Flexural and fatigue properties of polyester disk material for milled resin clasps

Jialin HAO\textsuperscript{1}, Natsuko MURAKAMI\textsuperscript{1}, Toshiki YAMAZAKI\textsuperscript{1}, Naohiko IWASAKI\textsuperscript{2}, Masaru YATAKE\textsuperscript{1}, Hidekazu TAKAHASHI\textsuperscript{2} and Noriyuki WAKABAYASHI\textsuperscript{1}

\textsuperscript{1} Removable Partial Prosthodontics, Oral Health Sciences, Graduate School of Medical and Dental Sciences, Tokyo Medical and Dental University, 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan
\textsuperscript{2} Oral Biomaterial Development Engineering, Graduate School of Medical and Dental Sciences, Tokyo Medical and Dental University, 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan

Corresponding author, Natsuko MURAKAMI; E-mail: n.murakami.rpro@tmd.ac.jp

To evaluate the flexural and fatigue properties of a polyester disk material used in milled resin clasps of removable partial dentures, experimental polyester disk (mPE), injection-molded polyester (iPE), and polymethyl methacrylate disk (mPMMA) were examined by three-point bending tests and cyclic fatigue tests at 0.75 or 1.50 mm deflection. The mPE exhibited significantly higher flexural strength than the iPE ($p<0.05$). Meanwhile, the mPMMA displayed higher flexural modulus and strength than the polyesters. The mPE exhibited a significantly lower residual strain than the iPE at the cyclic 0.75 mm deflection ($p<0.05$); however, microcracks were observed in the mPE at the 1.50 mm deflection. The mPMMA showed a high residual strain at the 0.75 mm deflection and fractured within 1,000 cycles at the 1.5 mm deflection. The higher flexural strength and lower residual strain of the mPE compared with the iPE suggest the advantages of milled resin clasps within a limited deflection.

**Keywords:** Removable partial denture, Thermoplastic resin, Resin clasp, Fatigue resistance, Residual strain

**INTRODUCTION**

Removable partial dentures (RPDs) are a noninvasive, cost-effective treatment modality in prosthodontics for restoring partially edentulous arches\textsuperscript{10}. The appearance of metallic clasps has long been a chief concern for patients requiring this prosthesis\textsuperscript{4}. Recently, the fabrication of retentive resin clasps by injection-molding thermoplastic resins has been popularized. Such clasps are referred to as “flexible dentures”\textsuperscript{10} or “non-metal clasp dentures”\textsuperscript{11}, and are expected to increase patient satisfaction owing to their improved appearance compared to conventional metallic clasp dentures\textsuperscript{12}. During denture insertion and removal, the resin clasps are repeatedly deformed\textsuperscript{13}. Therefore, conventional polymethyl methacrylate (PMMA)-based denture base resins are not applicable to retentive clasps, which are vulnerable to flexure. Currently, resin clasps are made of flexible thermoplastic resins such as polyamide\textsuperscript{8,17}, polyester\textsuperscript{8}, and polycarbonate\textsuperscript{8}, which commonly reveal a wider elasticity range than PMMA-based denture base resins\textsuperscript{10}. However, studies have shown that the thermoplastic resin clasps are still likely to permanently deform and even fracture with long-term use\textsuperscript{11,12}.

Denture bases milled by computer-assisted design/computer-assisted manufacturing (CAD/CAM) have shown improved mechanical properties compared with conventional heat-polymerized and injection-molded denture bases. The flexural strength, flexural modulus, and impact strength of the milled PMMA denture base disks were higher than those of the conventional heat-polymerized resin bases\textsuperscript{10} because the industrial fabrication of CAD/CAM disks minimizes the risk of developing porosities inside the block and prevents potential operator error during the polymerization procedure\textsuperscript{14}. However, to the best of our knowledge, there have been no reports on the mechanical properties of thermoplastic polyester disks used in milled resin clasps, and no studies that compare these clasps with commercially available injection-molded resin clasps\textsuperscript{10}.

We therefore evaluated the flexural properties and fatigue resistance of the experimental polyester disk (mPE) material specifically manufactured for fabricating resin clasps of RPDs. A material designed for clasps must be evaluated on the basis of its resistance to plastic deformation and fracture against cyclic flexural fatigue because retentive clasps are repeatedly flexed in patients’ mouths. The polyester material was chosen because it has been widely used as an injection-molded thermoplastic denture base incorporating resin clasps, which could accurately fit\textsuperscript{10} inside the mouth and resist the permanent deformation\textsuperscript{17} acceptable for clinical service. Furthermore, the polyester material reportedly adheres more strongly to auto-polymerized resins than other thermoplastic resins\textsuperscript{8,18}, which may be beneficial to denture repairs. This study evaluated the flexural and fatigue properties of an experimental polyester disk material for milled resin clasps and compared them with those of conventional injection-molded polyester for its application as resin clasps. The null hypothesis was that there is no difference in the flexural and fatigue properties between the experimental polyester disk and
the conventional injection-molded polyester.

MATERIALS AND METHODS

Specimen preparation
Injection-molded polyester (iPE; control) (EstheShot Bright, Nissin, Kyoto, Japan), an experimental polyester disk (mPE) (EstheShot Bright trial product, Nissin), and a polymethyl methacrylate disk (mPMMA; reference) (Bellezza PMMA DISK, Nissin) were used in this study (Table 1). Rectangular plates (30×30×2.5 mm) were manually injected using an injection-molding system (MIS-II, i-Cast, Kyoto, Japan) to prepare the iPE in accordance with the manufacturer’s instructions (90°C for 4 h; preheating conditions: 280°C for 20 min; injection pressure 1.0 MPa). The mPE disk (150×150×20 mm) was prepared by heating the same polyester pellets used to prepare the iPE plates under conditions similar to those for manual injection molding. The mPE and mPMMA plates were cut from their respective disks.

Three-point bending test
Each specimen was subjected to a three-point bending test using a universal testing machine (Autograph AGS-H, Shimadzu, Kyoto, Japan) with a 20 mm support span and a crosshead velocity of 1.0 mm/min after one week of storage in distilled water at 37°C (n=10 for each group). The flexural strength was calculated based on the load at a fracture point or the maximum load in the absence of a fracture, and a 0.02% yield strength was defined by the tolerance of the stress–strain curve and the 0.02% offset line by a software operation (TRAPEZIUM X, Shimadzu).

The flexural strength and elastic modulus were calculated using the following formulas:

Flexural strength=$\frac{3PL}{2bh^2}$ (1),
Elastic modulus=$\frac{FL}{4dth^3}$ (2),

where $P$ is the maximum load (N), $L$ is the support span (mm), and $b$ and $h$ are the specimen width and thickness (mm), respectively. Furthermore, $F$ is the load (N) at the proportionate point on the load–deformation curve, and $d$ is the deformation (mm) at load $F$.

Cyclic flexural fatigue test
For the cyclic flexural fatigue tests, all specimens had the same dimensions as those used in the three-point bending test and were stored in distilled water at 37°C for one week before the test. The fatigue tests were performed using a cyclic three-point bending with a 20 mm support span by sinusoidally loading the specimens at 2 Hz in water at 37°C for 15,000 cycles. Cyclic unipolar deflections from 0.075 to 0.75 mm and 0.15 to 1.5 mm were applied using an electromagnetic force micromaterial tester (MMT-250N, Shimadzu, Fig. 1). The deflection at 0.75 mm was calculated using the same strain as in a previous cantilever test6, assuming a resin clasp showing a 0.5 mm retention undercut. The specimen load and deflection were recorded during all 40 cycles and at every 1,000 cycles during the cyclic test using dynamic data acquisition software (DCS-100A, Kyowa Electronic Instruments, Tokyo, Japan). When the maximum load during the test decreased below 5 N, the mPE and mPMMA specimens were considered fractured, and the cyclic test was halted. The residual strain was calculated as follows (Fig. 2): A stress–strain curve was derived from a load–deformation curve. The flexural stress

Table 1  Materials used in this study

| Material | Abbreviation | Product name | Manufacturer | Processing method |
|----------|--------------|--------------|--------------|------------------|
| Polyester; polycycloalkylene terephthalate copolymer (PCAT)† | iPE | EstheShot Bright | Nissin, Kyoto, Japan | Injection molding |
| Polyester; PCAT | mPE | EstheShot Bright Experiment Disk | Nissin | Milling |
| Polymethyl methacrylate | mPMMA | Bellezza PMMA Disk | Nissin | Milling |
and strain at x mm deflection were calculated using the following formulas:

\[
\text{Flexural stress} = \frac{3P_xL}{2bh^2} \quad (3),
\]

\[
\text{Flexural strain} = \frac{6Xh}{L^2} \quad (4),
\]

where \(P_x\) is the load (N) at x mm deflection, \(L\) is the support span (mm), and \(b\) and \(h\) are the specimen width and thickness (mm), respectively. Thereafter, the regression line was calculated using the linear part of the stress–strain curve generated during unloading. Finally, the residual strain was defined as the difference between the strain calculated at 0 N during the first cycle and that calculated at every 1,000th cycle. Representative specimens were examined after the cyclic flexural tests using an optical stereomicroscope (SMZ 1000, Nikon, Osaka, Japan) with 50× magnification.

The data from all the tests were analyzed using the statistical software SPSS 11.5J for Windows (SPSS, Chicago, IL, USA). For the three-point bending test, the data on the flexural properties of each material were analyzed with the Kruskal–Wallis multiple comparison and pairwise tests. For the fatigue test, the residual strain of the mPE and mPMMA at each cycle were compared with those of the iPE specimen by the Mann–Whitney \(U\) test; All \(p\)-values less than 0.05 were considered statistically significant.

### RESULTS

The flexural properties of the materials are summarized in Table 2. Although none of the mPE or iPE specimens fractured during the three-point bending test, all the mPMMA specimens did. The mPE showed a significantly higher flexural strength (72.44±1.48 MPa) than the iPE (66.28±0.89 MPa, \(p=0.033\)), whereas there was no significant difference in the flexural modulus and 0.02% yield strength (\(p>0.05\)). The mPMMA showed a higher flexural modulus (2.93±0.08 GPa), flexural strength (123.40±4.23 MPa), and 0.02% yield strength (36.83±3.48 MPa) than the iPE and mPE (\(p<0.05\)).

Figure 3 shows the digital microscopic images of typical specimens after the cyclic flexural tests. No visible fracture or deformation was observed in any of the materials in the cyclic deflection at 0.75 mm, while obvious cracks were observed in the mPE and mPMMA at 1.5 mm. Figure 4 shows representative load–deflection curves generated during loading and unloading at the first and 5,000th cycle in the 1.5 mm deflection. Although the load required for the deflection at the 5,000th cycle was lower than that required for the deflection at the first cycle, the slopes of the load–deflection curves were essentially identical. However, the mPMMA required the highest load for deflection among all the materials, and it fractured within the first 1,000 cycles.

Figures 5 and 6 show the mean residual strain at each cycle in the cyclic deflections at 0.75 and 1.5 mm, respectively. For the cyclic deflection at 0.75 mm, the mPE showed a significantly lower residual strain (0.15±0.05%) than the iPE control specimen (0.21±0.06%, \(p=0.011\)), and the mPMMA showed significantly higher residual strain (0.29±0.09%) than the mPE (\(p<0.001\)) in the 15,000th cycle. For the cyclic deflection at 1.5 mm, the mPE showed significantly lower residual strain (0.76±0.31%) than the iPE (1.07±0.23%, \(p=0.004\)) until the 8,000th cycle; however, the residual strain of the mPE increased rapidly after 8,000 cycles.

![Fig. 2 Relationship between residual strain and stress–strain curves of the cyclic flexural test.](image)

**Table 2**  Flexural properties measured in three-point bending tests (Mean±SD)

| Properties             | iPE         | mPE         | mPMMA      |
|------------------------|-------------|-------------|------------|
| Flexural modulus (GPa) | 1.51±0.03*  | 1.46±0.04*  | 2.93±0.08b |
| Flexural strength (MPa)| 66.28±0.89* | 72.44±1.48b | 123.40±4.23c |
| 0.02% yield strength (MPa)| 27.20±1.95* | 28.81±1.83* | 36.83±3.48b |

Superscript letters indicate statistical differences per horizontal row (\(p<0.05\)).
Fig. 3  Digital microscopic images of specimens at 50× magnification after the cyclic flexural test at 0.75 (upper) and 1.5 mm (lower) deflection; iPE (left), mPE (center), and mPMMA (right). All specimens were taken after 15,000 cycles except mPMMA at 1.5 mm deflection, which was obtained after 373 cycles.

Fig. 4  Representative load–deflection curves from 0.15 to 1.5 mm deflection of iPE (left), mPE (center), and mPMMA (right). The blue curve shows the load required for deflection at the first cycle; orange curve, 5,000th cycle. In each cycle, upper and lower curves are at loading and unloading, respectively; mPMMA fractured before reaching 1,000 cycles.

Fig. 5  Residual strain at each cycle in the cyclic flexural test at 0.75 mm deflection. Asterisks show a significant difference compared with the residual strain of mPE at each cycle (p<0.05).

Fig. 6  Residual strain at each cycle in cyclic flexural test at 1.50 mm deflection. Asterisks denote a significant difference compared with the residual strain of mPE at each cycle (p<0.05).
DISCUSSION

The mPE was shown to have a higher flexural strength and lower residual strain compared to the iPE; therefore, the null hypothesis was rejected. The resin clasp of the RPDs milled from the mPE disk was more durable and showed higher resistance to deformation and flexural fatigue than the injection-molded RPDs.

Regarding the mPE for milling processing, conventional injection-molded iPE was used as a control, while mPMMA, which represents the most common disk material for denture bases, was used as a reference. This was because the mechanical properties of the disk of polyamide, which is one of the most common materials used for resin clasps, have not been reported. Therefore, we used PMMA disk material (mPMMA) as a reference represented the most common disk material for denture base. The mPMMA showed an elastic modulus value approximately double that of the polyesters, and such a high brittleness caused it to fracture after exceeding the flexural strength. In contrast, the polyesters were sufficiently ductile and deformed without fracturing. These results of injection iPE and mPMMA were in agreement with those of previous studies. The mPE showed significantly higher flexural strength than the iPE, suggesting that the former was more resistant to damage. However, clasps undergo permanent deformation and fatigue fracture under repeated flexures caused by denture insertion and removal and mastication. Therefore, it was necessary to evaluate the fatigue deformation and fracture resistance using a cyclic deflection test.

Repeated loading for 15,000 cycles, corresponding to approximately 10 years of denture usage assuming that RPDs are inserted and removed an average of four times per day, was selected as the test condition. Assuming that the resin clasp showed a 0.5 mm undercut for retention, the strain at the 0.75 mm deflection was lower than that at the 0.02% yield strength (which is the elastic-limit index). Whereas, the strain at the 1.5 mm deflection was higher than that at the yield strength, but lower than that at the flexural strength. As a result, these differences in deflection affected the fatigue resistance.

The cycle-dependent change in the mPE and iPE load–deflection curves indicated that the load required for deflection was reduced by the plastic deformation while maintaining the elasticity because the slopes of the load–deflection curves of the initial and 5,000th cycles were essentially identical. Therefore, the lower residual strain indicated higher fatigue resistance. For the cyclic deflection at 0.75 mm, the mPE showed higher deformation resistance and lower residual strain than the iPE. A previous study found that polyester denture base resins show the highest deformation resistances; thus, the mPE may have the highest deformation resistance among the thermoplastic denture base materials.

Furthermore, the mPE showed higher deformation resistance and lower residual strain than the iPE for up to 8,000 cycles, even for the cyclic deflection at 1.5 mm. Unexpectedly, the residual strain of the mPE rapidly increased when cracks formed, while the manual injection-molded specimens did not show a drastic change. This is because the actual deflection applied to the specimen changed for the cyclic deflection at 1.5 mm. Owing to the permanent deformation, the load–displacement curve at the 5,000th cycle clearly shows a particular moment during which no load was applied to the specimen between the specimen unloading and loading (Fig. 1-A). This demonstrates that even if the maximum deflection applied to the iPE and mPE specimens was identical, the actual deflection applied to the iPE specimen was lower than that applied to the mPE specimen owing to the higher plastic deformation. Therefore, as the number of load cycles increases, the cumulative deflection applied to the mPE was higher than that applied to the iPE, which could fracture the mPE.

The flexural and fatigue properties of the industrially injection-molded mPE and the manually injection-molded iPE were different, even though both specimens were prepared from the same polyester pellets. The cause of these differences was difficult to determine; however, the factors that influence the flexural properties of crystalline polymers include molecular weight, molecular orientation, crystallinity, and thermal history. Our results suggest that among these factors, the increased crystallization may have affected the deformation resistance. Although all the resins were melted and injection-molded at the same temperature and pressure, the change in temperature during mass production may have differed from that observed in the manually injection-molded materials, which may have affected the crystallization.

Since the mPE showed an elastic modulus similar to that of the manually injection-molded iPE, the existing design of the resin clasps should be retained to achieve the same retentive force as injection molding. The resin clasps milled from the high-deformation-resistance mPE disk have the potential to accurately fit with the abutment teeth and may retain the bracing and retentive force required for long-term service. In contrast, the use of undercuts larger than 0.5 mm for retention should be avoided because the deflection of the resin clasp would increase, thereby increasing the risk of plastic deformation and fracture. Although the mPMMA did not fracture for the cyclic deflection at 0.75 mm, it should not be used as a resin clasp because the high residual strain caused the mPMMA to fracture easily.

Finally, the limitations of this study must be acknowledged. First, the variations in resin clasp thickness, width, and length, which depend on the width of the abutment tooth and the gingival morphology, were not considered. Therefore, the 0.5 mm undercut is not applicable to all dentures. The resin clasps are affected not only by insertion and removal, but also by chewing. In addition, further studies are needed to determine the crystallinity and machinability of the polyester disk.
CONCLUSION

The polyester disk used in milled resin clasps showed a higher flexural strength and a lower residual strain against cyclic deflection than the injection-molded polyester. Milled polyester resin clasps show increased durability and better deformation resistance against repeated deflection for long-term clinical use, while showing limited application potential at high deflection. Therefore, the milled polyester resin clasps are excellent candidates for application in CAD/CAM RPDs.

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