Estimation on Residual Stress in Porcelain/Alloy System by Thermal Shock Test

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Computer calculations were used to simulate the effects of thermal shock on the stresses developed in porcelain-fused-to-metal (PFM) systems. A three-dimensional-finite-element model was employed to first calculate time-dependent temperature distribution in a beam shaped specimen $1.25-2.5 \times 8 \times 20$ mm. These were then used in a two-dimensional analysis of stresses in a beam consisting of 50 elements. Transient stresses in a beam consisting of 50 elements were calculated for porcelain, porcelain with an opaque layer, and porcelain-opaque-alloy system; the effects of porcelain thickness, thermal expansion coefficients, and quenching temperatures were evaluated. Experimental tests were also conducted for correlation with the theoretical calculations.

Results show that for $\alpha_m(\text{metal}) > \alpha_p(\text{porcelain})$, the highest stresses occur on the surface, and the interface stress is lower than $\alpha_m = \alpha_p$. When $\alpha_m < \alpha_p$, the surface stress is lower, and the interface stress is higher than $\alpha_m = \alpha_p$. When $\alpha_p - \alpha_m < 2 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$, cracks are more easily initiated in the porcelain surface rather than the interface. Theory and experiment are in general agreement.

Key words : Porcelain-fused-to-metal, Thermal Shock Test, Residual Stress

INTRODUCTION

The study of porcelain-fused-to-metal (PFM) restorations has included interest in the bond strength. Bending, pull-out, and push-out tests were adapted for this purpose. Anusavice *et al.*¹⁾ discussed eleven PFM bond tests and Asaoka and Kuwayama²⁾ computed interfacial shear stress distribution for pull-out tests using finite-element stress analysis. Dehoff *et al.*³⁾ compared the experimental observation of fracture from the finite-element stress analysis of a four-point flexural test for PFM. These studies show that the actual bond strength is very difficult to measure because the stress distribution along the interface is not uniform in every test specimen. Thus, recent research emphasis has shifted from measuring PFM bond strength to compatibility of PFM. Asaoka and Kuwayama^{4,5)} calculated the thermal residual stress in a PFM composite beam after firing using beam theory. Tesk *et al.*^{6,7)} conducted the effect of thermal expansion coefficient (α) and thickness ratio variations on gap displacement for a three-layered split ring. Dehoff and Anusavice⁸⁾ calculated residual stresses due to thermal contraction differences in PFM using semicircular arch. Walton and O'Brien⁹⁾ examined crack development due to thermal stress in porcelain, which had different values of thermal expansion coefficient, bonded to palladium-silver alloy. Anusavice *et al.*¹⁰⁻¹²⁾ employed a thermal shock test for disc shaped specimens to help determine the compatibility of PFM.

The objective of this study was to employ non-steady-state heat-transfer analysis and two dimensional composite beam stress analysis to calculate the effect of residual stress in

a PFM beam on the thermal shock resistance.

MATERIALS AND METHODS

Simulation Method—Time-dependent temperature distributions in a beam shaped porcelain 1.25 and $2.5 \times 8 \times 20$ mm, were simulated using a non-steady-state heat-transfer finite element method. The transient stresses in the beam arise from the differences in the local thermal contraction in the beam. These stresses can be calculated from the thermal contraction and the temperature distributions at any given time.

Fig. 1 Segment of an n -layer strip. E_1 , E_2 , E_{n-1} and E_n are elastic moduli; h_1 , h_2 , h_{n-1} and h_n , thicknesses of layer; α_1 , α_2 , α_{n-1} and α_n , linear coefficients of thermal expansion; and T_1 , T_2 , T_{n-1} and T_n temperatures.

In this study, the specimen is divided into n thin layers as shown in Fig. 1. There are no external forces acting on the specimen. That is,

$$\sum_{i=1}^n P_i = 0$$

where P_i is the axial load of the i th material. Then the relation between the moments of each element is given by :

$$\sum_{i=1}^n k_i P_i = \sum_{i=1}^n E_i I_i / \rho \quad \text{here, } k_i = \frac{h_o - h_i}{2} - \sum_{j=1}^{i-1} h_j$$

where E_i is the modulus of elasticity of the i th material; I_i , the moment of inertia of area; h_i , the thickness of the i th material; and h_o is the total thickness of the beam.

$$I_i = \frac{b h_i^3}{12}$$

where b is the strip width.

The next $(n-1)$ equations must be evaluated because all of the elements bond to each other.

$$\alpha_k T_k + P_k / (E_k b h_k) + h_k / 2\rho = \alpha_{k+1} T_{k+1} + P_{k+1} / (E_{k+1} b h_{k+1}) - h_{k+1} / 2\rho$$

where α_k and α_{k+1} are respectively the coefficients of thermal expansion of i th and $(i+1)$ th materials; T_k and T_{k+1} , the temperatures; and ρ is the radius of curvature of the beam. From these $(n+1)$ equations, $P_1 - P_n$ and $1/\rho$ can be calculate. Thus, the normal stresses (σ_{k1} and σ_{k2}) at the interface in material k can be calculated from P_k and $1/\rho$.

$$\sigma_{k1} = -E_k h_k / 2\rho + P_k / h_k b$$

$$\sigma_{k2} = E_k h_k / 2\rho + P_k / h_k b$$

If metal and porcelain have the same thermal expansion coefficient, internal normal stress does not develop from an equilibrium temperature change. Dynamic temperature change causes normal transient stress in the material. When the alloy has a different coefficient of thermal expansion from the porcelain, the normal internal stress is given by the sum of the equilibrium internal stress and the dynamic normal stress. The physico-mechanical properties of the porcelain in this study and the conditions are shown in Table 1.

Table 1 Physical and mechanical properties of porcelain and calculation conditions

Thermal conductivity	0.0025 cal/cm s°C
Density	2.5 g/cm ³
Specific heat	0.20 cal/g°C
Coefficient of thermal expansion	14 × 10 ⁻⁶ /°C
Modulus of elasticity	70 GPa
Time step increments	0.01 s
Number of time steps	500 steps

Experimental method—Ceramco¹ Gingival Al porcelain, Paint-O-Pake Al porcelain and three kinds of casting alloys² were used in this investigation. Specimens were made as follows: Porcelain powder was first weighed and placed in 10 × 30 mm stainless dies. The powder was preformed into useful shapes by a 4 ton (1.3 ton/cm²) force. The shaped powder was preheated to 650°C for five minutes on a stainless steel plate and made into a biscuit. The biscuit was baked on the porcelain investment up to 650°C in air, 650–930°C in vacuum and up to 1000°C in air. The specimen was then slowly cooled in the furnace. The quenching test specimen was modified to the shape to be used. Each specimen was then heated up to 800°C in air for 10 minutes in the furnace and slowly cooled in the furnace before the quenching test. The thermal shock test was then conducted. The specimen was held in the furnace at the quenching temperature for 20 minutes and then quenched into ice water. The cracks in the specimen were observed using an optical microscope. The magnification was about 50 and 100. If the specimen had no cracks, the quenching temperature raised 10°C and the same tests were continued.

RESULTS AND DISCUSSION

Porcelain—For the specimen which was held at 100°C and then quenched into ice water (0°C), the time dependent temperature distribution in the beam with 2.5 mm thickness (Specimen A) is shown in Fig. 2. The specimen has a large gradient of temperature in the

¹ Ceramco, Inc., East Windsor, NJ, USA

² Alloy A: Ultratek; Ni 80.48–Cr 11.39–Co 0.50–Al 2.22–Be 1.60–Fe 2.00–Mo 1.99; Metals for Modern Dentistry, Inc., Concord, CA, USA

Alloy B: Porcelain Metal 12K; Au 50–Pt 1–Pd 25–Ag 18–In 5–etc.; Sankin Industry Co., Ltd., Osaka, Japan

Alloy C: Porcelain Gold; Au 83–Pt 7–Pd 7–Ag 1–Ir 1–etc.; Sankin Industry Co., Ltd., Osaka, Japan

initial stages from the time of bathing in ice water. The temperature is gradually changed to a flat distribution, and eventually reaches to the uniform distribution at last. Figure 3 shows how temperature changes with time. The points A, B, and C represent the 1.25 and 0.625 mm from the surface of specimen A, and the center part of the beam with thickness 1.25 mm (Specimen B), respectively.

Fig. 2 Time dependent temperature distribution across the porcelain beam with 2.5 mm thickness quenching in 0°C ice water from heat-soak temperature 100°C.

Fig. 3 Temperature-Time curves for the porcelain beams with the thicknesses of 2.5 and 1.25 mm heat-soaked at 100°C and quenched in 0°C ice water.

Transient stresses were calculated from these temperature distributions as shown in Figs. 4 and 5. The vertical axis represents the transient stress and the horizontal axes represents time and distance from the surface of the specimen. The surface has tensile stress and the center part of the specimen has compressive stress. Maximum compressive stresses reached 26 MPa for specimen A, and 24 MPa for specimen B, respectively. Maximum tensile stresses were 65 and 55 MPa.

Fig. 4 Transient stress distribution profile for the porcelain beam with 2.5 mm thickness heat-soaked at 100°C and quenched in 0°C ice water.

Fig. 5 Transient stress distribution profile for the porcelain beam with 1.25 mm thickness heat-soaked at 100°C and quenched in 0°C ice water.

Figure 6 shows the relation between the transient stress and quenching temperature. The solid lines show the maximum tensile stresses at the surface of the specimen A at the 0.01, 0.5 and 1.0 s after quenched. This result shows clearly that the transient stress is directly proportional to the quenched temperature and confirms the work of Anusavice *et al.*¹²⁾ The tensile strength of porcelain will then be able to be estimated from the quenched temperature of thermal shock tests provided other properties are well known.

Figure 7 shows the relation between the transient stress and the coefficient of thermal expansion of porcelain. The results, of course, show that this relation is proportional. When

Fig. 6 Relation between transient stress and heat-soaked temperature for porcelain beam with 2.5 mm thickness. $\alpha_p = 14 \times 10^{-6}/^{\circ}\text{C}$; and $E_p = 70 \text{ GPa}$.

the porcelain has a low coefficient, the contraction of the porcelain is small and the internal stress is then low. The porcelain which has a low coefficient of thermal expansion has high thermal shock resistance. Figure 8 shows the experimental results of thermal shock tests for the specimen A. The surface cracks in (a), (b), (c) and (d) show the results of quenching from 125, 150, 200 and 250°C, respectively. The maximum tensile transient stresses, as estimated from the simulation results, reached 79, 94, 107 and 157 MPa. The patterns of these cracks show clearly the magnitudes of these transient stresses. The cracks were not initiated below 100°C quenching temperature. Figure 9 shows the results for specimen B. The transient stresses calculated are 69, 82, 110 and 137 MPa, respectively. Ten specimens, which have the same dimensions as specimen B, were tested. When quenched from 120°C, none of these specimens show cracks. When quenched from 130°C, the ten specimens had surface cracks.

Fig. 7 Relation between transient stress and linear coefficient of thermal expansion for the porcelain with 2.5 mm thickness quenched from heat-soaked temperature 100°C to 0°C ice water. $E_p = 70 \text{ GPa}$.

Fig. 8 Cracks development in the quenched porcelains with 2.5 mm thickness. (a) quenched into 0°C ice water from heat soaked temperature of 125°C ; (b) from 150°C ; (c) from 200°C ; and (d) from 250°C.

Fig. 9 Cracks development in the quenched porcelains with 1.25 mm thickness. (a) quenched into 0°C ice water from heat soaked temperature of 125°C ; (b) from 150°C ; (c) from 200°C ; and (d) from 250°C.

The tensile strength of this porcelain was then estimated about 70 MPa. It is concluded that the thermal shock test is an easy and reliable test for tensile strength of porcelain.

Porcelain-Opaque—The thermal expansion/shrinkage of the opaque porcelain above the glass transition temperature, is different from that of body porcelain. Below that temperature, thermal deformation of these two porcelains is very close. The elastic modulus of the opaque porcelain assumed to be close to that of the body porcelain. Six specimens with 1.25 mm thickness were made to combine body and opaque porcelain. The thickness of opaque porcelain was about 0.25 mm. For these specimens, surface cracks were observed in 2, 3 and 1 specimens with quenching temperatures from 120, 130 and 140°C, respectively. The maximum tensile stresses were calculated as 66, 72 and 77 MPa from the results of simulation. These strengths were nearly the same as those found for the body porcelain.

Porcelain-Opaque-Alloy—A deeper understanding of the data for compatibility in PFM can be acquired by comparing the difference of stress distributions between porcelain and PFM during the quenching test.

Several situations are considered: (A) Alloy and porcelain have the same coefficient of thermal expansion. Transient stress in the specimen is determined by the elastic moduli, the coefficients of thermal expansion, thickness and shape of the combined materials. The assumptions made on this calculation are that the beam has no internal stress before thermal shock test and the alloy and porcelains have the same value of the coefficients of thermal expansion; $14 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ (Whitlock *et al.*¹³), Fairhurst *et al.*¹⁴). The elastic moduli of alloy and porcelain are chosen 80 and 70 GPa, respectively (Nielsen¹⁵), Kase *et al.*¹⁶). Thickness of the porcelain and the alloy are 2.5 and 0.5 mm.

Figures 10 and 11 show the transient stresses at the surface and at the interface of the specimen A and B. Line A represents the transient stress at the surface of the porcelain-only specimen and lines B and C show the stresses at the surface and at the interface of the PFM systems. The specimen with the alloy has higher surface tensile stresses than the specimen without the alloy, but the interfacial stress is not so high. In this case, the surface cracks may

Fig. 10 Transient stress profile for porcelain A and porcelain A with the alloy heat-soaked at 100°C and quenched in 0°C ice water. $h_p = 2.5 \text{ mm}$; $h_m = 0.5 \text{ mm}$; and $\alpha_p = \alpha_m = 14 \times 10^{-6} / ^\circ\text{C}$.

Fig. 11 Transient stress profile for porcelain B and porcelain B with the alloy heat-soaked at 100°C and quenched in 0°C ice water. $h_p = 1.25$ mm; $h_m = 0.25$ mm; and $\alpha_p = \alpha_m = 14 \times 10^{-6}/^\circ\text{C}$.

then be more easily initiated than for the porcelain-only specimen. The specimen with greater thickness has higher transient stress at the surface.

Figure 12 shows the stress distribution at 0.01, 0.1 and 0.5 s after quenching for the specimen A. Under steady state conditions the internal stress is zero because temperature in the specimen is kept equal and the materials have the same coefficient of thermal expansion. For the quenching case, tensile stress is developed near both sides of the surface and

Fig. 12 Total transient stress profiles 0.01, 0.5 and 1.0 s after quenching Body-Opaque-Alloy composite beam in 0°C ice water from heat soaked temperature of 100°C. $E_p = 70$ GPa; $E_m = 80$ GPa; $h_p = 2.5$ mm; $h_m = 0.5$ mm; and $\alpha_p = \alpha_m = 14 \times 10^{-6}/^\circ\text{C}$.

compressive stress occurs at the center of the specimen. At the porcelain-alloy interface, tensile stress is not as high as the stress at the surface and not changed extremely with time.

(B) Alloy and porcelain have different coefficients of thermal expansion. In this case, the composite beam has residual stress under steady state conditions. The time dependent internal stress during quenching is represented as the sum of this residual stress and the transient stress. The residual stress is naturally changed due to temperature changes. The internal stress of the porcelain is closely related to the glass transition temperature. These physical properties are essential for calculating the residual stress in the porcelain. The coefficient of thermal expansion and elastic modulus are dependent on temperature. It is desirable to compute the residual stress in PFM under steady state conditions with these factors. However, in this simulation the elastic moduli and the coefficients of thermal

Fig. 13 Total transient stress profiles 0.01, 0.5 and 1.0 s after quenching Body-Opaque-Alloy composite beam in 0°C ice water from heat-soaked temperature of 100°C. $E = 70 \text{ GPa}$; $E_m = 80 \text{ GPa}$; $h_p = 2.5 \text{ mm}$; $h_m = 0.5 \text{ mm}$; $\alpha_p = 14 \times 10^{-6}/^\circ\text{C}$; and $\alpha_m = 15 \times 10^{-6}/^\circ\text{C}$.

expansion are presumed to be constant values below the porcelain glass transition temperature. The transition temperature is taken at 570°C.

Figure 13 show the results for the case of $\alpha_m > \alpha_p$. A solid line shows the stress at 0.01 sec after quenching, a broken line is for 0.5 s, and a fine line is the normal stress distribution across the beam under the infinite cooling time. The tensile stress at the interface is higher than for the case of $\alpha_m = \alpha_p$. The surface cracks will be then initiated with about a 10°C lower quenching temperature.

Figure 14 shows the case of $\alpha_m < \alpha_p$. In this case, surface tensile stress is low. The surface of the porcelain has the compressive stress under infinite cooling time. But, the porcelain-alloy interface has high tensile stress. In general, the fracture strength of the interface should be lower than the surface. The surface of porcelain is smooth and has not many defects. However, the interface has many small gas bubbles and defects with metal

Fig. 14 Total transient stress profiles 0.01, 0.5 and 1.0 sec. after quenching Body-Opaque-Alloy composite beam in 0°C ice water from heat-soaked temperature of 100°C . $E = 70 \text{ GPa}$; $E_m = 80 \text{ GPa}$; $h_p = 2.5 \text{ mm}$; $h_m = 0.5 \text{ mm}$; $\alpha_p = 14 \times 10^{-6}/^{\circ}\text{C}$; and $\alpha_m = 12 \times 10^{-6}/^{\circ}\text{C}$.

oxides. For this reason, it is preferable to use the alloy with a lower thermal expansion than that of porcelain. The critical point of the difference $\alpha_p - \alpha_m$ is determined for the interfacial fracture. The results of simulation show the difference of $\Delta\alpha = 2 \times 10^{-6}/^{\circ}\text{C}$ is the limit to provide protection against the interfacial cracks.

Thermal shock test was done for three kinds of fused alloys. For the beam with Alloy A, the surface cracks were observed with a quenching temperature 90°C of all seven specimens. The surface cracks were observed for Alloy B, that is, one sample had from 80°C and other two samples had from 100°C . All of the specimens with Alloy C had interfacial cracks for quenching from 130°C . Figure 15 shows the interfacial cracks for the beam with Alloy C. Thermal expansion coefficients of these alloys are 13.8 , 15.4 and $12.7 \times 10^{-6}/^{\circ}\text{C}$, respectively. These results are then in general agreement with the simulation results. For

Fig. 15 Interfacial crack development in the Porcelain-Opaque-Alloy C beam quenched in 0°C ice water from heat soaked temperature of 130°C .

more detailed discussion, it may be required that the residual stress before thermal shock tests are determined from temperature depended physico-mechanical properties.

CONCLUSION

Computer calculations were used to simulate the effects of thermal shock on the stresses developed in PFM systems. A three-dimensional-finite element model was employed to first calculate time-dependent temperature distribution. These were then used in a two-dimensional analysis of stresses in a beam consisting of 50 elements. Transient stresses were calculated for porcelain, porcelain with an opaque layer, and porcelain-opaque-alloy system. Experimental tests were also conducted for correlation with the theoretical calculations. The following conclusions were reached :

1. This study shows that thermal shock test is easy to perform and is a reliable method to measure the strength of porcelain. The influence of the coefficient of thermal expansion, thickness of the beam, and quenching temperature on transient stress were determined from the simulation.

2. The experimental test of porcelain with an opaque layer shows that the transient stress in the beam which was combined with two porcelains which have close physico-mechanical properties is not changed.

3. The coefficient of thermal expansion of alloy has a major influence on thermal shock resistance of porcelain-opaque-alloy beam. When $\alpha_m > \alpha_p$, higher stress occurs on the surface of porcelain, and the interface stress is lower than for $\alpha_m = \alpha_p$. When $\alpha_m < \alpha_p$, the surface stress is lower and interface stress is higher than $\alpha_m = \alpha_p$. When $\alpha_p - \alpha_m > 2 \times 10^{-6} \text{C}^{-1}$, cracks are more easily initiated in the porcelain surface. Theory and experiment are in general agreement.

For a more detailed consideration of this problem, it is highly desirable to use temperature dependent physico-mechanical properties.

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本号掲載論文の和文抄録

熱衝撃試験による金属焼付陶材中の残留応力の評価

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金属焼付陶材の熱衝撃試験をコンピュータシミュレーションした。すなわち、初めに3次元熱伝導有限要素法を利用して陶材中の温度分布を求め、その結果をもとに梁の理論から試験片内の内部応力を計算した。シミュレーションは陶材、陶材-オベーク陶材、陶材-オベーク陶材-合金の3種の試料について試みた。そして、板厚、熱膨張係数、焼き入れ温度と Transient stress との関係について調べ、以下の結論を得た。

α_m (金属) $>$ α_p (陶材) の場合、 $\alpha_m = \alpha_p$ の場合よりも陶材表面の応力は高く、界面での応力は低くなる。また、 $\alpha_m < \alpha_p$ の場合には陶材表面の応力は低くなるが、界面での応力は高くなる。金属焼付陶材の界面破壊を防ぐには $\alpha_p - \alpha_m$ が $2 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ より小さくなくてはならない。

熱膨張係数の異なる3種の合金に同じ陶材を焼き付け、熱衝撃試験を試みた結果はシミュレーションの結果と良く一致した。

接着性レジンによるアマルガムの窩洞への結合に関する研究

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アマルガム充填の際の辺縁封鎖性の向上や破折防止のための基礎的研究として、接着性レジンを窩壁に塗布してから、アマルガムを充填する術式について検討した。接着材としては酸無水物系モノマー、4-META を含む MMA-TBB 系レジン、および、リン酸エステル系モノマーを含むパナビア EX を使用した。これらのレジンを窩壁に塗布してからアマルガムを充填し、辺縁の漏洩防

止効果と接着強さを測定した。その結果、接着性レジンを塗布してからアマルガムを充填すると辺縁封鎖に対し有効であることが明らかになった。また、エナメル質を酸処理した後、4-META-MMA-TBB 系レジンを塗布し、アマルガムを充填した場合、水中浸漬1カ月後も高い接着強さが得られた。

各種コンポジットレジンとエナメル質の擦り合わせ摩耗に関する研究

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各種コンポジットレジン (P-10, Clearfil Posterior, Clearfil F II, Palfique, Microjar, Microrest AP, Silar, P-30) と牛歯エナメル質を連続往復水平運動型摩耗試験機を応用し、100,000 回の擦り合わせ摩耗試験を行うとともに、各種コンポジットレジンの諸性質 (フィラー含有量、ヌーブ硬度、圧縮強度、弾性率) を調べ、擦り合

わせ摩耗との関連性を検索した。

各種レジンの摩耗量は MFR 群に少なく、他のレジン間とに有意差が認められた ($P < 0.01$)。Microrest AP と Silar はほとんど摩耗されなかった。

レジンのフィラー含有量が多く、硬度が高く、さらに弾性率の高いレジン程、その摩耗量が多くなった。