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Elastic constants of two dental porcelains

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The development of stress that affects the bonding in porcelain-fused-to-metal (PFM) systems can be influenced by the temperature dependence of the elastic constants of both systems. Instead of using the normal, static procedure, e.g. determining the slope of a stress–strain curve, and measuring the lateral and vertical strains, in this study the sonic resonance technique was used to determine the elastic moduli for two dental body-porcelains. The sonic resonance technique involves the determination of both the flexural as well as the torsional resonance frequencies. From these values both Young's, *Y*, and shear moduli, *G*, are determined. Since two elastic constants are sufficient to describe completely the elastic response of isotropic materials, it was also possible to compute, by using *Y* and *G*, the bulk modulus, *B*, and the Poisson's ratio. Resonant frequency measurements taken at elevated temperatures resulted in correspondingly lower values for the elastic constants. Young's and shear moduli for two dental porcelains obtained in the range from 20°C (293 K) to 500°C (773 K) are presented in this study. These data may in the future be used for refined stress calculations in PFM systems.

1. Introduction

Since the introduction of porcelain-fused-to-metal (PFM) systems almost 20 years ago for dental prosthetic devices, there has been a continued attempt to improve the application of this combination of two entirely different materials.

Although metal–ceramic systems are in the meantime an approved method for reconstructive dentistry, there is still a certain risk of cracking or spalling of the veneered porcelain. These are thought to be mainly initiated by residual stress created by the manufacturing process in the entire construction of the appliance. The magnitude and tolerable limits of such stress remains a matter of uncertainty, especially for new PFM systems which appear with regularity. Because of the complexity of PFM-systems, residual stresses are attributed to a number of parameters. Beside other factors (see Table I), the development of the resultant stresses in systems considered can be influenced by the temperature dependence of the elastic constants of both materials. With regard to a refinement in evaluation of these crucial stresses and consequently to develop more stress-free PFM-systems in the future, it appears, it is quite necessary to determine exact behaviour of the elastic properties of the applied alloys and ceramics as well. Because stress initiation in glass ceramics like dental porcelains can only occur near and/or below the glass transition temperature of the material ($T_g \approx 570°$ C; 843 K), it is the purpose of this study to present data for the elastic moduli of two representative dental body-porcelains (A, B)‡ in the range from room temperature to 500° C (773 K).

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B = CERAMCO, Lot 2437, Ceramco, Inc, 20 Lake Drive, East Windsor, New Jersey 08520, USA.

TABLE I Factors affecting residual stresses in dental PFM systems due to manufacturing process (compiled by Whitlock *et al.* [2])

(a)	Thermal expansion
(b)	T_g, the glass transition temperature
(c)	Trapped residual stress (strain)
(d)	Temperature dependence of elastic modulus
(e)	Crystallinity
(f)	Thermal conductivity
(g)	Heat capacity
(h)	Number of layered components
(i)	Relative layer thicknesses
(j)	Ability to relieve stress

Casciani *et al.* [1] performed structural studies, using Raman spectroscopy, on very similar materials (opaque dental procelains) supplied by the same manufacturers. Although both of the opaque porcelains were basically glass ceramics, significant differences in crystalline phases and glassy nature were revealed. Providing the two body-porcelains applied in this investigation show the same differences in microstructure one may infer that the structural situation of either material is also reflected in different elastic moduli, hence different mechanical properties.

2. Experimental procedure

2.1. Specimens

Two commercially available dental body-porcelains (A, B) were chosen for this investigation. Both materials, each of them a glass ceramic, were delivered as powders. Specimens were prepared and fired as recommended by the manufactures. Subsequently the specimens were cut and ground to the desired dimensions of about 50 mm × 9.3 mm × 1.3 mm. Finally, in order to obtain a high accuracy in results, the surfaces were ground and polished. The grinding and polishing must be performed with great accuracy to obtain a shape as close as possible to a perfect rectangular bar. Extremely good parallelism of the opposite specimen surfaces is of high importance for the application of the sonic resonance technique, which is described in the following paragraph. The precision of the most important dimensions of the specimens, i.e. in thickness and width, was ± 0.004 mm.

2.2. Method and calculations

To obtain some characterization of the mechanical behaviour of a material, the Young's modulus is often calculated from a stress–strain curve, measuring the change in stress with the change in length over the elastic region. This method, however, is often not very applicable to brittle materials such as glasses and ceramics, because their range of elasticity is usually small and immediately followed by a sudden brittle failure when leaving that range.

Moreover, to determine the magnitude of the dynamic elastic response, it is necessary to separate this particular behaviour of the material from other time-dependent responses such as retarded elasticity and viscous creep. These become especially important at higher temperatures when one evaluates glasses and ceramics. Another side aspect of this study was to use a method that allows not only the determination of the elastic constants, but also some structural characteristics such as microcracking. This correlates strongly with the elastic moduli. That kind of information is difficult to obtain from quasi-static measurement techniques. Therefore, instead of the elastic procedure using the stress–strain curve the sonic resonance method was used in this investigation. This technique was originated by Forster [3] in 1937, and in the meantime researchers [4–7] have successfully applied this technique on a variety of different materials.

The experimental technique has been discussed in detail by Spinner and Tefft [8] and, more recently by Marlowe [9]. In addition, a comprehensive description of the necessary equipment has been provided by several authors [8–11] and will not be repeated here. A schematic diagram of the experimental arrangement employed in this study is shown in Fig. 1.

In brief, a wide range variable frequency oscillator was used to generate a sinusoidal electric signal which was converted to a mechanical vibration of the same frequency by the driver, a high-output piezoelectric phonograph cartridge.§ For room temperature measurements the mechanical movement was transmitted to a suspended specimen through cotton fibres. Platinum wire of 76 μm (0.003 in.) diameter was used to hold the specimen for elevated temperature determinations. In order to make those wires more flexible, they were annealed by pulling them slowly through a torch. A second suspension thread (or wire) at the other end of the specimen then conveyed the resultant excitation to another phonograph cartridge designated as pickup. The pickup needle re-converted

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§ ASTATIC: 62-1(M); Conneaut, Ohio 44030, USA.

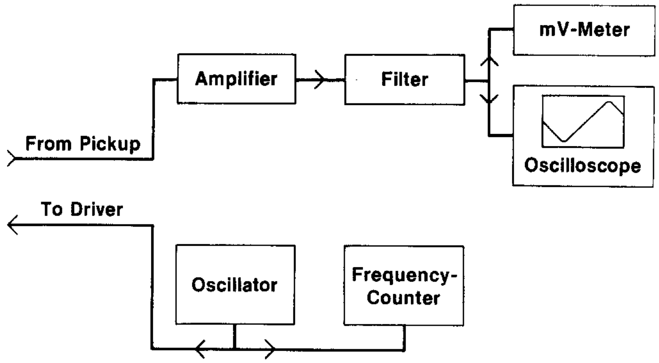

Figure 1 Instruments arrangement for dynamic resonance measurements.

the mechanical vibration to an electronic signal which was amplified, filtered, and passed into a voltmeter and on to the vertical plates of an oscilloscope. The electrical signal from the oscillator was also transmitted to a frequency counter. The method of suspending specimens for obtaining torsional as well as flexural vibrations is illustrated in detail in Fig. 2. It is important that the fibres are attached to opposite sides of the prismatic bar.

Elasticity measurements were made by varying the oscillator frequency until the suspended sample vibrated in resonance. Under such a condition the amplitude of vibration reached a maximum which was subsequently detected by both the oscilloscope and the voltmeter. The corresponding resonant frequency was taken as the average of three readings.

Before starting the measurements on the prepared dental ceramic specimens the entire experimental system was calibrated by means of a polycrystalline alumina resonance bar (NBS, SRM No. 718). The flexural and torsional resonant frequencies were then determined during heating and cooling over a temperature range from room temperature (20° C; 293 K) to 500° C (773 K). Data were taken at temperature intervals of approximately 50° C. After each temperature increase upon heating (or decrease during cooling) the equipment, including the specimen, was allowed to approach thermal equilibrium before the measurements were performed. A typical experimental run lasted about 24 h.

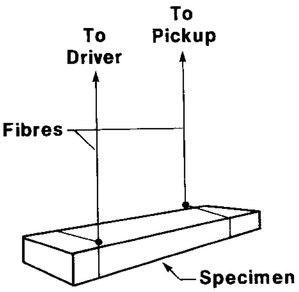

Figure 2 Method of suspending specimens for obtaining flexural as well as torsional resonance frequencies; from Spinner and Tefft [8].

As stated previously, the elastic moduli can be calculated from the resonant frequencies, the mass and the dimensions of a specimen. The equations used were those for prisms of rectangular cross-section proposed by Pickett [12] and later modified by Hasselman [13]. In order to obtain precise moduli data at room and elevated temperatures, two corrections were necessary. The first correction, concerning the resonant frequencies, was made for the effect of the platinum wires, used in the high temperature measurements. The equation used in this correction was:

$$\bar{f}_{pt} = (f_{c0}/f_{p0})f_{pt}, \qquad (1)$$

where f_{c0} is the resonant frequency at room temperature with the specimen suspended by cotton strings; f_{p0} is the resonant frequency at room temperature with the specimen suspended by platinum wires; f_{p_t} is the measured resonant frequency at temperature t, with platinum as support strings; and $\bar{f}_{p_t}$ is the corrected value at temperature t, for a specimen suspended with platinum wires.

The second adjustment, to correct moduli for the effects of dimensional changes due to thermal expansion at elevated temperatures was developed by Ault and Ueltz [14]:

$$Et = E_0(ft/f_0)^2/(1 + \alpha\Delta t), \qquad (2)$$

where the elastic moduli of any temperature, E_t, are expressed in terms of the value at room temperature, E_0, and the corresponding resonant frequencies, f_t and f_0. The factor $(1 + \alpha\Delta t)$ contains the coefficient of linear thermal expansion (α) and the difference between the temperature of

TABLE II Elastic constants of porcelains A and B at room temperature (20° C; 293 K)

Material	Young's modulus (GPa)	Shear modulus (GPa)	Bulk modulus (GPa)	Poisson's ratio
Porcelain A* (density 2.49 g cm^{-3})				
At start of run 1	68.0	28.5	37.1	0.19
At start of run 2	69.4	29.1	37.8	0.19
Porcelain B† (density 2.43 g cm^{-3})				
At start of run 1	69.2	29.0	37.4	0.19
At start of run 2	69.9	29.4	37.5	0.19

*A, VITA-porcelain.
†B, CERAMCO-porcelain.

measurement, t, and room temperature. For calculations in this study the thermal expansion data reported by Whitlock *et al.* [2] were used.

3. Results and discussion

Both of the dental porcelains investigated in this study were heated to the maximum test temperature twice. During these thermal cycles the resonant frequency measurements were conducted during heating and cooling. The results obtained from measurements at room temperature (20° C; 293 K) are shown in Table II. The experimental error involved in measuring the elastic moduli by the sonic resonance technique was studied by Marlowe and Wilder [15]. They reported the overall error for room temperature measurements should be less than 1.3 % with dimensional inaccuracy contributing the largest error. Spinner *et al.* [16] concluded that Young's modulus should be exact to four significant figures with the technique employed.

In the present study, an analysis of the possible sources of error leads to an inaccuracy of 1.5% for the determined values. The major sources of indeterminacy arise from possible uncertainties in dimensions and parallelism, density, thermal expansion and calibration.

From Table II it can be seen that there is no appreciable difference in the elastic behaviour between the material A and B before the first thermal cycle. Furthermore, the difference in the moduli data became even smaller after the first run and lies well within the experimental inaccuracy of the applied method. It should be emphasized that the presented sets of data are only valid for a given density and accordingly may vary with an increment or decrement in porosity.

The temperature dependence of both the Young's and shear modulus from room temperature to 500° C (773 K) is represented graphically in Figs. 3 to 8. In either case with increasing temperature both of the measured resonance frequencies shift to lower values. This results in correspondingly lower values for the elastic constants. Material A shows a drop in the elastic constants of 7% up to 500° C (773 K) whereas material B exhibits a decrease in its elastic response of only 5% up to the same temperature. This rate of decrease of modulus with temperature (namely a drop of about 1% in modulus with each increase in 100° C) is consistent with the observed modulus–temperature behaviour for a wide range of ceramic materials [17].

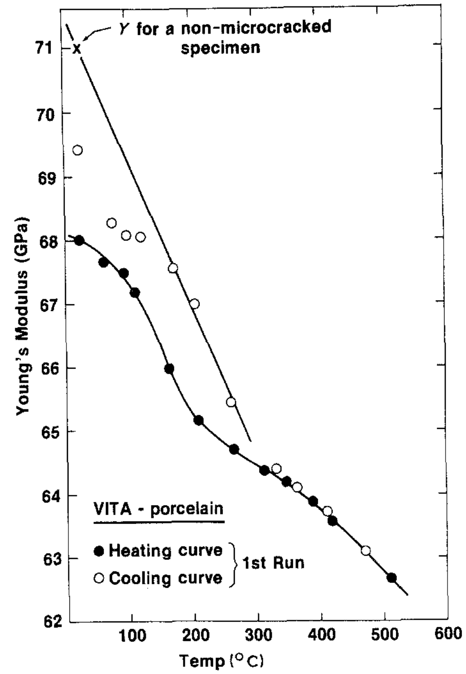

Figure 3 Young's modulus (Y)–temperature data for porcelain A; 1st run.

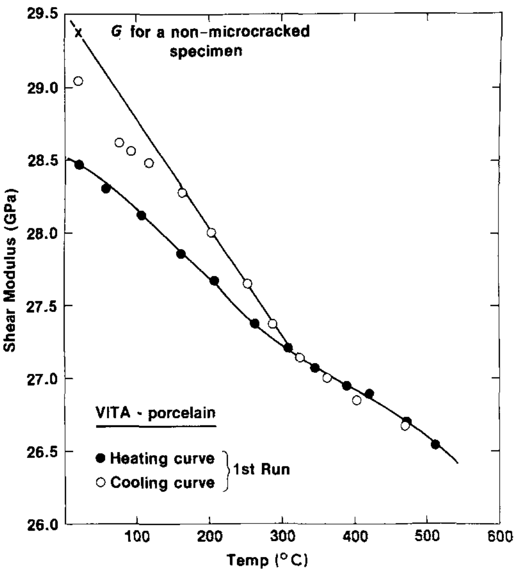

Figure 4 Shear modulus (G)–temperature data for porcelain A; 1st run.

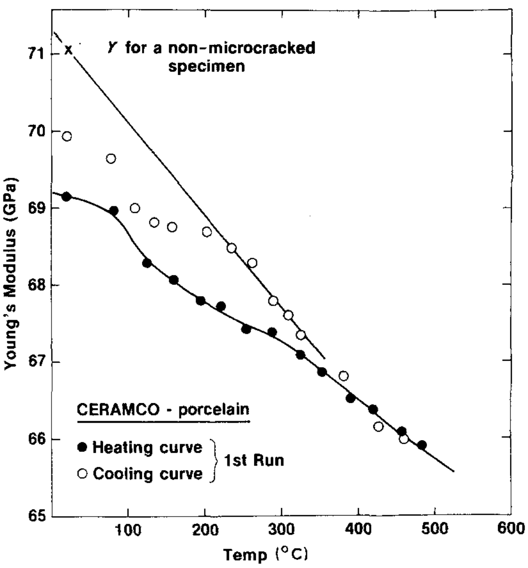

Figure 6 Young's modulus (Y)–temperature data for porcelain B; 1st run.

Besides this general behaviour, another important detail should be noted. The cooling curve, from the maximum test temperature down to room temperature, exhibits a hysteresis in the plots of elastic constants against temperature. As can be seen in Figs. 3 to 8, the departure between the heating and cooling curves occurs at about 350° C (623 K). The cooling curve then shows a range of a fairly linear increase in the elastic response with continuing cooling from near 200° C (473 K) down to room temperature, before it tends to level out. Such a difference in behaviour during the heating and cooling process, is characteristic for a microcracked material [18–23]. One possible interpretation of such a hysteresis in the elastic moduli–temperature data is that microcracks heal during heating and then some open again upon cooling below approximately 200° C (473 K).

The Young's modulus–temperature heating curves shown in Figs. 3, 5, 6 and 8, exhibit a fairly

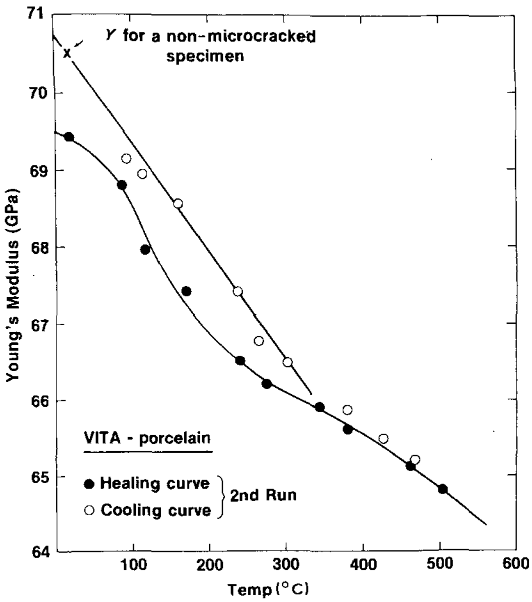

Figure 5 Young's modulus (Y)–temperature data for porcelain A; 2nd run.

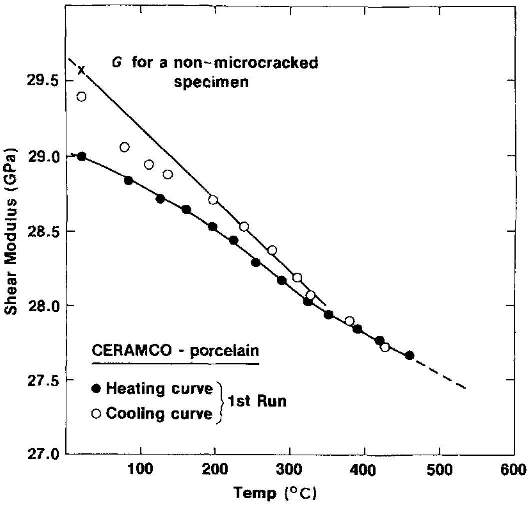

Figure 7 Shear modulus (G)–temperature data for porcelain B; 1st run.

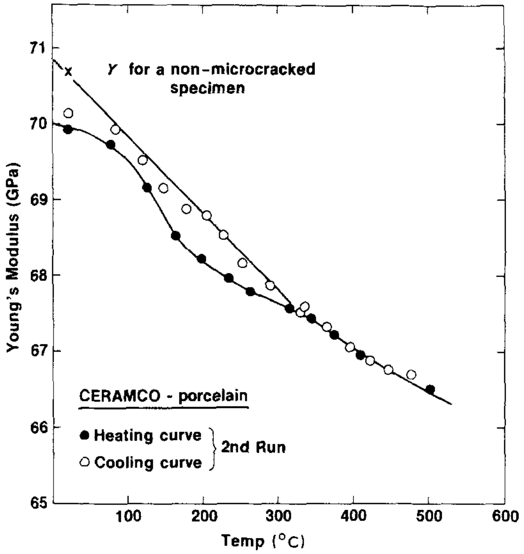

Figure 8 Young's modulus (Y)–temperature data for porcelain B; 2nd run.

sharp modulus drop within the first 200° C (473 K), followed by a range with a comparatively low slope before the rate of decrement in Young's modulus increases again from 300° C (573 K). It is very likely that this behaviour between 200° C (473 K) and 300° C (573 K) is attributed to microcrack healing. The reduction of the elastic modulus with the introduction of microcracks into a material has been discussed by several authors [24–28]. Hence, it follows that an increase in the number or size of cracks results in lower elastic moduli.

Crack formation in the materials investigated in this study is firmly established in the literature. It is well known that microcracks in ceramics can be attributed to a variety of mechanisms that produce localized internal stresses [23]. For example, phase transformations, differing thermal expansions in a multiphase body, thermal history or atmospheric effects can cause microcracking. Thus, it appears, further investigations are necessary in this case to establish the exact cause.

It should be noticed that after the first heating and cooling cycle, the elastic moduli at room temperature shift to higher values, whereas at the end of the second run they return to the same value obtained at the start of that cycle (see Table II and Figs. 3, 5, 6 and 8). This indicates definitely that during the first heating process an unknown number of microcracks heal and never open again. It is possible that the grinding procedure to which the specimens were subjected initiated some additional microcracking or microcrack growth which was subsequently removed during the first thermal cycle.

An attempt has been made to derive moduli data from the plots of elastic constants against temperature for a non-microcracked specimen. Assuming that basically all of the microcracks heal during heating, then upon cooling from the maximum test temperature down to about 200° C (473 K), the modulus–temperature behaviour would be that of a non-microcracked specimen. Hence, one should be able to extrapolate the approximately linear part of the cooling curve to room temperature. As may be seen from the corresponding Figs. 3 to 8, the values for an assumed non-microcracked body are only slightly higher (1 to 1.5%) than from the data actually obtained.

In extreme cases, the room-temperature modulus for a microcracked material may be only 10 to 20% of that observed for the corresponding non-microcracked material [29–32]. Thus, the extent of microcracking damage observed for the dental porcelains (this study) is relatively slight. (The Appendix gives a brief discussion of the quantitative relation between modulus decrement, the number density and size of microcracks.)

4. Conclusions

The resonance method was applied to determine the elastic constants of two dental body porcelains. For both materials data are presented for the temperature range 20° C (293 K) to 500° C (773 K). The maximum decrease in the elastic moduli in the temperature range mentioned before, is up to 7%. The recorded hysteresis in the elastic moduli–temperature curves indicates that both porcelains are slightly microcracked. All in all, materials A and B virtually show the same dynamic elastic behaviour. The determined elastic constants as a function of temperature, are now available for more precise stress calculations in PFM systems.

Appendix

Theories by Walsh [25], Salganik [26, 27], and Budiansky and O'Connell [28] relate the modulus decrement for a microcracked material to the number density and size of microcracks in the material. Each of the three theories mentioned above predict similar microcrack–modulus decrement behaviour, especially for a dilute concentration of microcracks [32].

For the Salganik theory [26, 27] in particular, the decrement in Young's modulus is given by:

$$Y = Y_0[1 - f(\nu_0)N\langle a\rangle^3], \qquad \text{(A1)}$$

where Y is the Young's modulus for the microcracked body, Y_0 is the Young's modulus for the non-microcracked body, N is the number density of microcracks, $\langle a\rangle$ is the mean microcrack radius, and ν_0 is the Poisson's ratio for the non-microcracked body. The function $f(\nu_0)$ is, in turn, given by:

$$f(\nu_0) = \frac{16(10-3\nu_0)(1-\nu_0^2)}{45(2-\nu_0)}.$$

Solving Equation A1 for the product $N\langle a\rangle^3$ gives

$$N\langle a\rangle^3 = \left(\frac{Y_0 - Y}{Y_0}\right)\left(\frac{1}{f(\nu_0)}\right). \qquad \text{(A2)}$$

For the dental porcelains studied here.

$$f(\nu_0) = 1.7855 \quad \text{for} \quad \nu_0 = 0.19$$

and

$$\frac{Y_0 - Y}{Y_0} \approx 0.015 \text{ (see Section 3)}.$$

For these $f(\nu_0)$ and $(Y_0 - Y)/Y_0$ values,

$$N\langle a\rangle^3 \simeq 8.401 \times 10^{-3}. \qquad \text{(A3)}$$

Using the Salganik theory¶, one easily obtains an estimate for the product $N\langle a\rangle^3$ from the measured values of Y, Y_0 and ν_0. In order to make further progress, one must obtain information about either the microcrack number density, N, or the mean microcrack radius, $\langle a\rangle$. As an aid in estimating $\langle a\rangle$, one may appeal to the experimental observation that microcracks induced by localized stresses are often one to several grain diameters in length [33–35]. However, for the dental porcelains considered in this paper, the observed grain size range is very broad (3 to 20 μm, with a small number of particles up to 100 μm), making it difficult to use such arguments to obtain a meaningful estimate of $\langle a\rangle$*. If, merely for the sake of example, we make the approximation:

$$\langle a\rangle \approx 5.0\,\mu\text{m} = 5.0 \times 10^{-4}\text{ cm},$$

then from Equation A3, the microcrack number density is:

$$N \approx 3.4 \times 10^4\text{ cm}^{-3}.$$

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¶For the theories of Walsh and of Budiansky and O'Connell, the microcracked Poisson's ratio, ν, is required, along with Y, Y_0 and ν_0. However, for the dental porcelains, ν may be calculated for the measured Young's (Y) and shear (G) modulus data.

*Microscopic observations of the microcrack size and number density also tend to be difficult because (1) the stress state of the surface of the specimen is not the same as the stress state in the interior of the specimen, so that the number and size of microcracks intercepting the surface may not be a representative sample of the bulk microcrack population, and (2) specimen preparation techniques almost invariably damage the specimen surface.

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