Creep on a Composite Resin in Water

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The compressive creep test of a composite resin (0-3.5 kg/mm² stress levels) was conducted in water for 500 h. Linear regressions were obtained between the creep strains and the compressive stress levels at various hours. It is possible to predict the creep strain of the composite from the regression when it reaches water absorbed equilibrium after 500 h.

The stress of the hygroscopic expansion was calculated from the linear regressions. The maximum stress due to the hygroscopic expansion of the composite was 0.74 kg/mm² at equilibrium of the water absorbed of the composite.

The linear regressions at several compressive stress levels were obtained within 30-50 hr in the strain-log time diagrams.

Key words : Compressive creep, Composite resin, Stress of the hygroscopic expansion

INTRODUCTION

Dental composite resins are widely applied in dental restorations, but their mechanical properties have not been completely evaluated for the high stress bearing posterior restorations. For one approach in an attempt to understand how the properties could be improved, the creep behaviors have recently been investigated¹⁻⁷⁾.

Ruyter and Øysaød¹⁾ investigated the compressive creep curves for several light cured composite resins characterized at several temperatures and stresses. Papadogianis, Boyer and Lakes^{2,3)} investigated the modulus and the creep strain of the composites at various stresses by the tortional test method. Cook and Watts³⁾ explained the compressive strain of the composites to be finally linear against log time in compressive creep. Bapna, Mueller and Knoeppel⁵⁾ tried to apply the creep compliance of the composites with the Voigt-Maxwell model. Ferracane, Matsumoto and Okabe⁶⁾ considered the effects of filler concentration and the extent of conversion in composite resins to time-dependent deformation. Oden, Ruyter and Øysaød⁷⁾ investigated the creep of the composites under static and dynamic compression in dry conditions and in water, and they showed that the creep deformation was of the same magnitude at the end of the test period, and also showed in water sorption, that the creep strain of the composite increased and the recovery of the composite decreased compared to the dry conditions.

In an oral environment the composite resins undergo hygroscopic expansion. This phenomenon takes a long time to reach equilibrium as water diffuses into the composite. The purpose of this study is to determine the behavior of the compressive creep of the composite in water and to determine the stress of the hygroscopic expansion of the composite from the

creep curves obtained.

MATERIAL AND METHODS

A chemically cured composite resin* was used as the experimental material. Specimens were prepared following the manufacturer's recommended mixing ratio and were mixed for 30 s. The mixture was injected by a syringe[#] into a mold of $2\phi \times 10$ mm and compressed with a glass plate. After 10 min the polymerized sample was removed and excess material was trimmed off. Then the sample was stored in an oven at 70–80°C for 24 h. Thereafter it was stored in a decicator with silica gel at 37°C for one week.

The measurements of dimensional changes were made throughout the experiment by means of an electronic micrometer[§] attached to the apparatus. When the specimen was loaded, distilled water was immediately poured into the stainless steel vessel of the apparatus as shown in Fig. 1, and so the specimen was kept in distilled water at 37°C.

Fig. 1 Testing apparatus.
A : electronic micrometer
B : stainless steel vessel
C : water reservoir bottle

* ADAPTIC (Batch No. GGO19) J and J Dental Products Co. East Windsor, NJ USA

[#] CRC Syringe (No. 210001) Centrix Inc. Stanford, CT USA

[§] MU-Checker (Type 122 M) Mitutoyo MFG. Co. Tokyo

The water uptake of the specimen seemed to reach equilibrium after 500 h as shown in Fig. 2. All the measurement were carried out for a period of 500 h.

The loads applied were selected at 3, 6 and 11 kg because of the specimen not being fractured or bent at these stresses.

Before every measurement a calibration was done using a 10 mm gauge block. The error of the apparatus was always within $\pm 2 \mu\text{m}$. The measurement of 0 kg/mm^2 of the stress level of the creep was made only with the electronic micrometer⁸⁾ (measuring load 40 g).

Three specimens in each condition were measured.

RESULTS AND DISCUSSION

The results obtained are given in Table 1 and Fig. 3. The dimensional change at 0 kg/mm^2 compressive stress showed that the specimen expanded continuously after start of measurement due to the water absorption. The strain under the compressive stress in Fig. 3 represent the total strain of the specimen involving instantaneous elastic, delayed elastic and plastic strain or creep strain associated with the stress, and also strain due to the water absorption. Thus some part of compressive strain might have been cancelled out by the hygroscopic expansion. The dimensional change at 1.0 kg/mm^2 was shown to be almost constant with time. The creep curve at 1.9 kg/mm^2 compressive stress showed the decrease in the specimen dimension after the start of the measurement, but the curve for the first few

Fig. 2 The curve of the water uptake of the specimen.

M_t : Rate of the water uptake at time t

M_∞ : Rate of the water uptake at equilibrium

Table 1 Results of the compressive creep of a composite obtained

compressive stress (kg/mm^2)	instantaneous strain (%)	strain after 500hrs (%)
0		0.29 (0.03)
1.0	-0.10 (0.02)	-0.06 (0.08)
1.9	-0.31 (0.06)	-0.50 (0.05)
3.5	-0.48 (0.06)	-0.93 (0.25)

() : standard deviation

hours showed somewhat expansion of the specimen by the effect of the hygroscopic expansion of the specimen. The specimen dimension decreased uniformly with time at the stress level of 3.5 kg/mm^2 .

The strain-log t diagrams of the composite at several stress levels in Fig. 4 to Fig. 7 showed linear portion within 50 h. These results coincided to the results of Cook and Watts⁴.

A linear regression was obtained between the creep strains after 500 h and the compressive stresses (Fig. 8);

$$Y = 0.2637 - 0.3543X \quad (r = 0.967)$$

Y : the creep strain after 500 h, X : the compressive stress, r : coefficient of correlation.

From the above equation, it is possible to predict the strain for a given biting pressure⁹ of the composite when it reaches water absorbed equilibrium after 500 h. It can be assumed

Fig. 3 Creep curves of a composite with various compressive stress levels.

Fig. 4 Strain-log t curve for a composite at 0 kg/mm^2 stress level.
 $30 \text{ h} > : Y = 0.0091 + 0.0390 \log X \quad (r = -0.944)$

Fig. 5 Strain-log t curve for a composite at 1.0 kg/mm^2 stress level.
 $50 \text{ h} > : Y = -0.1140 + 0.0198 \log X \quad (r = -0.825)$

Fig. 6 Strain-log t curve for a composite at 1.9 kg/mm^2 stress level.
 $50 \text{ h} > : Y = -0.4035 + 0.0210 \log X$ ($r = -0.743$)

Fig. 7 Strain-log t curve for a composite at 3.5 kg/mm^2 stress level.
 $75 \text{ h} > : Y = -0.5144 - 0.0692 \log X$ ($r = -0.953$)

Fig. 8 Linear regression obtained between the strain after 500 h and compressive stress levels.

that the rate of the water uptake of a composite is constant in the range of these stress levels because the weight of the specimens after 500 h immersion was almost constant at every stress level in the preliminary experiment. Therefore linear regressions to calculate the stress due to the hygroscopic expansion of the composite from the creep curves were obtained between the total strains and compressive stress levels at various testing period, as shown

Table 2 Linear regressions obtained and the stress of the hygroscopic expansion of the composite with various time

hr	1	3	5	10	20	50	100	200	400	500
A	0.0039	0.0047	0.0232	0.0369	0.0802	0.0905	0.1285	0.2067	0.2520	0.2637
B	-0.1661	-0.1676	-0.1726	-0.1808	-0.2043	-0.2184	-0.2435	-0.3244	-0.3393	-0.3543
r	-0.942	-0.954	-0.955	-0.961	-0.961	-0.963	-0.960	-0.990	-0.965	-0.967
stress of the hygroscopic expansion (kg/mm ²)	0.02	0.03	0.14	0.20	0.40	0.41	0.53	0.64	0.74	0.74

A : Constant, B : Regression coefficient, r : Coefficient of correlation (Y = A + BX)

Table 2. When $Y=0$ is introduced to the equation of Table 2, the stresses due to the hygroscopic expansion of the composite were given using the strain at various hours at every stress. Due to the increase in the rate of the water uptake of the composite, the stress of the hygroscopic expansion of the composite increased. The maximum hygroscopic expansion was estimated to be 0.74 kg/mm² when the water uptake of the composite had reached equilibrium. The stress due to the hygroscopic expansion of the composite is 3-6 times larger than the stress of the polymerization contraction^{10,11}.

CONCLUSIONS

Linear regressions were obtained between the creep strains and the compressive stress levels in the composite. It is possible to predict the creep strain of the composite when it reaches water absorbed equilibrium after 500 hr.

The maximum stress of the hygroscopic expansion of the composite was also calculated to be 0.74 kg/mm² at equilibrium of the water uptake of the composite.

The linear regressions at several compressive stress levels were obtained within 50 hr in the strain-log time diagrams.

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に比べクリープ破壊に対し長期間抵抗した。混合型高銅アマルガムは、破壊時のクリープ値が試験したアマルガム中でもっとも大きかった。これは、このアマルガムが連続的な荷重に対し他のアマルガムに比べてより柔軟性があるということを意味している。破壊時間はクリープ

速度の逆数にほぼ比例し、クリープ速度が減少すると破壊時間は急激に増加した。この結果は、以前に報告された一定期間内のクリープ値とアマルガム修復物の辺縁破折との相関をクリープ破壊の面から説明し、裏付けている。

象牙質処理剤がレジンの接着性に与える影響について

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Dentin cleanser によって象牙質の受ける変化が接着性にいかなる影響を及ぼすかについて、リン酸、グリシン含有ピルビン酸, EDTA による歯面清掃を行なった後の象牙質の物理的特性を計測し、これらとレジンの接着性の相関を検討した。その結果、レジンと象牙質との接着性は、cleanser によって象牙質に与える変化のうち Micro Vickers Hardness の低下と最も高い相関を示し

た。すなわち、cleanser によって残存象牙質硬さが 85% 以上に保たれる EDTA-2Na による処理が最も良好な接着性を示した。このことから、良好な接着性を得るためには smear layer のみを除去し、象牙質には全く影響を及ぼさない cleanser が理想的な材料であると結論付けられた。

Self Etching Dentin Primer の効果

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35%HEMA 水溶液に希釈されたジカルボン酸または、そのエステル構造物を含む市販及び試作の Self Etching Dentin Primer の効果を象牙質円柱窩洞内での可視光線重合型コンポジットレジンのコントラクションギャップの計測と象牙質平面に対する引っ張り接着強さによって評価した。

試作した primer はすべて市販のリン酸エステル系ボンディング材と、また市販品のセルフエッチングタイプの primer は付属のレジモノマーを併用し、全ての窩洞および被着面には、市販の光重合型コンポジットレジン

を充填または接着させた。

その結果、HEMA にメタクリロキシエチルコハク酸または、メタクリロキシエチルフタル酸を溶解した 2 種類の試作 primer が、今回用いられたボンディング材の象牙質に対する接着性を著明に向上させた。また、マレイン酸を含む市販の primer は、24 時間後の引っ張り接着強さが 11.9 ± 5.7 MPa と高い値を示したが、コンポジットレジンと象牙質窩壁との適合性を改善することはできなかった。

水中浸漬下におけるコンポジットレジンの圧縮クリープ

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コンポジットレジンの圧縮クリープを 4 種の異なる応力 (圧縮応力 $0 \sim 3.5$ kg/mm²) で水中浸漬下で 500 時間

にわたって行なった。得られた 4 種のクリープ曲線から、一定時間経過したときの各クリープひずみと圧縮応力と

の間には一次回帰式が成立した。そのためコンポジットレジン500時間後の吸水飽和状態になったときのクリープひずみは圧縮応力が分かれば予測可能となった。またこの回帰式を使い、各時間における吸水膨張応力を

求めた。最大吸水膨張応力はコンポジットレジンが吸水飽和したときに得られ、その値は 0.74 kg/mm^2 であった。各応力下で得られたクリープ曲線をひずみ対数時間曲線で表すと、初期の数十時間は直線となった。