

## Research Article

# Influence of Surface Treatment on Strength Distribution of Vita VMK 68 Dental Porcelains

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Weibull distribution function is the most commonly used statistical model for the investigation of mechanical properties of dental ceramics and design process with dental ceramics. However, it is still unclear whether the Weibull distribution function is the most appropriate function for fitting the strength data of dental ceramics with different surface treatments. In this paper, three-point bending test results of feldspathic body porcelain (Vita VMK 68) specimens with four different surface treatments are analysed. According to goodness-of-fit tests (Anderson-Darling test, Kolmogorov-Smirnov test, and Akaike information criterion), it is shown that the type of surface treatment has an important influence on deviation of strength distribution from perfect Weibull statistics. It is concluded that estimation of the most suitable statistical model for Vita VMK 68 is not only a material-dependent but also a process-dependent (machining of the specimens) procedure.

## 1. Introduction

The usage of ceramic restorations in restorative and prosthetic dentistry has been increased rapidly parallel to the advancements in mechanical design and manufacturing technologies.

The importance of ceramic restorations was extended related to the high demands for aesthetic and biocompatible materials. Although dental ceramics has advantageous material properties (aestheticism, corrosion resistance and abrasion resistance, low thermal conductivity, etc.) among other materials [1], their brittle nature causes difficulties in reliable application of dental ceramics [2]. Therefore, it is important to maintain the strength and toughness of dental ceramics without compromising aesthetics [3]. This introduces the main need of detailed analysis, improvement of mechanical properties (fracture toughness, strength, etc.), and reliability of ceramic restorations.

The strength of the ceramic restorations mainly depends on the surface roughness of the veneering material which is affected by the final preparation (grinding, polishing, glazing, etc.) [4–10].

Dental porcelains are used commonly as the porcelain veneers and intracoronal restorations. Moreover, the possibility of using in metal-ceramic and all-porcelain crowns and

bridges increased the application areas of dental porcelain. Vita VMK 68 is one of the most commonly used feldspathic body porcelain used for dental restorations. Surface modifications are fundamental for dimensional correcting the inadequate contours and improving the esthetic appearance and surface smoothness of porcelain restorations. For a reliable application, investigation of effect of surface roughness and surface treatment (grinding, polishing, and glazing) on the strength and fracture toughness of veneer ceramic materials becomes very critical. According to the bending experiments performed by using the ball-on-ring method, a relationship has been introduced between the average surface roughness  $R_a$  and the biaxial strength [4] of Vita VMK 68. Fischer et al. [5] investigated the relationship between the roughness value  $R_{max}$  which is defined as the maximum of the peak-to-valley height of the measure section and the flexural strength of Vita Akzent and showed a linear dependence of flexural strength on the roughness value  $R_{max}$ . Sato et al. [11] investigated the occurrence of microscopic fractures on the surface of ceramics during the grinding process and the effect of defect size on the fracture strength. They estimated the size of the cracks using the process zone size-fracture criterion and the Newman-Raju formula [12] and reported that the surface cracks caused by the grinding process decrease the strength.

Experiments performed with Vita VMK 68 [6] as glazed and polished with 1000-grit, 600-grit, and 100-grit with silicon carbide abrasive papers showed that the increase of roughness decreases the flexural strength.

Rosenstiel et al. [13] investigated the effect of polishing and glazing on fracture toughness of dental porcelain and obtained higher fracture toughness values for the polished specimens.

In almost all related studies in the literature, two-parameter Weibull distribution function was used for the characterization of strength distribution of feldspathic body porcelain, and the effect of surface treatment on the Weibull parameters was reported. For some dental ceramics (IPS Empres, Vita VMK 68, Vitadur Alpha Core, Vitadur Alpha Dentin, etc.), it has been shown that deviations from two-parameter Weibull distribution can occur [14, 15].

Unfortunately, there is no study in the literature which investigated the influence of surface treatment (surface roughness) of feldspathic body porcelain on deviation of strength data from the Weibull statistics. In this paper, the goodness-of-fit tests (Anderson-Darling test, Kolmogorov-Smirnov test, and Akaike information criterion) will be used in order to determine the deviation of strength distribution of four different set of specimens (glazed, polished with 1000-grit, 600-grit, and 100-grit) from the Weibull distribution. The physical explanations which stay behind the deviation of strength data from Weibull behaviour will be discussed. Moreover, three-parameter Weibull distribution is used for fitting the strength data of strength of set of specimens produced from Vita VMK 68, and the fitting capacity will be compared with the two-parameter Weibull distribution.

## 2. Materials and Methods

*2.1. Specimen Preparation and Testing.* The strength datasets of feldspathic body porcelain (Vita VMK 68, Bad Säckingen, Germany) with different surface qualities were taken from the literature with permission of Nakamura et al. [6]. Therefore, more detailed information about the specimen preparation and testing can be found there. Here brief information is given. The green body was produced by mixing the porcelain powder and water according to manufacturers' recommendation as ( $w/p$  ratio = 0.4) [6]. During molding, the excess water was removed by a tissue paper. In a digital furnace (Flagship VPF; JF Jelenko and Co., San Diego, CA, USA) [6], the specimens were sintered in a vacuum between the firing temperatures of 700°C and 930°C for 5 minutes. No thermal cycling was performed.

Thereafter, Nakamura et al. [6] polished the specimens with 2000-grit abrasive and carried out hand lapping in wet conditions until the final specimen dimensions are achieved. After keeping the specimens in a dry environment, the specimens were glazed. The specimen dimensions were measured by using slide callipers (Absolute Digimatic Solar Caliper 500–445 CD-S20C, Mitutoyo Corp., Kanagawa, Japan) [6] with an accuracy of  $\pm 0.02$  mm. Glazed specimens, are the original specimens and glazed groups is the control group. Thereafter, by polishing the glazed specimens with different abrasives, three addition group of specimens were produced.

As a result four groups of 30 specimens were produced as follows: (1) glazed, (2) polished with 100 grit abrasive, (3) polished with 600 grit abrasive, and (4) polished with 1000 grit abrasive.

The roughness measurements were performed with the machine (Surfcoder Model SE-3H; Shimadzu Corp., Kyoto, Japan) according to JIS (Japan Industrial Standard, B 0601, 1994) and the three-point bending tests were carried out with the universal testing machine (Autograph Model AG-500A; Shimadzu Corp., Kyoto, Japan) according to ISO Standard 6872 (1995) at room temperature under dry conditions by Nakamura et al. [6].

The flexural strength  $\sigma$  was calculated as given in

$$\sigma = \frac{3PL}{2bh^2}, \quad (1)$$

where  $P$  is the failure load,  $b$  is the width of the specimen, and  $h$  is the height of the specimen.

*2.2. Statistical Characterization of Strength of Ceramics.* Fracture of ceramics initiates from preexisting crack-like defects and flaws [16, 17]. These flaws may be volume flaws that occur during the sintering process of a ceramic material and/or surface flaws that appear during its machining process. The strength of a ceramic specimen is determined by the existing most critical crack in the volume or on the surface of the ceramic component. Here the most critical flaw is not always the largest flaw in the material. The size, orientation, and position of a crack determine whether a crack is the most critical flaw. The cracks are randomly distributed in the material. The cracks are randomly distributed in the material, and the position, size, and orientation of the most critical flaw show scattering. This scattering causes a variation of strength of ceramics from component to component, even if identical specimens are tested. Since the strength of ceramics varies from specimen to specimen, a probabilistic method is used for the design of advanced ceramics [18–22].

The Weibull distribution function [23, 24] is the most widely used statistical function for the characterization of strength distribution of advanced ceramics. The fundamental assumption of Weibull distribution is the weakest-link hypothesis (i.e., when the weakest volume element fails, the specimen fails). In its simplest form, the so-called Weibull distribution function for a uniaxial homogeneous stress state for a specimen of the volume  $V$  is given by Danzer [25]:

$$F(\sigma, V) = 1 - \exp \left[ -\frac{V}{V_0} \left( \frac{\sigma}{\sigma_0} \right)^m \right]. \quad (2)$$

The Weibull modulus  $m$  measures the scatter of the strength data,  $\sigma_0$  is the characteristic strength at which the specimen has a failure probability of 63.2%, and  $V_0$  is the unit volume. Equation (2) represents the so-called two-parameter Weibull distribution function. There is also a more comprehensive form of the function proposed by Weibull, the so-called three-parameter Weibull function, in which the stress  $\sigma$  is replaced by  $(\sigma - \sigma_{th})$  [26]. In this form of Weibull function,  $\sigma_{th}$  is the threshold stress below which no failure occurs. Threshold stress is also a measure of scatter of strength of identical ceramic specimens [27].

**2.3. Goodness-of-Fit Tests.** A goodness-of-fit test (GOF) measures the compatibility of a random sample with a theoretical probability distribution function. In other words, these tests show how well the distribution fits the selected data.

**2.3.1. Anderson-Darling Test (A-D Test).** The Anderson-Darling test is a statistical test of whether a sample of data comes from a population with a specific distribution [28]. It is a modification of the Kolmogorov-Smirnov (K-S) test which is distribution free in the sense that the critical values do not depend on the specific distribution being tested [29]. The Anderson-Darling test makes the use of the specific distribution in calculating critical values. This has the advantage of allowing a more sensitive test. The critical values for different types of distribution functions are available in the literature [30–32].

The formula for the Anderson-Darling statistic  $A$  of the ordered data  $\{Y_1 < Y_2 \dots Y_n\}$  is

$$A^2 = -n - S, \quad (3)$$

where

$$S = \sum_{i=1}^n \frac{2i-1}{n} [\ln F(Y_i) + \ln(1 - F(Y_{n+1-i}))], \quad (4)$$

where  $F$  is the cumulative distribution function of the specified distribution and  $n$  is the sample size. The hypothesis regarding the distributional form is rejected at the chosen significance level ( $\alpha$ ) if the test statistic,  $A^2$ , is greater than the critical value.

**2.3.2. Kolmogorov-Smirnov Test (K-S Test).** The Kolmogorov-Smirnov test (K-S test) is used to compare a sample with a reference probability distribution in order to decide whether the sample comes from a specific distribution [33]. The Kolmogorov-Smirnov statistic defines a distance between the empirical distribution function of the sample and the cumulative distribution function of the reference distribution. The Kolmogorov-Smirnov statistic can be calculated as

$$D = \max_{1 \leq i \leq n} \left( F(Y_i) - \frac{i-1}{n}, \frac{i}{n} - F(Y_i) \right), \quad (5)$$

where  $F$  is the theoretical cumulative distribution of the distribution being tested. The hypothesis regarding the distributional form is rejected if the test statistic,  $D$ , is greater than the critical value.

**2.3.3. Akaike Information Criterion (AIC).** Akaike information criterion [34] measures the goodness of fit of an estimated statistical model by linking the likelihood to a distance between the true (experimental) and the considered (Weibull) distributions. The AIC index which is used in a number of areas as an aid to choosing between competing models is defined as [22]

$$AIC = -2 \ln L + 2k, \quad (6)$$

TABLE 1: Arithmetic mean roughness values and their standard deviations of specimens of four groups [6].

Surface treatment	Arithmetic mean roughness $R_a$ ( $\mu\text{m}$ )
100-grit polished	$1.68 \pm 0.30$
600-grit polished	$0.9 \pm 0.34$
1000-grit polished	$0.38 \pm 0.16$
Glazed	$0.23 \pm 0.06$

where  $k$  is the number of free parameters in the model (for example,  $k = 2$  for two-parameter Weibull distribution). Another term  $\ln(L)$  is the maximized log-likelihood for a given model and can be calculated as

$$\ln(L) = \sum_{i=1}^n \ln f(Y_i), \quad (7)$$

where  $n$  is the number of data and  $f(Y_i)$  is the probability density function of the estimated distribution. The model with minimum AIC value is chosen as the best model to fit the data.

### 3. Results and Discussion

The results of the surface roughness measurements of four different test groups of the investigated feldspathic body porcelain, performed by Nakamura et al. [6], are given in Table 1.

While the feldspathic body porcelain polished with 100-grit abrasive has the highest roughness, the glazed porcelain has a very small roughness value. This shows that glazing makes the surface of the porcelain smoother. In other words, polishing with 1000-grit abrasive and glazing eliminates large surface defects on the surface. Moreover as the grit size increases, the standard deviation of the mean roughness decreases. The smallest standard deviation is reached when the surface is glazed. Nakamura et al. [35] investigated the effect of thermal cycling, performed by immersing the specimens 5000 times in water baths at temperatures of  $5^\circ\text{C}$  and  $55^\circ\text{C}$ , on roughness and flexural strength of Deguceram Gold and Vita Omega 900. In the case of Vita Omega 900, the thermal cycling increased the mean roughness for glazed and 1000-grit group, and a decrease in mean roughness was observed for 600-grit and 100-grit polished specimens.

In Figure 1, the Weibull plot of strength values of four different groups of samples of VITA VMK 68 [6] are represented.

As Nakamura et al. [6] stated in their article, the mean flexural strength increases as the mean roughness decreases. Now the question is that whether it is always possible to characterize the strength distribution of feldspathic body porcelain which has different surface roughness levels with the so-called Weibull distribution function. In the literature, the characteristic strength values have been used for the introduction of relationship between material strength and the surface roughness. In this paper, the effect of surface treatment (e.g., polishing, glazing) on the strength distribution

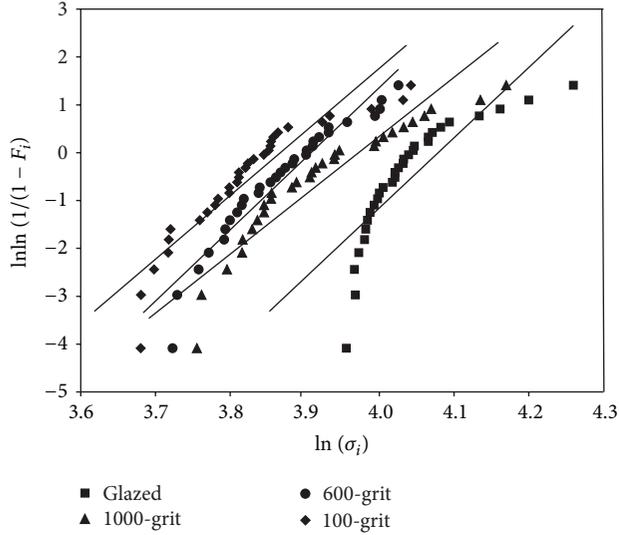


FIGURE 1: Three-point bending test results [6] of feldspathic body porcelain for four test groups.

behaviour of the feldspathic body porcelain VITA VMK 68 is of interest.

In Table 2, the goodness-of-fit test results applied to the Weibull cumulative distribution of the experimentally observed strength data is given. Basically, as the statistic values of Anderson-Darling test, Kolmogorov-Smirnov test, and Akaike information criterion increase, the data deviates more from the corresponding fitting distribution function.

According to all goodness-of-fit tests, the 600-grit polished group gives the smallest statistic values; the glazed group gives the highest statistic values.

Two-parameter Weibull critical values for the Anderson-Darling test statistic [31] and Kolmogorov-Smirnov test statistic [36] for samples containing 30 observations at significance level of  $\alpha = 0.1$  are  $A^2 = 0.64$  and  $D = 0.18$ . Comparing the test results with the critical values shows that the Weibull distribution is not a suitable distribution for fitting the strength data of specimens with glazed and 100-grit polished surfaces. In Figure 2, the empirical cumulative distributions and the fitted Weibull cumulative distributions of four groups (polished with 100-grit, 600-grit, 1000-grit, and glazed) are represented. The deviations from Weibull distribution function can be clearly seen graphically in Figure 2 for the 100-grit polished and glazed group.

This shows the critical importance of investigation of the surface treatment (e.g., surface roughness) on the behavior of the strength distribution of feldspathic body porcelain. Explanations for these results can be done by analyzing the effect of surface treatment on the surface flaw distribution and surface residual stresses of the specimens.

In ceramics, two types of fracture origins provoke failure of ceramic specimens, surface flaws (arising during machining), and volume flaws (arising during material processing). Experimental investigations show that advanced ceramics fail in general due to the surface defects [18, 19]. Mecholsky et al. [37] confirmed that normally surface flaws are the failure causing flaws for Vita VMK 68.

TABLE 2: Goodness-of-fit test results for Weibull distribution function of four different groups (polished with 100-grit, 600-grit, 1000-grit, and glazed condition).

Surface treatment	A-D Test	K-S Test	AIC
100-grit polished	1.054	0.181	186.19
600-grit polished	0.385	0.086	174.36
1000-grit polished	0.624	0.132	193.11
Glazed	2.157	0.203	196.63

However the main difference between the dental ceramics and the advanced ceramics is that dental materials are a quasibrittle material since the inhomogeneity size in the dental materials is not negligible compared to the size of the specimens. Feldspathic porcelains (e.g., Vitablocs II, Vitadur-Alpha dentin, Deguceram Gold, and Vita WMK 68) are porous materials, in which pore or grain is covered by small cracks. In Vita VMK 68 which is an example of Weinstein-type feldspar porcelain, microcracking occurs during cooling due to the differences in thermal expansion coefficients between leucite particles and the surrounding glass matrix. Since the thermal expansion coefficient of leucite particles is higher than that of surrounding matrix, they contract more than the surrounding glass. As a result, residual stresses appearing between leucite particles and glass matrix during cooling induce microcracks circumferential to the leucite particles [38, 39]. Cheung and Darvell [40] reported that minimum porosity is obtained at high sintering temperatures and high sintering times for Vita VMK 68. A group of pores and the cracks around the pores as well as their interaction will affect the final fracture rather than only the largest one as the weakest-link model (or the Weibull distribution) is postulated [41, 42].

For the specimens polished with 100-grit, the surface flaws cannot be eliminated totally. Then, both volume flaws and surface flaws on the surface or linked to the surface of the specimen may cause fracture. If failure occurs due to only surface flaws (microcracks) or only volume flaws (pores), then either the surface flaw or pore size distribution will govern the strength distribution. On the other hand, if both volume flaws and surface flaws may be the reason of failure, each individual flaw population will have its typical size distribution and lead to distributions with different Weibull parameters. As a result, the strength distribution cannot be modeled by one single flaw size distribution tail [15, 21].

Furthermore, since the inhomogeneity size in the Vita VMK 68 is not negligible compared to the size of the specimens, there may occur the interaction of flaws in the component. However, the main property of the Weibull statistics is that it is assumed that the cracks in the material do not interact.

In Figure 3 Weibull plot of strength of Vita VMK 68 polished with 100-grit is shown. It is evident that there are three different types of Weibull distributions which occur due to volume flaws, surface flaws, and their interaction.

During the glazing, the fusion of the glass on the surface of the dental porcelain provides a glossy surface layer and introduces some compressive stresses on the specimen

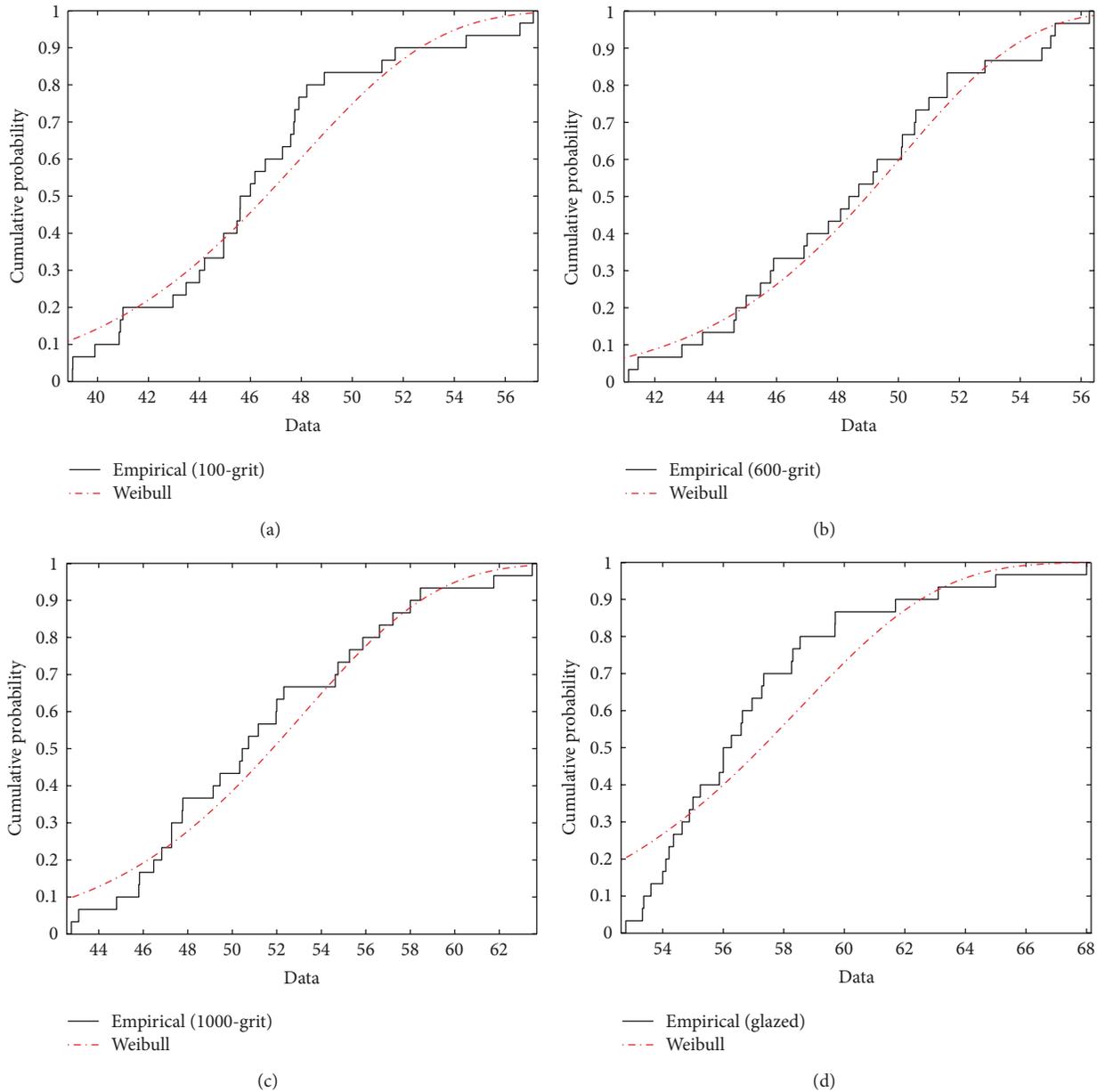


FIGURE 2: Empirical cumulative distribution function and the fitted Weibull cumulative functions of fracture data of four test groups.

surface. These compressive stresses result in a higher mean flexural strength but also at the same time they cause a threshold stress for the failure. Such surface compressive stresses cause a stress-dependent Weibull modulus. Moreover, glazing may also round the crack tip of the surface flaws. Furthermore, before glazing, the specimens were polished in wet environment. Such an environment may result in a subcritical-crack growth, in other words stable crack growth. These features may lead to the so-called R-curve effect [43].

In the case of existence of R-curve effect, stable crack propagation occurs before an unstable failure, and the crack growth resistance increases with crack extension [44].

Cesar et al. [45] verified the occurrence of R-curve effect in leucite-reinforced dental porcelains. The R-curve effect

influences the strength of components with large cracks (low strengths) but has no important effect on specimens with small defects (high strengths) [46]. The increasing crack growth resistance leads to an increase in the Weibull modulus  $m$  [46]. Therefore, when there is R-curve effect, it is expected that the specimens with low strength should have higher Weibull modulus than the specimens with high strengths.

In Figure 4, the Weibull plot of strength distribution of glazed VITA VMK 68 specimens is represented. As expected, the Weibull modulus is higher for the set of specimens with lower strength (containing larger cracks). This explains the main reason of the deviation of strength distribution of glazed Vita VMK 68 specimens from the perfect Weibull distribution.

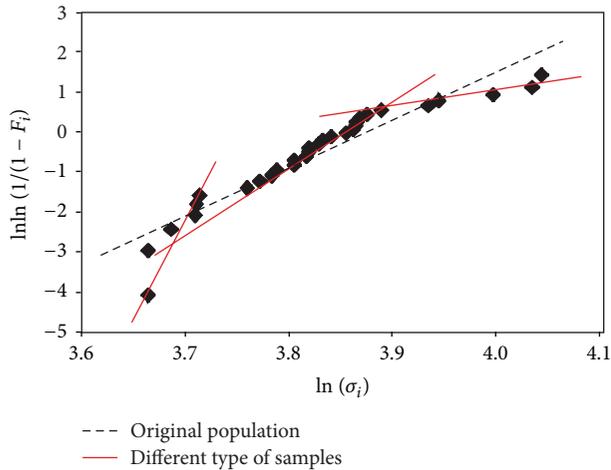


FIGURE 3: The Weibull plot of strength of Vita VMK 68 polished with 100-grit. The dashed line indicates the original population, and solid lines indicate volume and surface flaw populations.

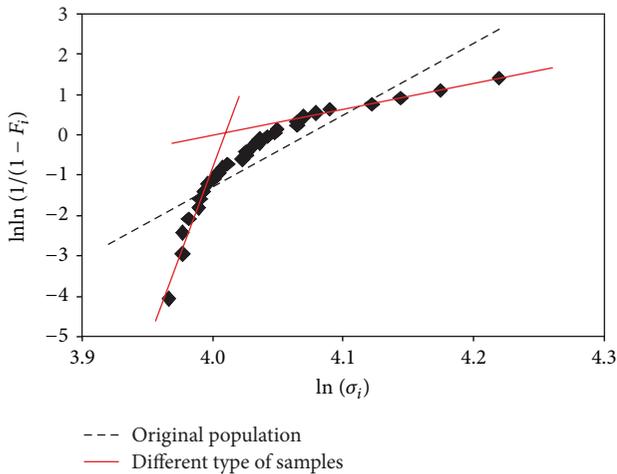


FIGURE 4: The Weibull plot of strength of glazed Vita VMK 68 specimens. The dashed lines indicate the original population, and solid lines show the effect of R-curve behavior.

As a result, when it is required to perform the reliability analysis of feldspathic body porcelain (Vita VMK 68) with Weibull statistics, the surface treatment of the component has to be also taken into account.

#### 4. Conclusion

In this paper, the strength distributions of four groups of feldspathic body porcelain (Vita VMK 68) were investigated. These groups differentiate from each other in a way that the surface machining process (polished with 100-grit, 600-grit, 1000-grit, and glazed condition) after the manufacturing is different. Three-point bending strength tests showed that as the roughness decreases, the mean flexural strength increases. According to the goodness-of-fit tests, the groups polished with 600-grit and 1000-grit abrasives can be characterized with Weibull distribution function. However, the Weibull

distribution function is not an appropriate function for fitting the strength data of group polished with 100-grit abrasive and glazed group. It was observed that the surface treatment (machining process) can cause deviation from Weibull statistics. It was concluded that, for 100-grit polished porcelain specimens, multimodal flaw distribution, and for the glazed group of specimens, the residual stresses appearing during the glazing process cause deviation of strength distribution from the Weibull distribution.

#### Conflict of Interests

The authors hereby declare that there is no conflict of interests with any financial organization regarding the material discussed in the paper.

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