JOURNAL OF MATERIALS SCIENCE **37** (2002) 425–429

In vitro wear of composite veneering materials

M. ROSENTRITT, M. BEHR, E. HOFMANN, G. HANDEL
Department of Prosthetic Dentistry, University of Regensburg, Germany
E-mail: martin.rosentritt@klinilc.uni-regensburg.de

Wear resistance of restorative composites is important for clinical longevity and aesthetics, especially in posterior areas. *In vitro* examinations may contribute to improvements in the durability of composites depending on a thorough understanding of the wear behaviour. Artificial wear was performed on four labour fabricated veneering composites in a two-body masticator with all-ceramic antagonists and a three-body wear testing device using two kinds of food bolus. After ageing the wear track was determined in comparison to the unworn surface with a profilometer. Wear was correlated with the total filler amount and universal hardness. After millet three-body ageing all composites showed material loss between 2 and 3.5 μm, after millet sheet/rice of about 50–130 μm. The resulting wear track after mastication was about 180–300 μm deep. The composites showed different wear behaviour according to the used wear method. Between the different wear mechanisms and hardness or filler content no clear correlation could be determined. The wear performance of composites is a complex phenomena, with various *in vitro* tests different wear results could be obtained. Contrary to the general opinion, hard or highly filled composites must not necessarily show highest wear resistance.

1. Introduction

The development of dental materials like e.g. new restorative composites, especially for veneering a new class of prosthodontic frameworks, so called fibre-reinforced composites (FRC), demand for fast examinations to evaluate their theoretical clinical usability. An important factor affecting the intra-oral performance of dental materials is the aspect "wear"-behaviour, commonly described by terms like attrition, abrasion, corrosion and fatigue, among others [1]. Due to the complexity of the clinical wear, laboratory simulation tests allow to consider single parameters of the phenomenon. Splitting the process may help to perform screening tests and to interpret parts of the whole wear process. The *in vitro* wear of dental materials is performed by a great variety of simulation devices [2]. Wear in occlusal contact areas (OCA) is simulated e.g. by two-body wear [3, 4] whereas wear of contact-free areas (CFA) often is performed with three-body tests using a food slurry [5, 6]. A correlation of the *in vitro* results with clinical data is described and verifiable [3, 5–7]. The clinical evaluation of wear is achieved by different systems as discribed by Smith and Knight [8], USPHS (Ryuge) [9], Leinfelder [10] or Moffa and Lugassy [11] and recently scanner-supported by Kunzelmann [12]. But good correlation does not necessarily mean, that the wear mechanisms associated with *in vitro* tests are the same mechanisms that caused clinical wear, even if they are showing a comparable amount of substance loss. The aim of this investigation was to compare the wear resistance of four light- and heat-curing laboratory processed dental composites. The influences of two- and three-body-wear conditions on wear performance as well as two kinds of food bolus should be examined.

2. Materials and methods

2.1. Two-body simulation

From the materials (Table I) rhombic specimen ($h = 2$ mm, $l = 15$ mm, $w = 10$ mm) were produced according to the manufacturer's instructions using the adequate polymerisation devices. The surface of the samples were abraded with sandpaper (final grid: 2400) to remove superficial oxygen inhibition and to flatten the surface. As a reference untreated bovine enamel was used. Bovine teeth were stored in 0.5% Thymol solution and cut in sample size 2 hrs before testing. Before artificial mastication the surfaces of all samples were investigated using a surface roughness testing device (Perthometer S6P, Perthen-Feinprüf, $l = 5.6$) and the total weight was determined (R160P, Satorius GmbH, Göttingen). 24 hrs after manufacturing the samples were fixed on a slope surface (45°) sample holder. The sample holders were mounted onto a ball bearing mounted slope surface of the oral simulator. In each mastication cycle, the antagonist (Empress 2, Ivoclar, $d = 5$ mm) was moving down and contacting the sample. A further lowering of the antagonist causes that both, holder and sample, were pushed in horizontal direction by the mastication force, resulting in a sliding movement of the antagonist sphere over the composite sample surface. By limiting the horizontal movement of the slope surface (2 mm) the resulting sliding direction and value were defined. This testing design tried to simulate a first occlusal impact contact and

TABLE I Overview of the tested materials

Material	BelleGlass	Conquest Sculpture	Sinfony	Targis
Batch no.	808472	830971	015	8610
Manufacturer	Girrbach, G	Jeneric Pentron, USA	Espe, G	Ivoclar, FL
Polymerisation	N_2, 140°C, 80psi, (10 min)	107°C, vacuum (15 min)	light, vacuum (15 min)	95°C (7 min), light (22 min)
Inorganic Filler Volume [%]	74	72	44	74
Hardness UHRD	363	347	259	450

further attrition of the dentition [4]. After 400 thermal cycles (5°C/55°C, 2 min each cycle, distilled water to rinse wear debris and simulate changing mouth temperatures) in combination with 160,000 mechanical cycles (with 20 N mastication force, frequency: 1 Hz) total weight and resulting abrasion gap were determined. Weight loss was calculated as difference between weight before and after simulation. The gap depth was measured in the middle of the wear trace in comparison to the unworn surface.

2.2. Three-body-simulation

All tests were performed with a wear testing machine (Willytec, München Germany) similar to the ACTA device described by de Gee [5]. With the help of the round sampler holder as a mould the specimens were fabricated as described in part 1. After preparation of the individual 12 chambers of the round ACTA sample holder with tribochemical treatment (Rocatec, Espe, Germany) all samples were adhesively luted (Variolink II, Ivoclar) onto the holder. 24 hrs after manufacturing the equipped sample holder was abraded round ($d = 50$ mm, $w = 10$ mm) with diamond wheels ($d = 16$ mm, $w = 6$ mm). The smaller width of the antagonist causes a 6 mm wear trace only in the middle of the samples. The unworn areas serve as control. Tests were performed using (A) millet seed (150 g) and (B) millet seed shells (30 g) and rice (120 g) which were ground in a rotating blade grinder (Moulinette, Moulinex) for 60 s. Both media were mixed with 275 ml distilled water and the food bolus was allowed to swell for 1 hr before starting the test (max. 200,000 cycles, 15 N, antagonist wheel 60 U/min, sample wheel: 130 U/min contra-rotating, resulting slip: −15%). After testing the resulting trace depth of the abrasion gap was measured using a roughness testing device (Perthometer S6P, Perthen-Feinprüf, $l = 5.6$, diamond). To show the abrasion development, during test (B) the wear track of all samples were measured after 50,000, 100,000, 150,000 and finally 200,000 cycles.

The universal hardness UHRD of two samples of each series was measured using the universal testing machine (Zwick 1446, Zwick, Ulm) in combination with a hardness measuring device (Zwick UHRD, testing force: 15 N, Zwick). Inorganic filler volume content was determined using thermal gravimetry (TG 1500, Rheometric Scientific, GB) by heating composite samples from room temperature to 600°C (heating rate 10 K/min) and keeping 600°C for 10 minutes. In reference to DIN 13922 the filler volume content was calculated as the weight of the remaining material. Statistical analysis was performed using Mann-Whitney-U-Test ($\alpha = 0.05$) and regression analysis (SPSS 6.01 Windows)[13].

3. Results

The total weight loss after artificial chewing was about 1% for all composites without significant differences between the single values. The abrasion rate using millet was about 2–3.5 μm with the composite Sinfony significant showing the lowest results (Fig. 1). A similar looking abrasion distribution could be found after artificial mastication though the abrasion results were about 70–200% higher. The values were between 180 and 300 μm (Fig. 2). Sinfony showed the significant lowest results in comparison to the other composites. For the control enamel an about 50% significant lower abrasion in comparison to the composite with lowest

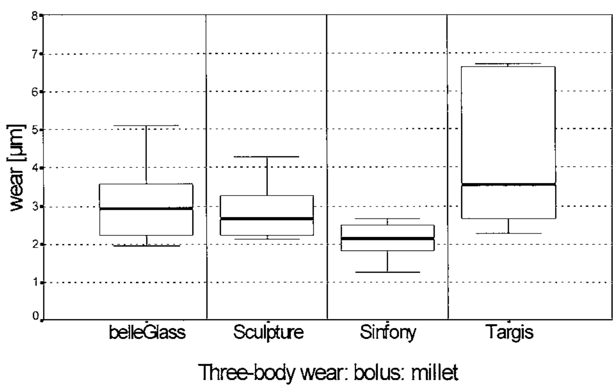

Figure 1 Three-body wear with millet (μm) (median, 25%/75% percentiles, min, max).

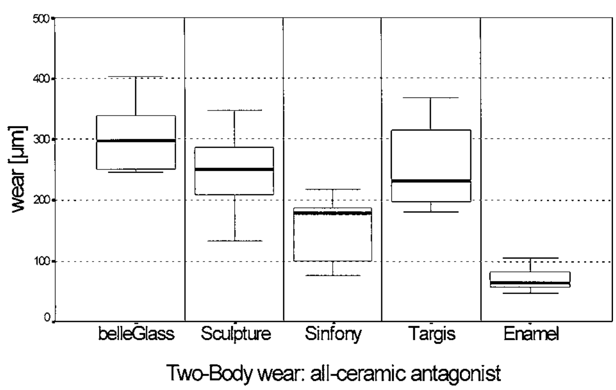

Figure 2 Two-body wear (μm) (median, 25%/75% percentiles, min, max).

TABLE II Statistical analysis of wear performance (Mann-Whitney-U-test, $P = 0.05$)

Two-body/three-body millet/three-body millet sheet-rice	BelleGlass	Conquest Sculpture	Sinfony	Targis
Conquest Sculpture	0.2135/0.7568/0.0909			
Sinfony	0.0059/0.0299/0.0000	0.0065/0.0052/0.0000		
Targis	0.3359/0.2660/0.0000	0.8065/0.3968/0.0000	0.0037/0.0045/0.0000	
Enamel	0.0026/-/-	0.0006/-/-	0.0034/-/-	0.0009/-/-

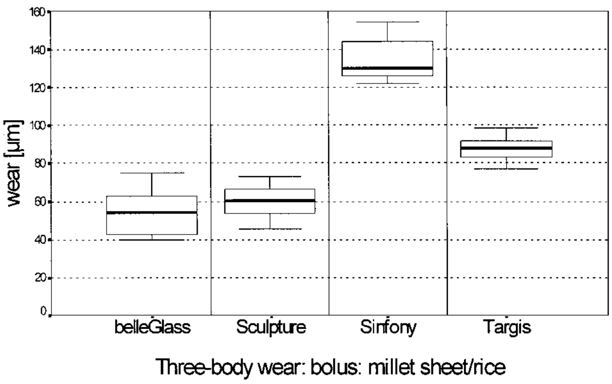

Figure 3 Three-body wear with millet sheet/rice (μm) (median, 25%/75% percentiles, min, max).

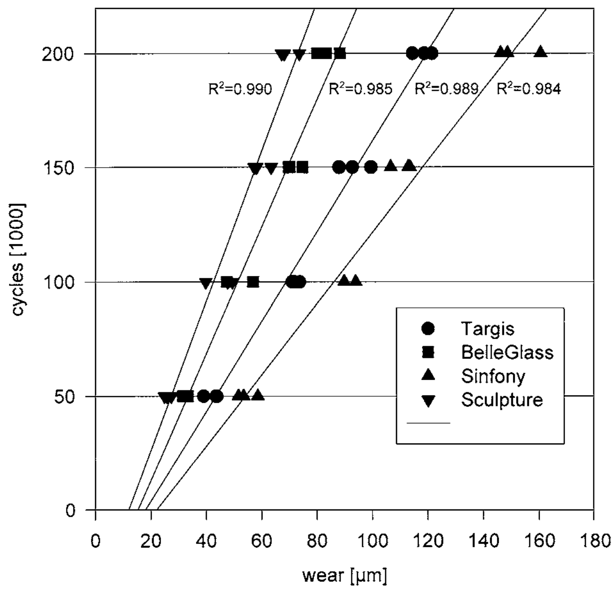

Figure 4 Three-body wear with millet sheet/rice after different wear cycles.

substance loss could be determined. The abrasion rate using millet sheet was about 20 times higher—between 50–130 μm—using just millet with highest abrasion for the composite Sinfony (Fig. 3). Only Conquest Sculpture and BelleGlass were not statistically different. The wear course after 50,000 to 200,000 cycles of three body wear with millet sheet/rice is showing a linear relationship between wear cycles and wear (Fig. 4). Statistical analysis is displayed in Table II. The UHRD hardness scale showed highest values (= highest hardness) for Targis, followed by BelleGlass, Conquest Sculpture and finally by Sinfony. All results are shown in Table I. Fig. 6 shows an exemplary wear track after three body millet/rice wear. The general inorganic filler content varied from 70–75 Vol%, only Sinfony showed values of 44 Vol% (Table I).

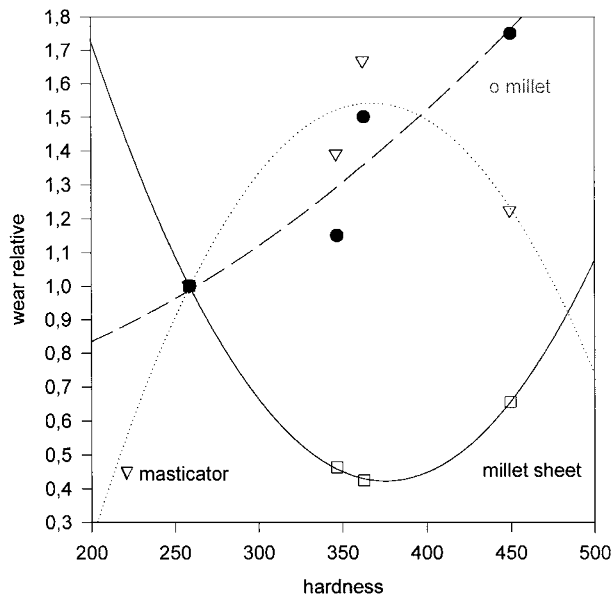

Figure 5 Correlation between relative wear and hardness.

4. Discussion

This investigation clearly distinguished the variety of results determined with different *in vitro* wear tests. After three-body millet abrasion wear rates lower than 5 μm could be determined, after three-body millet sheet/rice wear the amount of abrasion increased about 20 to 60 times and after artificial mastication about 100 times in comparison to three-body millet wear. The enamel reference tested in the artificial environment exhibited significantly higher wear resistance in comparison to the veneering composites.

The two systems of three-body wear were performed using identical wear parameters differing only in the kind of used food bolus. The composites' behaviour was distinct: the softest composite had lowest wear after three-body millet wear and highest wear of all tested materials after three-body millet sheet/rice testing. The results confirm investigations [14], that the *in vitro* wear pattern of the composites is affected by the nature of the food slurry. Differences in food bolus film layer between 2–10 μm [15] may contribute to this wear behaviour. Besides the amount, the type and design of fillers and the filler treatment may influence the wear behaviour of the composite. The interparticle spacing between the filler components is of special interest [16].

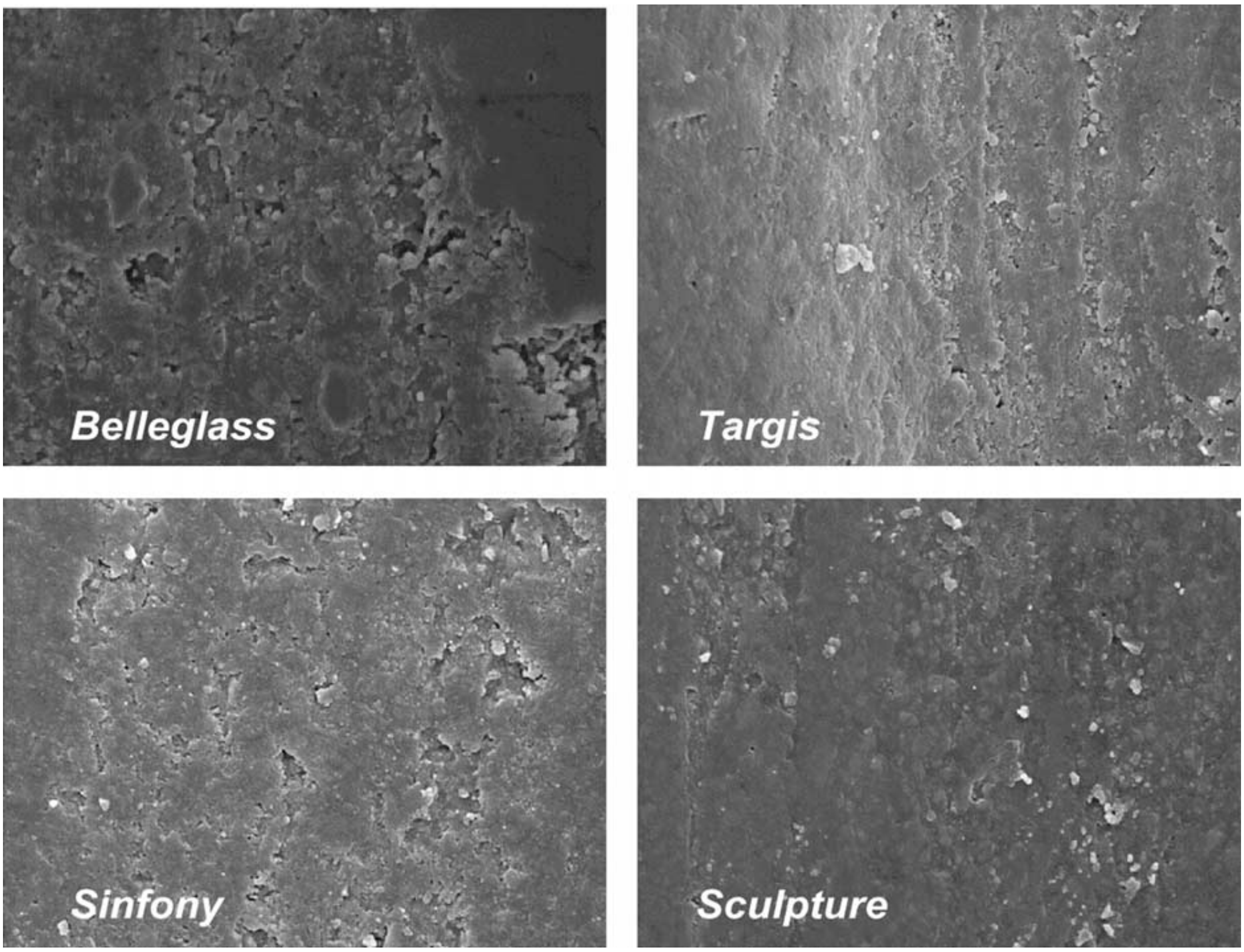

Figure 6 SEM photographs of composite surface after wear testing with millet sheet/rice.

Bayne *et al*. described that fillers protect the matrix from wear, so highly filled composites showed remarkable high wear resistance under abrasive loading [17]. The force transition between food bolus and filler particle seems to be more homogenous for spherical fillers in contrast to irregular fillers [18], force peaks at the interface filler-matrix were minimised. There were wear differences due to the filler shape (sperical-irregular) and additional influences due to the hardness of fillers [18]. Hart food bolus seemed to abrade the matrix and filler components nevertheless soft bolus only abrades matrix components of the composite. For three-body millet wear a linear correlation ($R^2 = 0.94$) between hardness and wear was found, whereas a logarithmic correlation ($R^2 = 0.99$) between hardness or filler content and wear could be determined for the three-body millet-sheet/rice simulation. SEM surface photographs show a smear layer on the soft composite, whereas for highly filled materials wear debris and chipping can be determined. Nevertheless the interface between filler and matrix, which is formed by silane coupling agents, is of importance. The debonding of fillers leads to protrusion and dislocation [16]. Regarding the comparable low total amount of wear after three-body millet testing, it is questionable whether a differentiation of composites by weight is possible and meaningful.

Comparing the results of three-body abrasion with the results after artificial mastication, wear behaved different. The two-body wear was about 200% higher than the wear after three-body millet testing. Although extremely different amounts of wear, similar ranking of the material was achieved for high abrasive two-body wear and 'mild' three-body millet abrasion: the softest material Sinfony showed the highest wear resistance. Nevertheless the mechanism of wear performance is different: SEM photographs showed a rough, smeared surface with chipping effects after artificial mastication and a scarred surface with debris after three-body wear. We suspect that this behaviour was caused by the ageing designs: artificial mastication and three-body millet wear caused attrition and sliding of the 'antagonist' on the sample, whereas three-body millet sheet/rice performed bolus abrasion. According to Ratledge *et al* . [19], the abrasion is correlated to the antagonist design and material. During mastication, the filler particles are fractured or dislodged, and can accelerate the wear process by acting as an abrasive. Problems with coupling agent effectiveness and hydrolysis have been proposed as possible weak links. Besides this the resulting wear debris which may act as an additional food bolus under artificial mastication conditions was rinsed away due to the continuing water cycling, whereas soft matrix was smeared. This may be the cause why the materials with higher filler level were not able to show any advantage over the low filled composite. Further investigations should show, that under long term and high loading conditions, the highly filled materials should perform higher wear resistance.

During wear testing the comparable filled materials Targis, Sculpture and Bellglass showed different results. This is suspected to be due to the different polymer system and the different conversion rate of the materials [20] due to polymerisation temperature, time and atmosphere. Pressure and a high temperature during polymerisation in combination with a non oxygen atmosphere were supposed to increase the degree of cure. A higher degree of cure can theoretical be achieved for microfilled composites in comparison to hybirds, due to their higher light transmission coefficient [21]. Hybrid composites seem to have higher localised wear resistance, and microfilled materials higher resistance to generalised wear [22]. The composition of the composite with polymer and filler components effects wear in different materials [23]. Further basic knowledge can be obtained only using experimental materials with known composition.

5. Conclusions

The wear performance of composites is a complex phenomena, with various *in vitro* tests different wear results could be obtained. Contrary to the general opinion, hard or highly filled composites must not necessarily show highest wear resistance. The stepwise approaching to the *in vitro* situation combining separate wear behaviour results may help to improve the knowledge of the complex clinical wear behaviour. The fast preclinical ranking shows differences between the composites and supports the choice of materials for further clinical application. Further developments of composite materials of both, filler and matrix components are necessary to improve the wear resistance of dental composites.

References

1. L. H. MAIR, T. A. STOLARSKI, T. R. W. VOWLES and C. H. LLOYD, *J. Dent.* **24** (1996) 141.
2. K. H. KUNZELMANN, *Trans. Acad. Dent. Mater.* **9** (1996) 89.
3. I. KREJCI, T. REICH, F. LUTZ and M. ALBERTONI, **100** (1990) 953.
4. M. ROSENTRITT, A. LEIBROCK, R. LANG, M. BEHR, P. SCHARNAGL and G. HANDEL, *Materials Testing* **39** (1997) 77.
5. A. J. DE GEE and P. PALLAV, *J. Dent. Suppl.* **22** (1994) 21.
6. J. R. CONDON and J. L. FERRACANE, *Dent. Mater.* **12** (1996) 218.
7. K. F. LEINFELDER and S. SUZUKI, *JADA* **9** (1999) 1347.
8. D. G. N. SMITH and J. K. KNIGHT, *Br. Dent. J.* **156** (1984) 435.
9. J. F. CVAR and G. RYGE. "Criteria for the Clinical Evaluation of Dental Restorative Materials" (United States Public Health Service, San Francisco, USA, 1971) p.1.
10. K. F. LEINFELDER, D. K. TAYLOR, W. W. BARKMEIER and A. J. GOLDBERG. *Dent. Mater.* **2** (1986) 198.
11. J. P. MOFFA and J. D. LUGASSY, *J. Dent. Res.* **65** (1986) 302 .
12. K. H. KUNZELMANN, R. PERRY, A. MEHL and R. HICKEL, *Deutsch Zahnärztl Z* **54** (1999) 575.
13. G. BROSIUS and F. BROSIUS. "SPSS Base System and Professional Statistics" (International Thomson Publishing, Albany, USA, 1995).
14. N. SATOU, A. M. KHAN, K. SATOU, H. SHINTANI, K. WAKASA and M. YAMAKI, *J. Oral Rehabil.* **19** (1992) 31.
15. P. PALLAV, A. J. DEGEE, A. WERNER and C. L. DAVIDSON, *J. Dent.* Res. **72** (1993) 56.
16. K. J. M. SÖDERHOLM and N. D. RICHARDS, *Gen. Dent.* **46** (1998) 256.
17. S. C. BAYNE, D. F. TAYLOR and H. O. HEYMANN, *Dent. Mater.* **8** (1992) 305.
18. S. SUZUKI, K. F. LEINFELDER, K. KAWAI and Y. TSUCHITANI, *Am. J. Dent.* **8** (1995) 173.
19. D. K. RATLEDGE, B. G. N. SMITH and R. F. WILSON, *J. Prosthet. Dent.* **72** (1994) 194.
20. J. L. FERRACANE, J. C. MITCHEM, J. R. CONDON and R. TODD. *J. Dent. Res.* **76** (1997) 1508.
21. M. KAWAGUCHI, T. FUKUSHIMA and K. MIYAZAKI. *ibid.* **73** (1994) 516.
22. K. KAWAI and T. TSUCHITANI, *J. Osaka Univ. Dent. Sch.* **34** (1994) 19.
23. M. W. BEATTY, M. L. SWARTZ, B. K. MOORE, R. W. PHILLIPS and T. A. ROBERTS, *J. Biomed. Mater. Res.* **40** (1995) 12.

Received 13 November 2000
and accepted 19 July 2001