Creep of resin veneer materials

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Abstract – Resin veneer materials differing in chemical composition and content of filler particles were investigated with regard to the property of creep. Different processing and curing methods were compared. Two methods of characterizing creep were used. Creep rates were determined on cylindrical specimens exposed to compressive stress. Creep was also measured by indentation testing on a separate series of specimens. The lowest creep rates were obtained for the light-cured specimens, whereas the micro-filled heat-cured products exhibited the highest values. The lowest creep rates of the unfilled acrylics occurred when processing was carried out in flasks. The results of the indentation testing showed a similar pattern. A correlation coefficient (R_s) of 0.900 was obtained for the 2 methods of creep characterization. The variations seen in creep implies that they would behave differently in clinical service.

Key words: dental resins, mechanical properties, processing methods, fixed prosthodontics.

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Resin facings of single crowns and fixed partial dentures may be exposed to prolonged stress in individuals with habits of bruxism or clenching. Especially the new micro-filled resin veneer materials, which according to the manufacturers can be used to partially cover incisal or occlusal aspects of the restoration, can be exposed to extensive and prolonged forces. If resin veneer materials with a high tendency to creep are subjected to such forces, a deterioration of the adaptation between the facing and the casting may result, and the retention of the veneer may be weakened, which can lead to fracture or loosening of the facing. Debonding of filler particles from the resin due to stresses can also occur (1).

Creep of dental resins has been determined *in vitro* by different methods. Cantilever bending (2–4), indentation testing (5–7), 3-point bending (5), compression of cylindrical specimens (8–11) and torsion testing (12) have been used.

It has been shown that creep in resins is related to the chemical composition and degree of conversion of the resin. The type, size, distribution and number of filler particles (9–11), the adhesion between the filler particles and the resin matrix, and sorption of water can also influence the time-dependent deformation of resins (13, 14).

Restorative composite dental resins have lower creep than heat-cured denture-base materials (9, 10). Also, it has been found that both chemically activated composites and light-cured composites have a comparatively low degree of conversion (20–50% of remaining unreacted groups), while heat-cured acrylic resin may contain less than 1% of unreacted groups (15).

Composite resins with different chemical composition and varying filler content have been introduced as prosthetic facing materials. New techniques of processing, including different polymerization methods, have been in use for some time. It is not known how these variations of the veneer resins affect their viscoelastic behavior.

The purpose of this investigation was to determine creep of different types of resin veneer materials processed by different methods commonly used by dental technicians.

Material and methods

Two methods for studying creep were utilized: static compression of cylindri-

Table 1. The products included in the investigation.

Product	Manufacturer	Batch no.	Color variant
Biodent K+B Plus	De Trey, Wiesbaden, FRG		
Powder: dentine		4*/20+	D-27*/D-21+
cervical		8*/7+	H-21*/H-39+
incisal		15*/12+	S-25*/S-13+
Liquid S		120*/112+	
Liquid N		4*/41+	
Protective coating		10	
SR-Isosit-PE	Ivoclar AG, Schaan, Liechtenstein		
Dentine		830175*/469S+	D-03*/D-6C+
Cervical		– /232S+	H-6C*/H-5C+
Incisal		– /517S	S-17*/S-21+
Cross-linking paste		–	
Catalyst		436S	
Separating fluid		1652 CE	
Triad K+B	De Trey/Dentsply, Wiesbaden, FRG		
Dentine		–	D-16
Neck		–	H-25
Incisal		–	S-16
Air barrier coating		–	
Model release agent		–	

* Denotes the materials used in the compressive creep test.
+ Denotes the materials used in the indentation test.

Table 2. The handling procedures used during the processing of the specimens.

	Powder/liquid ratio (weight)	Mixing method/time	Doughing time	Series
Free processing				
Biodent K+B Plus	2:1	+ 30 s	4 min	A
SR-Isosit-PE	++	+++ 30 s	–	C
Flask processing				
Biodent K+B Plus	2:1	+ 30 s	6 min	B
SR-Isosit-PE	++	+++ 30 s	–	D
Light-activated polymerization				
Triad K+B	–	– –	–	E

\+ Biodent K+B Plus was mixed by hand by a stroking action.
++ The dosage used for SR-Isosit-PE was 1 large measuring spoon of powder, 2 scale divisions of cross-linking paste, and 1 measuring spoon of SR-Isosit-PE catalyst.
+++ SR-Isosit-PE was mixed mechanically in capsules. The machine used was a Cap-Vibrator, Type CVU1, Ivoclar AG, Schaan, Liechtenstein.

Table 3. The processing and curing conditions used during the preparation of the specimens.

	External pressure (kPa)	Curing temp. (°C)	Curing time (min)	Liquid type	Series
Free processing					
Biodent K+B Plus	600	95	15	S	A
SR-Isosit-PE	600	120	6	–	C
Flask processing					
Biodent K+B Plus	0	100	+	N	B
SR-Isosit-PE	0	100	+	–	D
Light-activated polymerization					
Triad K+B	0	–	15++	–	E

\+ For the flask processing the instructions given for Biodent K+B Plus were also applied to SR-Isosit-PE; the clamped flask was immersed in water, heated to 100°C during a 30 min period, and then boiled for 30 min. The clamped flask was bench-cooled before deflasking.
++ The light source used for curing the light-activated resin was a Triad TM Model TCU-1 770W, Ser. No. 381, Part. No. 61075, Dentsply York Division, USA.

cal specimens, and examination of identations in the resin surface made by a Vicker's diamond pyramid.

Determination of compressive creep

Cylindrical specimens measuring 4.00 ± 0.03 mm in diameter and 8.00 ± 0.05 mm in length were processed from 3 different brands of veneer resins (Table 1). The heat-cured resins were processed either by the free technique or by the conventional flask technique (Tables 2, 3). A brass block with a thickness of 9 mm and with circular penetrating holes was used to produce the specimens. When the free technique was used, the soft resin was inserted into the holes by a modeling instrument before immersion into the water in a pressure vessel. When processing in a flask, the brass mold was embedded in gypsum in the flask, and the soft material was partly applied into the mold by a hand instrument, and partly by closing the flask in a conventional manner by the use of trial closures. For the preparation of the light-cured specimens a perforated block of Perspex was used. The soft resin was inserted into the holes by a procedure similar to the one used for the free technique.

The end surfaces of the cured specimens were ground in order to make them smooth and perpendicular to the long axis of the specimens. Grinding was carried out on water-proof silicon carbide papers (220–1000 grit) under water irrigation. The specimens were stored in distilled water at 37°C for 4 weeks. The dimensions (diameter and length) of the specimens were then measured in a profile projector at a magnification of ×30 (Nikon, 6C T2, Nikon, Japan). Six specimens that satisfied the dimensional requirements mentioned above were used.

The load was applied by a modified hardness tester (Sematic Durometer, AB Svenska Presisionsverktyg, Sweden). The specimens were placed between 2 steel rods mounted in the hardness tester. An extensometer (Instron G-51-16 M, Instron Instruments, USA), attached to the steel rods and connected to an amplifier (HBM KWS 3082, Hottinger Baldwin Messtechnik, FRG) and a printer (Chessel 301, Chessel, UK) recorded the compression of the specimens (Fig. 1). A load of 30 kg was applied to the specimens at 37°C for 48 h. This corresponded to an initial stress of 23.9 ± 0.4 MPa depending on the diameters of the specimens.

Strain was calculated after 1, 4, 8, 16,

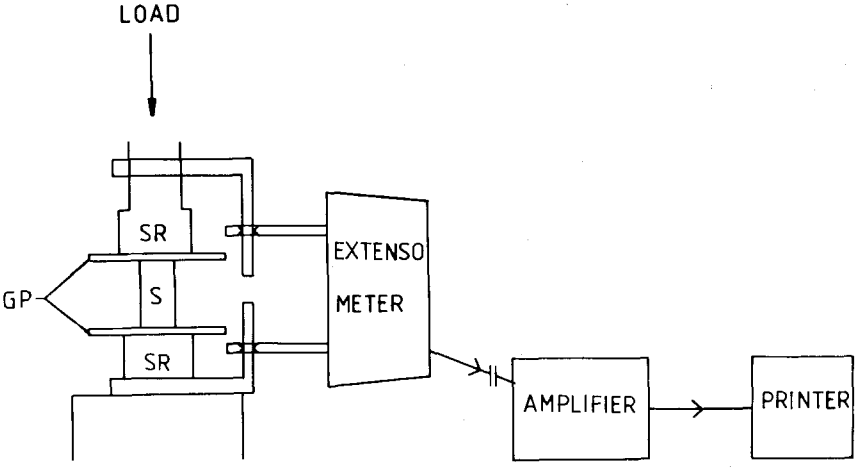

Fig. 1. The mounting of the specimens in the hardness tester and the arrangement of the recording system used during compression of the specimens. S: specimen; GP: glass plates; SR: steel rods.

24, 40 and 48 h of loading. The creep rate was expressed by the slope of the regression line for the strain-time data obtained between 4 and 48 h after the load had been applied (the secondary creep stage).

Determination of creep by indentation testing

In the compressive creep testing the stress may have differed somewhat since the diameters of the specimens varied slightly. This may have influenced the creep rates. For this reason creep was also studied by indentation testing. A new set of specimens was produced and arranged in similar test groups as before. The heat-cured specimens (15×10×3 mm) were made in gypsum molds. The light-cured specimens were cylindrical (diameter 12 mm and length 3 mm) and processed in Perspex molds.

After the specimens had been embedded in epoxy resin (Epofix, Struers, Denmark), they were handled similarly to the previous series of specimens with regard to conditioning and grinding. In addition, they were polished on polymer disks (Lam plan 415, Engis, UK) by the use of a diamond spray of grain size 14 μm (Hyprez five/star/diamond compound, Engis, UK) and ethanol/ethylene glycol as lubricant.

Vicker's diamond pyramid indentations were made in the polished surfaces at 37°C using a load of 196 N. In one series the load was applied for 30 s. In another the loading time was 120 min. The diagonals of the indentations were measured immediately after unloading using the profile projector.

The volumes of material displaced by the diamond pyramid were calculated. The (indentation) creep was expressed as the ratio between the volumes of displaced resin after 120 min and after 30 s of loading.

F-values were calculated for the creep rates and the volume ratios obtained for the groups of specimens by the use of a one-way analysis of variance. Two sample comparisons between the test groups were carried out by the Scheffés test for multiple comparisons. The Spearman rank correlation coefficient (R_s) for the results of the 2 test methods was also calculated. The statistical level of significance chosen was 5%.

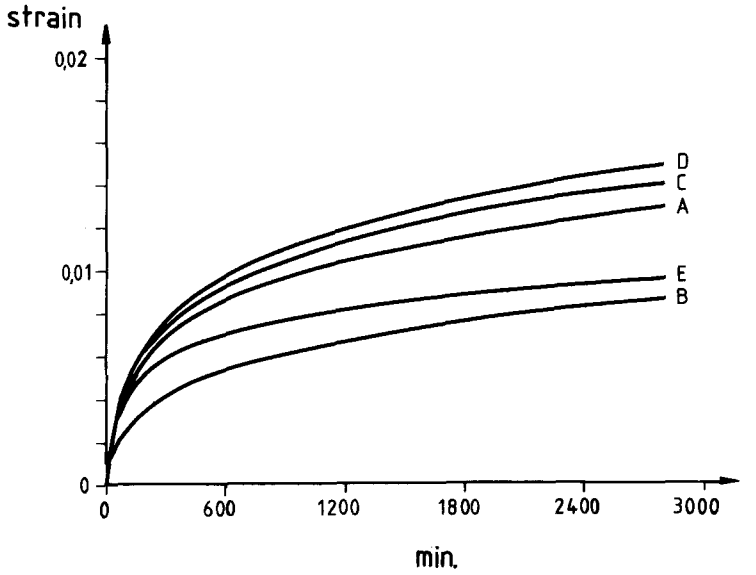

Fig. 2. Strain-time relationship for the dentine variant of the resins (see Table 2 or 3 for decoding of the test groups).

Results

Compressive creep

The shortening of the cylindrical specimens was in the range of 0.08–0.12 mm at the end of the full test period.

After 48 h of loading, the highest strain values were obtained for the SR-Isosit-PE specimens processed in flask (D), while the lowest values occurred in the group of Biodent K+B Plus specimens (B) processed similarly (Fig. 2).

An F-value of 74.17 ($df_1 = 4$, $df_2 = 85$, $p < 0.001$) was obtained for the creep rates of the 5 groups of specimens. The lowest creep rates were found in the light-cured specimens (E) (Fig. 3). The unfilled acrylics achieved the lowest creep rates of the heat-cured resins examined (A, B). The flask-produced specimens (B) had lower creep than the ones processed by use of the free technique (A) ($p < 0.05$). The microfilled resin SR-Isosit-PE obtained lower creep values when processed in accordance with the free technique (C) than when flask-processed (D) ($p > 0.05$) (Fig. 3).

No systematic differences were found between the 3 variants of the veneer resins (dentine, cervical, incisal).

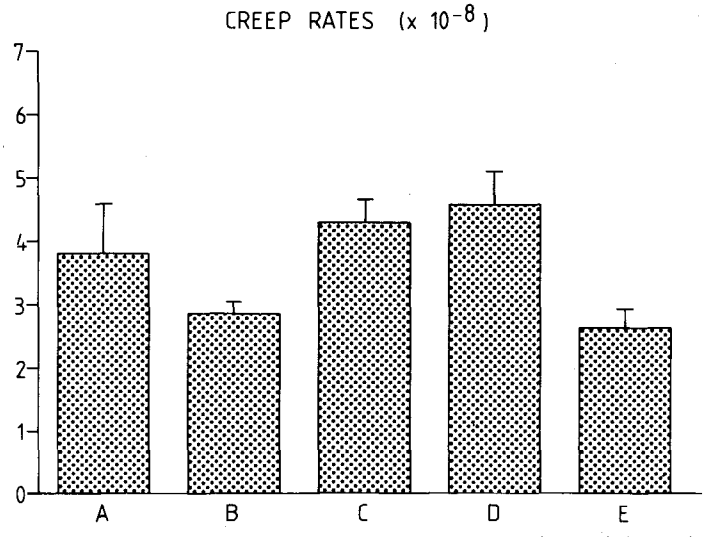

Fig. 3. The mean creep rates for the different series of specimens (n = 18) (see Table 2 or 3 for decoding of the test groups).

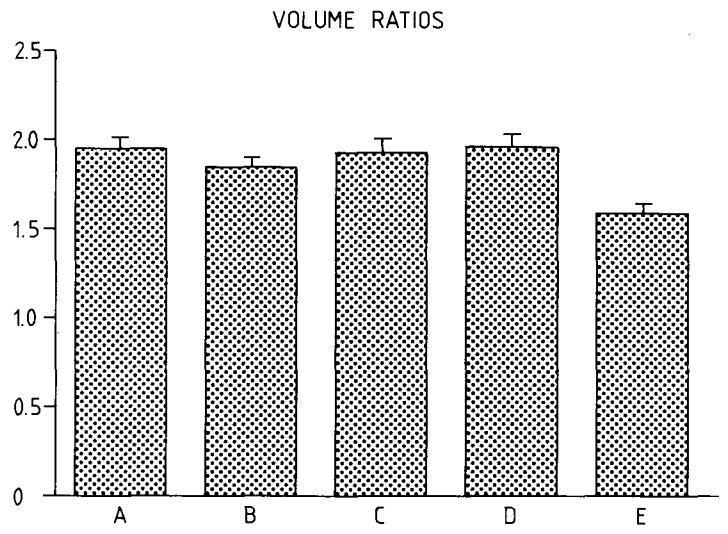

Fig. 4. The mean ratios of the displaced volumes for the indentation test during loading for 120 min and 30 s for the different series of specimens (see Tables 2 or 3 for decoding of the test series).

Table 4. The displaced volumes of resin (in mm^3) after indentation for 30 s and 120 min.

Series	Volume 1 (30 s)	Volume 2 (120 min)
A	$\bar{x}$ = 0.1010 s.d. = 0.0028	$\bar{x}$ = 0.1966 s.d. = 0.0042
B	$\bar{x}$ = 0.0897 s.d. = 0.0014	$\bar{x}$ = 0.1655 s.d. = 0.0047
C	$\bar{x}$ = 0.0630 s.d. = 0.0043	$\bar{x}$ = 0.1220 s.d. = 0.0093
D	$\bar{x}$ = 0.0632 s.d. = 0.0025	$\bar{x}$ = 0.1234 s.d. = 0.0062
E	$\bar{x}$ = 0.0267 s.d. = 0.0018	$\bar{x}$ = 0.0417 s.d. = 0.0022

Indentation testing

The mean volumes of displaced material ranged between 0.026 and 0.101 mm^3 after 30 s and between 0.041 and 0.196 mm^3 after 120 min (Table 4).

The distribution of ratios of displaced material (indentation creep) among the different test groups revealed an almost similar pattern of results as that of the creep rates (Fig. 4) ($F = 163.01$, $df_1 = 4$, $df_2 = 85$, $p < 0.001$).

A Spearman rank correlation coefficient of 0.900 was calculated for the results of the 2 test methods ($p = 0.083$).

Discussion

This investigation of compressive creep was carried out by the use of a constant load system. The diameters of the specimens varied to some extent, both within the same group and among different groups. Thus the specimens were subjected to slightly varying compressive stresses. The varying dimensions may be due to the fact that different molds consisting of brass and Perspex were used in the preparation of the specimens. In addition, the modeling technique used to fill the molds is difficult to standardize and thus differences in density of the inserted soft resin may have influenced the dimensions of the final specimens. The test groups consisted of different materials processed by different methods. The chemical composition, content of filler particles, curing methods, and polymerization cycles may affect polymerization contraction (16). However, it is unlikely that the differences in stress among the specimens influenced the creep rate to any major extent at the stress level used in this investigation. A low stress dependence has been shown for time-dependent deformations at low stress levels (< 23 MPa) for acrylic resins at 37°C, while at higher stress levels (> 40 MPa) the stress dependence is high (10).

The light-cured specimens obtained the lowest creep rates, while the heat-cured micro-filled specimens exhibited considerably higher values. This is most likely primarily explained by the different contents of filler particles, but also by the different chemical compositions of the 2 brands (11). According to the manufacturers Triad K+B contains 60% (by weight) microfillers while the Isosit resin has only approximately 30%. It has also been suggested that dental resins based on the monomer (UEDMA) used for Isosit may exhibit pronounced creep compared to other resins (11). Whether differences in the degree of conversion also influenced the viscoelastic behavior of the micro-filled resins of this study is not known.

The differences among the groups of unfilled acrylic specimens processed by the 2 techniques may be due to differences in composition and processing. Different monomer liquids are used for the 2 methods of processing. In addition, curing by the free technique takes place at 95°C for 15 min while the clamped flask is boiled in water at 100°C for 30 min. It has been shown that conversion of poly (methyl methacrylate) is influenced by the polymerization temperature (9). In addition, a shortening of the curing cycle may cause a considerable increase in residual monomer content in heat-cured denture-base materials (17).

The indentation testing was carried out to evaluate the results of the compressive creep test. The high value of the Spearman rank correlation coefficient showed that the results of the 2 test methods agreed well, although the correlation coefficient was not statistically significant ($p = 0.083$). It must be taken into account that in order to obtain a statistically significant coefficient, all 5 pairs of observations must have been ranked in the same order ($R_s = 1$), whereas in this case 4 out of 5 pairs were ranked similarly.

Resins that varied considerably in terms of hardness were compared and thus the different specimens were subjected to varying stress during indentation. However, by taking the ratios of the displaced volumes, the effects of the varying hardness and thus the displaced volumes on the calculated creep were reduced. The indentation creep test, roughly characterizing creep, seems to possess the advantage of being a simple test less affected by specimen design. Also, in compressive creep testing it can be difficult to ensure that axial loading is obtained and that friction between the contact surfaces of the specimens and the compression plates is minimized (9, 18). These difficulties are avoided in indentation testing.

The varying *in vitro* viscoelastic behavior of the prosthetic veneer resins suggests that they may behave differently in clinical service. Creep may play an important role in debonding of veneers and also in affecting the adhesion

between fillers and resin. The results of this investigation have shown that the light-cured highly filled brand and the flask-processed unfilled acrylic resin achieved the lowest creep. This should be taken into consideration when resin-veneered fixed prostheses are planned.

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