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Evaluation of subsurface damage in CAD/CAM machined dental ceramics

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Commercial ceramics for dental computer aided design/computer aided manufacture (CAD/CAM) restorations suffer from surface chipping defects and microcracking. The influence of CAD/CAM machining of dental materials on the mechanical strength and extension of the damage zone was studied. Two different commercial dental ceramics, a feldspathic porcelain and a glass–ceramic, were CAD/CAM machined according to dental practice. The extension of the damage zone was analysed by a stepwise erosion of the surface, and the biaxial flexural strength was measured. To simulate the adhesive fixing of ceramic inlays, the specimens were sealed using a light-curing monomer. The different machining behaviour is dominated by the microstructure of the investigated materials. Owing to the high amount of glassy phase, the feldspathic porcelain shows extensive microcracking and chipping defects. The extent of the damage zone can be determined as 40–60 μm. Sealing of the surface did not affect the flexural strength of the machined samples. The dominating response to machining of the glass–ceramic is crushing and crumbling with a major contribution of plastic deformation on a microscopic scale. The extent of the damage zone is less than 20 μm. These cracks can be bridged by sealing of the surface, resulting in a substantial increase in strength. © *1998 Chapman & Hall*

1. Introduction

The increasing aesthetic demands of patients and excellent biocompatibility have led to growing interest in using ceramics as restorative materials in dentistry. Usually, the restorations are fabricated by slip-casting, controlled crystallization of glass or hot-pressing of precrystallized glasses. A lost-wax process is used to shape the molten glasses or glass–ceramics [1]. The properties of these materials are highly dependent on the skills of the dental technician. In addition, process-dependent microdefects reduce the fracture strength and increase the time-dependent failure probability of restorations [2].

Efforts to automate the production of dental restorations have initiated the development of computer-aided design/computer-aided manufacture (CAD/CAM) units to process dental ceramics [3]. Computer-aided manufacturing of dental restorations helps to reduce these limitations by using ceramic materials which are manufactured under highly controlled conditions with small variations in microstructure. Using commercially available CAD/CAM systems, the prefabricated ceramic blocks are machined with diamond grinding wheels to fit into tooth cavities. In spite of increased machinability and physical properties, all presently available materials for CAD/CAM machining suffer from chipping defects, surface flaws and microcracking [4, 5]. These defects not only reduce the accuracy of fit of the restorations [6], but may be the predominant cause for the reduction of mechanical strength and lifetime.

The aim of the present study was to investigate the influence of CAD/CAM machining on dental ceramics concerning mechanical strength and the extension of the damage zone.

2. Experimental procedure

Two commercially available ceramic materials were used in this study: Vitablocs^R Mark II (Vita Zahnfabrik, Bad Säckingen, Germany), a fine-particle feldspathic porcelain with 80 vol % glass matrix, and Dicor^R MGC (DeTrey/Dentsply, Konstanz, Germany) a tetrasilicic fluormica glass–ceramic with 30 vol % glass matrix (see Table I). The materials were machined with the Cerec 1^R-system (Siemens, Bensheim, Germany), a dental CAD/CAM machining device with a high-speed diamond grinding wheel. To reduce surface tension, a combined detergent and lubricant (CEREC^R DENTAGRIND 2000, Siemens AG, Bensheim, Germany) was added to the cooling water. Average grain size of the diamond particles was 64 μm and the unloaded surface speed of the grinding disc attained 45 m s^{-1}. The samples were machined to the

TABLE I Material properties of the investigated ceramics

	Vitablocs^R Mark II	Dicor^R MGC
Glassy matrix	K_2O–Na_2O–Al_2O_3–SiO_2	K_2O–MgF_2–MgO–SiO_2
Crystalline phase	Orthoclase K[$AlSi_3O_8$] Albite Na[$AlSi_3O_8$]	Fluormica $KMg_{2.5}Si_4O_{10}F_2$
Crystalline phase (vol %)	20	70
Young's modulus	63 GPa	70 GPa
$KHN_{0.1}$ (Knoop hardness)	330	520
K_{Ic} (Fracture toughness)	1.2 MPa m$^{1/2}$	1.5 MPa m$^{1/2}$

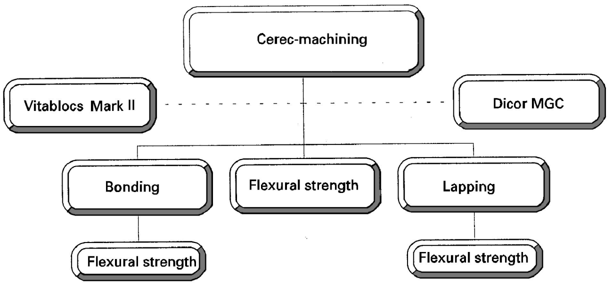

Figure 1 Flowchart of the investigated groups.

shape of a computer-generated rectangular test-inlay (10 mm × 10 mm × 1.4 mm) according to dental practice. To examine the surface morphology of the ground specimens, scanning electron microscopy (SEM, Leitz Isi Sr 50^R, Akashi, Japan) was used. The subsurface damage of the machined ceramics was evaluated using a confocal laser scanning microscope (CLSM, Carl Zeiss, Oberkochen, Germany). The strength of the machined samples was determined with the biaxial flexure test (ASTM Test: F394-78) [6] according to the "balls-on-three-balls" method [7] at a crosshead speed of 0.2 mm min^{-1} (Universal testing machine Zwicki^R, Zwick, Ulm, Germany).

To simulate the adhesive fixing of ceramic inlays in dental practice, the specimens were etched for 60 s (Vitablocs^R Mark II: 5% HF; Dicor^R MGC: HNO_3–HCl–HF mixture), silan Monobond S^R (Vivadent, Liechtenstein) and a thin layer of light-curing monomer (Heliobond^R, Vivadent, Liechtenstein) were applied. The bonding was light-cured for 20 s.

To detect the extension of the damage zone, the surface of the sample was lapped (PM2^R, Logitech, Scotland) in 20 µm steps with an Al_2O_3 abrasive (average grain size 3 µm) up to 100 µm. After each step, an average flexural strength was measured for 20 samples. Fig. 1 shows the experimental flow chart.

The sample data were analysed using the Wilcoxon-matched-pairs-test (SPSS^R 6.0.1 for Windows) and Weibull statistics [12]. The distributions of the strength values are investigated in boxplots. The lower boundary of the box is the 25th percentile, and the upper the 75th percentile. The line in the box represents the median. The mean 50% of the cases have values within the box. Cases with values less than 1.5 of box-lengths are marked with lines from the end of the box.

3. Results

The machined feldspathic porcelain exhibited brittle fracture mode with microcracking and large chipping defects. The sizes of the shell-shaped defects ranged laterally up to 40 µm (Fig. 2). In contrast, the glass–ceramic showed a combination of both plastically deformed areas and small brittle fractures. In Fig. 3, the ground furrows caused by the cutting disc can be seen. The distances (approximately 60 µm) correspond to the increment in positioning of the numerically controlled feed of the cutting disc. Compared to the feldspathic porcelain, the chipping defects were not predominant, material removal appeared to occur by crushing or crumbling. After lapping 50 µm, the surface morphology is comparable to that of the ground samples except that the removal appeared in a finer scale. Because of the opacity of the ceramic materials it was not possible to detect subsurface defects with the CLSM-method, only the surface defects could be investigated. The examination using CLSM showed a surface morphology of the machined samples similar to the SEM study (Figs 4 and 5). The section of the glass–ceramic vertical to the surface indicates furrows with a maximal depth of 5.7 µm, the section of the feldspathic porcelain shows chipping defects with a maximal depth of 6.5 µm.

Stepwise lapping of the feldspathic ceramic resulted in a significant increase of biaxial flexure strength, when surface layers greater than 40 µm were removed (Fig. 6). A further removal of the surface had no effect on fracture strength. Therefore, the extension of the damage zone can be estimated to be in the range of 40–60 µm. Bonding the surface resulted in a significant increase in flexural strength, but compared to the removal of the surface, the improvement was significantly minor. Lapping of the glass–ceramic up to 100 µm had no influence on flexural strength (Fig. 7), but bonding gave rise to a significant increase in strength of about 40%. The Weibull statistics (see Table II) resulted in a decrease of the modulus, *m*, after lapping of the feldspathic porcelain, whereas bonding increased the modulus. Lapping the glass–ceramic did not change the Weibull modulus, but after bonding of the surface an increase was observed.

4. Discussion

The two types of ceramics show a different machining behaviour according to Kelly *et al.* [5]. Material removal from the weaker feldspathic porcelain appears

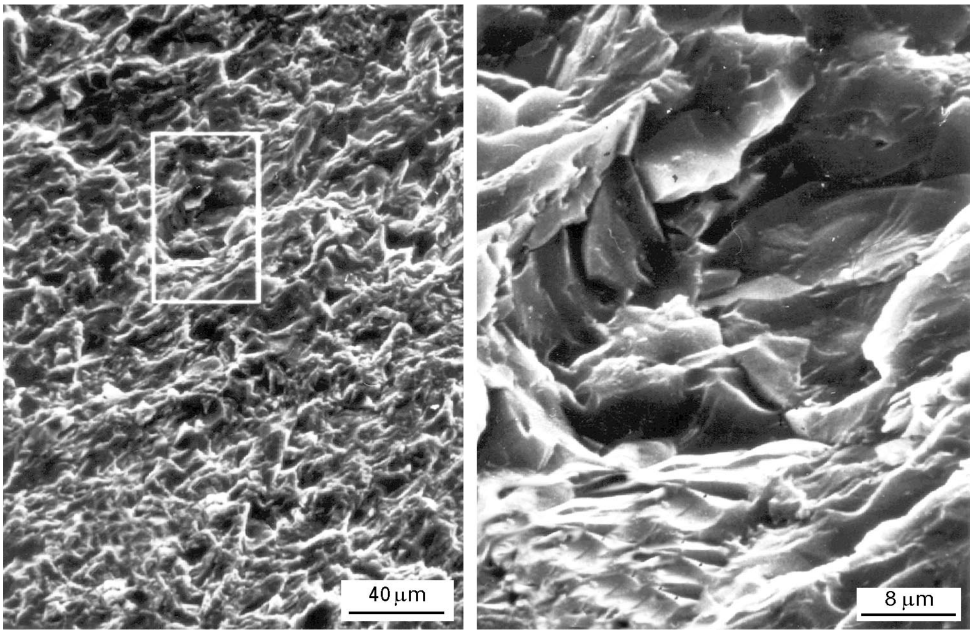

Figure 2 (a, b) Scanning electron micrographs of the machined surface of Vitablocs^R Mark II.

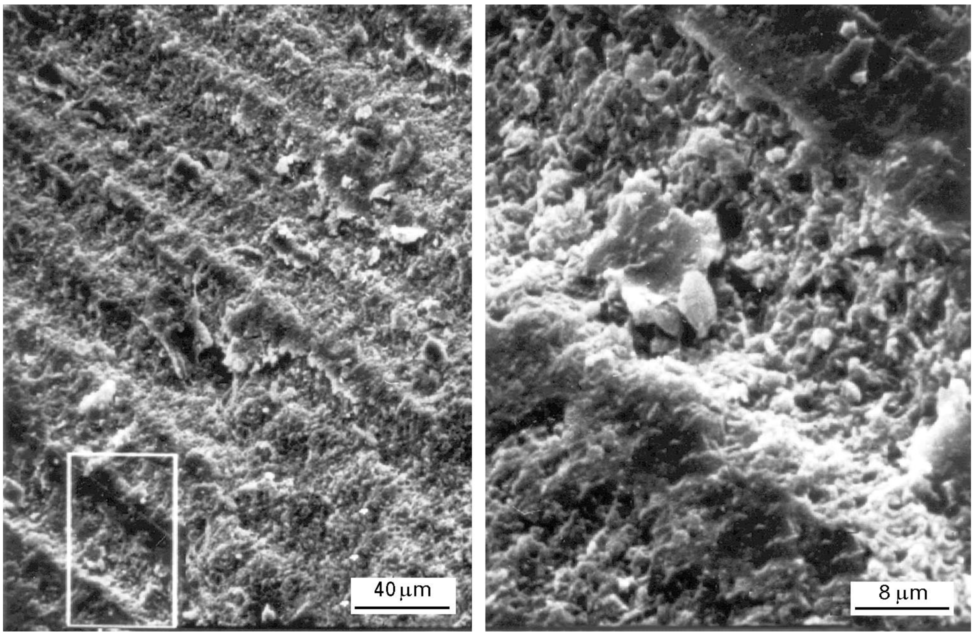

Figure 3 (a, b) Scanning electron micrographs of the machined surface of Dicor^R MGC.

to occur by microcracking or microchipping, whereas the predominant response of the glass–ceramic is crushing and crumpling. The grinding process of the glass–ceramic takes place by cleaving the micaceous crystals along their basal planes. Machining-initiated fractures follow the mica cleavage planes of the mica–glass interface; propagation across the basal planes is very difficult. Thus, fractures are repeatedly deflected and branched until the energy is consumed [10]. Therefore, a precise material removal by cleaving single layers is expected for the glass–ceramic. The surface morphology with the typical grinding furrows corresponds to this material property. However, the depth of the defects observed by CLSM did not differ from those of the porcelain (Figs 4 and 5). An explanation for this result could be the removal process of the porcelain. The removal occurs mainly in chipping areas parallel to the surface; compared to the lateral size, the depth of the defects is minor. Thus, deep grinding furrows were revised by a constant chipping of the surface. As a positive effect, this property results in a higher rate of material removal for the porcelain. For the glass–ceramic, studies showed a more difficult machining, a higher edge roughness and a reduced lifetime of the grinding discs compared to the feldspathic porcelain [9]. Additionally, the plastically deformed areas with indistinguishable grain boundaries reduce the material removal with increasing grinding cycles ("polishing wear") [5].

For all of the surface treatments examined, the mean fracture strengths of the glass–ceramic were significantly higher than the porcelain. This difference is caused by the interlocked microstructure of the glass–ceramic. As indicated earlier, the interlocked nature of the plate-like mica crystals results in a reinforcement of the material, whereas the porcelain with 80 vol % glassy matrix is lacking microstructural features which can stop crack propagation. Thus, a possible strengthening by the sanadine crystals seems to be of secondary importance. On the other hand, the high fracture toughness of the porcelain is explained by the strain field surrounding the sanadine crystals [10].

For the finish of hard and brittle materials such as ceramics, surface lapping is used. The advantages are the high precision and the exclusion of material defects bigger than the grain size of the abrasive (3 μm) caused by the treatment [11]. Therefore, the damage zone of the ground specimens was removed by lapping without inducing new microfailures, and the depth of the damage zone can be determined from the changes in flexural strength [11].

By comparing the flexural strength data for 50% failure ($\sigma_{0.5}$), 1% failure ($\sigma_{0.01}$) and the Weibull modulus, m, the effect of CAD/CAM machining on the mechanical properties can be discussed. For both ceramics, the flexural strength is dominated by machining-induced defects and can be improved by specific treatments.

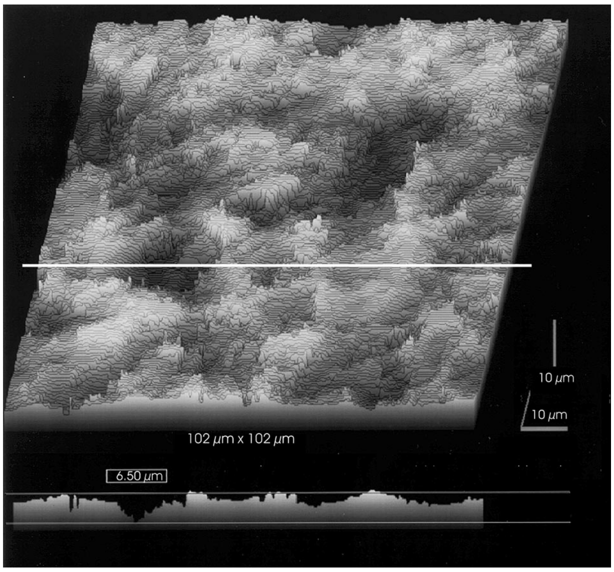

Figure 4 Confocal laser scanning micrograph of the machined surface of Vitablocs^R Mark II.

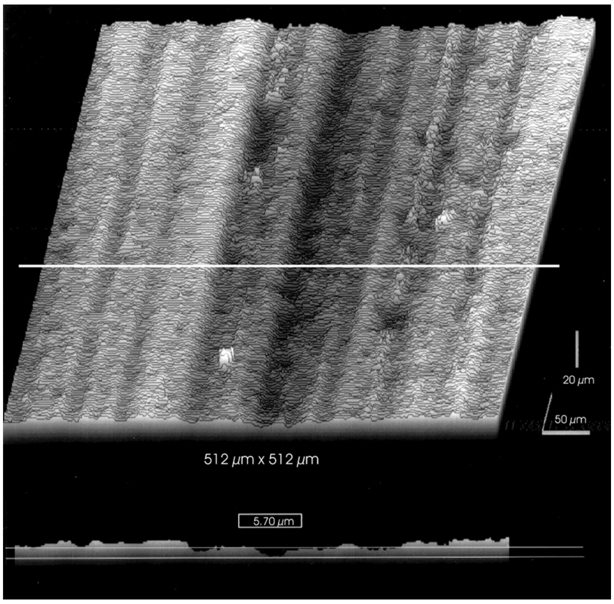

Figure 5 Confocal laser scanning micrograph of the machined surface of Dicor^R MGC.

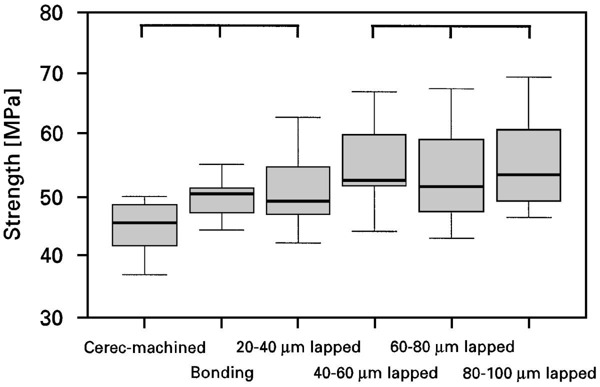

Figure 6 Boxplot of the Vitablocs^R Mark II groups. (┌┬┐) Groups showing no significant differences (Wilcoxon; $p < 0.05$).

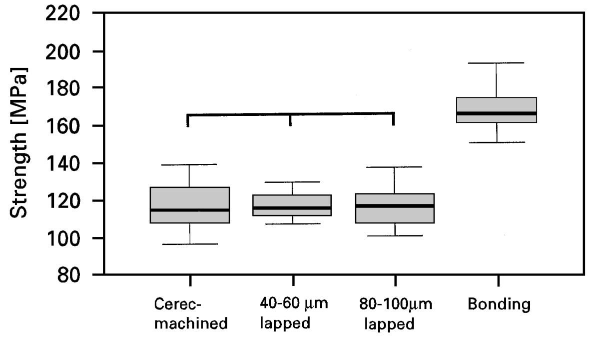

Figure 7 Boxplot of the Dicor^R MGC groups. (┌┬┐) Groups showing no significant differences (Wilcoxon; $p < 0.05$).

TABLE II Weibull statistics of the experimental data

	Vitablocs^R Mark II					Dicor^R MGC				
	σ_m (MPa)	S.D.	m	$\sigma_{0.01}$ (MPa)	$\sigma_{0.5}$ (MPa)	σ_m (MPa)	S.D.	m	$\sigma_{0.01}$ (MPa)	$\sigma_{0.5}$ (MPa)
Cerec-machined	45	4	11	31	45	116	13	9	74	118
40–60 μm lapped	54	7	8	32	54	114	11	9	72	115
80–100 μm lapped	55	7	6	33	56	115	11	11	80	116
Bonding	50	4	13	36	50	164	23	6	86	166

For the feldspathic porcelain, the major surface defects are located up to a depth of 40–60 μm. Lapping of this layer increases $\sigma_{0.5}$ by a factor of 20%. From the fact, that $\sigma_{0.01}$ is not affected by this lapping procedure, it can be concluded that there is at least a small number of cracks, which extend much deeper into the material. Consequently, the Weibull modulus is decreasing from 11 to 8.

By applying a small sealing layer (bonding) to the machined surface, narrow cracks can be bridged and both $\sigma_{0.5}$ and even more $\sigma_{0.01}$ increase. The Weibull modulus is higher compared to the machined surface.

Discussing the glass–ceramic, the surface defects after machining can be located in the range of a few micrometres. Lapping the surface with a mean grain size of 3 μm does not change the quality of the surface significantly. $\sigma_{0.5}$ is in the same range, as for the machined samples; the small increase in $\sigma_{0.01}$ indicates that there are only a few serious defects located in the outer surface layer.

A significant increase in strength can be observed for the sealing treatment (bonding). $\sigma_{0.5}$ is increased by a factor of 40% compared to the machined samples. The bonding material can infiltrate the small surface cracks and bridge them. From the fact that $\sigma_{0.01}$ is only increased by a factor of 16%, it can be concluded that there are additional failure origins which are dominating the fracture of the low-strength samples.

5. Conclusion

CAD/CAM machining of dental ceramics results in a subsurface damage, which is dominated by the microstructure of the material. The brittle feldspathic porcelain shows extended chipping and microcracking. The depth of the damage zone can be estimated to 40–60 μm by stepwise lapping of the machined surface. It is not possible to close the defects by bonding; only mechanical removal of the damage zone can improve the mechanical properties.

The dominant response of the glass–ceramic to CAD/CAM machining is crushing and crumbling together with plastic deformation in a microscopic scale. The extension of the surface defects can be located in the range of a few micrometres; lapping of the surface does not change the quality of the surface significantly. A substantial increase in strength can be observed after sealing the surface.

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