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Effect of transient stress on acoustic emission behaviour during firing of dental porcelain

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Acoustic emission activity was measured in the glass transition range of dental porcelain during firing. Transient and residual stresses in porcelain during cooling from a temperature higher than porcelain sag point and during re-heating of the tempered porcelain were calculated by computer simulation using a viscoelastic stress analysis. The detected acoustic emission event was discussed with the relative rules of the simulated transient stresses. High acoustic emission activity was detected at the temperature where the internal stress faded away for heating and build up for cooling. The low-level acoustic emission pulses were only detected in the following conditions: (1) in the temperature range where porcelain behaved like an elastic solid; (2) at temperatures higher than the deformation point of porcelain; (3) with a re-heating process of the porcelain without tempered stress. From these results, it was concluded that elastic energy is released related to transient stress in porcelain during viscoelastic deformation and can be detected by the acoustic emission method. The acoustic emission method is considered to be helpful in non-destructive testing in order to understand transient stress due to viscoelastic deformation of glassy materials in heat treatment.

1. Introduction

Currently, the use of porcelain-fused-to-metal (PFM) restorations represents a significant component of dental restorative practice, because PFM full-coverage restorations serve both the functional and cosmetic requirements of patients with carious, disfigured, or missing teeth. It consists of a cast metal coping on to which a porcelain veneer is fired. In this technique, opaque porcelain is fused against the casting to mask the metal. Because the opaque porcelain is reflective, it must be covered with at least 1 mm of body porcelain if reasonable aesthetics are to be achieved. However, the dental practitioner has been faced with the clinical problem of occasional failure of the porcelain–alloy system. Failures can be attributed to a variety of causes: clinical, processing and material deficiencies. Residual stress created by the manufacturing process in the entire construction of the appliance is thought to be one of the main factors in fracture. It is therefore important to elucidate the magnitude and tolerable limits of such stresses remaining in the porcelain and PFM restorations after firing; i.e. a deeper understanding of the mechanisms to build up residual stress in porcelain and porcelain–alloy system is needed in order to assess their strengthening method. However, the development of residual stress in porcelain has not been ascertained experimentally.

Acoustic emission (AE) is the transient elastic wave generated by release of elastic energy accompanying crack initiation, growth, plastic deformation, phase transformation, etc. We can detect AE activities related to transient stress development/dissipation when cooling/heating porcelain if elastic energy is released during the viscoelastic deformation of glassy materials such as dental porcelain. These experimental measurements may contribute to the understanding of the viscoelastic behaviour of glassy materials during heat treatment.

In this study, AE activity of dental porcelain was measured in the glass transition range during heating and cooling. Consequently, computer simulation was conducted to clarify the transient and residual stresses built up in the porcelain. Computed results were also compared with the measured AE activities.

2. Materials and methods

2.1. Preparation of the specimen

A dental porcelain in commercial use (Ceramco G body porcelain) was used. The cylindrical shape of the porcelain compact, 10 mm diameter and 2 mm thick, was formed by compression in a steel die. The porcelain compact was fired at 970 °C, according to the manufacturer's recommended procedure, and cooled in the furnace. Two kinds of specimen which had been cooled at different cooling rates were used for AE measurements: after the specimen was re-heated at 800 °C and held there for 5 min, (1) it was cooled sufficiently slowly in the furnace, or (2) it was removed from the furnace and cooled in ambient air at room

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temperature. The AE activity from the specimen was measured during re-heating at 0.3 °C s^{-1} to 700 °C and successively cooled at 0.13 °C s^{-1} in a furnace.

2.2. AE measurements

The method of suspending the specimen in order to obtaining AE signals in a furnace is shown in Fig. 1. AE signals were picked up at the top of a stainless steel bar (waveguide) and propagated through it to AE sensors. The instrument arrangement for AE signal measurement was similar to that given in detail by Sakamaki *et al.* [1] and as shown in Fig. 2. A two-channel acoustic emission measuring system was used. The acoustic emission from the heating/cooling specimen was transformed into an electrical signal by AE transducers with 200 kHz resonant frequency, and the signal was amplified by a total gain of 80 dB and was passed through a band pass filter with a range of 0.1–1 MHz. The AE data which consist of the AE envelope voltage, the AE event count, and the peak amplitude above the 20 μV threshold level, associated with the pre-amplifier input, were recorded on a pen recorder and digital printer, respectively. Only AE events which were detected by both of the transducers within a time lag of 8 μs were sent into the AE data memory to detect the AE in a temperature step of 10 °C.

2.3. Simulation

Transient and residual stresses in porcelain during cooling from temperatures higher than the porcelain sag point were simulated. The simulation procedure has been discussed in more detail in previous papers by Asaoka and Tesk [2, 3]. The following temperature-dependent factors were incorporated in the simulation: the elastic modulus (measured data by Käse *et al.* [4]), shear viscosity (Asaoka *et al.* [5]), and coefficient of thermal expansion (Asaoka *et al.* [5]). The cooling rate dependencies of the glass transition temperature, T_g, and the temperature distribution during cooling, were also included.

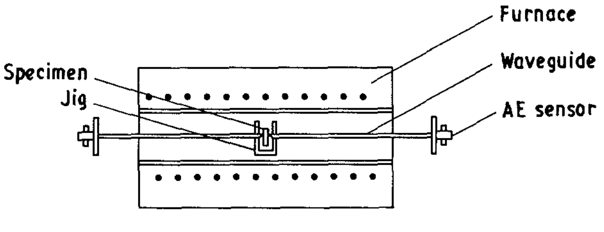

Figure 1 Method of suspending the specimen for obtaining AE signals in a furnace.

The porcelain was removed from the furnace at the heat-soak temperature to the ambient air and cooled according to an exponential curve as provided by Asaoka *et al.* [6]

$$T = T_a + A\exp(-mt) \tag{1}$$

where T and T_a are the temperatures of the porcelain and ambient air, respectively, A is a constant related to the heat-soak temperature, t is the time elapsed from porcelain removal, and m can be calculated from the heat capacity, the coefficient of heat convection and the ratio of surface to volume of the specimen.

When the surface temperature cools according to Equation 1 for $t > 0$, the temperature distribution in a slab which has zero initial temperature has an exact solution as follows [7]:

$$T = A\exp(-mt)\frac{\cos x\,(m/\kappa)^{1/2}}{\cos l\,(m/\kappa)^{1/2}} + \frac{16mAl^2}{\pi} \times \sum_{n=0}^{\infty}\frac{(-1)^n \exp[-\kappa(2n+1)^2\pi^2 t/4l^2]}{(2n+1)\,[4ml^2 - \kappa\pi^2(2n+1)^2]} \times \cos\frac{(2n+1)\pi x}{2l} \tag{2}$$

where κ is thermal diffusivity, l is the half thickness of the slab, and x is the distance from the midplane of the slab, $-l < x < l$. The thermal diffusivity of the porcelain was assumed to be 0.66 $mm^2\,s^{-1}$ as reported by Piddock [8].

For modelling the simulation, a composite beam was chosen having N thin, equal layers of porcelain. The temperatures which developed in the beam during cooling were calculated using Equation 2. Each layer has a unique elastic modulus, thermal expansion coefficient, and viscosity, as determined by its temperature. T_g in each layer was determined from its cooling rate using the equation proposed by Moynihan *et al.* [9] and T_g from a reference cooling rate. Creep relaxation of the porcelain in the glass transition temperature, was assumed to follow a Voigt model for cooling (see for example, McClintock and Argon [10]). Transient stresses were calculated by an incremental time-step method and with the temperature

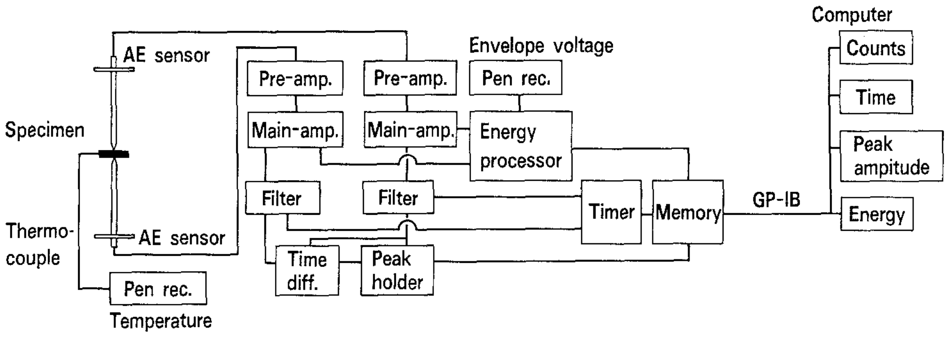

Figure 2 Instrument arrangement for acoustic emission measurements.

TABLE I Thermo-mechanical properties of the porcelain used in the simulation

Elastic modulus:		70.2 GPa at 30 °C
		66.7 GPa at 475 °C
Thermal expansion coefficient		
For cooling:		
	Liquid	$\alpha = 35 \times 10^{-6}\,°C^{-1}$
	$T > T_g$	$\alpha = [\alpha' + 0.07\,(T - T_g)] \times 10^{-6}\,°C^{-1}$
where, α' is the coefficient at T_g onset		
	$T < T_g$	$\alpha = (8.5 + 0.01\,T) \times 10^{-6}\,°C^{-1}$
For heating:		
	$T < T_g$	$\alpha = (8.9 + 0.02\,T) \times 10^{-6}\,°C^{-1}$
	$T > T_g$	$\alpha = [\alpha' + 0.30\,(T - T_g)] \times 10^{-6}\,°C^{-1}$
where, α' is the coefficient at T_g onset		
	Liquid	$\alpha = 35 \times 10^{-6}\,°C^{-1}$
Transition temperature		354 °C at 0.08 °C s^{-1} for cooling
		460 °C at 0.08 °C s^{-1} for heating
Viscosity		$\eta = 6.0 \times 10^{-4} \exp(30\,800/T)$ Pa s

from the heat-soak temperature to room temperature divided into 1 °C intervals. Internal stresses were calculated by equilibrium of internal moments and by the strain continuity required at the interfaces, with external forces set equal to zero.

The thermo-physical properties of the porcelain used in the simulation are shown in Table I. The transient stresses during re-heating at a constant rate of q °C s^{-1} of the tempered porcelain were calculated in order to use the following equation for the temperature distribution and the Maxwell model for creep deformation, as reported by Asaoka and Tesk [11]. Here, the temperature distribution in a slab which has zero initial temperature can be represented as follows, when the surface is kept at temperature qt for $t > 0$ [7]

$$T = qt + \frac{q(x^2 - l^2)}{2\kappa} + \frac{16ql^2}{\kappa\pi^3} \times \sum_{n=0}^{\infty} \frac{(-1)^n}{(2n+1)^3} \exp[-\kappa(2n+1)^2\pi^2 t/4l^2] \times \cos\frac{(2n+1)\pi x}{2l} \quad (3)$$

3. Results and discussion

3.1. AE activity of porcelain

Figs 3 and 4 show AE activities during heating to 700 °C and successive cooling of the porcelains which had been cooled in the furnace and in ambient air, respectively. Here, the AE events were counted at 10 °C intervals. The AE activity was low for the heating process of the porcelain with free-residual stress (slowly cooled) as shown in Fig. 3a. For the tempered porcelain, the AE activity increased from about 400 °C and reached a maximum number at 540 °C. At temperatures higher than 570 °C, no AE events were detected. The AE events for the cooling process were detected in the temperature range 650–350 °C. The number of AE events were dissimilar between Figs 3b and 4b because the total number AE events were different. However, these results are qualitatively considered to show a similar tendency, and the data had improved reproducibility with regard to temperature.

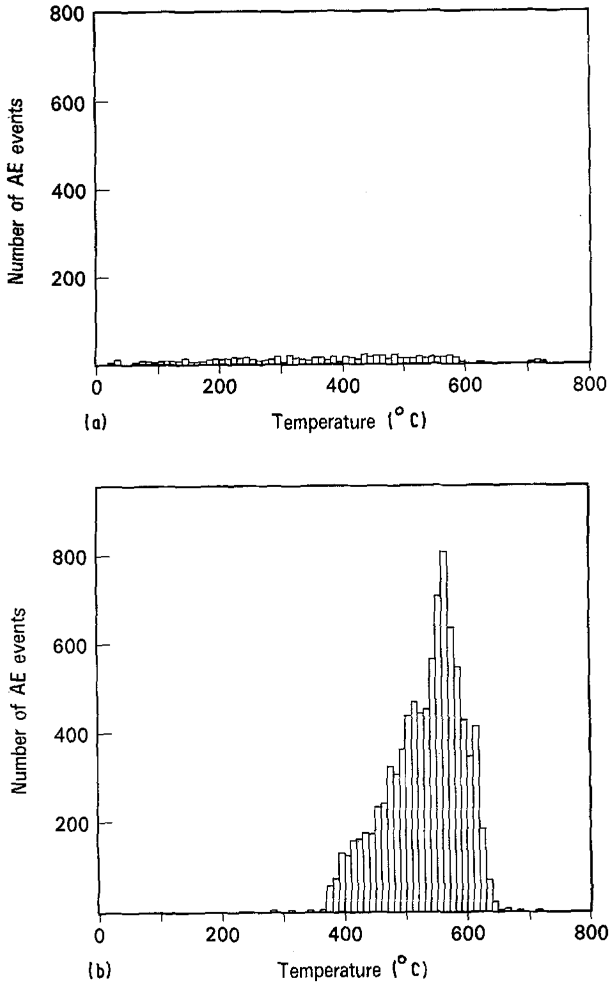

Figure 3 (a) The AE events during re-heating of the specimen which had been cooled in a furnace. The heating rate was 0.3 °C s^{-1}. The number of AE events was counted for each 10 °C interval. The total number of events was 571 pulses. (b) The AE events during cooling of the porcelain which had been re-heated to 700 °C. The cooling rate was 0.13 °C s^{-1}. The total number of events was 9085 pulses.

The temperature-dependence of the thermal expansion coefficients which were calculated at 20 °C intervals, were measured. The change in the actual coefficient of expansion is superimposed on the figures for AE activity, as shown in Fig. 4a. The onset of the glass transition temperature, T_g, of the porcelain was measured to be about 460° C. The strain point and annealing point were determined to be 480 and 550° C respectively from the viscosity of the porcelain which was measured by Asaoka *et al.* [5]. The viscous flow at temperatures higher than 520 °C was an effective factor in determining the total expansion, because the porcelain had a low viscosity at this temperature. The AE activity increased from the T_g onset to the annealing point. At higher temperatures than the deformation point, T_d, of porcelain, viscous flow easily

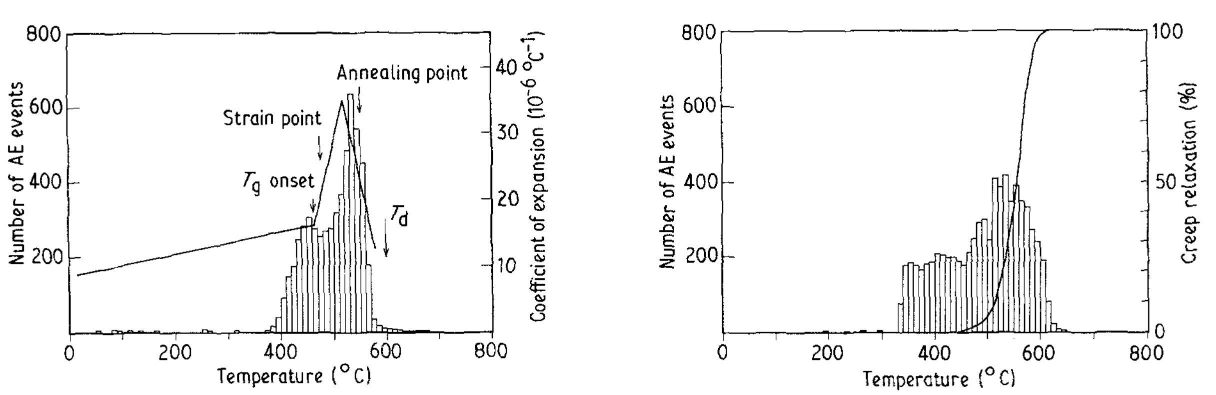

Figure 4 (a) AE events during re-heating of the tempered porcelain which had been cooled in ambient air at room temperature. The heating rate was 0.3 °C s^{-1}. The total number of events was 5551 pulses. (———) Actual coefficient of thermal expansion of the porcelain. Expansion was measured at the constant heating rate of 0.083 °C s^{-1}. Strain point and annealing point were calculated from the measured viscosity of the porcelain. T_g and T_d are defined as the point of the inflection of the slope of coefficient of expansion, and as the temperature where the expansion of the specimen due to thermal expansion is balanced by the negative viscous deformation due to the applied stress, respectively. (b) AE events during cooling of the tempered porcelain which had been re-heated to 700 °C. The cooling rate was 0.13 °C s^{-1}. The total number of events was 7050 pulses. (———) Creep relaxation (%) occurring within one temperature step of 1 °C at any temperature, depending on the cooling rate for the porcelain.

occurred by a low driving force for shear deformation, because the value of viscosity was low. The AE activity which was generated at higher temperatures than T_d was low enough in envelope voltage to be detectable. For residual stress-free porcelain (slowly cooled), the AE activity was low because the driving force (internal stress) to cause viscous flow of the porcelain was sufficiently low in the glass transition range.

The creep relaxation, ε_c, of the porcelain during cooling through the glass transition range, can be calculated as follows as reported in previous papers [3, 9]

$$\varepsilon_c/\varepsilon_0 = 1 - \exp(-Gt/\eta) \qquad (4)$$

where ε_0 is the strain of thermal contraction in 1 °C decrements of the surface temperature of porcelain, G and η are the shear modulus and viscosity of the porcelain, respectively, and t is the time elapsed for 1 °C decrements of temperature. The creep relaxation curve calculated from Equation 4 is superimposed on AE events in Fig. 4b. Viscous flow at higher temperatures than 600 °C helps the immediate relaxation of the strain mismatch which was generated by the temperature distribution in the porcelain. On the contrary, below 350 °C, the porcelain behaved as an elastic solid because of its high viscosity, and it had no

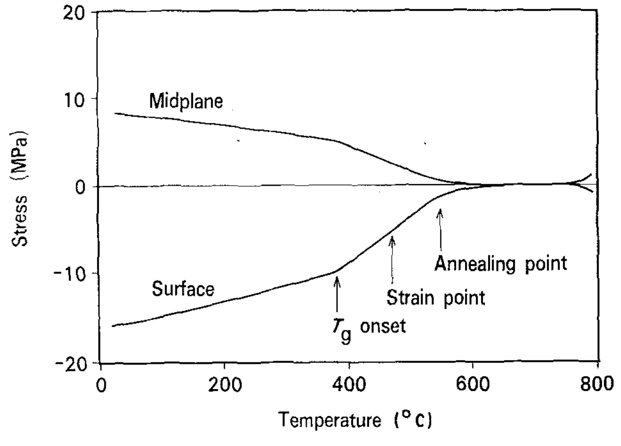

Figure 5 Simulated result of transient stresses near the surface and at the midplane of a porcelain slab 2 mm thick. The heat-soak temperature was 800 °C, and the specimen was cooled in ambient air at room temperature.

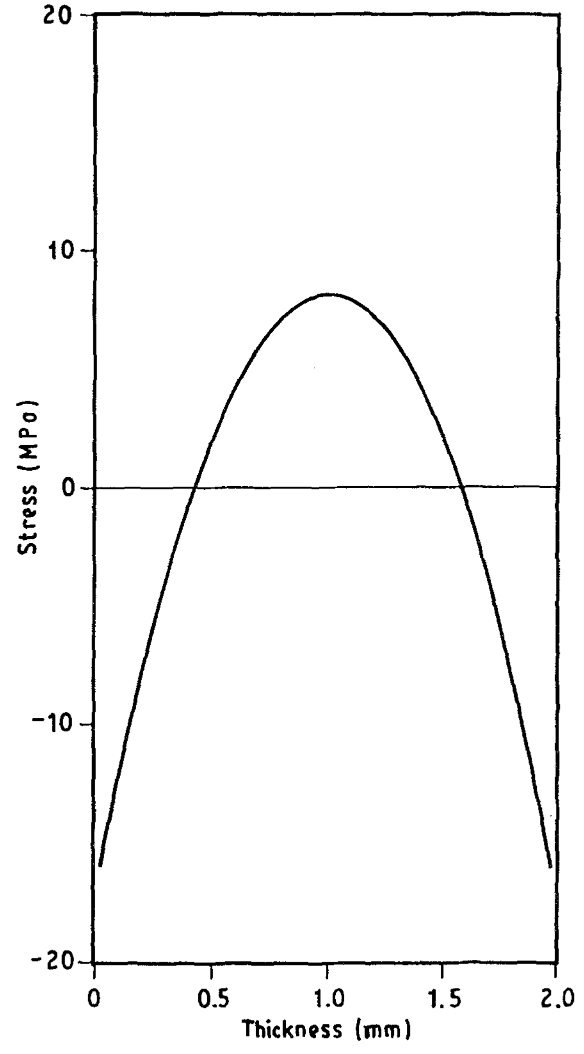

Figure 6 Residual stress distribution at room temperature in the porcelain slab after cooling.

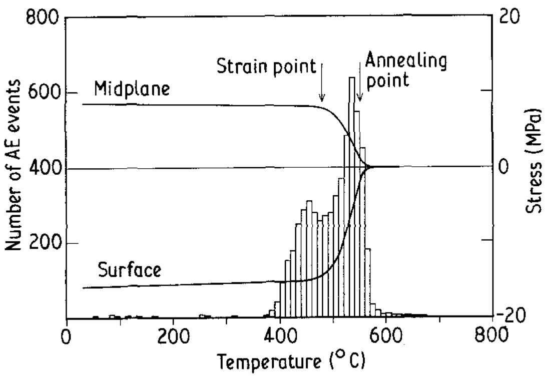

Figure 7 Simulated transient stresses for re-heating of the tempered porcelain slab with the residual stresses as shown in Fig. 6, superimposed on the AE events during re-heating of the tempered porcelain as shown in Fig. 4a. Transient stresses are shown for near the surface and the midplane.

stress relaxation. The AE activity was detected, related to viscoelastic behaviour in the glass transition range.

3.2. Transient stress in porcelain

When the porcelain samples, 10 mm diameter and 2 mm thick, were removed from the furnace held at 800 °C, and cooled in ambient air at room temperature, the constants A and m in Equation 1 were experimentally determined to be 800 °C and 0.0266 s^{-1}, respectively, from data measured by Asaoka *et al.* [6]. Residual stress in a tempered porcelain slab, 2 mm thick, was estimated by computer simulation. Fig. 5 shows the transient stresses in the surface and the midplane of the slab. Residual stress distribution at room temperature is parabolic, as shown in Fig. 6. The surface of the tempered porcelain had compressive residual stress. Fig. 7 shows the transient stresses during re-heating at the constant rate of 0.3 °C s^{-1} of tempered porcelain, superimposed on the AE activity as shown in Fig. 4a. The temperature where range of high AE events was in good agreement with the stress relaxation generated by viscous flow of porcelain.

5. Conclusions

AE measurements and computer simulations of dental porcelain during heat treatments have been carried out to clarify the relationships between the detected AE activities and the viscoelastic behaviour of the glassy materials. High AE activity was observed in temperature ranges where the internal stress in the porcelain built up and faded away. From measurements on the re-heated porcelain which had been tempered, the AE activity was found to increase from T_g onset to the annealing point. At higher temperatures than the deformation point, no AE events were detected. On the contrary, the AE activity of the cooling process became highly associated with the stress relaxation of the porcelain. The experimental results of AE activities related to viscoelastic deformation were confirmed by simulated transient stresses in heating/cooling porcelain.

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