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Strength and fracture origins of a feldspathic porcelain

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ARTICLE INFO

Article history:
Received 1 August 2011
Received in revised form
26 October 2011
Accepted 1 December 2011

Keywords:
Flaws
Strength distribution
Strength
Porcelain
Weibull modulus
Pores
Bubbles

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.

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 analyzed 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 [1–6]. 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

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doi:10.1016/j.dental.2011.12.005

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 (1170–1200 °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 $m^{1/2}$ ± 0.05 MPa $m^{1/2}$ (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 1/4-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 stereo-optical 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 had 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 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 $\bar{N}_V$ was estimated from Eq. (5.5) of Ref. [13]:

$$\bar{N}_V = \frac{\bar{N}_A}{\bar{D}}$$

where $\bar{N}_A$ is the number per unit area and $\bar{D}$ 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. $\bar{N}_A$ 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 Kα radiation with two theta scans from 5° to 65°. 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 ($KAlSi_3O_8$ or $NaAlSi_3O_8$) matched very well. There were enough distinct peaks that sanidine (19-1227[4]), orthoclase (19-0031), and albite (19-0460) could be identified. Mixed potassium and sodium feldspars such as anothorclase (10-361), and 0.5Na, $0.5KAlSi_3O_8$ (84-0710) also fit well. The feldspathoid nepheline, $NaAlSiO_4$ (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.

Fig. 1 shows the microstructure as revealed by the compound optical microscope on unetched test pieces. Here we

[1] Mark II for the CEREC® system, Vita Zahnfabrik, Bad Säckingen, Germany.

[2] Commercial 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.

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

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