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Fracture Statistics of Porcelain Ceramic: The Influence of Zirconia Additive and Sintering Temperature

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ABSTRACT

The knowledge of statistic characteristics in mechanical properties is important for designer in order to asses the reliability of the structure. Scatter characteristics of fracture strength (splitting strength) of porcelain toughened by zirconia and the effect of sintering temperature were investigated in this study. Many specimens were tested by Brazillian method to obtain the scatter data of fracture strength. The probability distribution of fracture strength is evaluated by using Weibull ddistribution function.

Fracture strength is increased with zirconia content having the highest value (43MPa) at 10 wt.% and then decreased .The same behavior was for Weibull modulus, having the highest value (39) for 10 wt.% Zirconia .

Fracture strength and Weibull modulus also affected by sintering temperature, they increased with the increasing of sintering temperature.

Keywords : Weibull modulus, fracture strength, porcelain ,zirconia ,sintering temperature .

أحصائيات الكسر في سيراميك البورسلين : تأثير اضافة الزركونيا ودرجة حرارة التلبيد

الخلاصـــــــــــة

ان معرفة الخصائص الاحصائية في الخواص الميكانيكية مهمه للمصمم لكي يخمن الاعتمادية في التركيب . تمت دراسة خصائص التشتت لمتانة الكسر(متانة الانفلاق) للبورسلين المقوى بالزركونيا وكذلك تأثير درجة حرارة التلبيد عليها. تم اختبار عدة نماذج لقياس متانة الكسر بالطريقة البرازيلية وكذلك تم دراسة توزيع الاحتمالية لمتانة الكسر حيث حسبت بدالة توزيع ويبل.ان متانة الكسر ازدادت مع اضافة الزركونيا وكانت اعلى قيمة (43MPa) عند نسبة وزن ١٠% ومن ثم ومن ثم تناقضت ، كذلك الحال بالنسبة الى معامل ويبل حيث كانت أعلى قيمة (٣٩) عند ١٠% زركونيا . اما عند زيادة درجة حرارة التلبيد فقد ازدادت كل من متانة الكسر ومعامل ويبل . متانة الكسر ومعامل ويبل ازدادت عند زيادة درجة حرارة التلبيد .

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INTRODUCTION

It is well documented that nominally indentical specimens of brittle materials such ceramics show a large variation of fracture stresses and in order to use brittle material as engineering ones, strength has to be characterized [1] .
Complicating factors affecting the strength of ceramics are manifested mainly in two main ways :
First , strength is generally time dependent in that the applications of tension stress on a component causes a gradual dimension of its capability to withstand further stress without rupture .
Second, there is a relatively large statistical variation in the strength of a batch of otherwise identical specimen [2,3] .
Davadge [4] found two reasons for this variation including:
1 – The sensitivity of the load measuring device and the accuracy of specimen dimensions measured in a mechanical property test.
2 – There is a genuine variation for specimen to specimen.
This variation could be related to the size variable in Griffth`s equation:

$$\sigma = K1c / \; Y \sqrt{a} \qquad \ldots (1)$$

where σ is fracture strength
K1c is fracture toughness
a is size of the critical defect (i.e origin of failure)
Y is a geometrical factor.
Hence the variation in strength should be related to the distribution in size of an appropriate microstructure feature which may be crack size, pore size or grain size.
This introduces the need for a statistical component in fracture analysis.
Fracture statistics should be based on a consideration of three elements which are:
1-Extreme value statistics.
2-Fracture mechanics.
3-Material microstructure.

Statistical properties of fracture behavior have been recognized as one of the important informations required for reliable design.
The particular function, which is based almost exclusively on the extreme value statistics, as developed by Weibull [5] .
Weibull`s function based on the weakest link theory, which assumes that a given volume of ceramic under a uniform stress will fail at the most severe flaw .
Also it is known to statisticians as Fisher-Tipper Type II distribution of smallest values or as a third asymptotic distribution of smallest extreme values [6] .
The Weibull distribution is given by

$$P = 1 - \exp\left[-V \left(\sigma - \sigma u \; / \sigma_0 \right)^m \right] \qquad \ldots (2)$$

Where :

P is the fracture probability for the stress σ , m is known as the Weibull modulus V is the volume of the specimen , σ_0 is a scaling parameter and $\sigma\mu$ is a threshold stress known below which the failure probability is zero .Equation (2) can be written in a linear form by the natural logarithm twice and rearranging this :-

$$\ln\ln (1/1\text{-}p) = \ln V + m \ln (\sigma - \sigma_0) - m \ln \sigma\mu \qquad \text{... (3)}$$

So a plot of lnln 1 / 1-p against ln σ should result in a straight line
The slope of which gives the (m) parameter when P the failure probability, is expressed as

$$P = J/ n+1 \qquad \text{... (4)}$$

Where J is the ranking position
n is the number of test .
Assuming that the strength results were ranked in a scending order .

Weibull modulus depends on many factors like particle size, particle size distribution ,densification process and strength satter factor [7] .

Al- Mohana [8] has determined the Weibull modulus of alumina toughneed by zirconia fibres. The values of the modulus were between 8-20 which increased with the increasing of zirconia fibres content having the highest value 10% and then decreased.

Dongfang et al[9] studied the Weibull modulus of a hydrotreating catalyst ($PCoMo/Al_2O_3$) they showed that there is a great possibility of improving the mechanical strength and reliability of solid catalyst with the optimization of the manufacturing process factors , having a Weibull modulus between 2-4 .

Hauert et al[10] have determined the Weibull modulus of angular alumina particles from experimental tensile data on high ceramic content metal matrix composite using a micromechanical model . They found that the modulus around 3-5 for this composite .

Borrero-Lopez[11] have studied the fracture strength of polycrystalline silicon wafers for the photovoltaic industry. The investigation was under the four point bending test, when generates high stresses both the surface and at the edges of the wafers. The characteristic strength and Weibull modulus each mode were 76-161 MPa and 1.6 – 11.5 respectively.

Qian et al[12] have recalibrated the Weibull modulus and the threshold fracture toughness in a three-parameter Weibull stress model for the cleavage assessment of the pressure project. The calibrated Weibull stress model based on the plasticity theory predicts an increased probability of fracture compared to that based on the classical plasticity.

Rosahl et al[13] studied a statistical approach for transferring fracture events across different sample shapes and investigets the capability of a noval calibration method to predict accurately fracture events at low temperature, and shown that the emergence of a threshold Weibull stress in the Weibull stress distribution is inherent in the fundamental assumptions of the Beremin Model .

Mohd et al[14] studied the scatter behaviour of fatigue limit and tensile strength of magnesium alloy, they found that scatter was very small and comparable to each other and the scatter of fatigue life was very large compared to that the fatigue limit.

The aim of this work is to study the effect of ZrO_2 content and sintering temperatures on the fracture strength and Weibull modulus of porcelain ceramic.

Experimental part

Materials

The starting materials of this study are Kaolin, Silica which obtained from Geological Survey Company. Feldspar obtained from Weigner Co. Germany and Zirconia from BDH.Co.England .

The chemical composing of Kaolin, Silica and Feldspar which done in the Laboratories of Geological survey company is listed in Table (1).

Table (1) Chemical compassion of Kaolin, Silica and Feldspar .

Oxide	Kaolin %	Silica %	Feldspar %
SiO_2	50.46	98.5	67.27
Al_2O_3	33.27	0.4	17.94
CaO	0.03	0.03	0.56
Fe_2O_3	0.67	0.51	0.16
MgO	0.41	0.03	------
Na_2O	0.24	------	2.56
K_2O	0.4	------	11.16
L.O.I	14.25	0.53	0.33

L.O.I is Loss on Ignition.

Procedure of Samples Preparation

To prepare porcelain ceramic the following starting materials were used ; 50% Kaolin 20% SiO_2 and 30% Feldspar .

The following procedure is used:

1. Mix thourghly the above contents in a porcelain Jar with balls for 24 hrs.
2. The mixed powder compacted in a steel die as discs of 30 mm in diameter using a load of 65 MPa .
3. The discs dried in an oven for 24 hrs at 100 ˚C to remove moisture .
4. The samples sintered at 1300˚C and for 2 hrs using a 5 ˚C/min as a heating and cooling rate.
5. The sintered discs then crushed and milled with balls to get less than 25 μm powders.
6. Four percents of ZrO_2 powder (5,10,15,20 wt.%) have been mixed and compacted with porcelain powder as the same above procedure.
7. The new discs sintered at 1250 ˚C and 1350 ˚C for 2 hrs with the same heating and cooling rate.

Strength test

Brazillian test is used to determine the fracture strength using the following formula

$\sigma = 2\, p / \pi\, dt$

where σ = fracture strength
ρ = Applied load
d = diameter of the disc.
t = thickness of the disc.

Results and Discussion

Bell – Shaped curve relating fracture strength to zirconia content and sintering temperature was reported that peaked around 10.wt % zirconia as shown in Fig.(1) fracture strength is increased with increasing sintering temperature , that may be due to the liquid glassy phase , where any access of temperature increases the viscosity of the liquid glassy phase, which fill the pores .

Filling the pores means decreasing the porosity which is inversely proportional with strength due to the following relation:

$$\sigma = \sigma_0\, e^{-bp} \quad \text{... (5)}$$

Where
σ = fracture strength .
σ_0 = strength at zero porosity.
p = porosity.
b = constant.

a fundamental assumption in Griffth`s equation for ceramic strength is that the flaws are atomically sharp (i.e the shape of the flaw is fixed). Some scientist have considered the pores themselves as an integral part of flaws i.e the length of the flaw is considered to be a pore diameter plus. one grain diameter or either side of the pore.[15]

This is based on the concept that cracks will propagate along grain boundaries until the next layer of grains is encountered.

effect in variation of pore shape and size that alter the stress field in the vicinity of the pores are not always considered .[16]

As shown in Figure (1) the strength is remarkedly increased with increasing zirconia content having the highest value at 10 wt% and then decreased. Pure zirconia however, exists in a tetragonal crystallographic from at temperature above 1000 ˚C but in a monoclinic from at lower temperature.

The transformation from one phase to the other on cooling , which is of a martenstic type, produces large volume increases (5%) and high shear strain .

when tetragonal zirconia is incorporated as a second phase into a host matrix , two different effects can occur depending upon whether the zirconia is retained as the tetragonal phase has transformed to monoclinic phase in the ceramic as fabricated .

In the first case one can get toughening plus strengthening , in the second one can find toughening without strengthening .

The increasing in volume of zirconia particles can stop the propagation of crack beside filling most of the pores in porcelain which have increased the strength and toughness of the porcelain ceramic [17] .

Typical data fitting the relation between lnln (1 / 1-p) against ln σ are shown in Figure (2-6) for different zirconia content an sintering temperature .

It is known that the low strength / probability part of the ceramic strength distribution is the key shown for mechanical reliability of ceramic bodies [18] . Clearly the larger the predicted strength, the higher the mechanical reliability, and have the better the industrial performance of the ceramic [19] .

The ultimate purpose of optimizing the mean strength as Weibull modulus is, therefore, to improve the ceramic mechanical reliability.

The Weibull modulus is estimated to be moderate values than the typical values found in the literature that usually range between 10 and 50 [20] .

Again the same behaviour of strength the values of Weibull modulus are increased as the sintering temperature increased and that may be due to the increasing in the fracture strength besides the decreasing the porosity casing by the existence of liquid glassy phase .

Introducing zirconia in porcelain samples has affecting the Weibull modulus too much, where the highest value again at 10 wt.% with 1350 ˚C sintering temperature which is three times the value of pure porcelain .

Bell- Shaped carves also relating for the Weibull modulus due to zirconia content and sintering temperature as shown in Figure (7).

The tendency with materials development is to produce ceramics with higher fracture strength. This has generally be achieved through the well known principles of producing uniform microstructure with a fine grain size and very low porosity.

In addition to high strength, a low scatter in strength is also desirable (higher Weibull modulus means lower strength variability).

CONCLUSIONS

In the present study, scatter behaviour of fracture strength of porcelain was investigated. The main conclusions obtained are summarized as follows:

1- The scatters data for fracture strength evaluated in this study could be sufficiently described by using Weibull distribution.
2- The fracture strength of porcelain ceramic is more influenced by sintering temperature and introducing zirconia in it. As a consequence the zirconia content increases the fracture probability of samples.
3- It has shown that the mean fracture strength, Weibull modulus and mechanical reliability of the ceramic can be improved significantly with sintering temperature and zirconia content.
4- Yeilds values for the Weibull modulus between (13 – 39).

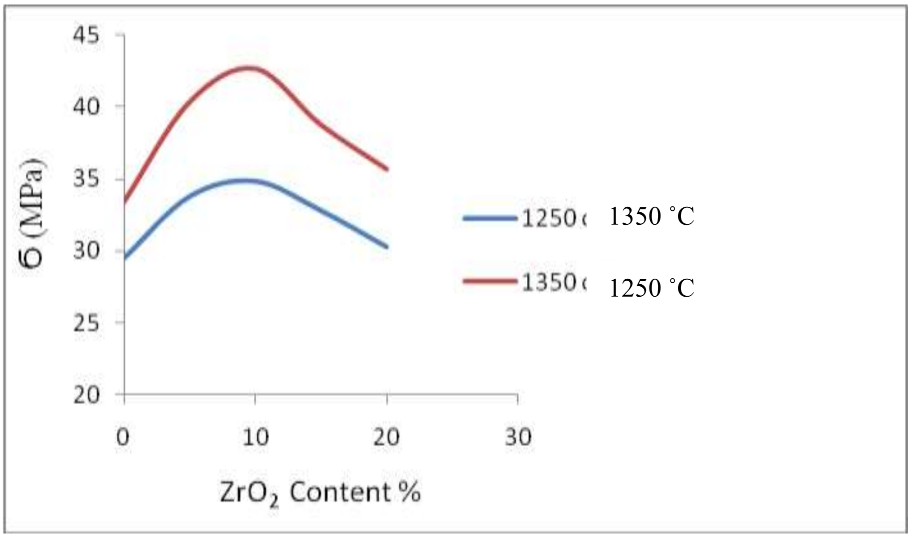

Figure (1) The effect of zirconia content on the strength of porcelain at different temperature.

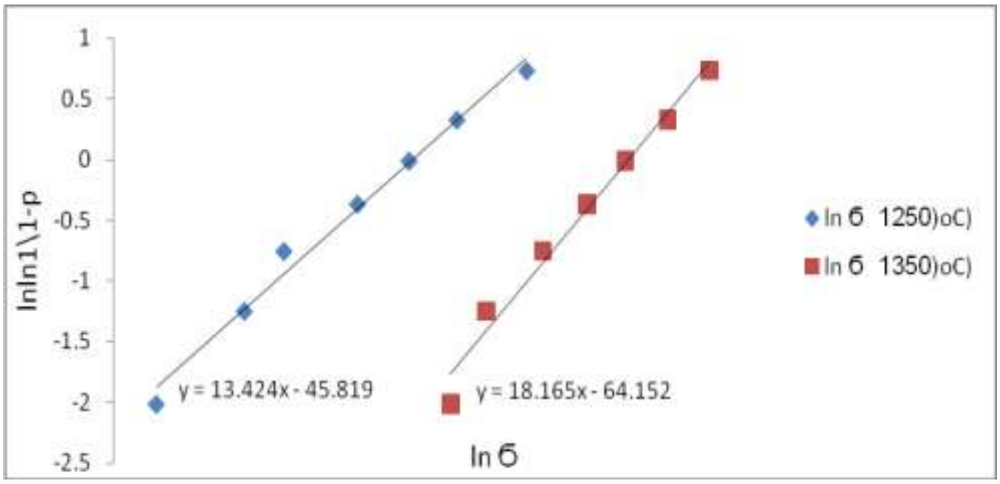

Figure (2) the weibull modulus of pure porcelain at different sintering temperature.

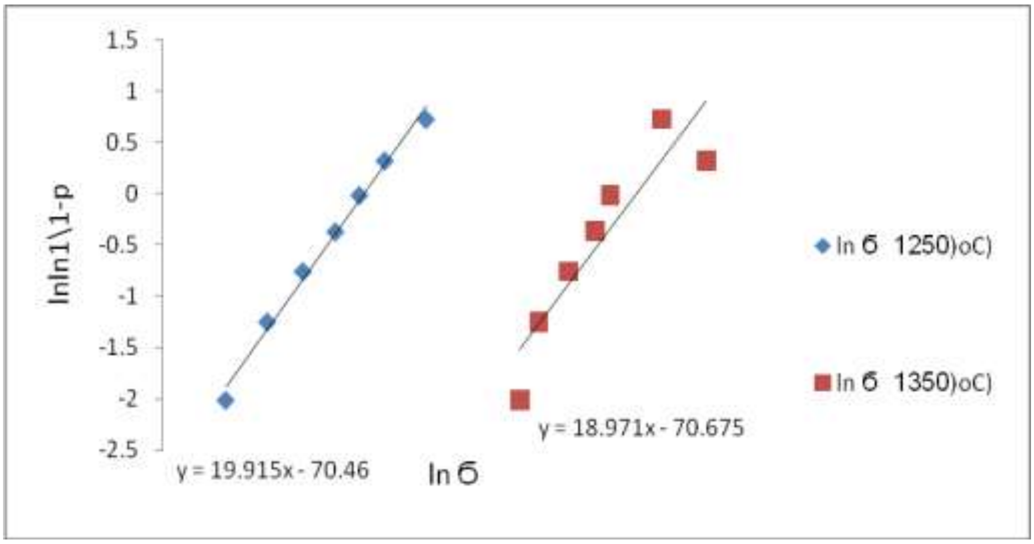

Figure (3) The effect of 5 wt % ZrO_2on the weibull modulus of porcelain at different temperature.

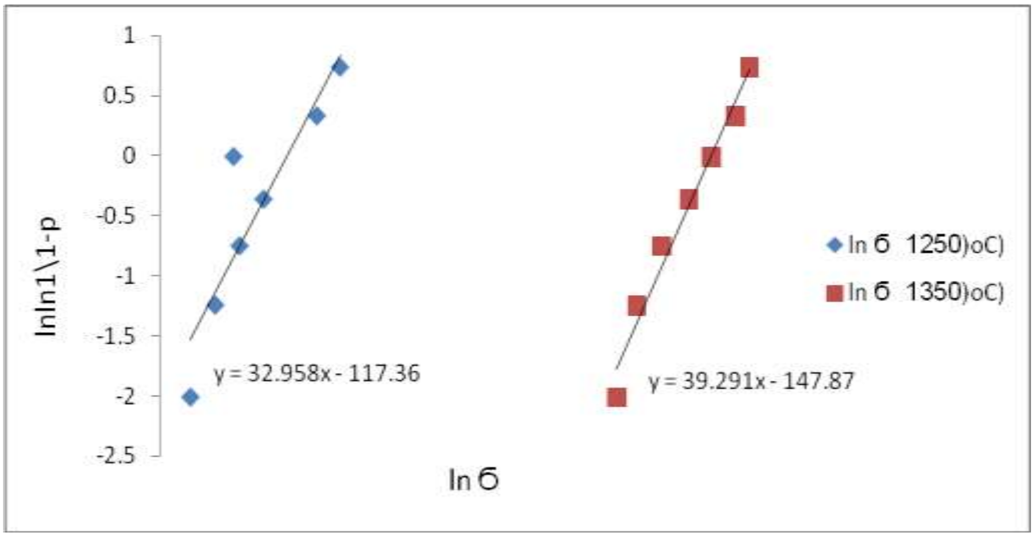

Figure (4) The effect of 10 wt % ZrO_2on the weibull modulus of porcelain at different temperature.

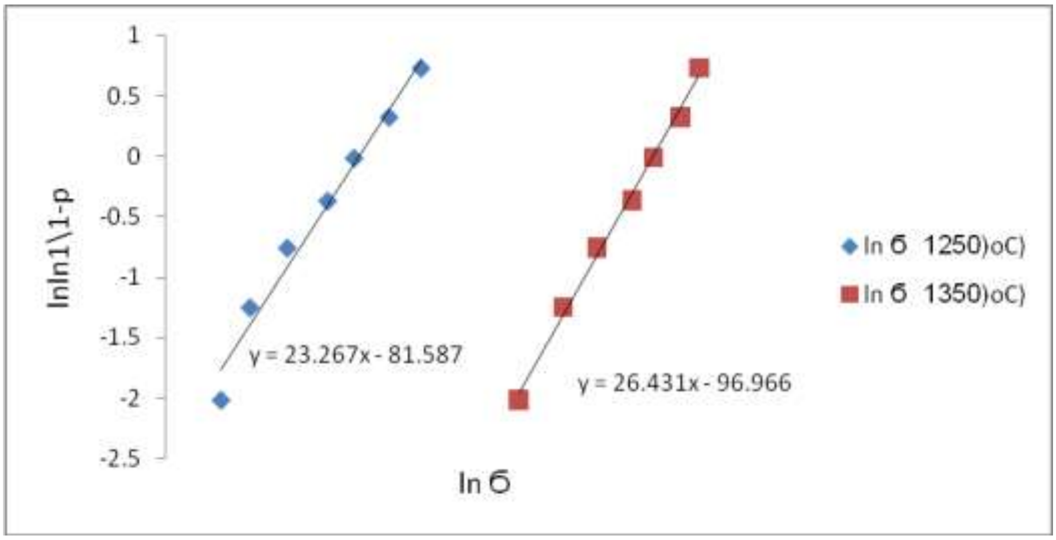

Figure (5) The effect of 15 wt % ZrO_2on the weibull modulus of porcelain at different temperature.

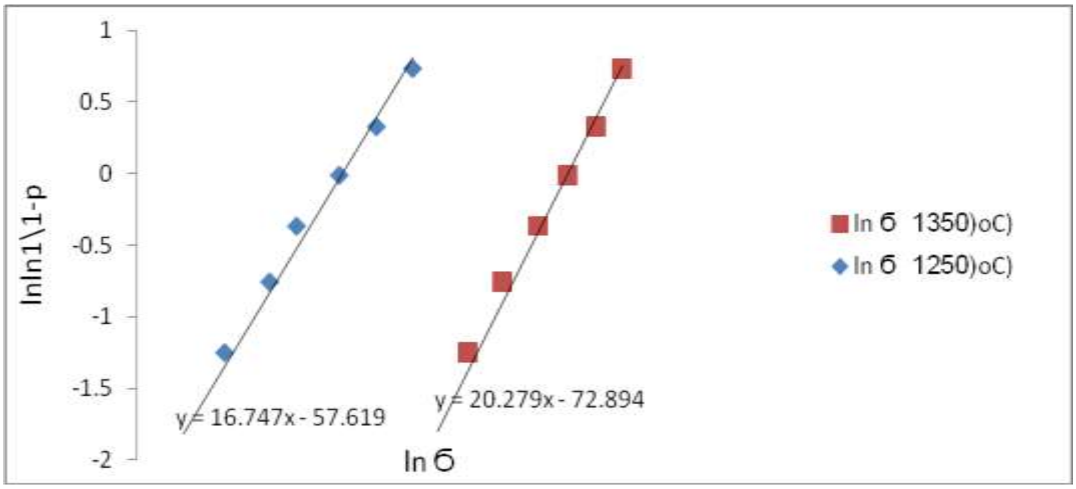

Figure (6) The effect of 20 wt % ZrO_2on the weibull modulus of porcelain at different temperature.

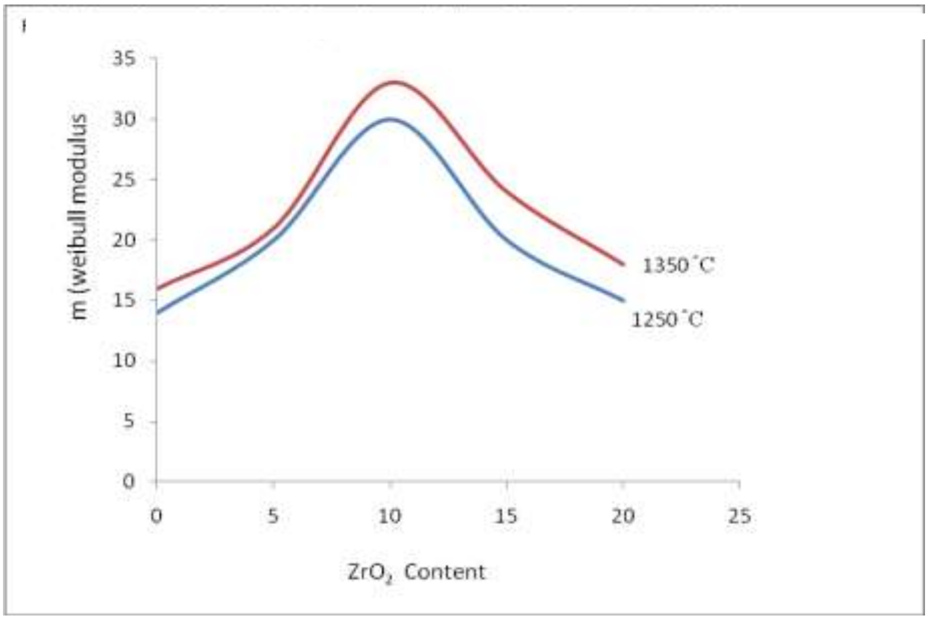

Figure (7) The effect of ZrO_2 Content on the weibull modulus of porcelain at different temperature.

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