Compressive Creep of Posterior and Anterior Composite Resins in Water

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The compressive creep and the recovery of commercially available composites, 2 kinds for posterior and 2 kinds for anterior use, in water were examined. In posterior composites, creep strain after 500h at a stress level of 8.3 kgf/mm² was significantly small (within 1 %). The results of the compressive creep test showed that posterior composites would be resistant to occlusal stress. Creep strain was higher in anterior composites than that in posterior composites because of their lower inorganic filler content. The rate of recovery of composites was rapid immediately after the creep test. The water sorption of composites after 500h test was almost constant at some reduced stress levels, especially in posterior composites.

Key words: Composite resin, Compressive creep, Water sorption

INTRODUCTION

Direct filling materials undergo a certain amount of deformation or creep when subjected to dynamic intra-oral stresses. In amalgam, when creep values are above some level in standardized testing, generally restorations with higher creep alloys experience greater marginal breakdown than those with lower creep alloys. The creep of dental composite resins should also be considered as being one of their most important mechanical properties¹), especially in use for posterior restorations as a substitute for dental amalgam. In the oral environment, composite resins undergo water sorption. Thus it is important to test these materials in water as their plasticity is increased by water sorption.

The results of creep tests have been reported previously²⁻¹²). In previous reports¹⁰,¹²) the authors estimated the stress of the hygroscopic expansion of a composite resin derived from the results of compressive creep tests at several compressive stress levels, and the water sorption of the composites was reconfirmed to be almost constant under some reduced compressive stress levels because of the low volumetric strain due to the high filler content of the composite.

The objective of this investigation was to determine the compressive creep of commercially available composites until reaching equilibrium in water uptake.

MATERIALS AND METHODS

Two kinds of composites for anterior use, CB* and SI**, and for posterior use, CP# and P50##,

* Photo Clearfil Bright, 0015B, Kuraray Co ltd. Osaka, Japan
** Silux, 012385, 3M, St Paul, MN, USA
# Clearfil Photo posterior, 0008A, Kuraray Co ltd. Osaka, Japan
## P-50, OCR4D, 3M, St Paul, MN, USA
were examined.

The composite was injected with a syringe\textsuperscript{*} into a teflon tube ($\phi$: 2 mm), and immediately irradiated for 90s with a light activation unit\textsuperscript{**}. Samples were then stored in an oven at 90°C for 24h to induce complete polymerization. The samples were cut with a low-speed saw\textsuperscript{§}, and were finished dimensions of $\phi$ 2 × 10 mm long. Thereafter, they were stored in a desiccator with silica gel at 37°C for at least one week.

The compressive creep test was carried out for a period of 500h with the same apparatus in water at 37°C as described previously\textsuperscript{10}). The stresses applied were selected at 0, 2.2, and 5.1 kgf/mm\textsuperscript{2} for anterior composites and at 0, 5.1, and 8.3 kgf/mm\textsuperscript{2} for posterior composites.

The stress level of 5.1 kgf/mm\textsuperscript{2} almost corresponded to the maximum stress level of normal occlusion\textsuperscript{13), and that of 8.3kgf/mm\textsuperscript{2} almost corresponded to the maximum stress level of unilateral occlusion\textsuperscript{13). The recovery and water sorption of the specimens were investigated over a period of 500h after completion of the creep test. All specimens were stored in distilled water at 37°C. Within 15 min following the creep test, the length of specimens was measured 5 times with a height gauge\textsuperscript{$\$$}, and the mean value was used to calculate the instantaneous recovery. The length of specimens were measured at fixed intervals, and recovery was calculated over a given period of time. The water sorption of the specimen during the testing was also measured within 15 min after completion of the test, and the gain in weight was indicated in terms of the water sorption. The water absorbed on specimens after the creep test was also measured at fixed intervals.

Three specimens in each condition were measured and the mean values were assessed.

\textbf{RESULTS}

\textit{Creep strain}

The creep strain and the recovery of the composites are shown in Table 1. Creep with a stress level of 0 kgf/mm\textsuperscript{2} indicates dimensional change by the hygroscopic expansion of the composite.

The anterior composites fractured during testing at a stress level of 8.3 kgf/mm\textsuperscript{2}, and therefore the creep test was performed with a maximum stress level of 5.1 kgf/mm\textsuperscript{2}. The creep strain at a stress level of 5.1 kgf/mm\textsuperscript{2} for SI was 2.89 %, a large strain, after the test (Fig. 1), and that at a stress level of 2.2 kgf/mm\textsuperscript{2} was 0.52 % after the test. The creep strain at the stress level tested in CB increased gradually from 100 to 500h, and final creep strains were 0.56 % at a stress level of 2.2 kgf/mm\textsuperscript{2} and 0.77 % at a stress level of 5.1 kgf/mm\textsuperscript{2}.

About 500h after completion of the creep test, the dimensional changes in recovery of these specimens approached that of their hygroscopic expansion.

For posterior composites, creep strains at stress levels tested were small from 100 to 500h. The dimensional change after completion of the creep test was within 1 % in CP and P50 at a stress level of 8.3 kgf/mm\textsuperscript{2}.

\textsuperscript{*} CRC Syringe (No. 21001), Centrix Inc., Milford, CT., USA
\textsuperscript{**} Dentacolor XS, Kurzer & Co. GmbH, Bad Homburg, Germany
\textsuperscript{§} Isomet, Buehler LTD., Evanston, IL., USA
\textsuperscript{$\$$} Digimatic Indicator (Type 543) Mitutoyo MFG. Co., Tokyo Japan
Table 1  Strain after 500h compressive creep test

<table>
<thead>
<tr>
<th>Name</th>
<th>$C_{rs}$</th>
<th>$I_s$</th>
<th>$R_{ir}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>CB</td>
<td>+0.39 (0.04)</td>
<td>0.50 (0.26)</td>
<td>0.69 (0.32)</td>
</tr>
<tr>
<td>SI</td>
<td>+0.59 (0.02)</td>
<td>0.52 (0.17)</td>
<td>2.89 (0.64)</td>
</tr>
<tr>
<td>CP</td>
<td>+0.13 (0.06)</td>
<td>0.29 (0.18)</td>
<td>0.29 (0.28)</td>
</tr>
<tr>
<td>P50</td>
<td>+0.19 (0.01)</td>
<td>0.29 (0.18)</td>
<td>0.29 (0.28)</td>
</tr>
</tbody>
</table>

unit: %, ( ) : S D and +values indicating expansion

$C_{rs}$ : creep strain after 500h creep test
$I_s$ : instantaneous creep strain
$R_{ir}$ : initial rapid recovery after creep test

Fig. 1 Creep and recovery curves for SI. ▲ and ● were measuring points at a stress level of 2.2 and 5.1 kgf/mm², respectively.

In recovery, the dimensional change in posterior composites almost coincided with that of their hygroscopic expansion of the specimens (Fig. 2).

**Water sorption**

As shown in Table 2, the water absorbed in CB after the test between stress levels of 0 and
Fig. 2 Creep and recovery curves for CP. ○ and □ were measuring points at a stress level of 5.1 and 8.3 kgf/mm², respectively.

Table 2 Water sorption of composites

<table>
<thead>
<tr>
<th>Name</th>
<th>Stress (kgf/mm²)</th>
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<tbody>
<tr>
<td></td>
<td>0</td>
</tr>
<tr>
<td>CB</td>
<td>W₅₀₀</td>
</tr>
<tr>
<td></td>
<td>R₅₀₀</td>
</tr>
<tr>
<td>SI</td>
<td>W₅₀₀</td>
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<td></td>
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<tr>
<td>CP</td>
<td>W₅₀₀</td>
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<td></td>
<td>R₅₀₀</td>
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<tr>
<td>P50</td>
<td>W₅₀₀</td>
</tr>
<tr>
<td></td>
<td>R₅₀₀</td>
</tr>
</tbody>
</table>

W₅₀₀: water sorption after 500h compressive creep test
R₅₀₀: water sorption after 500h recovery (500h creep + 500h recovery)
unit: wt%, and ( ): SD

5.1 kgf/mm² and in CP after the test between stress levels of 0 and 8.3 kgf/mm², were significant at P<0.1 by t-test. The other samples showed no significant in water absorption after the test at any stress level.

Water sorption between completion of the creep test and recovery, at a stress level of 5.1 kgf/mm² was only significant in SI at P<0.05 by t-test.

DISCUSSION

The creep strain of posterior composites after 500h at a stress level of 8.3 kgf/mm² was within 1 %, significantly small, and after completion of the creep test, the large recovery including an initial rapid recovery was observed as described previously. In the rate of water sorption in the specimens, there were no significant differences between completion of
the creep test and the recovery after 500h compared with those at a stress level of 0 kgf/mm².

A stress level of 8.3 kgf/mm² in compression is almost equivalent to maximum occlusal stress. This creep test was longer in duration and performed at a greater stress level than the standardized testing for amalgam from ADAS (creep after 7 days, stress of 3.8 kgf/mm² applied for 4h). The creep strain on the posterior composites including the instantaneous creep strain after 500h at a stress level of 8.3 kg/mm² was very small (within 1%); therefore the posterior composites tested would be resistant to occlusal stress.

The creep strain in anterior composites was higher than that posterior composites because of their lower content of inorganic filler as a reinforcing material; inorganic filler contents in SI and CB were about 40-45 vol%, while those in P50 and CP were over 70 vol %.

The creep rates between 100h and 500h were calculated. This creep rate was almost equivalent to the steady-state creep rate because creep curves of composites were flat in these areas. With the exception of SI, linear regressions were obtained for each composite. The order of the creep rate was $10^{-4}$/h at the stress levels measured (Fig. 3).

Water uptake after the creep test was almost constant at some reduced stress levels, especially in posterior composites. This was because of their low creep strain due to their high inorganic filler content.

Water uptake between completion of the creep test and the recovery in SI at a stress level of 5.1 kgf/mm² was significant at a level of $P<0.05$. This implies that the craze formation or microcrack would occur at the matrix/filler interface or at the matrix in a composite during the creep test as indicated by large creep strain. The increase in water sorption of the composites after testing under stress compared with the unloaded state may be used as an indicator of whether they were damaged during the test.
REFERENCES

臼歯部用と前歯部用コンポジットレジンの水中浸漬下のクリープ
平野 進, 平澤 忠
鶴見大学歯学部歯科工学科講座

市販の臼歯部用コンポジットレジンと前歯部用コンポジットレジン各々2種類について,その圧縮クリープと回復について水中浸漬下で研究した。
試験した臼歯部用コンポジットレジンの500時間後のクリープひずみは8.3kgf/mm²の応力下では1%未満であった。この結果, これら臼歯部用コンポジットは咬合応力に十分耐え得ることを示していた。前歯部用コンポジットレジンではフィラー含有量が臼歯部用に比べて少ないため, 臼歯部用にくらべてクリープひずみが大きかった。コンポジットの回復は試験直後に著しく大きかったが, 500時間後のクリープ試験後の試料の吸水率は, 臼歯部用コンポジットレジンでは, ある低応力下ではほぼ一定であった。

グラスアイノマーセメントの機械的性質の比較
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三種のグラスアイノマーセメントを選びて機械的性質を測定し, 各セメントの特性を検討することにより,今後の材料開発の参考とした。三種のセメントのうち一種は, ガラス繊維を含有した自硬性の試作材料で, 他の二種は市販の自硬性および光照射型セメントであった。バイアキシャルまたは通法の三点曲げ試験をおこない, 後者からは弾性係数も求めた。37℃, 相対湿度100%で24時間保存した試料を, 室温で試験した。市販のセメントが典型的な脆性破壊挙動を示したのに比し, 繊維を含有した試作材料では, 破壊過程を著しく遅延する効果が認められ, これはワイプレ係数が大きくなること, すなわちより安定した破壊挙動につながった。市販のセメントで得られた特性は, 象牙質に近似した弾性率と歯科用複合レジンに匹敵する強度であった。

アークイオンプレーティング法により Ni-50Ti 合金上に創成した TiN 膜の表面性状と耐食性
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北海道医療大学歯学部歯科工学科講座
¹Department of Orthodontics, Baylor College of Dentistry

アークイオンプレーティング法によりインプラント用 Ni-50Ti 合金上に創成した窒化チタン薄膜の構造と耐食性を調べた。X 線光電子分析装置を用いた角度分析により, 創成された窒化チタン薄膜は二層構造となっており, 最表層から TiOx, TiN (x>1), TiN と化学状態が変化していることが明らかとなった。また, コーティング層からニッケルは検出されなかった。0.9 % NaCl 溶液中におけるアノード分極曲線を測定したところ, 場化チタンでコーティングした Ni-50Ti 合金の不動態保持電流は自然酸塩電位から +500mV (vs. Ag/AgCl) まででは研磨状態の合金と比較しておよそ 1/100 となり, 耐食性が向上することが明らかとなった。しかし, 脱不動態電位が研磨状態の +1200mV から +500mV に低下し, 乳食感受性が高くなった。分極抵抗の測定から, 自