STRAIN CHANGES OF ACRYLIC RESIN SPECIMENS CURED BY THREE CURING CYCLES

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The acrylic resin was first introduced as denture base materials in 1937 and it is commonly used for denture base fabrication nowadays. Three different curing cycles (Conventional curing cycle, short curing cycle and long curing cycle) and three commercially available heat-activated acrylic resins (Vertex RS, Lucitone 199 and ProBase Hot) were investigated to find the curing cycle and material that showed the minimum shrinkage of the resin during polymerization process.
A brass master mold was fabricated and duplicated by additional silicone impression material. Stone molds were made by pouring of type Ⅲ dental stone (SILKY-ROCK YELLOW, Whip-Mix, Louisville, Kentucky). It was embedded in the flask. Strain gauge and thermocouple were embedded in the specimen. Strain gauge and thermocouple were connected to signal conditioning amplifier and data was recorded by pre-programmed software. The parameters ESmax (Maximum expansion strain), Sb (Strain measured just before deflasking procedure), Sa (Strain measured just after deflasking procedure) and Sf (Strain measured at the end of the experiment) were measured. ΔS was calculated from Sb and Sa (ΔS=Sb-Sa). In the experiment concerned about materials, the parameters 90-ESmax (Maximum expansion strain measured during early 90 minutes of curing procedure), 180-ESmax (Maximum expansion strain measured from 90 minutes to 180 minutes), Sb, Sa, ΔS and Sf were measured and the following conclusions were made.

1. The ESmax value of conventional curing cycle showed the largest value and the 180-ESmax value of Lucitone 199 showed the smallest value. 90-ESmax values showed no significant difference ($p<0.05$).
2. ΔS values of conventional curing cycle showed the positive values. ΔS values of short curing cycle and long curing cycle showed the negative values. All three materials cured by conventional curing cycle showed the positive values.
3. The Sf values of long curing cycle and ProBase Hot (cured by conventional curing cycle) showed the smallest values.

Key Words
Acrylic resins, Strain gauge, Curing cycle

Acrylic resin was first introduced as denture base material in 1937.[1] Various denture base materials[2-5] and processing methods[6-8] have been studied and developed in recent years. The heat-activated acrylic resin is commonly used for denture base fabrication thanks to its esthetic and physical properties and plastic manipulability.[9,10] But the heat-activated acrylic resin shows inevitable polymerization shrinkage. The volumetric shrinkage of the heat-activated acrylic resin is about 8%. But it will be distributed uniformly over all surfaces of the denture, so the fit of the denture to the master cast is not so seriously affected. The volumetric shrinkage, due to polymerization contraction, probably contributes very little to the linear shrinkage. It appears that thermal shrinkage of the resin is the chief contributor to the linear shrinkage phenomenon. The linear shrinkages of various denture base materials are reported as from 0.2 to 0.5 %.[11]

There are some discrepancies between the processed denture base and the master cast because of polymerization shrinkage. One of the principal factors contributing to denture retention is the adhesive action of a thin film of saliva between the palate and the fitting surface of the denture. The capillary forces of the salivary film are at a maximum when the distance between the denture surface and the basal seat is at a minimum.[12] Previous studies focused on dimensional changes of the processed acrylic resin. Takamata et al.[13] reported the adaptation of processed acrylic resin dentures by measuring gap between the processed denture and the master cast using the micrometer-slide measuring microscope. Sykora et al.[14] studied the adaptation of denture bases by the traveling microscope.

The strain gauge method is excellent in measuring linear shrinkages of materials. Real-time measurement of the linear shrinkage is possible during polymerization process. Kawara et al.[15] reported the shrinkage behavior of heat-cured resin with different processing methods by the strain gauge method. This study revealed that the shrinkage of heat-cured resin was mainly thermal shrinkage and low-temperature curing method showed average 64% shrinkage of that in the specimen processed by the conventional method.

In this study, three different curing cycles and three commercially available heat-acrylic resins cured by conventional curing cycle were investigated. The purpose of this study was to find the curing cycle and material that could minimize the shrinkage of acrylic resin during polymerization process.

MATERIAL AND METHODS

1. Curing cycles and materials

The curing cycles and the materials used in the experiment were listed in Table Ⅰ, Ⅱ.

The powder consists of poly (methyl methacrylate) and initiator and liquid contains monomer and activator. All tested materials were mixed according to the manufacturer' s instructions.

2. Mold fabrication

A brass master mold with an inner butt-joint cavity was made. Outer dimensions of the cavity were 40.0mm(length), 25.0mm(width) and 5.0mm(depth) as illustrated in Fig. 1.

Two side troughs were made to install a strain gauge and a thermocouple within the cavity. All ax-

Table Ⅰ. Curing cycles used in the experiment

Curing cycle	Curing temperature and time	Cooling
Conventional	90min. at 70℃ + 90min. at 100℃	Bench-cooling for 4hr. to room-temperature
Short	Insert flask in a boiling water(100℃) and curing for 20min.	
Long	24hr. at 70℃	

Table Ⅱ. Types, manufacturers and mixing ratio of three heat-cured acrylic resins

Resin	Composition	Manufacturer	Mixing ratio
Vertex RS	PMMA	Dentimex	3 (powder) : 1 (monomer) by vol.
Lucitone 199®	PMMA	Densply TRUBYTE	26g (powder) : 8ml (monomer)
ProBase Hot	PMMA	Ivoclar	22.5g (powder) : 10ml(monomer)

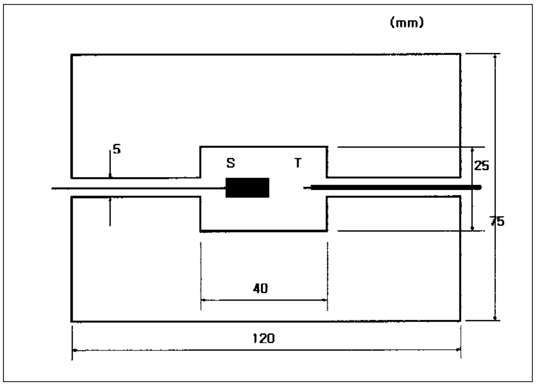

Fig. 1. Schematic illustrations of the prepared cavity in the brass master mold with the locations of sensors within the cavity.
S: Strain gauge, T: Thermocouple

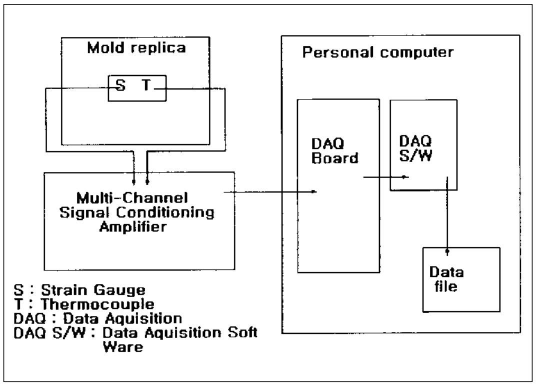

Fig. 2. Block diagram for computerized strain and temperature measurement system.

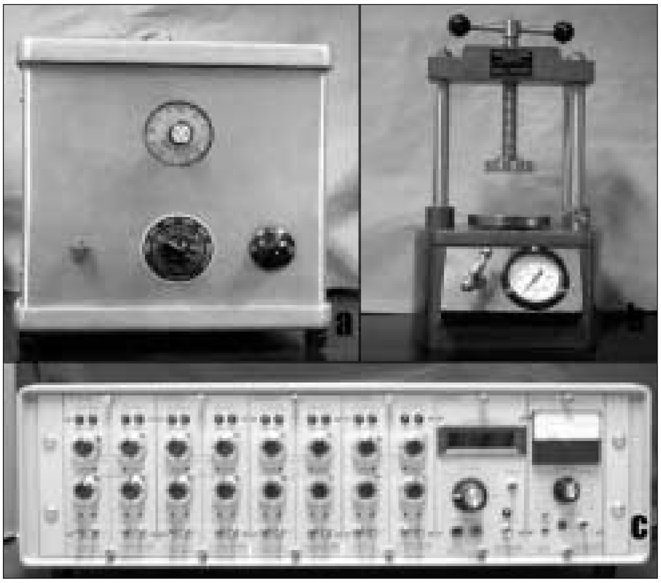

Fig. 3. Photographs of equipment used in the experiment.
(a) Controllable waterbath(Hanau Junior Curing Unit, Model No. 76-0, Teledyne Hanau, New York, U.S.A.)
(b) Oil-press (OSUNG IND. CO., Korea)
(c) Multi-channel signal conditioning amplifier (STRAIN GAUGE CONDITIONER 2120B, INSTRUNET DIVISION, Wendell, Atlanta, U.S.A.)

ial surfaces of a cavity had a 10-degree taper to ensure easy removal of the specimens after polymerization process. A polyvinyl siloxane (Exaflex, GC Inc., Tokyo, Japan) impression of the brass master mold was taken to make stone replicas. After the impression material set, the stone mold replicas were made by pouring of type Ⅲ dental stone (SILKY-ROCK YELLOW, Whip-Mix, Louisville, Kentucky). 50 stone molds and stone lids were made. 30 stone molds were made for the comparison of the curing cycle and 20 stone molds for the comparison of materials. Stone molds and stone lids were highly polished by using up to the 1000-grit sand paper. Each stone mold and stone lid was trimmed for embedding in the flask for upper denture. Each stone mold was embedded in a lower half of a flask by using plaster. After plaster set, excess was trimmed and plaster was highly polished using up to 1000-grit sand paper. Petroleum jelly was applied to surface of stone molds, stone lids. Stone lids were located on stone molds and plaster was poured in an upper half of the flask. So stone molds were embedded in a lower half of the flask and stone lids were embedded in an upper half of the flask.

3. Data acquisition system for strain and temperature measurement

A block diagram and actual strain and temperature measurement system were depicted in Fig. 2, 3.

A strain gauge (AE-11-S80N-120-EC, CAS Inc., Korea) and a thermocouple (K-type) were connected to the signal conditioning amplifier(Fig. 3c). Then, the analogue signals from the amplifier were collected and transformed into the digital signals by an analog-digital converting equipment (Instrunet 100B, INSTRUNET DIVISION, Wendell, Atlanta, U.S.A.). Digital data were recorded by pre-programmed software (DASYLab Ver5.5, National Instrument Inc., Austin, Texas U.S.A).

4. Strain and temperature measuring procedures

Before mixing pre-measured monomer and polymer, every exposed surface of stone mold was thoroughly coated with petroleum jelly as a separating medium to ensure as unhindered shrinkage of resin as possible. A strain gauge and a thermocouple were positioned on the bottom surface of the cavity as depicted in Fig. 1.

In the first experiment, three curing cycles listed in Table Ⅰ were used. In all curing cycles, Vertex RS was used. It was mixed according to the manufacturer`s instruction. After mixing, that was deposited in an airtight container. After 10 minutes of resin mixing, resin specimen was reached the dough stage. Resin specimen was trial-packed by oil-press (Fig. 3b) with 40kg/cm^2. The excess of resin was removed and the flask was clamped. The flask was submerged in a curing unit (Fig. 3a). Then the polymerization process was started according to each cycle and measurement was also started. After the polymerization process, the flask was bench-cooled to room temperature for 4 hour. Deflasking procedure was done and data was recorded more 15 minutes. In each procedure, data was collected 1-minute interval except in the short curing cycle. In the short curing cycle, 10-second interval monitoring was done in the curing stage.

In the second experiment, resin specimens were prepared from three commercially available heat-cured acrylic resins listed in Table Ⅱ. The conventional curing cycle was used in all materials. Cooling and deflasking procedure were identical to the first experiment.

5. Strain gauge calibration

The signal conditioning amplifier (Fig. 3c) used in this study has an internal shunt calibration circuit. Therefore, additional procedures for strain gauge calibration were not necessary. The strain gauges used in this study had no absolute temperature compensating capability. But, the strain changes for temperature range between 0℃ and 100℃ were less than -50$\mu\varepsilon$ as noted in its technical data sheet offered by the manufacturer. So no thermal compensation for measured strain data was done.

6. Thermocouple calibration

Thermocouple was submerged in 5 known temperature and each data was recorded. By statistical method linear regression, temperature was presented as a function of voltage data. Measured thermocouple voltage data was converted to temperature data.

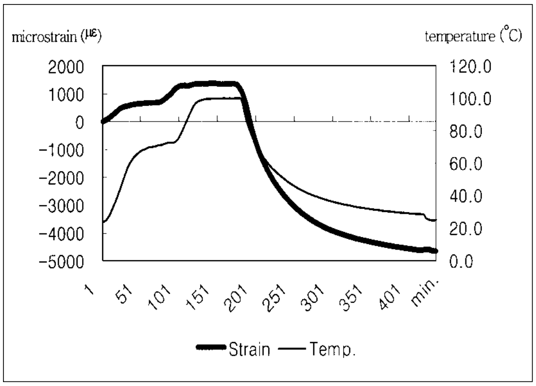

Fig. 4. The strain and temperature curves of conventional curing cycle.

7. Statistical analysis

The significant differences were examined by one-way analysis of variance (ANOVA) and the exact source of the differences was identified by the Duncan`s multiple range tests.

RESULTS

A strain and temperature curve for each curing cycle was shown in Fig. 4, 5, 6.

The origin of the horizontal axis corresponds to the moment at which flask is submerged in the curing unit. In the first experiment, the parameter ESmax, Sb, Sa, Sf and ΔS were measured. The parameter ESmax was the maximum expansion strain measured during polymerization process. The parameter Sb was the strain data measured just before deflasking procedure and Sa was data measured just after deflasking procedure. The parameter ΔS was calculated from Sb and Sa (ΔS=Sb-Sa). The parameter Sf (Final strain) was the strain measured at the end of experiment. Measured parameters were shown in Table Ⅲ.

The unit for strain value is microstrain ($\mu\varepsilon$). Measured ESmax for each curing cycle was depicted in Fig. 7.

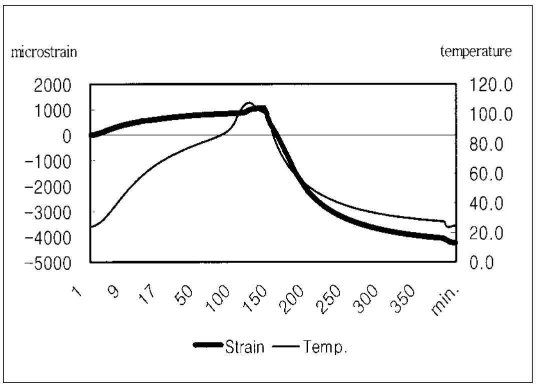

Fig. 5. The strain and temperature curves of short curing cycle.

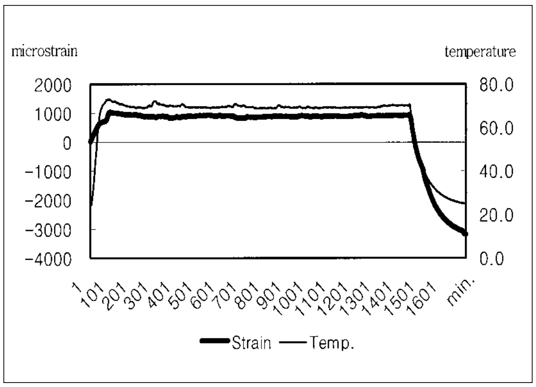

Fig. 6. The strain and temperature curves of long curing cycle.

Table Ⅲ. Mean measurements of strain parameters in the experiment($\mu\varepsilon$)

Curing cycle	n	ESmax	Sb	Sa	ΔS	Sf
Conventional	10	1509	-4631	-4582	49	-4660
Short	10	1158	-4099	-4112	-13	-4244
Long	8	1178	-3081	-3085	-4	-3183

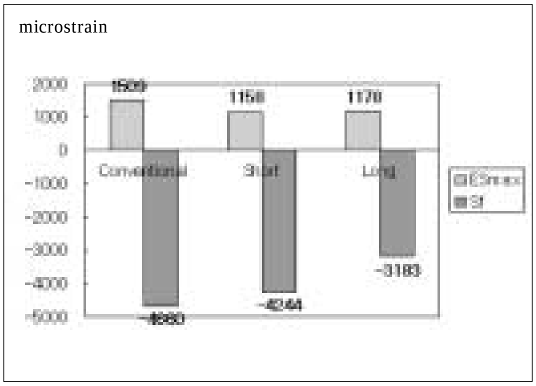

Fig. 7. Mean ESmax and Sf values for each curing cycle.

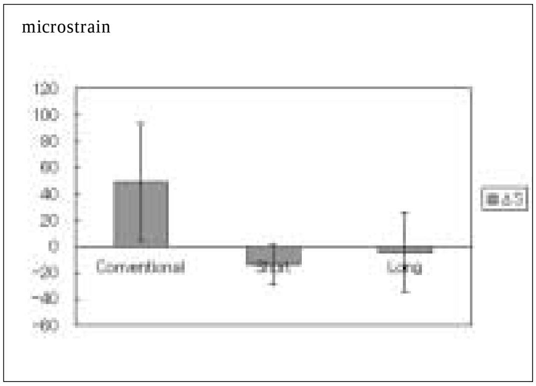

Fig. 8. Mean ΔS values of each curing cycle. ΔS value is the difference between Sb and Sa.

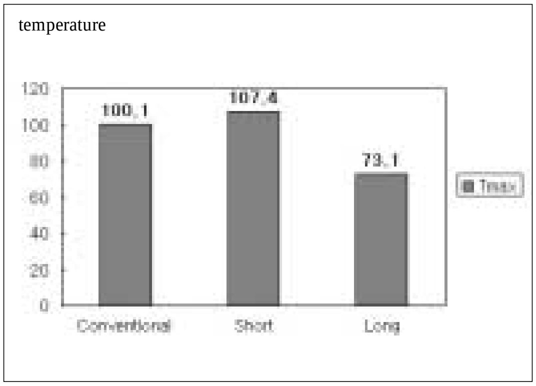

Fig. 9. Mean Tmax values of each curing cycle. Tmax means the maximum temperature measured during curing process.

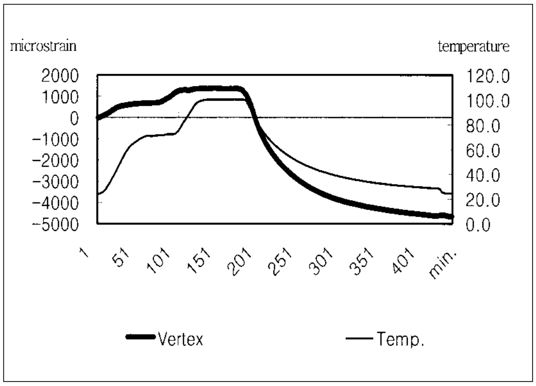

Fig. 10. The strain and temperature curves of the material Vertex RS.

The mean values of ESmax were 1509$\mu\varepsilon$, 1158$\mu\varepsilon$ and 1178$\mu\varepsilon$ for conventional curing cycle, short curing cycle and long curing cycle, respectively. Conventional curing cycle showed significantly higher the maximum expansion strain than the other two curing cycle ($p<0.05$). The mean values of Sb were -4631$\mu\varepsilon$, -4099$\mu\varepsilon$ and -3081$\mu\varepsilon$ for each curing cycle. Each value was significantly different and long curing cycle showed the lower shrinkage strain and conventional curing cycle showed the higher shrinkage strain ($p<0.05$). The mean values of Sa were -4582$\mu\varepsilon$, -4112$\mu\varepsilon$ and -3085$\mu\varepsilon$ for each cy-

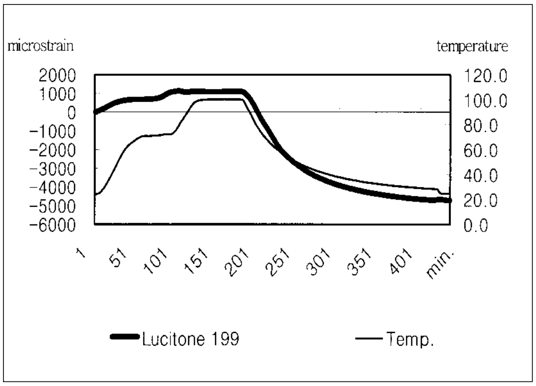

Fig. 11. The strain and temperature curves of the material Lucitone 199.

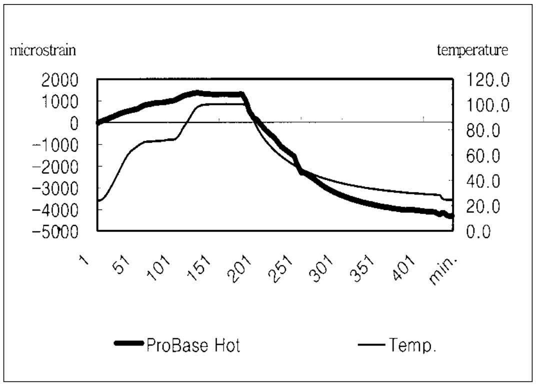

Fig. 12. The strain and temperature curves of the material ProBase Hot.

Table Ⅳ. Mean measurements of strain parameters in the experiment($\mu\varepsilon$)

Materials	n	90-ESmax	180-ESmax	Sb	Sa	ΔS	Sf
Vertex RS	10	972	1509	-4631	-4581	50	-4660
Lucitone 199	10	993	1124	-4708	-4655	53	-4722
ProBase Hot	8	995	1484	-4299	-4157	142	-4308

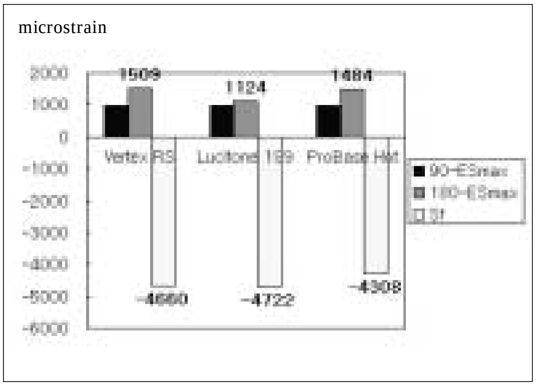

Fig. 13. Mean 90-ESmax, 180-ESmax and Sf values of each material.

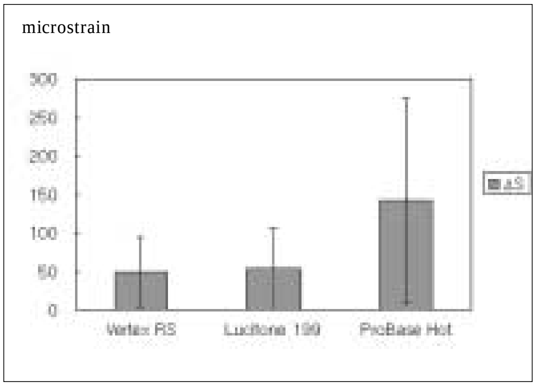

Fig. 14. Mean ΔS values of each material. ΔS value is the difference between Sb and Sa.

cle. Each value was also significantly different and long curing cycle showed the lower shrinkage strain ($p<0.05$). The mean values of ΔS were 49$\mu\varepsilon$, -13$\mu\varepsilon$ and -4$\mu\varepsilon$ for each cycle and depicted in Fig. 8. Positive strain changes were found in conventional curing cycle and negative strain changes were found in short and long curing cycles. Long curing cycle and short curing cycle showed no significant difference ($p<0.05$). The mean Sf values were -4660 $\mu\varepsilon$, -4244$\mu\varepsilon$ and -3183$\mu\varepsilon$. It was depicted in the Fig. 7. Long curing cycle showed the lower shrinkage strain and the each mean value was significantly different ($p<0.05$).

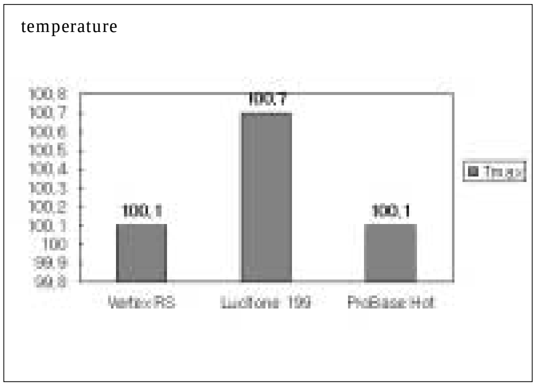

Fig. 15. Mean Tmax values of each material.

The parameter Tmax means the maximum temperature during the polymerization process and it was shown in Fig. 9. The mean values of Tmax were 100.1℃, 107.4℃ and 73.1℃ for conventional curing cycle, short curing cycle and long curing cycle.

A strain and temperature curves for each material was shown in Fig.10~12. Several critical parameters of each material were shown in Table Ⅳ.

The parameter 90-ESmax means maximum strain measured during polymerization process till 90 minutes. The parameter 180-ESmax means maximum strain measured during polymerization process from 90 minutes to 180 minutes. The mean values of 90-ESmax were 972$\mu\varepsilon$, 993$\mu\varepsilon$ and 995$\mu\varepsilon$ for Vertex Rs, Lucitone 199 and ProBase Hot.

Each value showed no significant difference ($p<0.05$). The mean values of 180-ESmax were 1509 $\mu\varepsilon$, 1124$\mu\varepsilon$ and 1484$\mu\varepsilon$ for each material and it was depicted in Fig. 13.

Lucitone 199 showed the lower expansion strain during from 90 minutes to 180 minutes than the other two materials ($p<0.05$). Vertex RS and ProBase Hot showed no significant difference ($p<0.05$). The mean values of Sb were -4631$\mu\varepsilon$, -4708$\mu\varepsilon$ and -4299$\mu\varepsilon$ for each material. ProBase Hot showed the lower shrinkage strain and the other two other materials showed no significant difference ($p<0.05$). The mean values of Sa were -4581$\mu\varepsilon$, -4655$\mu\varepsilon$ and -4157$\mu\varepsilon$ for each material. ProBase Hot showed the lower shrinkage strain ($p<0.05$). ΔS values were positive regardless of the materials. The mean values of ΔS were 49$\mu\varepsilon$, 53$\mu\varepsilon$ and 142$\mu\varepsilon$ for each material and these were shown in Fig. 14.

ProBase Hot showed the higher expansion strain ($p<0.05$). The mean values of Sf were -4660$\mu\varepsilon$, -4722$\mu\varepsilon$ and -4308$\mu\varepsilon$. ProBase Hot showed the lower shrinkage strain ($p<0.05$). Vertex RS and Lucitone 199 showed no significant difference ($p<0.05$) and these were shown in Fig. 13.

The parameter Tmax was also measured and it was depicted in Fig. 15. The mean values of Tmax were 100.1℃ (Vertex RS), 100.7℃ (Lucitone 199) and 100.1℃ (ProBase Hot).

DISCUSSION

In this study, the strain and temperature changes during polymerization process and bench-cooling stage were measured by strain gauge and thermocouple. Strain gauge method has been widely used in recent dental field. It appears to be well suited for real-time measurement of the polymerization process and provides a means for studying the kinetics of polymerization.[16] The strain gauge method can be classified as one of the linear measurement methods for polymerization shrinkage determination.[17] Stafford et al.[18] studied the strain levels obtained on poly(methyl methacrylate) dentures to calculate the magnitude and direction of the principal strain in the denture bases during biting and swallowing. Komiyama et al.[19] reported residual stress relaxation in heat-activated acrylic denture base resin fabricated by the polymer-monomer mixture method. To clarify the stress relaxation in the mold replica, specimens were removed from the mold by deflasking procedure.

The expansion strains were observed in curing stage and shrinkage strains in bench-cooling stage and deflasking stage regardless of curing cycles and materials. In conventional curing cycle, specimen was cured at 70℃ in early 90 minutes. About 66% expansion was developed in this period. And the rest expansion strain was occurred. Expansion strain nearly followed the trend of curing unit temperature setting (Fig. 4). Expansion strains of the short curing cycle were gradually elevated to ESmax (Fig. 5). Most of expansion strains of long curing cycle were observed in the early 90 minutes (Fig. 6). Conventional curing cycle showed the higher expansion strain. Short curing cycle showed the lower expansion strain and it was about 77% of that of conventional curing cycle. Short curing cycle and long curing cycle showed no significant difference. The expansion strains were measured at two periods. 90-ESmax values showed no significant difference. Lucitone 199 showed the lower 180-ESmax than the other two materials.

Short curing cycle showed the higher temperature in polymerization process (Fig. 9). In conventional curing cycle, temperature curve followed the setting temperature of cuing unit. Most of temperature elevation was shown in early 90 minutes. After curing was done, the flask was bench-cooled almost to the room temperature. Just after the flask was removed from the curing unit, strain and temperature decreased in all curing cycles and materials (Fig. 4~6, 10~12).

ΔS was calculated from Sb and Sa. In the experiment concern about material, all ΔS values were positive values regardless of materials used. ProBase Hot showed the higher value (Fig. 14). But in the experiment concerned about the curing cycle, only ΔS value of conventional curing cycle showed the positive value. Short curing cycle and long curing cycle showed the negative values (Fig. 8). However, ΔS value of long curing cycle was not so reliable because of large standard deviation. Positive ΔS means the expansion of materials during deflasking procedure. It may be postulated that non-horizontal curing stresses can be inhibited, so that it can not be transformed into measurable strain until the resin specimen was completely removed form the mold. So ΔS value may be related to the possible bending deformation of the specimens.

Sf means final shrinkage strain. Although there was the expansion strain in the curing procedure, Sf value is the factor that profoundly affects the adaptation of the denture base to the master cast. 1000$\mu\varepsilon$ indicates 0.1% distortion. So lower Sf value means the less distortion in the processed resin. Marx[20] explained the discrepancies by using a hypothetical cast that has 50mm between the alveolar crests. In the respect of the curing cycle, long curing cycle showed the least Sf value and short curing cycle also showed the lower Sf value than conventional curing cycle (Fig. 7). Sf value of long curing cycle was about 68% of that in conventional curing cycle and short curing cycle was 91% of that in conventional curing cycle. ProBase Hot showed the least Sf value and that was 91% of Sf in Lucitone 199 which showed the greatest Sf value among tested materials. But this difference was not so prominent in case of the curing cycle.

In this study, the fact that long curing cycle showed the least shrinkage was revealed. As concerned about materials, the material ProBase Hot showed the least distortion but difference was not so profound in the curing cycle. So if the curing cycle and material that shows the lower shrinkage would be used, more accurate denture could be made and this would result in more favorable clinical services of denture.

CONCLUSIONS

In this study, strain gauge and thermocouple were used to understand the behavior of the heat-cured resin during polymerization process. Three curing cycles and materials were investigated and the following conclusions could be made.

1. The ESmax value of conventional curing cycle showed the largest value and the 180-ESmax value of Lucitone 199 showed the smallest value. 90-ESmax values showed no significant difference ($p<0.05$).

2. ΔS values of conventional curing cycle showed the positive strain values. ΔS values of short curing cycle and long curing cycle showed the negative values. All three materials cured by conventional curing cycle showed the positive values.
3. The Sf value of long curing cycle and ProBase Hot (cured by conventional curing cycle) showed the smallest values.

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