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Strength and Fracture Origins of a Feldspathic Porcelain

George D. Quinn^a, Kathleen Hoffman^a, and Janet B. Quinn^a

^aADAF Paffenbarger Research Center, National Institute of Standards and Technology, Gaithersburg, Maryland, USA

Abstract

Objectives—To identify the strength limiting flaws in in-vitro test specimens of a fine-grained feldspathic dental porcelain.

Methods—Four-point flexural strengths were measured for 26 test specimens. The fracture origin site of every test specimen was studied using stereoptical and scanning electron microscopy. A fractographically-labeled Weibull strength distribution graph was prepared.

Results—The complex microstructure of the feldspathic dental porcelain included a variety of feldspars, tridymite, and a feldspathoid as well as pores/bubbles and residual glass. The relatively high flexural strength is due in part to the fine grain size. Fractography revealed five flaw types that controlled strength: baseline microstructural flaws, pores/bubbles, side wall grinding damage, corner machining damage, and inclusions. The baseline microstructural flaws probably were clusters of particular crystalline phases.

Significance—Each flaw type probably has a different severity and size distribution, and hence has a different strength distribution. The Weibull strength distribution graph blended the strength distributions of the five flaw types and the apparent good fit of the combined data to a unimodal strength distribution was misleading. Polishing failed to eliminate deeper transverse grinding cracks and corner damage from earlier preparation steps in many of the test pieces. Bend bars should be prepared carefully with longitudinal surface grinding whenever possible and edge chamfers should be carefully applied. If the grinding and preparation flaws were eliminated, the Weibull modulus for this feldspathic porcelain would be greater than 30. Pores/bubbles sometimes controlled strength, but only if they touched each other or an exposed surface. Isolated interior bubble/pores were harmless.

Keywords

flaws; strength distribution; strength; porcelain; Weibull modulus; pores; bubbles

1. Introduction

Fractographic analysis of dental porcelains is usually difficult due to the coarse microstructure and consequently rough fracture surfaces. This is true even for specimens broken under ideal conditions such as bend bars for strength testing. Results are often

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Corresponding author: George D. Quinn, ADAF, Stop 854-6, National Institute of Standards and Technology, Gaithersburg, MD 20899, USA, Phone: 301-975-5765, george.quinn@nist.gov.

⁴The numbers refer to the Joint Committee on Powder Diffraction Standards.

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analyses using Weibull statistics and the scatter in strengths is thought to be due to variability in the size and type of strength-controlling flaws. Verification of the latter is quite rare, however. Previous studies with lab scale test coupons have identified fracture origins as being occasional large pores, contact damage sites, leucite clusters, and only occasionally, (in older generation porcelains) unreacted quartz grains.[1,2,3,4,5,6] Experiments with Knoop indentation controlled precracks underscored how difficult unequivocal identification of fracture origins can be.[7] Electrical insulator porcelains often have unreacted quartz grain flaws, but these are usually not observed in modern dental porcelains. Systematic identifications of fracture origins in dental, electrical, or consumer whiteware porcelains test specimens are rare. The objective of the present study was to identify every fracture origin in 26 high-strength bend bars of a commercial feldspathic dental porcelain. Future fractographic analysis of clinical fractures can be aided by better knowledge of the origins in lab scale test coupons.

2. Materials and methods

2.1 Materials

A feldspathic porcelain with well-dispersed crystallites was used for this study.^{1, 2} It was a relatively strong pressed porcelain, making the fracture surfaces conducive to fractographic analysis. The material is described by the manufacturer [8] as consisting of natural feldspar materials with very fine crystalline portions homogeneously embedded in the surrounding glass matrix and fired at high temperature (1170°C – 1200°C). An average flexural strength of 154 MPa ± 15 MPa was reported by the manufacturer.[8] ³ The data is quoted as having been from another study [9] where the values were listed slightly differently as 154 MPa ± 12 MPa. The tests were done in three-point flexure with 1.5 mm × 3 mm × 20 mm specimens on 15 mm outer spans. The fracture toughness of this material by the single edged precracked beam method was 1.19 MPa m ± 0.05 MPa m (one standard deviation). [10]

This material was identified as “Porcelain 2” in our earlier study on the applicability of the Weibull statistics to dental materials strength analysis.[11] The bend bars were furnished by the manufacturer with no information provided about the grinding or polishing steps used. The bars appeared to have been polished since there were no machining striations on the ground surfaces. The edges were not rounded or beveled.

2.2 Methods

Flexural strength of 26 test pieces was measured in ¼-point, 4-point flexure with 3 mm × 4 mm × 28 mm specimens on a semi-articulating fixture with 10 mm and 20 mm spans. Bars of this short length were necessary since the bend bars were cut from CAD/CAM blanks. The crosshead speed was 0.2 mm/min and all testing was done in laboratory ambient conditions. (This crosshead rate produces stress or strain rates similar to those achieved with longer 3 mm × 4 mm × 40+ mm bend bars tested on standardized 20 mm × 40 mm bend fixtures). Every fracture surface of every test piece was examined with a stereoptical microscope at up to 300 magnification and also a scanning electron microscope (SEM) using procedures outlined in [12]. We did this since we initially did have difficulty characterizing some of the fracture origins. One specimen was selected for further intensive work with a field emission scanning electron microscope. In a few instances, fracture surfaces were

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²Commercial products and equipment are identified only to specify adequately experimental procedures and does not imply endorsement by the authors, institutions or organizations supporting this work, nor does it imply that they are necessarily the best for the purpose.

³The uncertainty type was not reported, but may be assumed to be one standard deviation.

etched with hydrofluoric acid (1 % for 20 s) to ascertain whether the origin tended to dissolve differently than the matrix. A compound optical microscope was used on a polished broken half of a specimen to examine the overall microstructure as well as to measure the approximate concentration of certain flaws per volume using quantitative microscopy techniques. A rough single estimate of the number of flaws per unit volume was made by counting the number of large flaws that were exposed per unit surface area on a 50 cm diagonal digital computer monitor image of the piece which had an exposed area of 4 mm × 17 mm. Only flaws larger than a few tens of micrometers were counted since these are the most likely fracture origins. The number of flaws of a certain size per unit volume $\overline{N}_v$ was estimated from equation 5.5 of reference 13:

$$\overline{N}_v = \frac{\overline{N}_A}{\overline{D}}$$

where $\overline{N}_A$ is the number per unit area and $\overline{D}$ is the average flaw diameter. Flaws of size 30 μm or greater were easily discernable when the exposed surface was magnified onto the computer monitor screen. $\overline{N}_A$ was estimated by counting the number of flaws exposed on the polished section and dividing by 4 mm × 17 mm. The microstructure of the polished specimens was also examined with the scanning electron microscope, with both unetched and etched specimens (1% hydrofluoric acid for 20 s).

The crystalline phase assemblage was evaluated by X-ray diffraction analysis with copper K_α radiation with two theta scans from 5 degrees to 65 degrees. An aluminum reference was scanned over the same range for comparison.

3. Results

X-ray diffraction revealed the material had multiple crystalline phases. There was considerable overlap of a number of the peaks making analysis difficult. Nevertheless, a number of potassium and sodium feldspars (KAlSi₃O₈ or NaAlSi₃O₈) matched very well. There were enough distinct peaks that sanidine (19-12274), orthoclase (19-0031), and albite (19-0460) could be identified. Mixed potassium and sodium feldspars such as anorthoclase (10-361), and 0.5Na, 0.5KAlSi₃O₈ (84-0710) also fit well. The feldspathoid nepheline, NaAlSiO₄ (35-4201), a silica-under-saturated aluminosilicate, matched with several peaks that were not accounted for by the feldspars. Monoclinic trydimite (18-1170) was also present and its distinct 100% peak at 21.6° was observed and was not accounted for by any other phase. Most of the other trydimite peaks overlapped those of other phases. There was no leucite present. In summary, at least three potassium and sodium feldspar phases were present as well as nepheline and trydimite.

Figure 1 shows the microstructure as revealed by the compound optical microscope on unetched test pieces. Here we are not so concerned with evaluating average grain size, but rather the presence of large (e.g., > 30 μm) potentially strength-limiting flaws. The most common irregularity was a very smooth round bubble-pore. They created interesting optical effects if they lay just beneath the surface. Other irregularities appeared as white specks, some of which are probably intentional coloration additives. From a count of the number of such features per unit area (18 pores of size 40 μm or larger and 8 other flaws of size 30 μm or larger) over large polished section area, the flaw densities were estimated to be 6.6 pores/mm³ and 3.9 other flaws/mm³. Figure 2 also shows the microstructure, but as imaged by the SEM. There are substantial amounts of both crystalline and matrix glassy phases. Some regions preferentially etched away, suggesting different phase assemblages were present. This is not unlike what happens with leucite porcelains.

Figure 3 shows a typical fracture surface which had clear markings such as hackle lines, compression curl, and a distinctive fracture mirror. There was no difficulty finding a fracture origin site, but as discussed below, the origin flaw itself sometimes was hard to characterize.

Figure 4 shows the Weibull strength distribution graph. The Weibull modulus estimate obtained through a Maximum Likelihood Estimation (MLE) procedure⁵ was a very respectable 18.0 and the characteristic strength estimate was 115 MPa. A large Weibull moduli like this and a linear-appearing data set are often assumed to imply a single flaw type is active, but the fractographically-labeled Figure 4 reveals this material is much more complex. No less than five flaw types were active in the 26 specimens.

Pores, denoted by P in Figure 4, acted as strength limiting flaws in five specimens, but only if they touched an external surface or another pore. Figure 5 shows several examples. The symmetry and smoothness of these pores, which were of the order of 20 μm to 50 μm in diameter, suggests they occurred as the result of trapped gas during processing. They are more aptly termed “pore/bubbles” and they are common in dental ceramics. Figure 6 shows several possibilities for the location of pore/bubbles. Many are harmless since a smooth spherical bubble is not a sharp flaw. It is a simple stress concentrator with locally enhanced stress that is only factor of two greater than the nominal stress. On the other hand, if the pore/bubble breaks through to a surface or touches another pore/bubble, there are small sharp cusps that are extremely vulnerable sites. Cracks easily form from the cusps and grow into the bulk.

The dominant flaw type at the high strength end of the distribution was initially very difficult to identify. It appeared to be a microstructural irregularity of some sort. We carefully examined both fracture halves of all ten specimens that broke from such origins. Figure 7 shows examples of what we came to characterize as “baseline microstructural flaw (BMF),” one category of flaws in the Guide [12] and also described by Rice as “mainstream microstructural features.”[14] These were 30 μm – 90 μm diameter origins that were not obviously abnormal features such as inclusions or large pores, but instead were localized irregularities in the normal microstructure. These origins were volume-distributed. Energy dispersive spectroscopy analysis revealed the origins had a mix of silicon, aluminum, potassium, and sodium which was entirely consistent with the normal microstructure. The backscattered electron microscopy images also revealed no apparent chemistry differences. However, the microstructure appeared subtly different than that of the mainstream microstructure. A detailed inspection with the field emission scanning electron microscope around one such flaw did not clarify matters. We suspect that these flaws are local concentrations of one or two of the five crystalline phases present in the material. It would be hard to imagine that there could be a perfect dispersal and ideal randomness of the five separate phases that we identified by X-ray diffraction. It is hard to discriminate the BMFs by EDS spectroscopy since the chemistry of the feldspars and the feldspathoid are very similar. Only a selected area diffraction analysis of the grains at the origin could verify the hypothesis that there is a different local phase assemblage, but such analysis was beyond the scope of this study.

Two specimens had inclusions associated with the fracture origin, but in one case the inclusion was tiny and incidental. In specimen I shown in Figure 8, the inclusion was large enough to qualify as a strength-limiting flaw, but even in this instance, there was a nearby BMF. In the polished specimens, occasional brightly reflecting zirconium and titanium

⁵The MLE approach is preferred in many cases over the simpler linear regression (LR), because the confidence intervals for the MLE parameter estimates are tighter than those from LR. In accordance with the customary procedures, unbiasing factors are applied to the estimate of the modulus, and the Weibull modulus is labeled MLE-ub.

inclusions were detected and their chemistry was identified by EDS in the SEM. They are probably coloring agents or opacifiers.

The low strength end of the Weibull distribution graph was dominated by two specimen preparation flaw types, side wall (SW, n=5) grinding damage and corner damage (Cor, n=4). Figure 9 shows examples of the SW flaws. Identification of these was difficult, and they initially were confused with BMFs. Side wall grinding damage was not expected and was masked by the final polishing of the specimen surfaces. Gradually we came to recognize the telltale overlapping semi-elliptical cracks that are typical of grinding damage [12]. This interpretation was confirmed by the observation of stray grinding grooves on the side wall surfaces of most of the affected specimens. These telltale traces were oriented vertically along the specimen sides indicating the specimens were ground transversely at some point during preparation. Final polishing was meant to eliminate such damage. The polishing made the specimens look very nice, but deeper residual grinding crack damage lurked beneath the surface.

Four other specimens had edge origins such as shown in Figure 10. The edges were not beveled or rounded and irregularities or chips acted as strength limiting-flaws.

4. Discussion

Multiple crystalline phases with a residual glass matrix are common in dental porcelains, and it can be challenging to identify all of the phases. Barriero and Vicente [15,16] showed examples where leucite and various feldspars such as sanidine, orthoclase, and monoclinic, were all present depending upon the processing conditions. They relied upon meticulous X-ray diffraction work, scanning electron microscopy coupled with emission spectroscopy, differential etching, and reflection optical microscopy to make the identifications. With reference to the classic ternary phase diagram of Schairer and Bowen [17,18] for $K_2O-SiO_2-Al_2O_3$, Barriero and Vicente noted that these phases are “reconstructive above 800°C, but are sluggish and irreversible. Consequently, some high temperature structures are quenchable as metastable phases.”[15] Spatial variations in the crystalline phases could be attributed to the use of different starting frits.[15] It has also been remarked that spatial variations in the glass matrix composition can affect local properties in dental porcelains if multiple frits are used as starting powders.[11]

Some researchers have expressed skepticism about the unbridled use of the Weibull distribution [e.g., 19], but there is ample justification for its use.[1,20] Jayatilaka and Trustrum [21,22] and later Danzer [23] have shown that the Weibull distribution can be derived from first principles for brittle materials if the right side tail of the flaw size distribution has a power law dependence on flaw size. Multiple flaw populations complicate things considerably, but they can be analyzed using censored statistics⁶ as described in many references [24,25,26,27] and in three documentary standards. [28,29,30] The skeptics are right, however, if researchers simply fit a line through data on a strength distribution graph and infer that strength is controlled by one flaw type. Only a fractographic analysis can verify or disprove the latter.

It would be incorrect to scale strengths for the combined distribution to other configurations and sizes on the basis of a unimodal distribution. Each strength distribution for each flaw type would have to be scaled separately. Although censored statistical analysis can in

⁶One cannot simply separate the data subsets and analyze them separately. Specimens that fail from type A flaws still contain a type B flaw of an unknown, but greater, strength. Similarly, specimens that fail from type B flaws contain a type A flaw of unknown but greater strength. Specimens that break from type B flaws must be included as censored data in the ranking of strengths of type A flaws and vice versa. See Ref. 25 (Jakus et al. 1981).

principle permit such scaling, it is usually only done for two flaw types. It is impractical for five concurrent flaw populations. As discussed by Service et al. in 1985 [25], thirty fractures from each flaw type should be obtained for good estimates of the individual flaw distribution parameters. In the present case, that would mean that about 80 specimens total would be needed to obtain 30 BMF flaws, 150 specimens to get 30 for the pore set, and as many as 780 specimens to see the inclusion distribution more clearly.

An additional complication in the size scaling of strength using Weibull statistics arises from the behavior of the pore/bubble flaws. They are volume-distributed features, but they are harmless if isolated in the interior. The stress concentration factor for an isolated smooth pore is simply a factor of two. It is a simple stress *concentration* matter, however, and the flaw does not *intensify* stresses to anywhere near the magnitude that occurs around a sharp crack. On the other hand, if the pore/bubble is in proximity to another pore/bubble, or a free surface, the stress concentration can be much greater and a crack can pop-in the weak ligament. The situation is much worse if the pores touch the surface or each other such that sharp cusps form. A sharp crack will easily pop in from them. Weibull strength scaling for size theory assumes that flaws do not interact with each other or with free surfaces. Modifications to the conventional Weibull strength size scaling theory would have to be developed for size scaling of strength for such flaws. Conventional Weibull analysis implicitly makes an assumption that flaws are randomly distributed and do not interact. This is not an isolated nor trivial matter, since pore/bubbles are common in dental restorative materials. A companion paper [31] explores these matters in more detail and offers a number of examples of pore/bubbles for dental and non-dental ceramics and resin composites.

In our earlier study, [8] the strength of this porcelain (#2) was compared to another feldspathic porcelain (#1) made by a different manufacturer. A direct strength comparison was hampered by the different sizes of test pieces used in the respective strength tests. We used the conventional Weibull approach to scale the strengths so that the strengths could be compared on the basis of a common specimen size. The size scaling failed to account for the strength differences and it was argued that porcelain 2 was stronger than porcelain 1, irrespective of the specimen sizes. This conclusion is still valid on the basis of a comparison of the microstructures and flaw sizes of the two porcelains. Furthermore, if the enhanced Weibull modulus (> 30) of the present porcelain for its material flaws alone were used in the size scaling computation, the disparity in strengths between the two porcelains still would not be accounted for by the specimen size differences. In other words, porcelain 2 (with its finer microstructure and pressing step in processing) is stronger than porcelain 1, even if the size difference is taken into account.

In this study, the baseline material flaws controlled the strength of the strongest specimens. It is believed that the flaws are clusters of one or two crystalline phases. Since the feldspar and feldspoid phases are very similar in chemistry, it is difficult to discriminate between them on the fracture surfaces in the SEM. The local variations in the phases are probably intrinsic to this material since a perfect randomization or mixing of the phases would be impossible. Some of the feldspar phases are monoclinic (sanidine, orthoclase) and others are triclinic (microcline and the mixed potassium-sodium variants). Nepheline is hexagonal and tridymite is monoclinic. It is possible that differential thermal expansions in various crystallographic directions might cause grain boundary microcracking. This is consistent with the appearance of many of the fracture origins. Tridymite, a polymorph of silica, was also detected by X-ray diffraction. Large grains of another polymorph, quartz, is a notorious fracture origin in many porcelains due to the beta to alpha phase conversion which entails a large displacive transformation during cool down from high temperatures.[32] The transformation causes microcracking around the quartz grains if the grains are of the order of 50 μm or larger. Quartz was not detected in the dental porcelain evaluated in this study,

but it is possible that large tridymite grains could be susceptible to the same problem since it also undergoes displacive transformations during cool down. Nevertheless, we did not see isolated large grains with cracking around them, a characteristic feature of the quartz fracture origins in electrical porcelains. We intend to do additional studies of the BMF flaws in cooperation with the material manufacturer.

What would be the strength distribution of this material if the grinding and edge flaws were eliminated? This is not too difficult a problem to answer since censored statistical analyses are available and even standardized for cases of concurrent flaw populations [27,29]. Figure 11 shows the results of such a reanalysis. The preparation flaws would have a Weibull modulus of about 10 and a characteristic strength of 126 MPa. The material flaws have a characteristic strength of 119 MPa, not much greater than the 115 MPa of all the data combined (Figure 3), but an astonishing Weibull modulus of 35. This estimate of the Weibull must be tempered by the fact that it was not corrected for bias associated with the maximum likelihood estimation analysis. Unbiasing factors, which are less than 1.0, are known for unimodal strength distributions as discussed in C 1239 and ISO 20501, but they are not available for cases of a censored statistical analysis of concurrent flaw populations. If an unbiasing correction was applied to the red line in Figure 11, then the lower left of the line would shift more to the left and line up better with the data. If we arbitrarily use the unbiasing factor of 0.914 for a unimodal distribution for $n = 16$ specimens (from Table 1 of C 1239), then the revised estimate of the Weibull modulus would be 32.

Finally, it is not known whether the flaws identified in the lab scale bend bars are causes of clinical fractures. This is an open question at the present time. We now know what occurs in bend bars and we can be on the lookout for similar flaws in fractured restorations.

5. Conclusion

The apparent good fit of all the flexural strength data to a simple Weibull distribution was misleading. A comprehensive fractographic analysis revealed that five flaw populations were active. The flaws were: pores/bubbles that contacted the surface or other pore/bubbles; baseline material flaws probably associated with local crystalline phase variations; inclusions; side wall transverse grinding damage; and corner damage. Polishing the specimens did not remove all subsurface grinding damage from earlier grinding. Specimen edges should be bevelled or rounded. If the grinding and edge flaw types were eliminated by more careful specimen preparation, this feldspathic porcelain would have a modest improvement in the four-point flexure strength (115 MPa to 119 MPa), but more importantly, a dramatically improved Weibull modulus (18 to greater than 30).

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Figure 1.
(a) Microstructural irregularities revealed on polished, unetched surfaces. (b) shows a close-up of some of the irregularities.

Figure 2. SEM secondary electron images of the microstructure. (a) An unetched polished section. (b) An etched surface. The ellipses highlight regions of different microstructure since they etched very differently.

Figure 3.

Fracture surface of a bar E (119.3 MPa) with the origin marked by white arrows. (a) An optical photo of the entire fracture surface after gold coating. (b) An SEM close-up of the mirror. The origin flaw is shown in Figure 5d.

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Figure 4.

The fractographically labeled Weibull strength distribution. SW denotes sidewall grinding damage; P, Pore; BMF, Baseline Microstructural Flaw; I, Inclusion; and Cor, Corner flaw. The Weibull modulus, m and the characteristic strength, σ_0 , were evaluated using Maximum Likelihood Estimate (MLE) with unbiasing correction for m .

Figure 5.

Examples of pore/bubble fracture origins. (a) and (b) are secondary electron and backscattered (BS) electron images of specimen D (120.1 MPa), respectively. (c) A BS image of a triple pore that touches the surface in specimen J (111.5 MPa). (d) A BS image of a double pore in specimen E (119.3 MPa).

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Figure 6. Pore/Bubbles may either be harmless or can be strength limiting flaws, depending upon their proximity to external surfaces (a) or each other, (b). The dashed lines show possible crack pop-in stages.

Figure 7. Backscattered SEM images of “baseline microstructural flaws (BMF).” (a) Specimen H (122.9 MPa), (b) Specimen A (120.6 MPa), and (c) Specimen F (111.8 MPa).

Figure 8.
Backscattered SEM image of the fracture surface of specimen V, with a zirconium containing inclusion (solid circle) near a BMF flaw (dashed ellipse).

Figure 9. Side wall damage in piece K. (a) A optical image of the gold coated fracture surface and (b) A backscattered electron SEM close-up of the origin site. (c) An optical image of the side wall surface of the same specimen K (94.6 MPa). The two horizontal white arrows show stray vertical grinding striations. The slanted arrow shows another one right at the origin site.

Figure 10.

Corner damage in specimen N (114.2 MPa). (a) An optical image of the side wall of the specimen and a band of vertical grinding damage near the corner. (b) An SEM close-up of the fracture origin in the same specimen.

Figure 11.

Weibull strength distribution graph for the material flaws and the specimen preparation (side wall and corner) specimens analyzed by censored statistical analysis. The Weibull moduli estimates are biased, and therefore are denoted as: MLE -b.