Effects of Cyclic Loading on Viscoelastic Properties of Soft Lining Materials

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Received March 3, 2003/Accepted June 26, 2003

The cyclic loadings during mastication supposedly accelerate degradation of soft lining materials. The purpose of this study was to investigate the effects of cyclic loading on viscoelastic properties of soft lining materials. Two plasticized acrylics, two silicones and one isoprene-based monomer contained elastomer were selected. Cylindrical-shaped specimens, 10 mm x 10 mm, were prepared. Twenty specimens were subjected to cyclic loading in a water bath at 37°C; another twenty specimens were without cyclic loading. The viscoelastic properties were measured using a creep-meter. The cyclic loading significantly decreased the heights of the two materials, the instantaneous elastic displacements of one acrylic liner and the viscous flows of three materials. The cyclic loading affected mainly delayed deformations. Therefore, cyclic loading was a useful method for evaluating the durability of soft lining material.

Key words: Lining materials, Viscoelasticity, Fatigue test

INTRODUCTION

The elderly population of Japan is markedly increasing. In general, alveolar ridges of elderly people are often highly absorbed and oral mucosa covers the ridge occasionally showing thin and poor resilience. Patients suffering from these problems complain of pain during mastication. Therefore, soft lining materials are expected to relieve these problems, and studies have been conducted testing their efficiencies.

Many soft lining materials have been marketed during the past several years. The soft lining materials on the market are classified according to their compositions such as plasticized acrylic base resin, silicone base elastomer, fluoroelastomer, polyolefin and isoprene-based monomer contained elastomer. The plasticized acrylic base resin and silicone base elastomer are the most common soft lining materials. Moreover, these two types of soft lining materials are also subclassified according polymerization method: self-curing, heat-curing and photo-curing. The self-curing and the photo-curing types are mainly used for chair-side treatment and the heat-curing type is used in dental laboratories.

However, there are problems of soft lining materials such as heavy loss of resilience, growth of germs, poor color stability and adhesive strength to the resin...
base\(^1\). The causes of these problems are mainly because of saliva\(^5\), food\(^6\) and temperature\(^7\). Therefore, the degradation of soft lining materials has been evaluated by longer water or stained solution storage\(^7\)\(^\sim\)\(^11\) and thermal cycling\(^7\). In addition, an accelerated aging chamber exposing to visible light and water spray was employed to evaluated durability of soft lining materials\(^12\)\(^,\)\(^13\). Moreover, the cyclic loadings during mastication supposedly accelerate degradation of soft lining materials. However, there are few studies\(^14\) regarding the effect of cyclic loading on properties of soft lining materials. The purpose of this study was to investigate the effects of cyclic loading on viscoelastic properties of five commercial soft lining materials.

**MATERIALS AND METHODS**

**Soft lining materials**
The soft lining materials examined in this study are listed in Table 1. Two plasticized acrylic base soft lining materials, one self-curing (Coe-soft, GC-America, Alsip, IL, USA: GCS) and the other heat-curing type (Super-soft, GC-America, Alsip, IL, USA: GSS), two silicone rubber base soft lining materials, one heat-curing (Molloplast-B, Detax, Ettlingen, Germany: DMB) and the other self-curing type (Sofreliner, Tokuyama, Tokyo, Japan: TSR) and one isoprene-based monomer contained elastmer (Clearfit LC, Kuraray, Okayama, Japan: KCF) were selected.

**Specimen preparation**
Cylindrical-shaped specimens, 10 mm in height and 10 mm in diameter, were prepared. All tested materials were polymerized according to the manufacturer’s instructions.

GCS and GSS were mixed at a P/L ratio of 5.00 g/4.00 ml. TSR was automatically mixed with a dispensing gun (Dispenser II, Tokuyama, Tokyo, Japan). DMB and KCF were provided as a one-paste system. The specimens of GSS and DMB were prepared by the dental flaking method. A cylindrical-shaped wax pattern was made using a stainless steel mold, 10 mm in height and 10 mm in diameter. The pattern was embedded in a flask (Varsity Upper, Buffalo, NY, USA) using gypsum (Zostone, Shimomura Sekko, Tokyo, Japan). After setting the gypsum, the wax pattern was removed and the dough of GSS and DMB was packed. The flask was immersed in preheated water in a boiling bath (EWL5518, Kavo, Biberach, Germany) at

<table>
<thead>
<tr>
<th>Product</th>
<th>Manufacturer</th>
<th>Curing Procedure</th>
<th>Lot No.</th>
<th>Code</th>
<th>Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coe-soft</td>
<td>GC-America*</td>
<td>Self-curing</td>
<td>L0108171</td>
<td>GCS</td>
<td>Plasticized acrylic</td>
</tr>
<tr>
<td>Super-soft</td>
<td>GC-America*</td>
<td>Heat-curing</td>
<td>0010171</td>
<td>GSS</td>
<td>Plasticized acrylic</td>
</tr>
<tr>
<td>Molloplast-B</td>
<td>Detax**</td>
<td>Heat-curing</td>
<td>010629</td>
<td>DMB</td>
<td>Silicone</td>
</tr>
<tr>
<td>Sofreliner</td>
<td>Tokuyama***</td>
<td>Self-curing</td>
<td>690722</td>
<td>TSR</td>
<td>Silicone</td>
</tr>
<tr>
<td>Clearfit</td>
<td>Kuraray****</td>
<td>Light-curing</td>
<td>011112</td>
<td>KCF</td>
<td>Isoprene-based elastomer</td>
</tr>
</tbody>
</table>

*Alsip, IL, USA  **Ettlingen, Germany  ***Tokyo, Japan  ****Okayama, Japan
74°C for 30 min, heated to 100°C at 0.87°C/min and maintained at 100°C for an additional 10 min. Subsequently, the flask was cooled to room temperature.

The fresh mixtures of GCS and TSR were packed into the stainless steel mold. The two opened sides were pressed tightly with glass plates through the intermediary polyethylene sheets, and this mold was heated in an incubator (ITD-20H, ALP, Tokyo, Japan) at 37°C for 10 min.

The fresh mixture of KCF was also packed into the same stainless steel mold in the same aforementioned manner except for the use of a silicone sheet. After removal of the glass plates and silicone sheets, the mold was placed in a light polymerizing unit (a-Light, Morita, Kyoto, Japan) for 5 min exposure to light from two opened sides. The specimen was removed from the mold, and additionally polymerized in the same unit for another 5 min.

Forty-five specimens of each material were prepared. All the specimens were stored in the incubator with silica gel at 37°C for 24 hr. After storage in the incubator, weights (\( W_0 \)) and heights (\( H_0 \)) of all specimens were measured using a balancer (AE240, Mettler Toledo, Greifensee, Switzerland; minimum reading 0.01 mg) and a creep-meter (Rheoner RE3305, Yamaden, Tokyo, Japan; minimum reading 0.01 mm), respectively.

**Storage conditions**

Twenty pairs of specimens of each material were randomly assigned following 4 storage periods of water immersion. One specimen of each pair was mounted on an electromagnetic force micro-material tester (MMT-250N, Shimadzu, Kyoto, Japan) in a water bath at 37°C for cyclic loading. The testing conditions for the cyclic loading were as follows: a sinusoidal cyclic unipolar load (i.e., haver-sines cyclic load) of 2 Hz for frequency, a minimum pressure of 19.6 kPa (0.20 kgf/cm²), and maximum pressure of 196 kPa (2.0 kgf/cm²) (Fig. 1). The cyclic loading was performed during water storage of 1.75, 3.5, 7.5 and 22.5 hr, therefore, the number of cyclic loadings were 12,600, 25,200, 54,000 and 162,000, respectively. The other specimen of each pair was immersed in an identical water bath simultaneously. Therefore, the storage condition was classified as with or without cyclic loading and the storage period as shown in Table 2. Five specimens of each material were stored under each condition.

After storage, weights (\( W_i \)) and heights (\( H_i \)) of each specimen were measured with the aforementioned method and calculated the percentage change of height (\( Ch \)).

\[
Ch = \left| \frac{(H_0 - H_i)}{H_0} \right| \times 100
\]

<table>
<thead>
<tr>
<th>Table 2 Selected factors and levels for storage conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Factor</td>
</tr>
<tr>
<td>Cyclic loading</td>
</tr>
<tr>
<td>Immersion period</td>
</tr>
</tbody>
</table>
CYCLIC LOADING EFFECT ON SOFT LINING MATERIAL

Viscoelastic properties

After storage, viscoelastic properties were measured by a creep test using a creep-meter. In this study, the ball-shaped probe was employed to eliminate the effect of deformation during measurements. The testing conditions for the creep test were performed using a 3-mm diameter ball-shaped probe at a cross-head speed of 1 mm/sec until the load reached 1.96 N (200 gf) and maintained for 60 sec (Fig. 2). The amount of applied load for measurement was not changed among materials to evaluate the total displacement as an indication of deformation. A personal computer (WK-C650A, Wakamatsu, Tokyo, Japan) connected to the creep-meter divided the amount of displacement into three parts using the Voight four-element model: instantaneous elastic displacement, delayed elastic displacement and viscous flow using software (Creep Analysis, Yamaden, Tokyo, Japan). A typical result of the creep test and obtained viscoelastic properties are illustrated as Fig. 3. The instantaneous elastic displacement indicates the initial elastic deformation, the delayed elastic...
displacement indicates that following elastic deformation after initial deformation and the viscous flow exhibits continuous displacement due to viscosity.

The remaining 5 specimens of each material without water storage were also measured as a control.

Water sorption and solubility
All specimens after storage were dried in another incubator (DX300, Yamato, Tokyo, Japan) at 37°C until reaching a constant change of weight of less than 0.2 mg within 24 hr. Subsequently, each specimen was weighed again \( W_2 \). In general, the water sorption and solubility were evaluated from the change of weight per surface area\(^{19}\). In this study, the water sorption and solubility were evaluated based on volume because the top and bottom surfaces of the specimen were covered with plungers during cyclic loading. Water sorption \( (W_s) \) and solubility \( (S_o) \) were calculated using the formulæ shown below.

\[
W_s = \frac{(W_1 - W_2)}{V}
\]

\[
S_o = \frac{(W_0 - W_2)}{V}
\]

\( V \) is volume of the specimen determined by dimensions before storage.

Statistical analysis
Changes of height, viscoelastic properties (the amount of instantaneous elastic displacement, delayed elastic displacement and viscous flow), and water sorption and solubility were analyzed with the two-way ANOVA selecting the cyclic loading and storage period as factors and Tukey's HSD test using statistical software (JMP 5J, SAS, Tokyo, Japan). The significance level was set at 0.05.

RESULTS

Change of height
The changes of height are shown in Fig. 4. The ranges of height changes were 0.0-
CYCLIC LOADING EFFECT ON SOFT LINING MATERIAL

Table 3 Results of two-way ANOVA of each material

<table>
<thead>
<tr>
<th>Material</th>
<th>GCS</th>
<th>GSS</th>
<th>DMB</th>
<th>TSR</th>
<th>KCF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Change of height</td>
<td>O</td>
<td>O</td>
<td>O</td>
<td>O</td>
<td>O</td>
</tr>
<tr>
<td>Instantaneous elastic displacement</td>
<td>O</td>
<td>O</td>
<td>O</td>
<td>O</td>
<td>O</td>
</tr>
<tr>
<td>Delayed elastic displacement</td>
<td>O</td>
<td>O</td>
<td>O</td>
<td>O</td>
<td>O</td>
</tr>
<tr>
<td>Viscous flow</td>
<td>O</td>
<td>O</td>
<td>O</td>
<td>O</td>
<td>O</td>
</tr>
<tr>
<td>Total displacement</td>
<td>O</td>
<td>O</td>
<td>O</td>
<td>O</td>
<td>O</td>
</tr>
<tr>
<td>Water sorption</td>
<td>O</td>
<td>O</td>
<td>O</td>
<td>O</td>
<td>O</td>
</tr>
<tr>
<td>Solubility</td>
<td>O</td>
<td>O</td>
<td>O</td>
<td>O</td>
<td>O</td>
</tr>
<tr>
<td>Delayed deformation</td>
<td>O</td>
<td>O</td>
<td>O</td>
<td>O</td>
<td>O</td>
</tr>
</tbody>
</table>

LXP: The interaction of cyclic loading and storage period
O: P<0.05

Fig. 5 The viscoelastic properties after various storage conditions.

76.4% (GCS), -0.3-17.3% (GSS), -0.2-0.5% (DMB), 0.0-7.1% (TSR) and 0.0-1.7% (KCF). In general, the heights of tested materials decreased after cyclic loading; the heights also decreased with longer loading period. The results of two-way ANOVA are summarized in Table 3. The cyclic loading significantly affected the changes of height of GCS, GSS and KCF. The storage period significantly affected the changes of height of GCS, GSS, TSR and KCF. An interaction between cyclic loading and storage period was found on GCS, GSS, TSR and KCF; the cyclic loading increased the changes of height with a longer loading period.

Viscoelastic properties

The obtained viscoelastic properties are shown in Fig. 5. The viscoelastic properties of each material without water storage are also exhibited in Fig. 5 as storage P0.

The examined soft lining materials were classified as three types: elastic material with almost no viscosity (DMB), elastic material with slight viscosity (TSR and KCF), and elastic material with viscosity (GCS and GSS).
The instantaneous elastic displacements varied among materials; those of GSS increased 0.23-0.40 mm, those of DMB slightly increased 0.63-0.67 mm, whereas the instantaneous elastic displacement of GCS, TSR and KCF decreased 0.78-1.63 mm, 0.94-1.37 mm and 0.77-0.80 mm, respectively. The cyclic loading significantly decreased the instantaneous elastic displacement of GCS, however, it did not change the instantaneous elastic displacement of the other materials. The immersion period significantly decreased the instantaneous elastic displacement of GCS and TSR, and significantly increased the instantaneous elastic displacement of DMB and GSS.

The delayed elastic displacement of GCS, GSS, DMB, TSR and KCF were 0.26-0.71 mm, 0.69-0.98 mm, 0.03-0.05 mm, 0.07-0.26 mm and 0.18-0.20 mm, respectively. In general, the delayed elastic displacements of GCS, GSS and TSR decreased with longer immersion period. The interaction of cyclic loading and storage period was found on TSR; more cyclic loading showed a greater decrease of TSR.

The absolute values of viscous flow except GCS were smaller than those of instantaneous elastic displacement and delayed elastic displacement. The cyclic loading, the immersion period and their interactions significantly decreased the viscous flow of GCS, DMB and TSR; the viscous flow of GCS with cyclic loading rapidly decreased after the initial period whereas that without cyclic loading gradually decreased according the longer storage period. The viscous flows of TSR and DMB were very small (0.00-0.05 mm) and these values were not significant from a clinical point of view.

The total displacement of GCS (1.15-2.88 mm) was twice that of other materials which were 0.68-1.67 mm. The cyclic loading, immersion period and their interaction significantly decreased the total displacement of GCS.

To clarify the initial elastic deformation and following elastic and viscous deformation of soft lining materials, the ratio of delayed elastic displacement and viscous flow to the total displacement was calculated as a delayed deformation. The delayed deformations are indicated in Fig. 6. The delayed deformations with cyclic loading except DMB were smaller than those without cyclic loading. A significant effect of cyclic loading was found regarding GCS, GSS and TSR. Therefore, cyclic loading...
accelerated the decrease of delayed deformations. Moreover, the delayed deformation showed a tendency to decrease with longer storage period.

**Water sorption and solubility**

The results of water sorption and solubility are summarized in Figs. 7 and 8. The cyclic loading slightly increased the water sorption and the solubility of all tested materials. The water sorption increased 33.7-72.8 mg/cm³ (GCS), 11.9-14.6 mg/cm³ (GSS), 6.1-6.4 mg/cm³ (DMB), 3.34-6.30 mg/cm³ (TSR) and 0.24-0.54 mg/cm³ (KCF) after storage. The immersion period significantly increased the water sorption of GCS and TSR. The water sorption of all materials except TSR showed similar values of solubility for identical materials. The water sorption of TSR increased significantly with a longer immersion period. The solubility increased 42.0-72.2 mg/cm³ (GCS), 11.0-14.9 mg/cm³ (GSS), 6.38-7.00 mg/cm³ (DMB), 1.46-1.82 mg/cm³ (TSR) and 0.40-0.59 mg/cm³ (KCF). The immersion period significantly increased the solubility of GCS and GSS.

**DISCUSSION**

Soft lining materials suffer from repeated stress of biting force during mastication. Stress of the complete denture's basal surface during mastication has been reported 49-177 kPa (0.5-1.8 kgf/cm²). Therefore, an amplitude of 196 kPa (2.0 kgf/cm²) was employed as the maximum stress in this study. The frequency of mastication has been reported to be 1.00-1.78 Hz. In addition, an unfavorable effect of heating on the plastic materials was considered when a high frequency of cyclic loading was applied. Therefore, a cyclic loading frequency of 2 Hz was selected in this study. Chewing cycles of complete denture wearers are estimated to be 1,800 cycles per day. Consequently, periods of cyclic loadings were employed for 1.75, 3.5, 7.5 and 22.5 hr which corresponded to denture insertion for 1 week, 2 weeks, 1 month and 3 months, respectively, based on the number of mastication cycles. In addition, the cyclic loading test was performed in 37°C water to mimic the actual oral cavity.
The soft lining materials are expected to absorb, relieve and uniformly distribute the biting force during mastication. These functions are contributed by viscoelastic properties. Penetration depth is often used as a mechanical property test in a specification such as ISO 10139-2:1999. In addition, a hardness test such as the Shore A hardness test has been employed by many researchers. However, these properties do not display the viscoelastic properties. For measuring viscoelastic properties, dynamic and static measurements are suggested. Dynamic measurements are generally performed using a very rapid movement. However, actual mastication movement is not so rapid. Therefore, static viscoelastic measurement is more desirable. The creep-meter is able to measure displacement precisely. Consequently, the creep-meter is suitable for static viscoelastic measurement.

In this study, the water sorption and solubility were evaluated based on volume. Therefore, the water sorption and solubility of the specimens with the cyclic loading increased when these values were calculated based on the surface area due to the coverage of the plunger on the top and bottom surface. The water sorption and solubility obtained in this study increased slightly after cyclic loadings. These findings suggest an increase of water sorption and solubility after cyclic loadings.

Grant et al. reported that DMB was most elastic, GCS was most viscous and GSS was intermediate between these two materials. Wagner et al. suggested that GSS was more elastic and viscous than DMB, and DMB showed no viscosity. Murata et al. reported that acrylic resin materials showed viscoelastic properties and the silicone materials exhibited elastic properties. The viscoelastic properties without cyclic loading in the present study were almost equal to these previous findings. The self-curing acrylic is usually less polymerized than the heat-curing acrylic. Therefore, the mechanical properties of self-curing soft lining materials (GCS) were more affected by the water storage than those of the heat-cured type (GSS). These changes were mostly considered due to loss of plasticizers and unpolymerized monomers. Moreover, additional polymerization during water storage was also possible. As a result, the viscoelastic properties of self-curing acrylic soft lining materials lost a delayed deformation during the water storage. Silicone and isoprene-based soft lining materials do not contain the plasticizers, therefore the water sorption and solubility were small.

Cyclic loading mainly affected the change of height and viscous flow. Moreover, cyclic loading influenced the delayed deformation of soft lining materials. The effects of cyclic loading on metallic materials are well known such as fatigue, while the effect of cyclic loading on some ceramic materials is considered to be negligible. However, the effects of cyclic loading on plastic materials are more complicated. The effects of cyclic loading in the present study were not clearly identified as the effect of loading duration or the number of cyclic loading. The specimens were compressed and recovered during cyclic loading. The residual monomers and plasticizers might be excluded during the compression process and there was a possible uptake of water during the recovery process. Perhaps these two processes caused the degradation of soft lining materials to accelerate. Therefore, the cyclic loading
influenced the viscoelastic properties especially the delayed deformation such as the delayed elastic displacement and the viscous flow of soft lining materials.

The decrease of delayed deformation after cyclic loadings indicated reduced stress distribution effects of soft lining materials. Moreover, the water absorption per surface area was suggested to increase after cyclic loadings. These unfavorable changes were problems of degeneration of soft lining materials. Therefore, evaluation of viscoelastic properties after cyclic loading was effective to evaluate the durability of soft lining materials. However, further research must be done to identify the effect of loading duration and the cyclic effect on soft lining materials.

REFERENCES


