal-free restorations[12]. Flexural and compressive properties of 72 types of CR have been investigated by *in vitro* three-point bending tests and the flexural modulus and flexural strength shown to increase with an increase in filler content up to 60 vol%[13]. These CR are also influenced by multi-factorial factors such as filler size, shape, and the component of resin matrix other than filler content. Consequently, a clear cause of the increase in flexural properties has not strictly been clarified *in vitro*.

In silico analysis is one possible solution to the need for rigorous comparison of multi-factorial influences. The micro-structure of CR containing irregular fillers has been modelled using the micro-CT (computer tomography) imaging technique and the relationship between physical properties and the volume contents of fillers was explored by three-dimensional finite element analysis (3D-FEA)[14]. Despite the fact that geometrically realistic finite element models have exhibited similar trends in experimental results in terms of Young's modulus, to date there has been no way to directly predict flexural strength.

To predict flexural strength, an appropriate failure criterion for CR is required. Generally, maximum shear stress and von Mises stress have been used as the failure criteria for ductile materials, while maximum principal stress and strain have been applied to brittle materials[15].

In the case of CR, mixed failure criteria are estimated because CR with 0 vol% fillers (100 vol% resin matrix) are ductile materials and those comprising 100 vol% fillers are brittle materials[16]. The Drücker-Prager criterion has been proposed as one of the failure criteria for CR[16]. Although this criterion can represent residual stress during the curing process and stress relaxation of resin matrix from a long-term perspective[17], this criterion assumes the presence of filler particles without resin matrix. In addition, one dimension of the specimen has been modified[18], and the use of 3D-FEA for CR has been extensively refined[19-22].

Correlation between the flexural strengths and volume contents of fillers has been evaluated by 3D-FEA using failure criteria comprising the von Mises stress for the resin matrix and maximum stress for the silica fillers at micro-scale. Analytical results using this approach showed good agreement with experimental results[23]. However, the von Mises criterion provides unrealistic results, with low peaks in stress[17], albeit offering ease of implementation. In addition, there have been no comparison studies investigating the use of

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failure criteria to predict the flexural strength of CR at the macro-scale.

The aim of this study was to investigate a failure criterion to predict flexural strengths of CR by 3D-FEA.

MATERIALS AND METHODS

Three-dimensional CAD model

Three-dimensional CAD models composed of three rods and a specimen were created using the SolidWorks Simulation software package (SolidWorks, Concord, MA, USA). According to ISO 4049:2009[18], two rods were arranged in parallel, with 20 mm between centers, and a third rod was centered between and parallel to the others. Three rods in combination can be used to give a three-point loading to a specimen with dimensions 2×2×25 mm.

Mechanical properties of specimens

The mechanical properties of the specimen for each filler volume content, using 3D-FEA, are shown in Fig. 1. Young's moduli and Poisson's ratio were calculated by a modified McGee-McCullough model[14]. For Young's moduli, the constant values ε and α used for the calculation in the modified McGee-McCullough model were 2 and 1, respectively. The constant values for Poisson's ratio were 1 and 2.3697, respectively. Pure Young's moduli and Poisson's ratios of fillers and the resin matrix were 72 and 3 GPa; and 0.16 and 0.45, respectively[14].

Three-point bending test

Using experimental CR containing irregular silica fillers and 2,2-bis[4-(2-hydroxy-3-methacrylyloxy-propoxy)-phenyl]-propane (Bis-GMA)/triethyleneglycol dimethacrylate (TEGDMA), flexural strengths were measured according to ISO4049:2009 using a universal testing machine (EZ-SX, Shimadzu, Kyoto, Japan) with crosshead speed 1.0 mm/min (n=10). Contents (monomer/filler) of the experimental CR were 68.8/31.2 (50.0/50.0), 59.8/40.2 (40.0/60.0), 49.7/50.3 (30.0/70.0), and 26.3/63.7 (20.0/80.0) vol% (mass%), respectively. Compositions of monomers were Bis-GMA (75 mass%), TEGDMA (25 mass%), benzoyl peroxide (BPO) (1.4 mass%), dl-camphorquinone (CQ) (0.7 mass%), and aldehyde compound and dibutylhydroxytoluene (2.17 mass%). Irregular silica glasses (D99: 57.2 μm, D50: 5.7 μm) with surface modification by γ-methacryloxyproyl trimethoxysilane (MPS)[24] were used as fillers. Each specimen was light-cured for 10 s with the light-emitting-diode curing light (Pencure 2000, Morita, Kyoto, Japan) which had a maximal light density of 2,000 mW/cm^2.

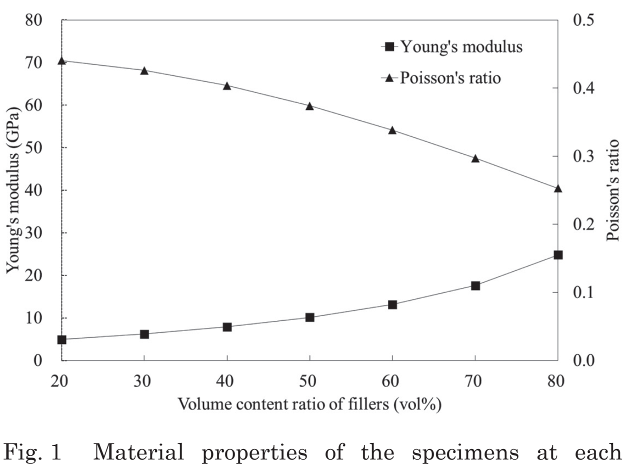

Fig. 1 Material properties of the specimens at each volume content, calculated by the modified McGee-McCullough model.

3D-FEA

For simulations of three-point bending test, a 'contact' condition[25], which accepts possible sliding, was set at the interfaces between the three rods and the specimen. Two rods were fixed, and a static load was applied to the third rod in a downward direction at the center of the specimen, until failure criteria (maximum principal (MP) stress, MP strain, XZ shear stress, and von Mises stress) reached given thresholds. The rods and the specimen were divided into the finite element meshes of tetrahedrons with 16 nodes, with dimensions 0.258 mm. The 3D-FEA was performed using an advanced function of the CAD software (SolidWorks). Distribution of each failure criteria was observed and the location indicated the maximum value of each failure criteria was compared. Flexural strengths were defined when the failure criteria, calculated using the fracture loads of experimental CR with 50.3 vol% fillers, exceeded given thresholds. All flexural strengths, for CR ranging from 20 to 80 vol% in 10-vol% steps, were calculated by 3D-FEA. Coefficients of variations (standard deviation/mean)[26] for each failure criteria were calculated and stress and strain distributions in specimens were observed.

Statistical analysis

Flexural strengths obtained from the *in vitro* three-point bending test were compared statistically with the values determined by *in silico* analysis using Pearson's correlation coefficient test (PASW Statistics 18, IBM, Somers, NY, USA).

RESULTS

Flexural strength resulting from in vitro testing and in silico analysis

The flexural strengths of experimental composites containing 31.2, 40.2, 50.3, and 63.7 vol% filler were 114.8±10.5, 128.6±6.9, 138.8±4.4, and 180.1±17.3 MPa, respectively (Fig. 2). Thresholds for calculating the flexural strength *in silico* of MP stress, MP strain, XZ shear stress, and von Mises stress were 137.9 MPa, 0.01161, 61.1 MPa, and 140.8 MPa, respectively. They were significantly correlated with those obtained through *in silico* 3D-FEA using the failure criteria of MP strain (96.6, 117.2, 146.3, and 201.3 MPa; Pearson's correlation test, r=0.992, p<0.01), while there was no significant correlation for MP stress (146.8, 147.3, 147.2, and 147.0 MPa; r=0.045, $p>0.05$) and negative correlations for XZ shear stress (165.1, 157.1, 148.0, and 137.3 MPa; r=−0.964, $p<0.05$) and von Mises stress (153.5, 147.8, 146.3, and 138.8 MPa; r=−0.979, $p<0.05$) (Fig. 3).

Failure criteria

Coefficients of variations (standard deviation/mean)

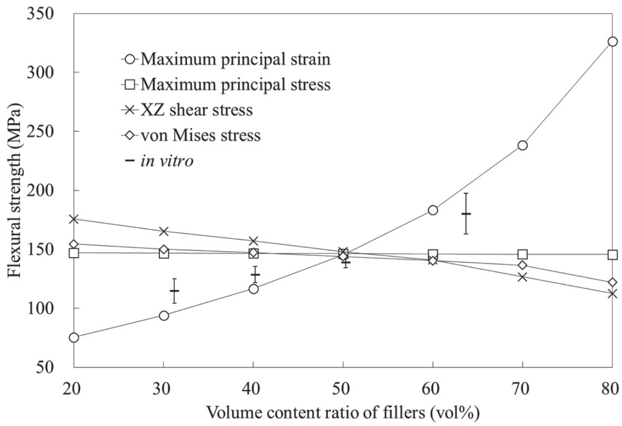

Fig. 2 Flexural strengths obtained by *in vitro* testing and *in silico* analysis.

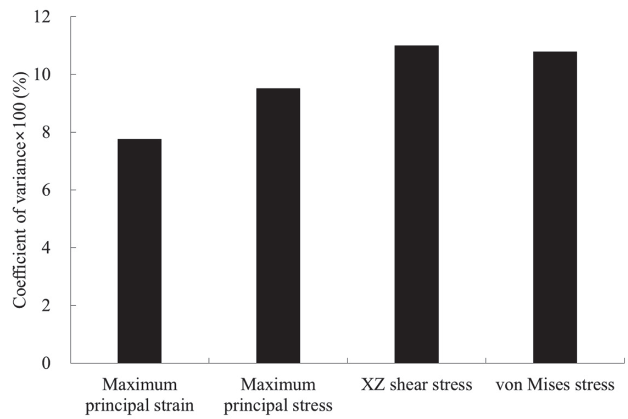

Fig. 4 Coefficient of variance (standard deviation/mean).

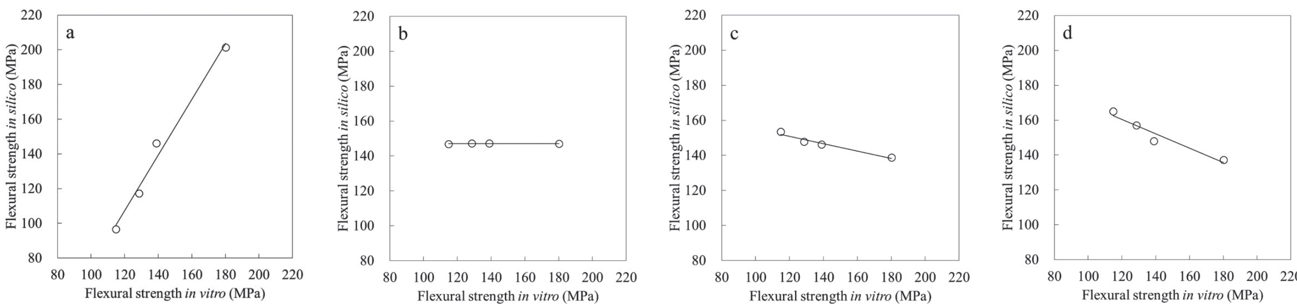

Fig. 3 Correlation between *in vitro* and *in silico* flexural strengths of (a) MP strain, (b) MP stress, (c) XZ shear stress, and (d) von Mises stress.

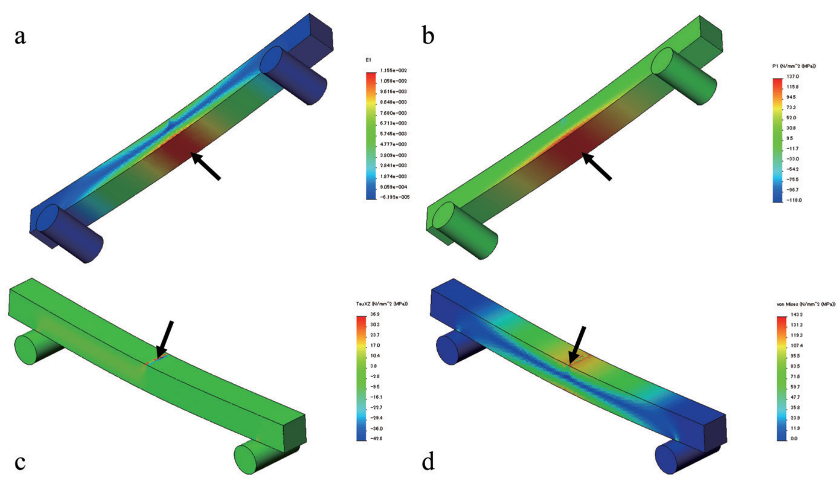

Fig. 5 Stress/strain distribution of model obtained by 3D-FEA.
Black arrows indicate the location at which the maximum value of failure criteria were observed: (a) MP strain, (b) MP stress, (c) XZ shear stress, and (d) von Mises stress.

of MP strain, MP stress, XZ shear stress, and von Mises stress were 7.785, 9.505, 10.994, and 10.782%, respectively (Fig. 4). The coefficient of variation in MP strain was the lowest of the failure criteria.

Stress/strain distribution

Figure 5 shows strain/stress distribution of each failure criteria. The model of the third rod for loading has been hidden to show the strain/stress distribution clearly. Maximum MP strain and MP stress values were observed at the bottom center of the models in a longitudinal direction (Figs. 5a and b). Maximum XZ shear stress was observed at the top center of the model and concentrated in a small area (Fig. 5c). Maximum von Mises stress value was observed at the top center of the model while the stress concentration was observed at both the top and bottom of the model (Fig. 5d).

DISCUSSION

In terms of three-point bending analysis, Young's modulus and Poisson's ratio calculated using the modified McGee-McCullough model were used as material properties in 3D-FEA. The flexural strengths were predicted using four types of failure criteria used as thresholds: MP strain, MP stress, XZ shear stress, and von Mises stress. The prediction of the flexural strengths from the multi-factorial factors of fillers such as size, type, and geometric layout of the fillers is useful for creating new designs for composite materials and for their optimization.

Despite slight differences between the flexural strengths *in silico* and *in vitro* at 31.2 and 63.7 vol%, the flexural strengths obtained using MP strain *in silico* strongly correlated with those obtained *in vitro*. One of the reasons for the slight difference found at 63.7 vol% was defects in the CR such as unbonded filler/matrix interfaces and micro bubbles in the resin matrix of experimental CR have been shown to negatively affect material properties of CR[14]. The other slight difference at 31.2 vol% suggests that the material characteristics of CR shifted from brittle to ductile behavior, with a non-linear load-deflection curve. Assuming the intended end-product use is in the clinic, the contents of fillers used in this study ranged from 20 to 80 vol% because lower and higher filler contents have differing viscosities[14,27] and lead to unsatisfactory handling properties of CR before light curing. In addition, higher filler contents exhibit lower fatigue resistance[28]. No significant or negative correlations for MP stress, XZ stress, and von Mises stress were caused by the change in yield stress of specimens as the volume contents of fillers increased.

The lowest coefficient of variance for MP strain values means that this failure criterion was appropriate as the threshold for predicting the flexural strength of CR because its coefficient of variance tended to be nearly invariant with changes in the volume content of fillers. These results agree well with the result of a survey on failure criteria of laminate composites carried out by the American Institute of Aeronautics and Astronautics[29].

With regard to the investigation of maximum shear stress theory[15], XZ shear stress was selected in this study as the threshold because it has the greatest stress, compared with XY and YZ shear stresses. The maximum tensile stress value during the three-point bending test has been observed to occur at the bottom center of the specimen in a longitudinal direction[16], while crack initiation in experimental CR has been observed through fractography to occur at the bottom center of specimens[30]. Fractured surface of the specimens after *in vitro* three-point bending tests may be affected by the direction of an initial crack similar to the highly filled resin composites[31]. The MP stress distribution (Fig. 5b) showed a similar trend to conventional studies *in vitro*. These trends suggest that the maximum shear stress and von Mises stress values observed at the top center of the model (Figs. 5c and d) were not appropriate for use as failure criteria in three-point bending analysis *in silico*.

A limitation of this study was a lack of simulation of multi-factorial properties such as size, type, and geometric layout of the fillers. Multi-scale analysis[32,33] enables the investigation of the influence of the multi-factorial properties of fillers at micro- or nano-scale on the physical properties at macro-scale[34]. The *in silico* analysis established in this study, which replaces theoretical analysis with a composite theory such as the modified McGee-McCullough model, in a multi-scale analysis used to calculate the material properties of CR at macro-scale, is applicable to the investigation of the above-mentioned factors, while conventional approaches are not[14,16,17].

Modeling of CR micro-structure using nano-CT imaging technology[35] is ongoing, to investigate the influence of size, type, shape, or geometric layout of fillers on the flexural strength at macro-scale. Molecular dynamics simulation[36] may be an effective approach to determining the influence of silane coupling ratio on flexural strength.

CONCLUSION

The *in silico* analytical model established in this study was found to be effective to predict the flexural strengths of CR incorporating various silica filler contents by MP strain, suggesting its possible usefulness for optimization of the mechanical properties of CR.

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CONFLICTS OF INTEREST

The authors declare no conflicts of interest.

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