Fatigue Properties of Acrylic Denture Base Resins

Koichi FUJII

Department of Dental Materials Science, Kagoshima University Dental School 1208-1 Usuki, Kagoshima 890, Japan

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Observations were made of fractured surfaces caused by flexural and tensile fatigue tests made in polymethyl methacrylate denture base resins (PMMA). In addition, the changes in dynamic viscoelastic and tensile properties of the materials along with fatigue propagation were investigated.

In the tensile and flexural fatigue tests, both the fractured surfaces, which had striations on their surfaces and cracks near the fractured section, closely resembled each other in appearance. On the other hand, all of the tensile properties, such as elastic modulus, toughness and tensile strength, decreased with the increase of the number of stress cycles in the fatigue test. The storage modulus (E') of the material decreased gradually along with fatigue propagation over the whole range of temperatures tested. The loss modulus (E'') and mechanical loss tangent ($\tan\delta$) increased slightly.

The fatigue limit of four commercial denture base resins varied widely from one product to another.

Key words: Acrylic resin, Fatigue, Dynamic Viscoelastic property

INTRODUCTION

Polymethyl methacrylate (PMMA) has been widely used in dentistry. Although it has been generally employed as a denture base in order to function more satisfactorily in the mouth¹⁾, it is considered that this material is relatively weak in durability, such as under the conditions of repeated flexural stress and strain, and in light resistance^{2,3)}. Most fractures of the acrylic dentures are caused by repeated stress or strain during mastication. In particular, upper complete dentures often fail during use⁴⁾. Therefore, many studies on flexural fatigue have already been reported⁵⁻¹⁸⁾. However, the mechanisms of fatigue failure have yet to be sufficiently clarified since they are closely related to environmental factors.

The purpose of the present investigation was to elucidate the changes in the dynamic viscoelastic and tensile properties of PMMA along with fatigue propagation.

MATERIALS AND METHODS

Materials

1) Experimental resins (pure PMMA)

The materials used in this study, together with the composition, mixing proportions of monomer to polymer and curing conditions are listed in Table 1. The doughs were formed into metal molds for each specimen for the tensile and flexural fatigue tests and were polymerized using a hot press according to the curing conditions shown in Table 1. Thereafter, they were allowed to cool down to room temperature. The specimens employed in the tensile fatigue test were corrected into the size as shown in Fig. 1, then rubbed down

Table 1 Materials investigated and curing conditions

Material	Type	Code	L(ml)/P(g) ratio		Initiator	Cross linking agent	Temperature (°C)	time (min)	Pressure (kgf/cm ²)
Experimental resin (pure PMMA)	Heat cured	A	8*	16*	BPO**	0	70→100**	60→60	50
		B	8*	16*	BPO**	EGDMA**	70→100**	60→60	50
Commercial dental base resin	Pour	C	8	16	—	—	70→100**	60→60	—
		D	7	10	—	—	45	15	3
		E	7	10	—	—	45	15	3
		F	9	15	—	—	50	20	1.5

*1 Monomer and polymer used in this studies are methylmethacrylate (1st class reagent) and polymethylmethacrylate (Acricon).

*2 Benzoyl peroxide (1st class reagent) concentration used as initiator is 0.5 wt% to monomer.

*3 Ethyleneglycol dimethacrylate (1st class reagent) concentration used as cross linking agent is 3 wt% to monomer.

** Polymerization was carried out by processing for 1 hour at 70°C and then keeping for 1 hour at 100°C, using the hot press.

** Polymerization was carried out by processing for 1 hour at 70°C and then boiling for 1 hour in water.

Fig. 1 Specimen shape used for the tensile fatigue test.

with a wet silicone carbide paper (# 600) and polished using Al_2O_3 powder ($0.3 \mu\text{m}$ in diameter). Furthermore, the specimens were treated by heating them for 5 hours at a temperature of $110 \pm 5^\circ\text{C}$, prior to testing to remove any internal stress and decrease the scattering of the results. Flexural fatigue specimens were also polished using the same method as described for tensile fatigue specimens.

2) Commercial dental resins

For the heat cured resin, all the specimens were prepared by boiling them according to the dental curing techniques as shown in Table 1 and by using the same mold as for the tensile fatigue specimen. Three commercial pour resins were also employed. Polymerization was carried out in PTFE molds according to the manufacturers' recommendations shown in Table 1. After curing, the external shape of both specimens was corrected and finished to a smooth surface using No. 600 emery paper by hand. All the pour resin specimens were stored in a desiccator for a month at $23 \pm 1^\circ\text{C}$ before testing for neglecting the changes of residual monomer within a material.

C: Acron, GC Dental Ind. Co., Tokyo, Japan; D: Modern Core, Sankin Ind. Co., Osaka, Japan; E: Palapress, Kulzer, Wehrhelm, Germany; F: Poly base-S, Nissin Dental Products Inc., Kyoto, Japan

Fig. 2 Apparatus used for the flexural fatigue test.

Methods

1) Flexural fatigue testing

The apparatus used was an experimental device mainly designed for flexural fatigue tests, as shown in Fig 2. This instrument, which was a cantilever type, was able to give a constant repeated strain to the specimens by means of an eccentric cam attached to the rotating shaft of the motor with a rod joined to it.

Tests were carried out at a span of 35 mm, a deflection of 8 mm at the free end of the specimen and a repeated flexure of 30 Hz in air at $23 \pm 1^\circ\text{C}$. Only material A, which was not treated by heating, was tested and its specimen size was 40 ± 0.5 mm in width and 1 ± 0.1 mm in thickness, considering an area of denture base in clinical application.

2) Tensile fatigue testing

The apparatus used was a modification of the fatigue testing machine^{*}, to which the repeated constant stress was added by means of unbalanced mass method¹⁰⁻¹³. Tests were performed by applying a repeated stress of 30 Hz and adding a tensile mean stress to a specimen in air at $23 \pm 1^\circ\text{C}$ until each specimen failed, and an S-N curve was plotted.

3) Tensile testing

Tensile tests were performed with a crosshead speed of 0.5 mm/min using a universal testing machine^{**} in air at $23 \pm 1^\circ\text{C}$.

^{*} UF-15, Shimadzu Mfg. Co. Kyoto, Japan

^{**} Auto-graph AG-100, Shimadzu Mfg. Co. Kyoto, Japan

4) Measurement of dynamic viscoelastic properties

The apparatus used was a direct reading dynamic viscoelastometer^{*}, which permitted the angle of phase difference ($\tan\delta$) between the stress and strain to be measured¹⁹⁻²². All measurements were performed at a frequency of 1 Hz over a temperature range of 23 to 110°C. The temperature was increased at a rate of 0.5°C/min. The storage modulus E' and loss modulus E'' were calculated using the following equations:

$$E' = |E^*| \cos\delta$$

$$E'' = |E^*| \sin\delta$$

and

$$E^* = \frac{1}{8} \times \frac{1}{A \times D - K} \times \frac{L}{S} \times 10^{12} \text{ (dyn/cm}^2\text{)}$$

where A was the proportional constant of the dynamic viscoelastometer, D was the dynamic load and K , L and S were the proportional constant, the length and the cross-sectional area of the test pieces, respectively. Rectangular test pieces, 4 ± 0.1 mm in width, were prepared by cutting them from the specimens to which a repeated stress of 1.9 kgf/mm² was applied for 0, 10^5 , 10^6 and 1.5×10^7 times in the tensile fatigue test.

5) Two dimensional photoelastic stress analysis

This analysis was done to examine the general stress distribution in specimens used in the tensile fatigue test, using a photoelastic apparatus²³.

The material used as test pieces was an epoxy resin with an elastic modulus of 3.07×10^{10} dyn/cm² at 23°C. Their shapes were geometrically similar with that of the tensile fatigue specimens and three times the original size, and they were used after polishing with No. 1200 emery paper. The test pieces were heat-treated for 4 hours at a temperature of 90°C and then allowed to cool down for 12 hours to room temperature in an electric furnace.

6) Determination of residual monomer

Measurement of the residual monomer was performed using a high performance liquid chromatograph^{**}, incorporating two steel columns. After polymerization, the material (0.1 g) was weighed and placed into 10 ml of tetrahydrofuran (THF). For pour resin, materials used were stored in a desiccator for a month at $23 \pm 1^\circ\text{C}$ after polymerization. A sample of each solution (10 μl) was injected into the chromatograph and three measurements were taken at a temperature of $37 \pm 0.1^\circ\text{C}$. A standard solution of monomer (MMA) was prepared for the purpose of calibration and the residual monomer (%) was calculated from the area under the curve at the peak produced by MMA.

7) Microscopic observation

After specimens underwent fatigue failure, their fractured surfaces were investigated using a microscope.

* Rheovibron DDV-III-C, Orientec Co. Tokyo, Japan

** LC-3A, Shimadzu Mfg. Co., Kyoto, Japan

Fig. 3 Cracks developed from the surface of tensile side of the specimen in the flexural fatigue test (in cross-section)

Fig. 4 Comparison with each surface between the compressive side (a) without cracks and the tensile side (b) with cracks in the flexural fatigue test.

RESULTS

Figures 3 and 4 show the surfaces of a specimen on which a repeated strain was applied 2.7×10^7 times in the flexural fatigue test on material A without the heat treatment. The crack was observed on the surface at the tensile side of the specimen. This information will be a useful key in regard to the growth of cracks. Striations were also observed on the fractured surface of a lot of the specimens as shown in Fig. 5, and were detected for the specimen on which the strain was repeated 1.267×10^7 times.

Isochromatics was seen in the epoxy resin specimen as shown in Fig. 6, when a 67 kgf tensile load was added in air at $23 \pm 1^\circ\text{C}$. The principal stresses (σ_x and σ_y) were calculated using the results measured by means of the construction of the isochromatics and isoclinics, as shown in Fig. 7. From these results, it can be seen that stress concentration within the specimens used in the tensile fatigue test were produced in the middle (a and b) of each curved line.

Fig. 5 The striations developed from the surface of tensile side of the specimen used in the flexural fatigue test, on a fractured surface.

Fig. 6 Isochromatics in an epoxy resin specimen, when 67 kgf tensile load was applied to the specimen in air at $23 \pm 1^\circ\text{C}$.

Figure 8 is the S-N curves obtained from each tensile fatigue test of materials A and B. The addition of ethyleneglycol dimethacrylate (EGDMA) to base monomer (MMA) improved the fatigue limit of material A.

Figure 9 shows the S-N curves of material A with a mean stress (σ_m) of 1.5, 1.9 and 2.5 kgf/mm². As shown in Fig. 10, the increase of the mean stress brought a decrease in the fatigue limit.

Figure 11 shows the stress-strain curves obtained from the tensile test for each specimen when 1.9 kgf/mm² of stress was applied repeatedly for 10^4 , 10^5 and 10^6 times along with

Fig. 7 Distribution of principal stresses (σ_x and σ_y) along line a-b shown in Fig. 6, obtained by shear difference method: The symbols $\blacksquare$ and $\bullet$ show σ_x and σ_y , respectively.

Fig. 8 Variation of stress amplitude (σ_a) with the number of stress cycles in tensile fatigue test in air at $23 \pm 1^\circ\text{C}$: The symbols $\bullet$ and $\blacksquare$ show materials A and B, respectively.

Fig. 9 Variation of stress amplitude (σ_a) with the number of stress cycles for material A in the tensile fatigue test in air at $23 \pm 1^\circ\text{C}$: The symbols ●, ■ and ▲ show fatigue behavior at each mean stress of 1.5, 1.9 and 2.5 kgf/mm^2 .

Fig. 10 Relationship between mean stress (σ_m) and stress amplitude (σ_a): The symbols □ and ■ are at 10^5 and 10^6 cycles.

Fig. 11 Stress-strain curves of material A obtained before and after the tensile fatigue test: The numbers 0, 10^4 , 10^5 and 10^6 indicate the number of stress cycles.

Fig. 12 Variation of tensile properties with the number of stress cycles for material A: The symbols ■, ● and ▲ show the elastic modulus, tensile strength and toughness, respectively.

fatigue propagation. According to the increase in the number of stress cycles, the elastic modulus, toughness and tensile strength decreased, as shown in Fig. 12.

Figure 13 shows the variation in the viscoelastic properties (E' , E'' and $\tan \delta$) with the temperature, when measured by using the test pieces cut from the same specimens as described in Fig. 11. With the increase in the number of stress cycles, the storage modulus

Fig. 13 Variation of dynamic viscoelastic properties with temperature of material A : The symbol ● shows the specimen before the fatigue test and ▲, ■ and ○ at 10^5 , 10^6 and 1.5×10^7 cycles, respectively.

(E') gradually decreased over the whole range of temperatures tested and the loss modulus (E'') slightly increased, together with the mechanical loss tangent ($\tan \delta$), as shown in Fig 14.

Figure 15 shows the S-N curves of material C measured in air at $23 \pm 1^\circ\text{C}$ and in water at $37 \pm 1^\circ\text{C}$. The fatigue limit of the specimens soaked in water was smaller than that of the material tested in air.

Figure 16 shows the S-N curves for materials D, E and F in air at $23 \pm 1^\circ\text{C}$. The pour resins had low fatigue limits, compared with the heat cured acrylic resin, and their fatigue limits varied widely.

DISCUSSION

Flexural fatigue test

A repeated strain of 0.98%, much below the proportional limit, was applied to the specimen. In the case of a repeated strain, it was seen that the stress produced within a specimen decreased with time because of the stress relaxation caused by the flow of the material. Therefore, fatigue resistance may be slightly increased. In regard to appearance of the failure surface, many researchers have already reported a similar tendency^{4,6,8)}.

Tensile fatigue test

1) Tensile fatigue behavior of acrylic denture base resin

The endurance limit of high polymers has commonly employed a fatigue strength at 10^7 applications as a tentative criterion for judgment²⁴⁾. In clinical applications, it is known that the average number of times that a person chews is over five hundred thousand per annum²⁵⁾

Fig. 14 Variation of dynamic viscoelastic properties with the number of stress cycles: The open and closed notations are at the temperatures of 37°C and 80°C.

Fig. 15 Variation of stress amplitude (σ_a) with the number of stress cycles for material C in the tensile fatigue test: The symbols ● and ■ are measured respectively in air at $23 \pm 1^\circ\text{C}$ and in water at $37 \pm 1^\circ\text{C}$.

and that the life span of dentures is about ten years. Therefore, if it is assumed that the fatigue limit of materials is given at 10^6 cycles, it was 2.15 kgf/mm² for material A and 2.28 kgf/mm² for material B. Furthermore, the addition of EGDMA brings an increase in the value of this fatigue limit. This is probably because it produced a crosslinked structure between the molecules. The results in the present study relatively low when compared with the fatigue limits reported by earlier researchers²⁶⁻²⁸. This may be due to the temperature rise generated within a specimen because the testing speed (30 Hz) was comparatively higher

Fig. 16 Variation of stress amplitude (σ_a) with the number of stress cycles in the tensile fatigue test in air at $23 \pm 1^\circ\text{C}$: The symbols $\blacktriangle$, $\bullet$ and $\square$ show the materials D, E and F, respectively.

than that of earlier investigations. The increase in the mean stress brought the decrease in the fatigue limit as shown in Fig. 10. Also, this result may be closely related with the friction between molecules.

In addition, it was considered that the fatigue failure was induced by the development of cracks in the specimen surfaces and/or a flaw within the specimens, as shown in Figs. 17, 18 and 19. The fractured surface possessed two surfaces, a comparatively smooth surface with striations and a rough surface with many uneven spots. From these results, it can be supposed that the fracture of specimens would occur swiftly when the growth of cracks attained to a certain length along with fatigue propagation.

2) Changes of tensile properties together with fatigue propagation

Specimens in which the number of stress cycles was permitted to increase had a decrease in the tensile properties, such as the elastic modulus, tensile strength and toughness, and it was proved that the decrease of these properties was already commenced at 10^4 stress applications, as shown in Figs. 11 and 12. These results agree well with the results of previous work²⁸⁾. But, the elastic limit and Brinell hardness reported in previous work increased a little according to the number of stress cycles. In the case of the appearances of each fractured surface which underwent 10^4 , 10^5 and 10^6 stress applications, these were similar to the fracture surface obtained after the tensile test for the specimen that did not undergo repeated stress. However, there was not striations on the fractured surface, and the fractured surface had a tendency to become a rough surface with a lot of uneven spots when the number of stress cycles was increased. It can be seen that these results are probably made by the development of many cracks along with fatigue propagation.

3) Changes of dynamic viscoelastic properties along with fatigue propagation

It is generally known that the viscoelastic properties of polymers are affected remarkably by their experimental conditions, such as temperature, loading speed and strain^{29,30)}. One of the purposes of this investigation was to clarify the changes of dynamic viscoelastic properties of denture base materials with fatigue. The frequency used in this study was 1 Hz in consideration of real chewing³¹⁾. As the number of stress cycles increased, the storage

Fig. 17 Magnification of striations on the fractured surface,

modulus (E') and glass transition temperature (T_g) of the denture base resins decreased gradually over the entire range of the temperatures tested, compared with that of the specimens without fatigue, as shown in Fig. 14. On the other hand, the loss modulus (E''), which indicates energy lost as frictional heat between molecules, increased slightly with the increase of stress cycles and the same statement was true for the mechanical loss tangent ($\tan \delta$). It should be noted that these results were also obtained by cracking, as shown in Figs. 19 and 20.

4) Fatigue behavior of four acrylic denture base resins

Material C, which was soaked in water, presented the lowest fatigue resistance when compared with materials stored in air. Its reduction rate was about 11.8%. This result indicates that the absorbed water may act as a plasticizer. And this same result was also obtained by an increase in temperature from 23 to 37°C.

Residual MMA monomer of the material used in this investigation is listed, together with the tensile strength (σ_t) and stress amplitude (σ_a) in Table 2. A significant correlation was estimated between them, and the coefficient of correlation was -0.91 for the tensile strength (σ_t) and -0.84 for the stress amplitude (σ_a).

From these results, it would appear that the fatigue limit is influenced by the amount of

Fig. 18 Magnification of striations developed from a flaw within a specimen.

Fig. 19 The development of cracks on the specimen surface near the fractured section.

residual monomer included within each material.

CONCLUSION

Changes in mechanical and dynamic viscoelastic properties of polymethylmethacrylate denture base resins along with fatigue propagation were investigated using a universal fatigue testing machine, a universal tensile testing machine and a direct reading dynamic viscoelastometer. The summary is as follows :

- 1) Fracture surfaces caused by repeated flexure closely resembled those of tensile fatigue in appearance.

Fig. 20 The development of cracks on the specimen surface together with fatigue propagation: The symbols (a), (b) and (c) are at 10^4 , 10^5 and 10^6 cycles, respectively.

- 2) By increasing the mean stress applied to the specimen, the fatigue limit was decreased
- 3) Along with fatigue propagation, the elastic modulus, toughness and tensile strength were reduced.
- 4) Along with fatigue propagation, the storage modulus (E') and glass transition temperature (T_g) were gradually decreased over the whole range of the temperatures tested and also the loss modulus (E'') and mechanical loss tangent ($\tan \delta$) increased slightly.
- 5) The fatigue limit was improved by the addition of a cross-linking agent.
- 6) Heat cured acrylic resin had a high fatigue limit, compared with that of the pour resins that were tested
- 7) Water absorbed into heat cured resin caused the fatigue limit to decrease.
- 8) There was a negative correlation between the amount of residual monomer included within the materials tested and the fatigue limit.

Table 2 A comparison of tensile strength, stress amplitude and residual monomer for materials used

Material	Tensile strength σ_a (kgf/mm ²)	Stress amplitude α (kgf/mm ²)(at 10 ⁶)	σ_a / α	Residual monomer (%)
A	8.74 (0.52)	2.15	0.240	0.41
B	8.51 (0.18)	2.28	0.268	0.40
C	7.95 (0.06)	2.40	0.302	0.21
D	7.09 (0.67)	1.62	0.228	1.75
E	6.97 (0.73)	1.13	0.162	1.36
F	6.02 (0.21)	1.52	0.252	1.95

() : SD

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ポリカルボン酸を含む歯科用セメントと歯質との接着機構 電位差滴定からみたカルシウム共存下におけるセメントポリマーの高分子電解質挙動

飯岡淳子*, 荒木吉馬**, 松田浩一*, 大野弘機**

*東日本学園大学歯学部歯科保存学第II講座

**東日本学園大学歯学部歯科理工学講座

種々な濃度のカルシウムイオンが共存するポリアクリル酸およびポリカルボン酸系セメントから分離したポリマー水溶液の電位差滴定をNaOH溶液で行った。その結果、これら高分子電解質は、カルシウムイオンが共存しない場合にはいずれも弱酸の挙動を示した。ところが、ポリマー中のカルボン酸基の濃度に対して1/2当量以上カルシウムイオンを共存させると、各ポリマーは強酸型

の挙動を示した。つまり、このことはアルカリによってカルボン酸を中和してゆくと、解離したカルボキシレートイオンが共存するカルシウムイオンと強く結合するを意味している。

したがって、このような挙動は、セメントと歯質の間で起るとされる化学的接着の機構として有力なものと考えられる。

義歯床用材料 (PMMA) の疲労特性に関する研究

藤井孝一

鹿児島大学歯学部歯科理工学講座

義歯床用材料 (PMMA) に関して、曲げ疲労試験及び引張疲労試験を行った。さらに引張疲労試験に伴う粘弾性と引張特性の変化を調べた。

その結果、疲労試験後の破面の比較では、曲げ疲労による破面は、引張側の表面からクラックが開始し、その後のクラックの進行を表わす striation が観察され、破壊にいたるという様相を示す。一方、引張疲労の場合にも類似の傾向が認められる。

疲労に伴う粘弾性の変化については、疲労が進むと貯蔵弾性率 (E') は、測定されたすべての温度領域で低下

する。一方、損失弾性率 (E'')、損失正接 ($\tan \delta$) はわずかに増加する傾向を示した。また、弾性率、引張強さ、靱性などは、 10^4 回の繰り返し数で、すでに低下を示していた。これらの結果は、疲労に伴うクラックの発生が材料の劣化を招いたものと考えられる。

義歯床用材料として使用されている4種類の市販品の疲労特性を調べた。流し込み型レジンと熱重合型より小さく、材料間で大きく異なる。このばらつきの一因として、材料組成の違い、未反応物質の量の違いなどが考えられる。

屋外直接暴露試験によるエpiteーゼ用シリコンラバーの変色に関する研究

鷹股哲也*, B. Keith Moore**, Varoujan A. Chalian***

*松本歯科大学歯科補綴学第一講座

**Dental Materials, School of Dentistry, Indiana University

***Maxillofacial Prosthetics, School of Dentistry, Indiana University

エpiteーゼ用材料はその使用目的から、顔面皮膚との色調の調和、皮膚類似性の柔軟性と自然感、生体親和性と物理化学的な安定性、成形が容易で彩色に優れていることなどの諸性質が必要とされる。特に、顔面補綴では、機械的な劣化以上に、変色が原因で再製作を余儀なくさ

れる場合が多いといわれる。変色の要因あるいは原因はいろいろ考えられるが、これらを検討するには、着色していないベースポリマーと着色・彩色の施されているエラストマーについてそれぞれ考慮しなければならない。本研究では、着色・彩色のされていないベースポリマー