# Mechanical Properties and Bond Strength of Silicone-based Resilient Denture Liners

Koichi FUJII<sup>1</sup>, Hiroyuki MINAMI<sup>2</sup>, Hiroyuki ARIKAWA<sup>1</sup>, Takahito KANIE<sup>1</sup>, Seiji BAN<sup>1</sup> and Mitsuko INOUE<sup>1</sup>

<sup>1</sup>Department of Biomaterials Science, Graduate School of Medical and Dental Sciences, Kagoshima University, 8-35-1 Sakuragaoka, Kagoshima 890-8544, Japan

<sup>2</sup>Department of Fixed Prosthodontics, Graduate School of Medical and Dental Sciences, Kagoshima University, 8-35-1 Sakuragaoka, Kagoshima 890-8544, Japan

Corresponding author, Koichi Fujii E-mail:fuji@dentb.hal.kagoshima-u.ac.jp

Received July 5, 2005/Accepted October 28, 2005

To evaluate the ease of manipulation and durability of 11 commercially available silicone-based resilient denture liners, extrusion force, hardness, weight change, and bond strength were determined. Extrusion force from the cartridge of each material ranged from 0.25 to 1.26 MPa at an extrusion rate of 1 cm/min. Durometer hardness, after set materials were stored in distilled water at  $37^{\circ}$  for one day, ranged from A5.9 to A47.7, and after four weeks their values increased by 4.0 to 275%. Bond strength ranged from 1.01 to 2.88 MPa after set materials were stored in distilled water at  $37^{\circ}$  for one day, but decreased to 0.59 to 1.99 MPa after 10,000 thermal cycles. These results suggested that except for one material, the rest of the evaluated materials exhibited good handling properties – for example, mixing and spreading of material can be done easily. However, some materials exhibited inadequate durability for clinical service, because hardness increased during storage and/or bond strength decreased after thermal cycling.

Key words: Silicone-based denture liner, Mechanical properties, Bond strength

#### INTRODUCTION

Soft denture liners have often been used for longtime denture wearers, who have developed excessive resorption of alveolar bone and localized lesions of alveolar mucosa<sup>1,2)</sup>. This is because soft denture liners are able to relax and/or proportionate masticatory pressure on the supporting tissue, as well as improving denture retention by utilizing their viscoelastic properties<sup>1,3)</sup>.

Based on their chemical structure, soft lining materials can be classified as acrylic-based, siliconebased and fluoroethylene copolymer<sup>4,5)</sup>. In terms of relining procedure, the denture base can either be relined by the direct method (*i.e.*, cured-in-mouth) in the dental clinic, or by the indirect method in the dental laboratory. Although acrylic-based soft denture liners indicate the most marked improvement in masticatory function because they exhibit better viscoelastic properties  $^{6,7)}$ , they have disadvantages such as unpleasant odor and taste, and irritation to the soft tissue inside the mouth $^{8)}$ . These drawbacks are caused by the monomer contained in acrylic-based soft denture liners. As such, they are used only on a short-term or temporary  $basis^{9,10)}$ . On the other hand, silicone-based denture liners have often been used on a long-term basis because they are more resilient and more resistant to aged deterioration than acrylic-based denture liners $^{11-13)}$ . However, several problems have been notably associated with the use of these materials before and after setting. These problems arise from varying degrees of fluidity and consistency<sup>14)</sup>, change in hardness<sup>15)</sup>, water sorption and solubility<sup>16,17)</sup>, oral colonization by Candida albicans<sup>18,19)</sup>, poor tear strength<sup>20)</sup>, and deterioration of bond strength to denture<sup>21,22)</sup>.

Recently, new brands of silicone-based resilient denture liners have been placed on the market. We have reported on the setting characteristics of seven silicone-based resilient denture liners: four new brands versus three established silicone-based liners<sup>14</sup>. Although it is essential that an appropriate brand is selected based on the purpose of clinical usage, it is important too to have a good knowledge of the durability of these materials. The purpose of this study, therefore, was to investigate the physical and mechanical properties and bond strength of new silicone-based resilient denture liners in comparison to seven established silicone-based denture liners, and to clarify the durability of the materials examined.

# MATERIALS AND METHODS

#### Materials

Cartridge-type, silicone-based resilient denture liners used in this study are listed in Table 1, together with their codes, manufacturers, lot numbers, working and setting times, and inorganic substance contents. Each material was auto-mixed and set using a dispenser (Cartridge dispenser II, GC, Tokyo, Japan) and tip (Mixing tip II, S (Pink), GC, Japan) according to manufacturer's instructions. Materials

668

## MECHANICAL PROPERTIES OF SILICONE DENTURE LINER

Material	Code	Manufacturer	Lot No.	Working time	Setting time (min)		Inorganic substance
				(s)	Direct	Indirect	(wt%)
EVATOUCH SUPER NORMAL	ET-SN	$NEO^1$	ER37C III	<90-120	<5	< 30	29.5 (0.14)
EVATOUCH SUPER SLOW	ET-SS	$NEO^1$	ER38C III	<120-180	$<\! 6$	< 30	29.7 (0.12)
PERMAFIX-KOHLERR	PF-K	$KOHLER^{2}$	21102	60-105	5 <	30 <	48.0 (1.51)
RELINE Extra Soft	RL-ES	$\mathrm{GC}^3$	403041	<120	5 <	30 <	41.3 (0.24)
RELINE Soft	RL-S	$\mathrm{GC}^3$	306031	<120	5 <	30 <	58.3 (1.16)
RELINE Ultra Soft	RL-US	$\mathrm{GC}^3$	402051	<120	5 <	30 <	37.5 (0.3)
SOFRELINER MS	SL-MS	TOKUYAMA <sup>4</sup>	48834	60-90	5 <	20 <	41.6 (0.37)
SOFRELINER S	SL-SS	TOKUYAMA <sup>4</sup>	16024	60-90	5 <	20 <	37.3 (0.26)
SOFRELINER TOUGH M	SL-TM	TOKUYAMA <sup>4</sup>	01534	60-90	5 <	20 <	65.8 (2.72)
ZEATY Reliner Soft	ZR-S	Panasonic-dental <sup>5</sup>	402003	60-80	3	4 (35°C)	58.3 (0.76)
ZEATY Reliner Super Soft	ZR-SS	$Panasonic-dental^5$	402004	90-120	3	4 (35℃)	32.8 (1.41)

Table 1 Materials used in this study

<sup>1</sup>NEODENTAL CHEMICAL PRODUCTS CO. LTD., Tokyo, Japan

<sup>2</sup>KOHLER MEDIZINTE CHNIK, Neuhausen, Germany

<sup>3</sup>GC CORPORATION, Tokyo, Japan

<sup>4</sup>TOKUYAMA DENTAL CORPORATION, Tokyo, Japan

<sup>5</sup>Panasonic-dental Co., Osaka, Japan

used are commercially available, including marketable goods such as ET-SN, ET-SS, ZR-S, and ZR-SS. They could be divided into three categories as described in JIS T6520-2000<sup>23)</sup>, *i.e.*, Type A (hard): ET-SN, RL-S, SL-TM, and ZR-S; Type B (medium): PF-K, RL-ES, and SL-MS; and Type C (soft): RL-US, SL-SS, and ZR-SS.

# Extrusion force, extrusion energy and apparent viscosity

Maximum extrusion force and energy - the cumulative work necessary for extruding - were determined when each material was auto-mixed in air at room temperature using the dispenser and tip (inside diameter of tip end: 4.8 mm) in a universal testing machine (Techno Graph TG-50kN, Minebea, Nagano, Japan). Cross-head speeds (i.e., extrusion rates of material) were 1, 2, and 3 cm/min, and distance limit was 8 mm at which the flow condition of each material contained a 4-mm distance that coincided with a constant and maximum extrusion force<sup>13)</sup>. Extrusion energy was obtained by calculating the area under the extrusion force-distance curve. Furthermore, an apparent viscosity  $(\eta_a)$  was calculated from the extrusion force and cross-head speed that produced a steady-state flow condition on the extrusion forcedistance curve. The equation used in the calculation is shown below – a formula interpreted broadly from Hagen-Poiseuille's law<sup>24)</sup>:

$$\eta_{a} = (\pi \text{ pr}^{4})/8\text{QL}$$

where  $\pi$  is the ratio of the circumference to its diameter, p is the applied pressure to material (extrusion force, MPa), r is the radius calculated from the total cross-sectional area of the cartridge (cm), and Q and L are the flow quantity (cm<sup>3</sup>/s) and extruded length (cm) of the material respectively.

# ${\it Hardness}, \ weight \ change \ and \ inorganic \ substance \ content$

(): S.D.

From the start of mixing, Durometer hardness  $(H_{DA})$  – as described in JIS K6253-1997 $^{25)}$  – was measured regularly at stipulated time intervals.

Specimens (20 mm in diameter and 6 mm in height) were prepared using a polyvinyl chloride resin mold and stored in distilled water at  $37^{\circ}$ C for four weeks after setting. At the same time, change of specimen's weight before and after the storage in distilled water was examined using an electronic balance (FR-200MKII, A & D, Tokyo, Japan), and was expressed as a value divided by the superficial area of the specimen.

As for inorganic substance, it was determined by heating the specimen – following its  $H_{DA}$  measurement – inside a porcelain crucible in an electric furnace at 700°C for 30 minutes. Heating was repeated until change of specimen's weight was less than 0.01 g. Inorganic substance content (%) was then given as a weight ratio before and after heating.

## Tensile strength and tear strength

Tensile strength and tear strength were determined at room temperature using a universal testing machine (Techno Graph TG-50kN, Minebea, Nagano, Japan) at a cross-head speed of 254 mm/min. Specimens for tensile test were set in a stainless steel mold with dumbbell shape  $(50 \times 15 \times 1 \text{ mm}; \text{ fractured}$ neck section: 5 mm in width, 8 mm in length) in air at 37°C, and thereafter were stored in distilled water at 37°C for 24 hours. Tensile strength was obtained by dividing the maximum tensile load obtained by the fractured cross-sectional area.

Tear strength was evaluated according to ASTM D1938-92<sup>26,27)</sup>. Specimens for tear test were set in a PTFE mold with rectangular shape  $(75 \times 25 \times 1 \text{ mm})$ .

They were conditioned in the same manner as those of tensile test, and were cut into trouser-shaped strips with a 50-mm long cut prior to testing. Maximum force (F) during tearing at notch tip was recorded in tension at a separation rate of 254 mm/ min, and F was divided by the specimen thickness (d) to give tear strength (Ts=F/d).

# Bond strength

A square rod  $(10 \times 10 \times 35 \text{ mm})$  of PMMA material (Acrylite, Mitsubishi Rayon, Tokyo, Japan) was stored in distilled water at 37°C for 48 hours. An end of the rod was polished with waterproof sandpaper (#180) and dried, and then treated using a primer according to manufacturer's instructions. The #180-grade sandpaper was used because the resultant effect coincides with the average surface roughness that was obtained by polishing PMMA rods with the carbide bur which was packed together with each product in the case.

For each material, six specimens were prepared by filling the material into a 2-mm gap for bonding between the two PMMA rod ends that were butted together using a flame. After setting in air at 37 °C, the specimens were stored in distilled water at 37 °C for 24 hours and divided into two groups prior to testing. One group was used in an adhesion test, and the other group was used in thermal cycling test for 5,000 and 10,000 cycles – whereby specimens were dipped alternately for one minute into each water bath at the temperatures of 4 and  $60^{\circ}$ C. Bond strength in tension of all specimens in both groups was determined using a universal testing machine with a cross-head speed of 20  $mm/min^{21,28,29}$ . Bond strength was calculated by dividing the maximum tensile force by the section area of the specimen applied with a soft liner. Surfaces of the debonded specimens were inspected under  $\times 10$  magnification with a stereoscopic microscope (SMZ-10, Nikon, Kanagawa, Japan) to determine mode of failure: adhesive, cohesive, or mixed mode. Adhesive failure referred to an overall separation at the interface between a soft denture liner and PMMA resin; cohesive failure referred to tearing within a soft denture liner; and mixed failure referred to both.

Three specimens were used in all measurements. Values obtained among the eleven materials for each measurement were compared using one-way analysis of variance (ANOVA) and Fisher's LSD test at a significance level of 0.05. The effects of extrusion rate, storage period and thermal cycling were investigated using Student's t-test (p=0.05).

#### RESULTS

Fig. 1 shows the extrusion force of the eleven materials tested in this study, which ranged from 0.18 to 1.26 MPa at the extrusion rate of 1 cm/min, and

their values increased by 61 to 115% as the extrusion rate increased to 3 cm/min (p<0.05). Extrusion energy (at 1 cm/min) was 86 to 364 N·cm (Fig. 2). Apparent viscosity decreased with increase of extrusion rate, as shown in Fig. 3. Durometer hardnesses  $(H_{DA})$  increased with increasing storage time, and ranged from A19.2 to A56.5 after storage for four weeks (p < 0.05) (Fig. 4). Weight change of the materials after four weeks ranged from 0.13 mg/cm<sup>2</sup> for ZR-SS to 0.56 mg/cm<sup>2</sup> for SL-SS (Fig. 5). Tensile and tear strengths of each material ranged from 1.06 to 4.53 MPa and from 4.3 to 101.7 N/cm respectively, after storage in distilled water at 37°C for one day following setting (Figs. 6 and 7). Bond strength ranged from 1.01 to 2.88 MPa after storage in distilled water for one day, but decreased by 10-63% after 10,000 thermal cycles, except for SL-SS; conversely, that of SL-SS increased by 59% (p<0.05) (Fig. 9). In terms of failure mode, most portions of debonded surfaces demonstrated cohesive failure or adhesive failure, as shown in Fig. 11.

## DISCUSSION

The composition of each material used in this study is not known, because they are commercially available items including some marketable goods. As a result, it was not possible to discuss fully the characteristics essential to clinical practice – namely fluidity, hardness, water absorption, tensile strength and tear strength, and bond strength. Nonetheless, these characteristics were investigated in the light of some key considerations in an oral environment, in particular water storage and temperature change.

Extrusion force, extrusion energy and apparent viscosity

Figs. 1 and 2 show the extrusion force and extrusion



Fig. 1 Extrusion force (MPa) of 11 silicone-based resilient denture liners. Same letter over columns denotes no significant differences among their values of each material (p>0.05).

670



Fig. 2 Extrusion energy (N-cm) of 11 silicone-based resilient denture liners. Same letter over columns denotes no significant differences among their values of each material (p > 0.05).

energy of the eleven materials tested in this study. There was meaningful correlation (r=0.994) between extrusion force and extrusion energy at 1 cm/min. However, as material was extruded, its force or energy was not directly proportional to increase in extrusion rate, but rather increased at a lower rate. This result conformed well to a previous report<sup>14</sup>. All materials used might have thixotropic characteristics such as shear-rate thixotropy<sup>30</sup>, despite the setting process. Nonetheless, it is necessary to investigate this aspect of their behavior in greater detail in the future. However, it could be seen that these materials were non-Newtonian, and exhibited pseudoplasticity (structural viscosity) rather than thixotropy. This was because as shown in Fig. 3, ap-



Fig. 3 Variation of apparent viscosity (MPa·s) with extrusion rate for 11 silicone-based resilient denture liners. There is a significant difference among their values obtained for three extrusion rates of each material (p < 0.05).

parent viscosity tended to decrease as extrusion rate increased. In addition, SL-TM and ZR-S — which included a larger inorganic substance — showed a relatively lower apparent viscosity, which could be attributed to the different type and molecular weight of prepolymer used. Generally, for materials with relatively low apparent viscosity (such PF-K, RL-US, SL-TM, ZR-S, and ZR-SS), they have a high fluidity and can be easily handled and spread on the mucosal surface of denture base. However, in this study, we could not determine quantitatively the influencing factors on fluidity.

#### Hardness

After each set material was stored in distilled water at 37 °C for one day, Durometer hardness ( $H_{DA}$ ) ranged from A5.9 to A47.7. Their values increased with increasing storage time, and after four weeks the range was A19.2 to A56.5 (Fig. 4). Rate of increase in hardness was 4.00 to 275%, and was more than 20% for four materials (RL-ES, RL-US, SL-SS and ZR-SS) that were relatively soft materials classified as type B (medium) or type C (soft) (JIS T6520- $2000^{23}$ ). Variation in hardness increase rate amongst the materials could be attributed to the rate of chemical reaction after setting. In addition to these setting characteristics, ease of sorption or solubility of soft denture liner would have an influence on hardness increase behavior too. This is because when immersed, plasticizers or other soluble materials leached out of the liner material while the interface of matrix resin and inorganic filler particles absorbed water<sup>17)</sup>. The net balance of these leaching and absorbing behaviors then affected the ultimate hardness. Although the hardness measurement method in this study differed from that described in JIS T6520-2000, it is given in the quality standard that hardness change within four weeks should be less than 20%. Moreover in clinical practice, it would be unacceptable for hardness to change too much in a



Fig. 4 Durometer A hardness  $(H_{DA})$  of 11 silicone-based resilient denture liners. Each material was stored in distilled water at 37°C for 4 weeks.

patient's oral cavity. On the other hand, there was an inadequate correlation (r=0.538) between H<sub>DA</sub> and inorganic substance. This suggested that mechanical properties such as H<sub>DA</sub> depended strongly on the type, molecular weight, and crosslink density of the prepolymer used, in addition to the volume and particle size distribution of the filler, although the details were still not known in this study.

Inoue et  $al.^{4)}$  reported on the H<sub>DA</sub> of six soft denture liners, which were stored in air at 37℃ for 24 hours after setting. The values were A2.0-A3.7 for acrylic type, A2.7-A39.5 for silicone type, A36.2 for fluoro type, and A66.2 for olefin type. Further, they measured the hardness of oral soft tissues of seven patients using a trial hardness tester and reported hardness values of about 1-40, taking into consideration the individual variability and thickness of these oral soft tissues<sup>31)</sup>. Besides, hardness values were also obtained for eight tissue conditioners stored in air and water at 37°C for 24 hours, and they were 1.5-7.0 and 2.0-9.0 respectively.  $H_{DA}$  values obtained in this study were slightly higher than those of silicone type reported by Inoue et al., and might be similar or slightly higher than those of oral soft tissues mentioned above. This might be because the different measuring instruments were used for oral soft tissues. However, it was noted that in the study of Inoue *et al.*, hardness values determined by the Durometer and the trial hardness tester were similar for acrylic-type soft denture liners, although different representation units were used in both measuring instruments.

Currently, it remains a question how much hardness is required for a long-term silicone-based denture liner. However, whenever it is used in clinical practice, it is indisputable that the soft lining material helps to stabilize the denture base and improve its occlusion<sup>32,33)</sup>. Therefore a functionally gradated denture – which possesses the characteristic of increasing hardness value from the tissue surface to the palatine surface of denture – would be recommended for providing comfort to denture-wearing patients.

### Weight change

Weight change of the materials examined, except SL-MS, increased with increase of storage time (p<0.05), and their values after four weeks ranged from 0.13 mg/cm<sup>2</sup> for ZR-SS to 0.56 mg/cm<sup>2</sup> for SL-SS (Fig. 5). Presently, there is no JIS (Japanese Industrial Standard) specification for water absorption of soft denture liners (JIS T6520-2000). However, if JIS specification for acrylic denture base resins (JIS T6501-1993)<sup>34)</sup> were to be used as a guide, sorption value after seven days should not be more than 32  $\mu$ g/mm<sup>3</sup> (0.784 mg/cm<sup>2</sup>). Although water absorption of the eleven materials obtained in this study conformed to JIS (T6501-1993) specification for



Fig. 5 Weight change (mg/cm<sup>2</sup>) of 11 silicone-based resilient denture liners. Each material was immersed for 30 min after start of mixing in distilled water at 37℃, and then stored for 4 weeks.

acrylic denture base resins and was similar to the values of some studies $^{35-37)}$ , it might be unacceptable for soft denture liners. This is because an increasing trend in weight change, most probably caused by water absorption into the material, would definitely lead to swelling, distortion, coloring, stench, hardening, debonding, and bacterial habitation. Weight change for SL-MS showed a decreasing trend as storage time increased. It could probably be due to loss of either filler or a leachable material, such as plasticizer loss in the case of acrylic materials. Thus weight change of a soft denture liner, which could be caused by high-level water sorption and/or solubility, would influence its physical and mechanical properties, hardening behavior, and bond strength.

#### Tensile strength and tear strength

Like rubber materials, tensile and tear properties are fundamental properties of silicone-based resilient denture liners. Tensile strength provides information on the ultimate strength of a material in tension, whereas elongation gives the degree of deformation a material can be subject to until it breaks. Tear strength, on the other hand, provides data on the ability of a material to resist the progress of failure at the site of stress concentration. Together, tensile and tear properties serve as an important guide to clarify the mechanism of a debonding mode between soft denture liner and denture base resin, although only compressive and/or shear stresses are built up in the soft denture liner during occlusion by a denture.

Figs. 6 and 7 show the tensile strength and tear strength of the eleven materials examined, which were stored in distilled water at  $37 \,^{\circ}$ C for one day after setting. Tensile and tear strengths ranged from 1.06 to 4.53 MPa and from 4.3 to 101.7 N/cm respectively, and were similar to the values of several studies<sup>20,38,39</sup>. ET-SN, RL-S, SL-TM, and ZR-S were

672



Fig. 6 Tensile strength (MPa) of 11 silicone-based resilient denture liners. All materials were immediately immersed in distilled water at 37℃ after setting and stored for 1 day. Same letter over columns denotes no significant differences among their values of each material (p>0.05).

classified as hard- or medium-type materials, and their tensile strength values were 2.5-4.3 times greater than that of ZR-SS, which exhibited the lowest tensile strength amongst the soft-type materials (PF-K, RL-US, SL-SS, and ZR-SS). Indeed, the tensile strength of ZR-SS was approximately half of that of PF-K, RL-US, and SL-SS. As shown in Fig. 7, tear strength of SL-TM was the highest whereas that of ZR-SS was the lowest. It can be seen that the tear strengths of ET-SN, ET-SS, and SL-TM were 14.8-23.7 times greater than that of ZR-SS. Further, for both tensile strength and tear strength, only RL-ES showed relatively larger values amongst all the other materials.

Fig. 8 shows the typical external appearances of



Fig. 8 Typical failure mode of the materials after tearing.



Fig. 7 Tear strength (N/cm) of 11 silicone-based resilient denture liners. All materials were immediately immersed in distilled water at 37°C after setting and stored for 1 day. Same letter over columns denotes no significant differences among their values of each material (p>0.05).

some specimens after tearing, where arrows indicate the starting point of failure. For RL-S, SL-MS, ZR-S, and ZR-SS, which were of relatively lower tear strength, the fracture direction was almost on the extension line of the 50-mm long cut line of the trouser-shaped strips. As for ET-SN, ET-SS, RL-ES, and SL-TM, which were of higher tear strength, complicated fracture pattern was obtained. The difference could arise from differences in chemical structure, such as the degree of crosslinking and prepolymer type. In this study, it was found that both tensile and tear strengths had a fairly positive correlation (r=0.520). Besides, the correlation coefficient between  $H_{DA}$  and tensile strength was an adequate r=0.723, whereas that between  $H_{\text{DA}}$  and tear strength was merely r=0.391. However, fracture mechanism of tearing specimens is not still known, further investigation is necessary.

### Bond strength

In clinical practice, failure of silicone-based resilient denture liners is often attributed to a breakdown of the bonding between soft denture liner and denture base resin. Thus, a soft denture liner must not only provide the requisite strength for bonding with denture, but also acceptable durability of its bonding. Adhesion test was used in this study, and the mode of failure was evaluated. This was an efficient method for ranking the bonding capability of each material because tensile force was directly applied in a direction perpendicular to the bonding area between both materials, although it might not simulate the clinical forces that induce separation between soft denture liner and denture base resin.

Fig. 9 shows the bond strength of the specimens



Fig. 9 Bond strength (MPa) of 11 silicone-based resilient denture liners. All materials were immediately immersed in distilled water at 37°C after setting and stored for 1 day. Then, they were thermocycled in water baths at temperatures of 4 and 60°C for 5,000 and 10,000 cycles.

that were stored in distilled water at  $37^{\circ}$ C for one day, and which were subjected to 5,000 and 10,000 cycles in a thermal cycling test. Bond strength ranged from 1.01 to 2.88MPa after storage in distilled water for one day, and showed a decreasing trend as the number of thermal cycles increased, except for SL-SS. In particular, bond strength values decreased by 10-63% after 10,000 cycles, whereas that of SL-SS increased by 59%. The results of this study were similar to or slightly higher than those obtained before and after thermal cycling by Kulak-Ozkan *et al.*<sup>22)</sup>. although a direct comparison of both studies could not be made because of differing test conditions, such as specimen size, cross-head speed, and temperature of thermal cycling. If a bond strength of 4.5 kgf/  $\rm cm^2~(0.44~MPa)$  as already  $\rm reported^{22,28,29)}$  is considered an acceptable criterion in clinical practice, then all the materials examined in this study would have a sufficient bond to PMMA resin.

Thermal cycling had some adverse effect on the bond strength of the materials examined, except for SL-SS. A significant reduction in bond strength after thermal cycling was due to water absorption as shown in Fig. 5, thereby leading to swelling and consequent stress buildup at the bonding interface. Otherwise, loss of a leachable material caused the viscoelastic properties of soft denture liners to change, hence making the materials stiffer. Coefficients of correlation between bond strength and tensile strength, and tear strength, and  $H_{\text{DA}}$  were 0.805, 0.479, and 0.902 respectively, which were estimated when materials were stored in distilled water at 37  $^{\circ}$ C for one day after setting (Fig. 10). Meaningful correlation among bond strength, tensile strength, and  $H_{DA}$  implied that the inherent mechanical properties of soft denture liners directly affected the bond



Fig. 10 Interrelation among bond strength, tensile strength and Durometer hardness.



Fig. 11 Percentage of cohesion failure (%) after adhesion test for 11 silicone-based resilient denture liners. A 0% denotes an adhesion failure. Bar at column top shows the standard deviation.

between soft denture liner and PMMA resin, although the primers used for adhesion took a substantial role in bonding both materials at the interface. It can therefore be said that bond strength to PMMA resin depended on the balance between the bonding capability of the primer and the magnitude of mechanical properties mentioned above.

Fig. 11 shows the failure modes after debonding. For the debonded surfaces of ET-SN, ET-SS, RL-S, SL-MS, and ZR-SS, cohesive failure occupied a greater percentage. As for PF-K, RL-ES, RL-US, SL-TM, and ZR-S, adhesive failure occupied the most part. Thus, in this study, materials which were chiefly of cohesive failure behavior had a bond strength that was more than 2 MPa, except SL-TM and ZR-SS (Fig. 10). SL-TM possessed a bond 674

strength of 2.22 MPa and relatively larger values of tensile and tear strengths and  $H_{DA}$  (as compared with the others), but showed a larger percentage of adhesive failure. Conversely, ZR-SS possessed bond strength of 1.21 MPa and relatively lower values of the mechanical properties mentioned above, but showed a large percentage of cohesive failure. Thus, in addition to inherent mechanical properties, various failure modes might occur due to differences in bonding capability at the interfaces among soft denture liner, primer, and PMMA resin as an adherend. Indeed, more detailed investigation is required.

Further, the fracture mode of all materials tended to lean toward adhesive failure as the number of thermal cycles increased, except RL-US and SL-SS. It could be because water directly infiltrated the bond site, thereby leading to swelling and consequent stress buildup at the interfaces of soft denture liner, primer, and PMMA resin, as stated previously. After storage in distilled water at 37°C for one day, RL-US and SL-SS exhibited a large percentage of adhesive failure, but were altered toward cohesive failure after 10,000 thermal cycles. This could be explained by a slow and mild chemical reaction proceeding at the bond site. As conjectured by an increase of  $H_{DA}$  in Fig. 4, it could be supposed that the bond strength at the bonding site between soft denture liner and primer was increased by the hardening of soft denture liner. Hence, as the chemical reaction proceeded by itself, the region of cohesive failure became larger.

Fluidity, hardness, weight change, tensile strength, tear strength, and bond strength were examined in this study, and their properties were different among the eleven soft denture liners evaluated. Based on the results of this study, only one material could not sufficiently satisfy all the requisite properties demanded of soft denture liners. Although selection of soft denture liner cannot be based on any single property in clinical practice, selection of the material should also be considered in the light of the situation being used.

## CONCLUSIONS

In the present study, the physical and mechanical properties, as well as the bond strength of 11 silicone-based resilient denture liners were investigated. Results obtained were as follows:

1. All materials examined showed a pseudoplastic behavior.

2. Both Durometer hardness (H<sub>DA</sub>) and weight change increased with increasing storage time in distilled water at 37°C for up to four weeks. Their values after four weeks ranged from A19.2 (ZR-SS) to A55.5 (RL-S) for H<sub>DA</sub>, and from 0.131 (ZR-SS) to 0.557 (SL-SS) mg/cm<sup>2</sup> for weight change. Rate of H<sub>DA</sub> increase was more than 20% for four materials (RL-ES, RL-US, SL-SS and ZR-SS).

3. Tensile strength and tear strength, after storing in distilled water at 37°C for one day, ranged from 1.06 (ZR-SS) to 4.53 (RL-S) MPa, and from 4.3 (ZR-SS) to 101.7 (SL-TM) N/cm, respectively. Mutual relationship between tensile strength and tear strength was not established in this study.

4. Bond strength, after storing in distilled water at 37°C for one day, ranged from 1.01 (SL-SS) to 2.88 (RL-S) MPa. Debonded surfaces exhibited two failure modes: adhesive and cohesive failures. After 10,000 thermal cycles, bond strength decreased by 10 to 63% — except for SL-SS, and failure mode was also altered to mixed failure.

5. Adequate correlation was obtained among bond strength, tensile strength, and  $H_{\rm DA}.$  Coefficient of correlation between bond strength and tensile strength was 0.805, and that between bond strength and  $H_{\rm DA}$  was 0.902.

#### ACKNOWLEDGEMENTS

The authors would like to thank NEO Dental Chemical Products Co. and Panasonic Dental Co. for generously providing the materials used in this study.

# REFERENCES

- Kawano F, Ohgri T, Koraniii A, Matsumoto N, Uchikawa T. Influence of lining design of three processed soft denture liners on cushioning effect. J Oral Rehabil 1999; 26: 962-968.
- Taguchi N. A clinical case report of complete dentures using resilient denture liners. J Jpn Prosthodont Soc 2004; 48: 811-814.
- Murata H, Taguchi N, Hamada T, McCabe JF. Dynamic viscoelastic properties and the age changes of long-term soft denture liners. Biomaterials 2000; 21: 1421-1427.
- 4) Inoue K, Itonaga A, Tsuruda H, Terao T, Yamashita H, Tsukada G, Oku J, Arikawa H, Kanie T, Fujii K. Hardness and elastic modulus in compression of denture base soft lining materials determined by a modified experimental hardness tester. JJ Dent Mater 1993; 12: 346-351.
- Braden M, Wright PS, Parker S. Soft lining materials

   A review. Eur J Prosthodont Rest Dent 1995; 3: 163-174.
- Murata H, Taguchi N, Hamada T, Kawamura M, McCabe JF. Dynamic viscoelasticity of soft liners and masticatory function. J Dent Res 2002; 81: 123-128.
- Tamura F, Suzuki S, Mukai Y. An evaluation of the viscoelastic characteristics of soft denture liners. J Prosthodont 2002; 11: 270-277.
- Okita N, Hensten-Pettersen A. In vitro cytotoxicity of tissue conditioners. J Prosthet Dent 1991; 66: 656-659.
- 9) Harrison A. Temporary soft lining materials. A review of their uses. Br Dent J 1981; 151: 419-422.
- 10) Park S -K, Lee Y -K, Lim B -S, Kim C -W. Changes

#### FUJII et al.

in properties of short-term-use soft liners after thermocycling. J Oral Rehabil 2004; 31: 717-724.

- 11) Jin C, Nikawa H, Makihira S, Hamada T, Furukawa M, Murata H. Changes in surface roughness and color stability of soft denture lining materials caused by denture cleansers. J Oral Rehabil 2003; 30: 125-130.
- 12) Kanie T, Terao T, Arikawa H, Kadokawa A, Hamano T, Fujii K, Inoue K. Deterioration of the soft lining materials with the denture cleansers. J Jpn Prosthodont Soc 1992; 36: 488-495.
- 13) Dınçkal Yanikoğlu N, Yeşil Duymuş Z. Comparative study of water sorption and solubility of soft lining materials in the different solutions. Dent Mater J 2004; 23: 233-239.
- 14) Fujii K, Arikawa H, Kanie T, Ban S, Inoue M. Setting characteristics of silicone-based resilient denture liners. Dent Mater J 2004; 23: 566-571.
- 15) Parr GR, Rueggeberg FA. In vitro hardness, water sorption, and resin solubility of laboratory-processed and autopolymerized long-term resilient denture liners over one year of water storage. J Prosthet Dent 2002; 88: 139-144.
- McCabe JF. A polyvinylsiloxane denture soft lining material. J Dent 1998; 26: 521-526.
- 17) Waters MG, Jagger RG, Winter RW. Water absorption of (RTV) silicone denture soft lining material. J Dent 1996; 24: 105-108.
- 18) Nikawa H, Jin C, Makihara S, Egusa H, Hamada T, Kumagai H. Biofilm formation of Candida albicans on the surfaces of deteriorated soft denture lining materials caused by denture cleansers *in vitro*. J Oral Rehabil 2003; 30: 243-250.
- 19) Bulad K, Taylor RL, Verran J, McCord JF. Colonization and penetration of denture soft lining materials by Candida albicans. Dent Mater 2004; 20: 167-175.
- 20) Dınçkal Yanikoğlu N, Denızoğlu S. An investigation of the tear energy of five soft lining materials. Dent Mater J 2003; 22: 444-451.
- Al-Athel M, Jagger R, Jagger D. Effect of ageing on the bond strength of a permanent denture soft lining material. J Oral Rehabil 2002; 29: 992-996.
- 22) Kulak-Ozkan Y, Sertgoz A, Gedik H. Effect of thermocycling on tensile bond strength of six siliconebased, resilient denture liners. J Prosthet Dent 2003; 89: 303-310.
- Japanese Industrial standard, JIS T6520-2000: Longterm soft lining materials for removable dentures.
- Nakagawa T. Rheology, 2nd ed, Iwanami Zensyo, Tokyo, 1978, pp. 80.

- 25) Japanese Industrial standard, JIS T6253-1997: Hardness testing methods for rubber, vulcanized or thermoplastic.
- 26) ASTM Designation, D1938-02: Standard test method for tear-propagation resistance (trouser tear) of plastic film and thin sheeting by a single-tear method.
- 27) Lai JH, Wang LL, Ko CC, Delong RL, Hodges JS. New organosilicon maxillofacial prosthetic materials. Dent Mater 2002; 18: 281-286.
- 28) Kawano F, Dootz ER, Koran A, Craig RG. Comparison of bond strength of six soft denture liners to denture base resin. J Prosthet Dent 1992; 68: 368-371.
- 29) Hong G, Murata H, Hamada T. Relationship between plasticizer content and tensile bond strength of soft denture liners to a denture base resin. Dent Mater J 2004; 23: 94-99.
- Nakagawa T. Rheology, 2nd ed, Iwanami Zensyo, Tokyo, 1978, pp.103-105.
- 31) Inoue K, Itonaga A, Tsuruda H, Arikawa H, Kanie T, Fujii K, Hamano T, Kishita C, Nagaoka E. Comparison of the hardness of tissue conditioners and oral soft tissue measured by a modified hardness tester. Dentistry in Japan 1993; 30: 374-378.
- 32) Shim JS, Watts DC. An examination of the stress distribution in a soft-lined acrylic resin mandibular complete denture by finite element analysis. Int J Prosthodont 2000; 13: 19-24.
- 33) Hayakawa I, Hirano S, Takahashi Y, Keh ES. Changes in the masticatory function of complete denture wearers after relining the mandibular denture with a soft denture liner. Int J Prosthodont 2000; 13: 227-231.
- 34) Japanese Industrial standard, JIS T6501-1993: Acrylic denture base resins.
- 35) Kawano F, Dootz ER, Koran A, Craig RG. Sorption and solubility of 12 soft denture liners. J Proshet Dent 1994; 72: 393-398.
- 36) Hayakawa I, Keh E -S, Morizawa M, Muraoka G, Hirano S. A new polyisoprene-based light-curing denture soft lining material. J Dent 2003; 31: 269-274.
- 37) Muraoka G, Takahashi H, Hayakawa I. Effects of cyclic loading on viscoelastic properties of soft lining materials. Dent Mater J 2003; 22: 251-261.
- 38) Waters MGJ, Jagger RG. Mechanical properties of an experimental denture soft lining material. J Dent 1999; 27: 197-202.
- 39) McCabe JF, Carrick TE, Kamohara H. Adhesive bond strength and compliance for denture soft lining materials. Biomaterials 2002; 23: 1347-1352.