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CHAPTER 2

The influence of surface roughness on porcelain strength

2.1 Abstract

Objectives: In order to adjust occlusion, the functional surfaces of porcelain restorations are often ground and mechanical machining is even an essential part of the CAD-CAM process for these restorations. The aim of this study was to investigate the influence of the finishing procedures on the biaxial flexure strength of four commercial porcelains.

Methods: Four commercial porcelains of which two are used for metal-ceramic restorations (Flexo Ceram Dentine and Vita VMK68) and two for veneers and inlays (Duceram LFC Dentine and Cerinate BODY) are used in this study. For each porcelain, sixty discs (ø =22 mm, h = ~ 2.0 mm) were produced using twelve different finishing procedures. Twenty discs were left untreated, twenty discs were milled, using a high-speed diamond disc, and twenty discs were machined in a high-speed grinding/polishing device. Half of the samples were glazed. In each of these six groups, half of the samples were stored for 16 hrs at 80° C in a 4% acetic acid solution. The biaxial flexure strength was determined using the ball-on-ring method. In each group the roughness of the surface was determined and examined via SEM.

Results: With the exception of Flexo Ceram Dentine, a significant correlation was found between the roughness of the surface and the biaxial strength: the smoother the surface, the stronger the sample. The differences in biaxial strength may be attributed to the stress concentration of an applied load due to the roughness of the surface caused by the mechanical finishing or chemical action. The fact that the strength of Flexo Ceram Dentine was not affected by the different surface treatments is probably due to the size of the leucite particles, which apparently induce more stress concentration than the surface flaws and the roughness of the surface.

Significance: It was concluded that surface roughness determines the strength of a porcelain material, except where the inner structure of the material causes greater stress concentration than that caused by the combination of surface roughness and surface flaws.

2.2 Introduction

The essential step in CAD-CAM production of porcelain restorations is the mechanical machining of ceramic materials. Machining reshapes the surface and introduces surface flaws. The roughness produced by the finishing procedure and the introduction of surface flaws may be accompanied by a reduction in strength. It is indeed customary to expect that, within certain ranges, an increase in surface roughness will lead to a decrease in strength. According to the literature, strength is determined by the shape, sharpness, size and depth of the surface flaws as well as by internal defects [1]. Various techniques such as glazing have been proposed to strengthen the material after the introduction of surface flaws. The effectiveness of such strengthening mechanisms is disputed. Studies on dental porcelains both support and refute the strengthening effect of a glaze layer on porcelains [2-4].

The objective of the present study was to establish criteria for the influence of the surface finishing on the strength of dental porcelain. It was hypothesized that the surface roughness obtained by the different finishing procedures will concentrate an applied stress.

As a consequence of the stress concentration, failure will occur at a lower stress. The flaws which cause failure may not occur randomly, but rather may be concentrated around points with higher stress, as a result of surface roughness. For this reason, the influence of finishing procedures on the roughness and the biaxial flexure strength of four commercial porcelains were investigated.

2.3 Material and methods

Four commercial porcelains were investigated. Two of these are used for metal-ceramic restorations (Flexo Ceram Dentine, Elephant, Hoorn, the Netherlands and Vita VMK68, Vita Zahnfabrik H. Rauter GmbH & Co KG, Bad Säckingen, Germany), and two for veneers and inlays (Duceram LFC Dentine DA1, Ducera, Rosbach v.d.H, Germany and Cerinate Body, Den-Mat Corporation, Santa Maria, CA, U.S.A.).

For each porcelain sixty discs (ø = 22 mm, h = ~2.0 mm) were produced, using a stainless steel die with an internal diameter of 25 mm and a height of 3 mm.

The specimens were condensed with the condensing liquid, prescribed by the producer, by means of a vibration blotting technique. They were turned during blotting in an effort to avoid internal differences in the degree of porosity. All specimens were fired according to the manufacturer's instructions in a programmable vacuum furnace (Austromat 3001, Dekema, Freilassing, Germany). The fired discs were randomly divided into six groups:

Group 1, untreated. Group 2, glazed by heating the specimen to the glazing temperature prescribed by the manufacturer and holding it at that temperature for 1 min. Group 3, milled in the Celay milling machine (Mikroma Technologie AG, Spreitenbach, Switzerland), using a diamond disc with a diameter of 17 mm at a rotation speed of 80,000 rpm. Group 4, as 3 and also glazed and held at the prescribed temperature for 1 min. Group 5, ground/polished using a grinding/polishing machine, (WOCO, Wolfgang Conrad, Clausthal – Zellerfeld, Switzerland). Group 6, as 5 and also glazed and held at the prescribed temperature for 1 min.

Finally, half of the samples in each group were stored for 16 hrs at 80° C in a 4% acetic solution in accordance with DIN 13 925. The treatment in acetic acid was intended to create a surface roughness such as might develop through erosion due to the exposure to the different acid fluids in vivo.

The dimensions of the specimens were measured with a digital micrometer (Digimatic, Mitutoyo Corporation, Japan). The diameter of the discs was measured at two diametrically opposite points and the nominal mean value was recorded. The thickness of the discs was measured at five places in the center of the disc and the lowest value was recorded. The biaxial load was determined by means of the ball-on-ring method on a servomechanical testing instrument (Instron 6022, Instron Limited, High Wycombe, UK) at a cross-head speed of 0.1 mm/min at room temperature (20° C). The loading ball in the ball-on-ring device was made of stainless steel and had a diameter of 5 mm. The ring, which had a radius of 8.0 mm, consisted of twenty stainless steel supporting balls with a diameter of 2.5 mm. The primary virtue of the test fixture used in this study was that the lack of perfectly plane and parallel surfaces was compensated for in the fixture design. The supporting balls rested on an enclosed silicone ring that responded like a liquid. In this way, all the supporting balls provided the same load. The observed fracture loads and specimen dimensions were used in the calculation of the stress at fracture, as described in the literature [5], and in ASTM Standard [6] for biaxial flexure strength. In the calculations, a value of 0.25 for Poisson's ratio was used. The biaxial flexure strength distributions were hypothesized to follow the Weibull distribution.

The characteristic value of the biaxial flexure strength of each group of five specimens was determined. We then turned to the broken specimens of the biaxial flexure strength test.

Since the roughness of the five specimens of a material with a specific surface finishing did not show significant differences, the assumption was made that a single sample was characteristic of the whole set. From each group we selected a piece of the disc whose biaxial flexure strength was closest to the characteristic level of that group.

These were used for a roughness determination, by means of a profilometer (Perthometer equipped with feeler RHT 6-250, Perthen GmbH, Hannover, Germany).

The average degree of roughness R_a was determined in accordance with DIN 4768. The nominal mean value of five values of R_a was determined, measured at distance of 50 μm from one another.

The same samples were evaluated by scanning electron microscopy (SEM, Philips XL 20, Eindhoven, the Netherlands), in order to obtain an image of the broken test specimens. The images were taken in a standardized manner in the area where tensile stresses were highest during loading and perpendicular to the fracture line, i.e. the line between the fracture surface and the surface of the highest tensile stress. The surface of the highest tensile stress was placed at a 45° angle to the horizontal. In order to describe the roughness, characteristic rough spots (typical irregularities) were examined. Since the width of all rough spots was less than 50 μm, on the photomicrograph the apparent distance between the highest and the lowest point of a characteristic rough spot over a distance of 50 μm was measured and compared with the roughness data. This height difference was hypothesized as the dominant factor for the stress concentration.

Using Emrcnisa finite element linear elastic stress analysis (EMRC, Troy, Michigan, U.S.A.) the concentration of an applied stress caused by a characteristic rough spot on the fired and glazed Duceram LFC specimen was simulated.

Since the height of the roughness was small in comparison with the thickness of the discs, the stress concentration in the vicinity of the rough spot is a local stress concentration problem according to Peterson [7]. In the model of the surface layer, the tensile stress in the surface layer was considered constant and equal to the maximum tensile stress.

2.4 Results

Biaxial flexure strength

Table 2.1 shows the nominal mean value, the standard deviation, the Weibull characteristic strength and the Weibull shape factor of the biaxial flexure strength for the five specimens in each group.

Roughness

Table 2.2 presents the data on the surface roughness R_a. The SEM examination of the samples shows characteristic rough spots due to finishing, which occurred at least once in each 250 μm by 250 μm square. The apparent height difference over a distance of 50 μm displayed by a characteristic rough spot when measured by SEM was plotted as a function of the roughness R_a for Duceram LFC in Fig. 2.1. A regression analysis with the SSPS package version 8.0 (SSPS, Illinois, U.S.A.) was performed with the roughness R_a as the dependent and the difference in height of a characteristic rough spot as the independent variable. A linear relation was found to be significant ($P < 0.05$).

Table 2.1

The biaxial flexure strength of the finished materials. The nominal value, the standard deviation, the Weibull's characteristic level and the Weibull's shape factor (n=5; C.L. is the Weibull's characteristic value; S.F. is the Weibull's shape factor)

	Cerinate BODY				Duceram LFC				Flexo Ceram Dentine				Vita VMK68			
	Mean value MPa	Standard deviation MPa	Weibull's C.L. MPa	S.F.	Mean value MPa	Standard deviation MPa	Weibull's C.L. MPa	S.F.	Mean value MPa	Standard deviation MPa	Weibull's C.L. MPa	S.F.	Mean value MPa	Standard deviation MPa	Weibull's C.L. MPa	S.F.
Untreated	81.1	23.5	92.8	3.0	42.3	15.4	49.6	2.3	53.5	9.0	58.8	5.4	67.5	1.5	66.9	40.9
Untreated, 16 hrs 4% acid	69.5	34.9	81.1	1.6	56.5	4.0	56.2	16.5	60.6	5.7	62.4	14.2	74.6	10.1	79.9	6.7
Untreated, glazed	82.3	9.9	84.7	8.3	25.5	2.0	25.7	23.5	58.3	3.9	60.0	13.1	73.0	27.9	83.3	2.1
Untreated, glazed, 16 hrs 4% acid	64.6	4.9	68.3	12.0	79.8	20.4	89.5	3.5	52.8	9.2	56.2	5.0	79.5	7.5	82.3	9.5
Milled	53.3	22.2	62.0	2.0	51.1	7.2	53.8	7.1	61.6	6.7	66.3	7.6	68.3	9.4	71.4	1.6
Milled, 16 hrs 4% acid	59.9	7.1	63.4	9.6	66.5	3.9	68.0	23.7	66.5	9.5	71.4	7.8	74.8	15.3	80.9	5.1
Milled, glazed	96.2	9.2	100.4	10.5	89.2	15.4	97.3	5.6	57.9	3.7	59.3	18.4	77.0	8.2	79.4	13.9
Milled, glazed, 16 hrs 4% acid	104.6	20.0	113.6	5.9	87.7	14.1	94.3	6.4	58.2	5.1	58.7	13.1	77.1	13.0	81.1	7.0
Ground/polished	68.1	7.3	72.5	8.9	72.7	11.9	80.1	6.0	68.9	3.0	70.7	16.6	104.0	9.2	110.3	8.9
Ground/polished, 16 hrs 4% acid	105.1	18.6	113.3	6.8	96.0	12.1	104.3	7.7	63.7	6.7	67.4	8.2	94.0	14.9	98.3	9.0
Ground/polished, glazed	103.5	34.4	116.5	2.8	106.1	34.8	115.8	3.1	60.7	5.8	62.4	12.0	73.9	6.1	75.9	11.2
Ground/polished, glazed, 16 hrs 4% acid	117.6	38.0	136.2	3.0	139.6	26.6	151.6	5.2	59.8	9.3	61.8	9.3	87.0	10.5	91.5	10.1

Table 2.2

The surface roughness R_a of the finished materials (n=5; R_a is the average roughness height in μm)

	Cerinate BODY		Duceram LFC		Flexo Ceram Dentine		Vita VMK68	
	Mean value (μm)	Standard deviation (μm)	Mean value (μm)	Standard deviation (μm)	Mean value (μm)	Standard deviation (μm)	Mean value (μm)	Standard deviation (μm)
Untreated	1.8	0.5	4.5	0.7	2.0	0.6	6.8	6.0
Untreated, 16 hrs 4% acid	0.8	0.1	5.0	1.0	2.9	0.9	2.5	1.1
Untreated, glazed	0.5	0.2	4.4	0.1	0.5	0.1	0.9	0.1
Untreated, glazed, 16 hrs 4% acid	0.8	0.2	2.0	0.4	0.9	0.7	0.8	0.1
Milled	3.7	0.8	2.4	0.8	2.0	0.2	2.1	0.2
Milled, 16 hrs 4% acid	1.9	0.3	1.8	0.2	2.0	0.1	1.4	0.2
Milled, glazed	0.9	0.6	2.9	1.3	1.2	0.2	0.8	0.1
Milled, glazed, 16 hrs 4% acid	0.8	0.0	1.0	0.6	0.9	0.1	0.6	0.1
Ground/polished	0.6	0.1	1.0	0.1	0.5	0.1	0.7	0.1
Ground/polished, 16 hrs 4% acid	0.6	0.2	0.6	0.1	0.5	0.0	0.8	0.1
Ground/polished, glazed	0.3	0.0	0.5	0.1	0.6	0.2	0.5	0.0
Ground/polished, glazed, 16 hrs 4% acid	0.5	0.2	0.4	0.1	0.3	0.0	0.3	0.1

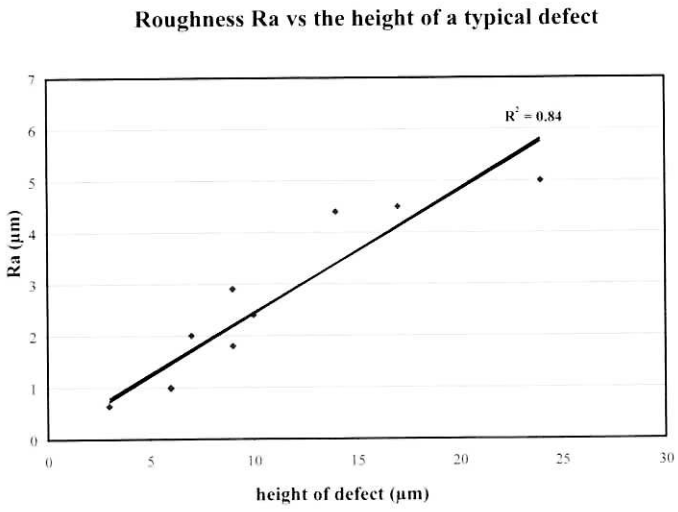

Fig 2.1 For Duceram LFC, the surface roughness as a function of the height difference of a characteristic rough spot.

Relation between surface roughness and porcelain strength

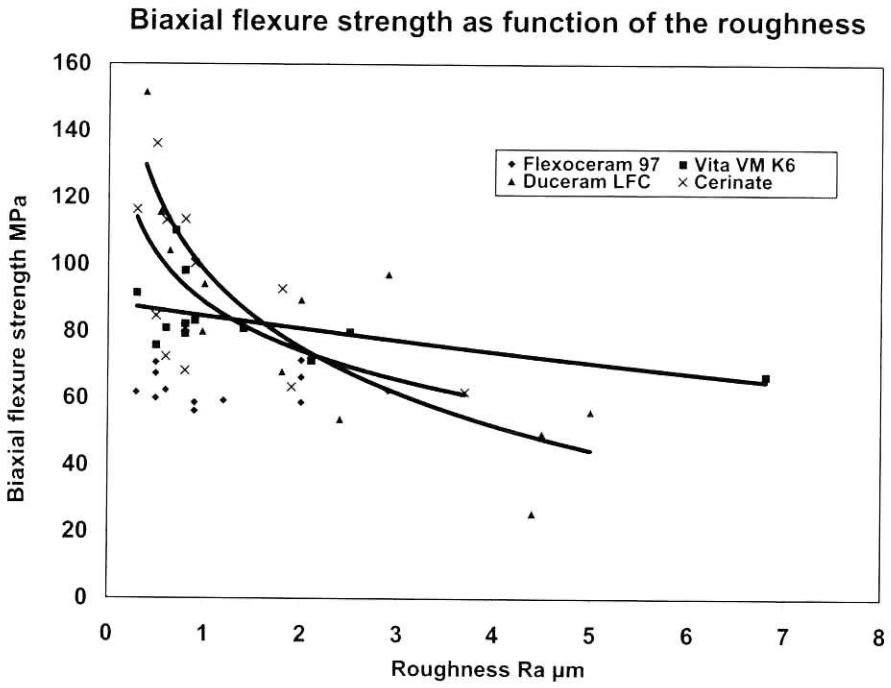

Fig. 2.2 The characteristic levels of the biaxial flexure strength as a function of the roughness R_a of the different types of finishing of the four materials

In Fig. 2.2, the characteristic levels of the biaxial flexure strength are plotted as a function of the roughness R_a of the four groups representing the four materials.

A regression analysis with the SSPS package version 8.0 was performed for the four materials with the biaxial flexure strength as the dependent and the roughness R_a as the independent variable.

Significant correlations were found, except in the case of Flexo Ceram Dentine ($P < 0.05$).

Effect of glaze on porcelain strength

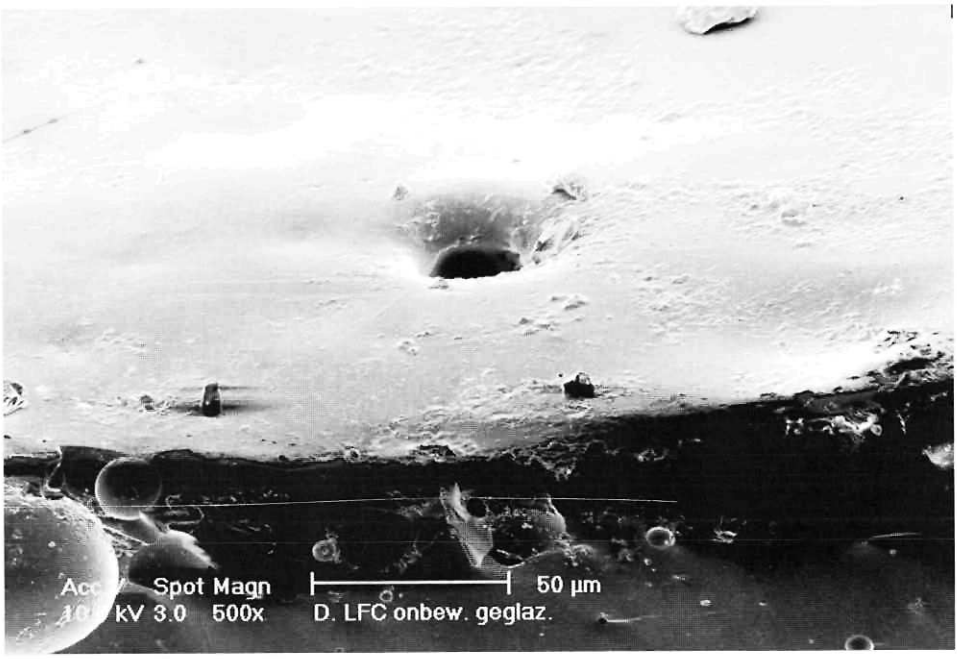

Fig. 2.3 A characteristic rough spot of fired and glazed Duceram LFC

Fig. 2.3 is a SEM photomicrograph of a characteristic rough spot on a broken fired and glazed Duceram LFC disc. In the samples that were only fired no defects of this kind appeared. In this group of specimens the glazing displayed a significant reduction in strength.

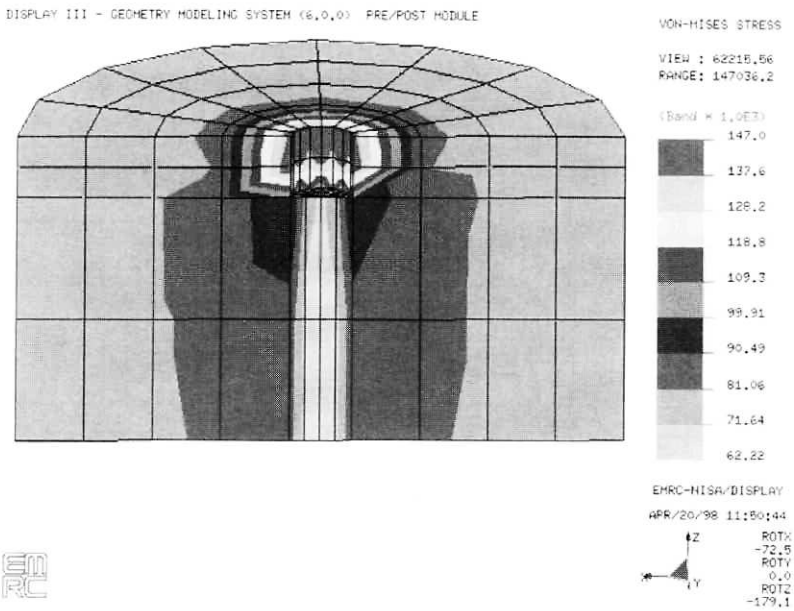

Fig. 2.4 Finite element analysis of the stress concentration of an applied stress caused by a characteristic rough spot of a fired and glazed Duceram LFC disc.

Fig. 2.4 is a finite element analysis plot of the stress concentration caused by a characteristic rough spot on a fired and glazed Duceram LFC disc, as shown in Fig. 2.3.

The applied stress used in the finite element analysis was 75 MPa, resulting in a stress of 137.6 to 147.0 MPa at the corner edges of the defect. The Von Mises stress at this spot is a tensile stress along the surfaces of the defect.

Fig. 2.5 SEM photomicrograph of fired and glazed Duceram LFC with cracks on the points with the highest stress concentration caused by glazing defects.

Fig. 2.5 is a SEM photomicrograph of a broken fired and glazed Duceram LFC disc with cracks at the points of the highest stress concentration.

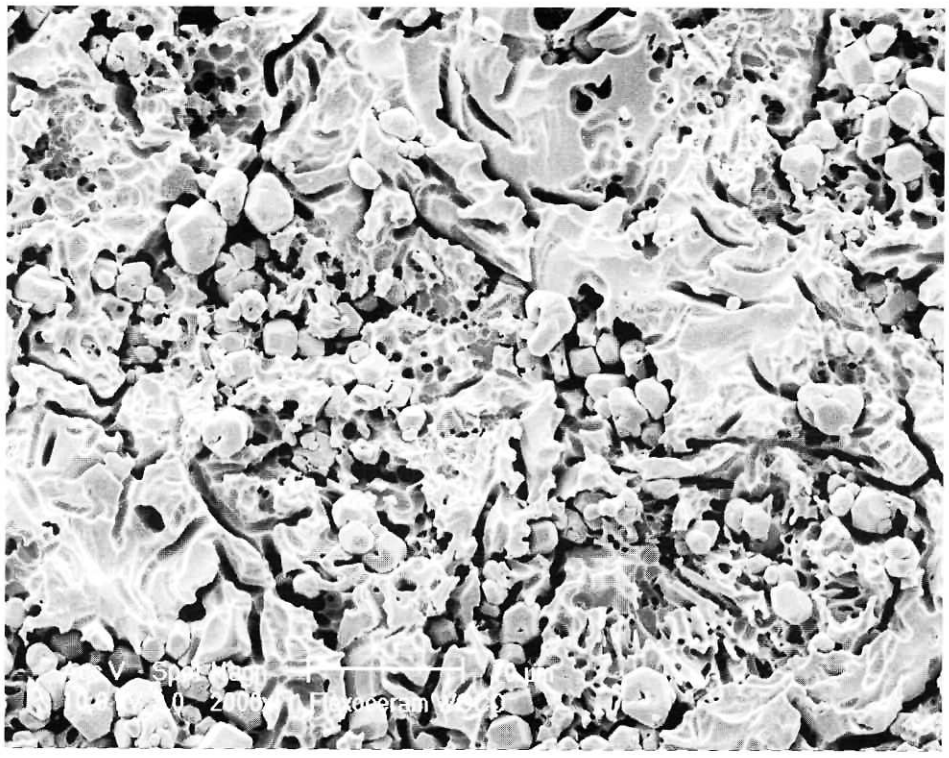

Fig. 2.6 SEM photomicrograph of an etched specimen of a broken Flexo Ceram Dentine disc.

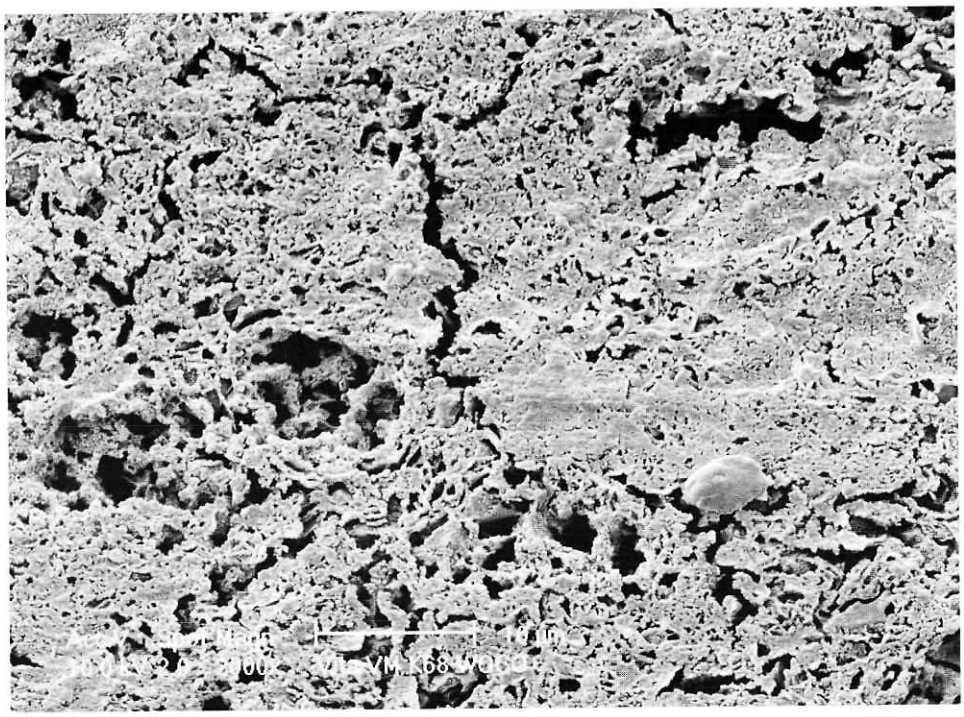

Fig. 2.7 SEM photomicrograph of an etched specimen of a broken Vita VMK 68 disc.

A comparison of the photomicrographs in fig. 2.6 and 2.7 clearly shows that the leucite particles in the Flexo Ceram Dentine are larger.

2.5 Discussion

The tentative conclusion is that the stress concentration due to the roughness of the surface caused by the different surface treatments is responsible for the differences between the biaxial flexure strength of the different sample groups. In the literature [8], the failure of many materials, including ceramics, has been attributed to the propagation of a large system of densely distributed cracks, rather than to a single precisely defined fracture. The number of cracks and microcracks is extremely large and, according to the literature, their location and orientation are random. Irwin [9] demonstrated that stress intensity is related to a crack shape in a particular location with respect to the loading geometry.

Microcracks have hardly influenced the measured surface roughness data with the method used in this study. The point of the feeler of the profilometer had an angle of 90 degrees with a radius-tip of 3 μm, too big to detect these microcracks.
The finishing procedures influence the existence of microcracks and residual stress. For example, glazing and treatment in acetic acid could round the crack tip of possibly microcracks. The change in crack length and crack tip radius would change the strength of the material. The finishing procedures, however, produce also a certain surface roughness. Surface roughness will lead to a non-uniform stress distribution and concentrate locally an applied stress due to the shape differences in the surface layer, as shown in Fig. 2.4. The distributed cracks may not develop or propagate randomly, but occur or propagate at points with higher stress as a result of the surface roughness. In Fig. 2.5, the points with the highest stress concentration are those with cracks, confirming that crack initialization can start at stress concentration points caused by surface roughness. This hypothesis is also supported by the work of Mecholsky *et al.* [10], who loaded samples with grinding grooves and gouges both perpendicular and parallel to the loading direction.

In the case of the samples with the tensile axis perpendicular to the grinding direction, this resulted in lower fracture strength and flaws resulting in failure generally being situated parallel to the grinding direction.

Grinding grooves or gouges parallel to the tensile axis will not cause stress concentration, while those perpendicular to the tensile axis will do so. This stress concentration will result in lower fracture strength and in failure-causing flaws being situated on the points with the highest stress parallel to the grinding direction.

The height difference of a rough spot over a distance of 50 μm will dominate the stress concentration.

The roughness R_a proved to be an indication for the height difference of a characteristic rough spot (Fig. 2.1).

In the dental porcelains studied here the relation between the biaxial flexure strength and the roughness R_a - with the exception of Flexo Ceram Dentine - supports the hypothesis that surface roughness will concentrate an applied stress, resulting in a lower biaxial flexure strength (Fig. 2.2).

In this study, the biaxial flexure strength of the four commercial porcelains for untreated versus untreated glazed, milled versus milled glazed and ground/polished versus ground/polished glazed reflects the different effects of glazing (Table 2.1). This is supported by studies on dental porcelains that both support and refute the strengthening effect of a glaze layer on porcelains [2-4].

The glazing of the untreated Duceram LFC samples resulted in a significant reduction in strength. Characteristic rough spots (Fig. 2.3) were found in the glazed samples and such spots did not appear in the untreated ones.
Subsurface pores in the untreated samples probably caused these defects. The considerable stress concentration caused by these rough spots (Fig. 2.4) may explain the lower biaxial flexure strength.

The glazing of milled and ground/polished Duceram LFC samples did not cause such defects, but rather had a smoothing effect on the roughness of the surface, which ultimately served to strengthen the material.

The conclusion that the stress concentration caused by higher or lower surface roughness after glazing is the dominant factor may be supported by the findings of Griggs *et al.* [4]. In that study flaws were created by means of a Vickers indenter. The researchers found no statistically significant improvement in biaxial flexure strength after glazing, as Giordano *et al.* [3] did, in the case of overglazing.
This could be due to the slightly lower depth-to-width ratio of the indentation after glazing, resulting in an only slightly lower stress concentration.

The roughness of the surface had no effect on the biaxial flexure strength of Flexo Ceram. The fact that the strength of Flexo Ceram Dentine was not altered by the various surface treatments may be due to the size of the leucite particles. Flexo Ceram Dentine and Vita VMK 68 are good examples of the Weinstein-type feldspar porcelains, which sometimes form microcracks upon cooling, due to the differences in thermal expansion between leucite particles and the surrounding glass matrix. During cooling, the leucite particles contract more than the surrounding glass, and above a critical particle size, the stress created during cooling can induce microcracks circumferential to the leucite particles. This process has been well described in the literature [11]. A comparison of the SEM photomicrographs clearly shows that the leucite particles in Flexo Ceram Dentine (Fig. 2.6) are larger than those in Vita VMK 68 (Fig. 2.7).

Apparently these particles induce more stress concentration than the defects in the surface, which may explain the fact that in this study the biaxial flexure strength of Flexo Ceram Dentine was independent of the roughness of the surface.

Fracture surface analysis according to Mecholsky [3] confirmed the hypothesis that surface flaws are the failure causing flaws for Vita VMK 68, Duceram LFC and Cerinate Body. Flexo Ceram Dentine showed the failure causing flaws, as assumed in the structure of the material.

The results of this study indicate that surface roughness determines the strength of a porcelain material, except where the material has an inner structure which causes an even larger stress concentration than that caused by the combination of surface roughness and flaws.

Further research is needed to find a material for the outer layer of the restoration which, in combination with surface treatment, produces a surface which remains smooth. Given proper surface treatment, such a material would not require crack-stopping properties, and *in vivo*, the surface of the material would remain smooth, resulting in long-lasting restorations.

In view of the improved performance recorded for the Duceram LFC Dentine ground/polished, glazed specimens using the acetic acid treatment, this is a very real possibility.

2.6 Acknowledgement

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