A New Procedure of the Tensile Fatigue Test for Dental Materials

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A new tensile fatigue test using a stainless steel screw was introduced. A transparent PMMA rod was selected as the test material. A hole was drilled and tapped into the rod; a screw was put into the rod with dental cement or without cementation. Three types of cyclic loads were applied to the test piece; the number of times the load was applied when the initial crack was observed and when the final fracture occurred were recorded. Surface observation showed that actual fatigue fractures occurred in the test piece. The preferred applied load waveform was the cyclic havers-triangles load because of the small coefficient of variance. Fatigue strengths of test pieces with and without cementation were different. Cementation improved the fatigue properties, and the type of cement had an effect on the fatigue properties. Although the number of final fractures minus the number of initial cracks was the constant regardless of the type of cement used.

Key words: Fatigue test, S-N curve, Waveform effect

INTRODUCTION

The amount of chewing by a normal human being is estimated to be more than one million chews per year. The dental material used in the mouth must resist not only a single large force but also repeated small forces during mastication. The repeated stress sometimes results in fatigue fractures. Thus, mechanical properties of a dental material must be tested not only by a static method but also by a dynamic method. The fatigue property, by which life of the material is predicted and the structural design will be planned, is one of the typical properties revealed by the dynamic test method; this property is tested by tensile or torsional force¹⁾. Due to the difficulty in preparation of the test piece, only a few studies have reported fatigue properties of dental materials, especially brittle materials, e. g., a dental cement²⁻⁴⁾, a dental amalgam⁵⁻⁷⁾, a composite resin⁸⁻¹⁰⁾ and so on. We developed a new method for the tensile fatigue test using a stainless steel screw. In this study, we introduced the test procedures and evaluated a suitable test condition.

MATERIALS AND METHODS

A commercial transparent polymethylmethacrylate (PMMA) rod (6mm in diameter and 20mm in length) was used as the test material. This rod was drilled and tapped for the preparation of a screwed hole; the diameter and the depth of the hole and the tapped pitch and height of the thread were 1.6mm, 4.5mm, 0.2mm and 0.45mm, respectively. A stainless

steel screw (SUS304)^{#1} corresponding to the screwed hole was inserted into the acrylic rod luted with either a zinc phosphate cement (ZP, P/L : 1.45g/0.5ml)^{#2}, a zinc oxide eugenol cement (ZOE, P/L : 1.0g/0.3g)^{#3}, or without any cement (Fig. 1). All test pieces were stored on a bench in the laboratory ; the fatigue test was performed 24 hours after preparation. The test piece was mounted in a digital servohydraulic testing machine^{#4} with a universal chuck and a holding jig (Fig. 2). In this study, a stress-controlled cyclic load was used.

A sinusoidal cyclic unipolar load (i. e., haversine cyclic load) of 2 Hz was employed to obtain the stress-number diagram of the test specimens without cementation. The number of times the load was applied when an initial visible fatigue crack was observed with a loupe of 5 magnification was recorded as N_c ; the number of times the load was applied when the test piece was fractured was recorded as N_f . The applied maximum tensile stress varied from 40MPa to 5MPa ; the applied minimum tensile stress was constantly 0.6MPa.

To estimate the effect of waveforms of cyclic load on the test pieces without cementa-

Fig. 1 Test piece of the acrylic specimen and the stainless steel screw.

Fig. 2 Test piece set in the fatigue test machine.

^{#1} Nitoh Seikoh, Tokyo, Japan

^{#2} Elite 100, GC, Tokyo, Japan

^{#3} Eugedyne, Showa Pharm., Tokyo, Japan

^{#4} Model-8500, Instron Corp., MA. USA

tion and with ZP cementation, the haver-sines, haver-squares and haver-triangles cyclic loads (maximum tensile stress: 18.8MPa, minimum tensile stress: 1.3MPa) of 1Hz were selected (Fig. 3). A haver-triangles cyclic load of 1Hz was also applied to the test piece with ZOE cementation for comparison of the various luting materials.

The static tensile strengths of test pieces with ZP cementation and without any cementation were tested with a 490N/min load speed.

The fractured surfaces of the test pieces were observed with a microscope §.

All test procedures were done in an ambient laboratory atmosphere ($23 \pm 2^\circ\text{C}$ temperature and $(50 \pm 10)\%$ relative humidity).

RESULTS

Figure 4 shows a typical situation of this fatigue test when a crack of the test piece was propagated. The initial crack growth was always observed at the end of the fixed screw ; the

Cyclic unipolar waveform

Fig. 3 Three applied waveforms of cyclic load.

Fig. 4 The initial crack was observed from the terminal edge of the stainless steel screw and the crack propagated laterally.

§ Versamat, Union Kohgaku, Tokyo, Japan

crack developed laterally with an increase in the number of times the load was applied. The obvious fatigue striations and the final fracture surface were observed by a microscope (Fig. 5).

The stress-number diagram is shown in Fig. 6. The applied stress represents the applied maximum load divided by the test specimen's cross-sectional area which was the result of subtracting the tapped hole's cross-sectional area from the PMMA rod's cross-sectional area. Within the range of the applied stress used in this study, there was no horizontal line at high cycles.

The effects of the waveforms and the luting materials on the N_c and the N_f are summarized in Table 1. Regarding the waveform, there was a significant difference among the three waveform's means of their N_c s and N_f s with ZP cementation by the one-way analysis of variance (ANOVA); that of the haver-triangles cyclic load was significantly larger than the others by the Scheffe multiple comparison test ($p < 0.01$) (Fig. 7); there was

Fig. 5 Fracture surface of tested specimen applied with a 2Hz cyclic load: obvious fatigue striation (S) and final fracture surface (F).

Fig. 6 S N diagram: applied maximum load varied from 40MPa to 5MPa, minimum load was 0.6MPa.

Table 1 Summary of effects of applied stress waveform and cement materials on fatigue strength ; Nc : number of applied load when an initial crack was observed, Nf : number of applied load when the final fracture occurred. (-) : without cementation ; (ZOE) : cementation with the zinc oxide eugenol cement ; (ZP) : cementation with the zinc phosphate cement

waveform	(-)		ZOE		ZP	
	Nc	Nf	Nc	Nf	Nc	Nf
Haver-sine	67 ± 29	440 ± 210			390 ± 70	1125 ± 130
Haver-square	80 ± 79	506 ± 315			260 ± 82	2425 ± 746
Haver-triangle	147 ± 50	1020 ± 88	327 ± 64	4339 ± 690	1923 ± 433	5911 ± 375

n=3, mean + S. D.

Fig. 7 Effect of waveform on the Nc and the Nf; the Nc means the number of times the load was applied when an initial crack was observed; the Nf means the number of times the load was applied when the final fracture occurred. (-) : without cementation ; (ZP) : with the cementation using zinc phosphate cement.

no significant difference between the mean of the Nc or the Nf obtained from the haver-sines load and the Nc or the Nf from the haver-squares load, respectively. In addition, there was no significant difference among the three waveform's means of the Nc without cementation by the one-way ANOVA ; there was a significant difference among those of the Nf without cementation. The coefficient of variances (CV) of the Nc without cementation using the cyclic haver-sines, haver-squares and haver-triangles loads were 43.3%, 99.2% and 34.3%, respectively ; the CVs with ZP cementation were 18.0%, 31.5% and 22.5%, respectively. The CVs of the Nf without cementation using the cyclic haver-sines, haver-squares and haver-triangles loads were 47.7%, 62.2% and 8.5%, respectively ; the CVs with ZP were 11.5%, 30.8% and 6.3%, respectively.

Compared with the luting materials which applied the haver-triangles cyclic load, the Ncs or the Nfs with cementation were significantly larger than the Ncs or the Nfs without cementation, respectively (Fig. 8). With respect to ZOE cementation, the Nf was approximately four times larger than the Nf without cementation ; however, the Nc was approximately twice as large than the Nc without cementation. Regarding ZP cementation, the Nf

was approximately six times larger than the N_f without cementation ; however, the N_c was thirteen times larger.

The mean and standard deviations of the static tensile strength without cementation were 15.1MPa and 2.4MPa, respectively ; those with ZP cementation were 26.7MPa and 2.6MPa, respectively. The tensile strength with ZP cementation was 1.5 times larger than that without cementation. The fracture surface obtained from the static tensile test was very rough compared with the fracture surface obtained from the fatigue test (Fig. 9).

DISCUSSION

In the conventional tensile fatigue test, the crack propagates from the surface to the inside¹⁾ ; however, in this new test, the crack propagates from the internal hole to the outside. As a result, it is rather difficult to compare the results of the conventional test with those of

Fig. 8 Effect of luting materials ; (-) : without cementation ; (ZOE) : cementation with zinc oxide eugenol cement ; (ZP) : cementation with zinc phosphate cement.

Fig. 9 Fracture surface.

- (a) fracture surface of an applied cyclic load of the maximum of 10MPa haversines
- (b) fracture surface resulting from the static tensile strength test

this method. By this method, the condition of the outer surface of the test specimen will not affect the result. Preparation of the test piece for this method was not difficult, and it was also possible to embed a screw directly into the test material.

This new tensile fatigue test is a precrack fatigue test because the terminal edge of the screw acts as a precrack. As a result, the crack propagation of each tested specimen was initiated from the terminal edge of the screw. On the other hand, with the photoelastic test, some researchers¹¹⁻¹³⁾ reported that an applied stress caused stress concentration around the terminal edge of the screw post. Our observation of the initial crack position agreed with a previously reported position of stress concentration.

A PMMA rod was used as the test material because of its easy machinability and excellent transparency; as a result, a uniform shape of the screw hole could be prepared. The SUS 304 stainless steel used in this study is very popular for dental application, being employed for example, in screw posts for core build-up. The initial crack growth could be visually detected without a special apparatus. Observation of the fractured surfaces revealed an actual fatigue fracture such as that shown in Fig. 4.

Fatigue strength is little affected by a frequency in the low frequency range¹⁴⁾, and our preliminary experiment showed no difference between the fatigue properties when 1Hz or 2Hz cyclic loads were applied; therefore, the 1Hz cyclic load was employed for the experiment of waveform effect because of the easy detection of the initial crack; the 2Hz cyclic load was employed for the S-N diagram because of the reduction in experimental time.

The shape of the S-N diagram obtained from this study was similar to that of previous reports on the PMMA^{15,16)}. The following equation was calculated with the obtained S-N diagram¹⁷⁾.

$$\log S = 1.71 - 0.17 \log N_f$$

$$\log S = 1.68 - 0.22 \log N_c$$

The S in the equation is the maximum applied stress. As a result, the fatigue parameters of the Nc and the Nf were 4.5 and 6.0, respectively. This indicated amplitude of the maximum applied stress effected the final fracture more strongly than the crack initiation. Moreover, a ten-percent decrease of the applied stress caused a 1.6 times larger the number of times the cyclic load was applied when an initial crack was observed; however this decrease caused a 2.0 times larger the number of times the cyclic load was applied when the final fracture had occurred.

The waveform is one of the influential factors of the fatigue properties. According to the obtained figures, the mean of the Nc and the Nf of the cyclic haver-triangles load was the largest; in addition, the coefficient of variance (CV) of the Nf of the cyclic haver-triangles load was the smallest. In terms of the structural mechanism of the test machine, a cyclic haver-sines load is usually selected for applied fatigue stress. However, the best applied stress was considered to be the cyclic haver-triangles load because of the small coefficient of variance.

The applied load of the tested specimen without cementation acted mainly as tensile stress; however, the applied load also acted as torsional stress because of a small amount of play between the tapped hole and the screw. As a result, the Nc and the Nf of the test pieces without cementation were significantly small. For the same reason, the static tensile strength

without cementation was weaker than that with ZP cementation. These results suggested that the screw cemented or directly embedded into the test material was more favorable because the test piece was then free from torsional stress. With respect to the luted test specimen, applied force initially destroyed the cement layer between the acrylic specimen and the screw ; applied force secondly destroyed the acrylic specimen. The mechanical properties, such as compressive strength and diametral strength, of the zinc oxide eugenol cement were weaker than those of the zinc phosphate cement¹⁸⁾. The fatigue strength of the zinc oxide eugenol cement is considered to be weaker than that of the zinc phosphate one. Consequently, the Nc with ZOE cementation was smaller than that with ZP cementation. After the crack was initiated, it propagated regardless of the type of cement used. The remainder of the Nc from the Nf with ZOE cementation was, therefore, almost equal to that with ZP.

As seen from the obtained results, this new tensile fatigue test caused a fatigue fracture. With this test, it is possible to distinguish differences among not only the tested materials but also the cement material luting the screw. This testing method is applicable to dental materials, especially brittle materials, e. g., dental cement, dental amalgam, dental gallium alloy, composite resin, dentin of the tooth and so on.

CONCLUSION

A new tensile fatigue test method with a stainless steel screw was introduced. By this method, actual fatigue fractures occurred in the acrylic test specimen. The number of times the cyclic load was applied the crack was initiated and when the final fracture occurred varied with the applied waveform. The more favorable waveform of applied load was considered to be the cyclic haver-triangles load. Luting of the screw with a cement strengthened the fatigue properties ; therefore, it was considered better for the screw to be cemented or be directly embedded into the tested material. The type of cement had an effect on the fatigue properties ; however, the difference of numbers of times of applied load between the initiate crack and the final fracture with cementation using zinc oxide eugenol cement and that using the zinc phosphate cement was nearly the same.

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本号掲載論文の和文抄録

Nd₂Fe₁₄B 磁石および SmCo₅ 磁石の腐食挙動

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小型で強力な希土類系磁石は、歯科補綴や矯正の装置に用いられるようになってきた。このような応用を考える上で希土類系磁石の口腔内環境下で吸引力の変化と腐食について明らかにしておく必要がある。そこで Nd₂Fe₁₄B 磁石および SmCo₅ 磁石を 37°C の 1% 塩化ナトリウム水溶液、1% 乳酸水溶液、0.05% 塩酸水溶液、0.1% 硫化ナトリウム水溶液、人工唾液に浸漬させ、吸引力の経時的変化、イオン溶出、重量変化、アノード分極特性

を調べた。またステンレス鋼に磁石を入れレーザー溶接により密封された磁性アタッチメントの耐食性を調べた。密封されていない希土類系磁石の耐食性は不良であった。特に 1% 乳酸水溶液中では、激しく腐食され、吸引力も低下し溶出イオン量も多かった。磁性アタッチメントの耐食性は良好であり、レーザー溶接を行ったステンレス鋼のカプセルは防錆効果があると考えられた。

in vivo および *in vitro* におけるリン酸八カルシウムの相変態

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リン酸八カルシウム[Ca₈H₂(PO₄)₆・5H₂O, 以下 OCP と略す]を ddY 系雄性マウスの両大腿部筋膜上に 3 週間インプラントした。また、OCP 粉末を 37°C に保持した人工体液および 1% NaCl 溶液中に、1 日、1 週間、3 週間浸漬した。所定期間経過後、試料は走査型電子顕微鏡、X 線回折、赤外分光分析により状態分析した。X 線回折の結果より、マウスに 3 週間インプラントした OCP は

すべて結晶性の低いアパタイトに転化していた。また、1% NaCl 溶液中に浸漬したものではアパタイトに転化していた。赤外分光分析結果より、これらのアパタイトには炭酸が含有されていた。一方、人工体液中に浸漬したものでは OCP はほとんど変化しなかった。これは、人工体液中の Mg イオンが OCP からアパタイトへの相変態を遅延しているためと考えられる。

歯科材料引張疲労試験の新しい方法

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ステンレス鋼製スクリューを測定試料に埋め込み、スクリューと測定試料に繰り返し引張力を与える引張疲労

試験を考案した。試験試料として PMMA 棒を選び、これにネジきりをし、スクリューを歯科用セメントで合着

した試料と合着しない試料を製作した。負荷波形としてサイン波、矩形波、三角波について検討した。PMMA 試料を用いたため、亀裂の発生が観察でき、亀裂発生までの繰り返し応力回数と、試料の破断までの回数を記録した。破断面を観察したところ、破壊疲労を示す表面性状が観察された。負荷波形としては、測定値の変動係数が

小さい三角波形の負荷が望ましいと考えられた。疲労強度はセメント合着により改善され、セメントの種類によりその程度は異なった。しかし、亀裂発生から破壊までに与えられた繰り返し応力の回数はセメントの種類の影響を受けなかった。

歯科修復物の辺縁封鎖性の新しい評価法

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辺縁封鎖性評価のための新しい試験装置を開発した。この試験装置は熱サイクル負荷のための流路と漏洩検出のための流路からなっており、両流路は被験修復歯で隔絶される。熱サイクル負荷系にはトレーサーとして使用したローダミン B の冷熱水溶液を循環させておき、6つの電磁弁の開閉で試験片に熱サイクルを与えられる。試験片に熱サイクルを与えながら、検出系を循環する蒸留

水中に漏洩してくるトレーサー量を定期的に分光光度計で測定した。銀合金インレー修復、アマルガムあるいはグラスアイオノマー充填修復について漏洩データを実験式で解析したところ、漏洩挙動をかなりよく説明でき、熱サイクルに対する完全封鎖保持期間として辺縁封鎖性を推定評価できた。

Ni-Ti 合金鑄造体の機械的性質に及ぼす組成および純度の影響

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Ni-Ti 合金鑄造体の機械的性質および変態点に及ぼす組成およびチタンの純度の影響について、引張試験および示差走査熱量測定により検討した。鑄造用インゴットの組成は、チタンの原子パーセントで 49.0 から 49.2 % とし、比較的純度の高い 3 種類のチタンを材料として使用した。

その結果、Ni-49.0 Ti はやや脆い性質を示し、Ni-

49.2 Ti は低い見かけの耐力および高い伸びを示した。残留ひずみは、チタンの組成が高くなるほど増加した。また、チタンの純度のわずかな低下が、Ni-Ti 合金の機械的性質および変態点に影響を及ぼした。すなわち、変態点が下がることにより、見かけの耐力が高くなり、残留ひずみが小さくなり、伸びが減少した。

フィラー含有量の異なるコンポジットレジンの中浸漬下における圧縮クリープと回復

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フィラー含有量の異なるコンポジットレジンを作製し、その圧縮クリープと回復について水中浸漬下で研究

した。500 時間後のコンポジットレジンのクリープひずみはコンポジットレジン中のフィラー含有量の増加にと