

Influence of Nano Barium - Titanate Glass Coating Layer on the Dielectric Properties of Zirconia

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Abstract

Dielectric materials used in electrical energy storage devices, must have a high dielectric constant with low dielectric losses. In this work, new method was used to improve the dielectric properties of 3Y-TZP (3% mol yttria tetragonal zirconia polycrystalline) ceramics, by adding different percentages of nano barium titanate (BaTiO_3) to glaze mixture. 3Y-TZP specimens were sintered at a 1450°C , and the glaze layer was fired at 800°C . BaTiO_3 increased from 40 wt% to 60% resulting an increase in the crystalline size. As well, both dislocation density and lattice strain were decreased down to 175×10^8 lines / mm^2 and 12×10^{-3} , respectively. The dielectric constant values were doubled up to 75.6 for glass-coated specimens when adding 60 wt% BaTiO_3 . This increase was accompanied by an increase in tangent losses value up to 0.026. Electrical breakdown E_{br} of specimens was measured with the number of repeat breakdown and at different ramp rate of the voltage RRV. The $E_{br\text{max}}$ is 55.7 kV / mm at RRV 5 kV / s for coated specimens without BaTiO_3 . A drop in E_{br} has reached a minimum value of 41.7 kV/s at the same RRV. Various phenomena accompanied the E_{br} , including electromechanical, electrothermal and electrical treeing.

Keywords: Zirconia, Nano Barium Titanate, Glaze, Dielectric constant, Electrical Breakdown, Electrical Treeing.

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تأثير طبقة طلاء نانو تيتانات الباريوم - زجاج على خصائص العزل الكهربائي للزركونيا

شهاب احمد زيدان

قسم العلوم التطبيقية – الجامعة التكنولوجية

الخلاصة

يعتبر ثابت العزل الكهربائي العالي ومعامل الفقدان العزلي الواطئ من متطلبات العوازل الكهربائية المستعملة في ادوات خزن الطاقة الكهربائية. استعملت طريقة جديدة في تحسين خصائص العزل الكهربائي للزركونيا 3Y-TZP باضافة نسب مختلفة من تيتانات الباريوم النانوية الى خليط التزجيج. لبدت عينات الزركونيا بدرجة حرارة 1450°C ونضجت طبقة التزجيج بدرجة حرارة 800°C . لوحظ ان تزايد اضافة تيتانات الباريوم من 40 wt% الى 60 wt% ادت الى زيادة الاحجام البلورية وتناقص كل من كثافة الانخلاعات والانفعال الشبكي. تضاعفت قيمة ثابت العزل الكهربائي الى 75.6 نتيجة تعزيز طبقة التزجيج باضافة 60 wt% تيتانات الباريوم. بالمقابل بلغ ظل زاوية الفقد العزلي 0.026 و تناقصت قيمه متانة العزل الكهربائي من 55.7 kV/mm الى 41.7 kV/mm فضلا عن ظهور التأثيرات الكهروميكانيكية والكهروحرارية وظاهرة التشجر الكهربائي.

كلمات مفتاحية: زركونيا، تيتانات الباريوم النانوية، تزجيج، ثابت العزل، متانة الانهيار، التشجر الكهربائي.

Introduction

A small amount of Y_2O_3 is added to zirconia to stabilize the high-temperature cubic phase, otherwise, when cooled from the sintering temperature, the zirconia undergoes a destructive phase transition from tetragonal to monoclinic. However, it is determined that partially stabilized zirconia (PSZ) (that is, contains fewer additives than needed to build 100% cubic crystals) is more resistant to thermal shock than the fully stable form [1].

Zirconia is classified as a good electrical insulator, however, the dielectric properties depends on the phases of zirconia. Among different phases tetragonal zirconia has high dielectric

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constant compared to other phases. Structural properties of zirconia are affected by the variation in crystallite size that leads to phase transition [2].

Yttria-stabilized zirconia materials are pivotal components in wide range of domestic and industrial ceramic products, including microwave telecommunications, capacitors, wear part ceramics, piezoelectrics, fiber optic ferrules, molten metal filters and oxygen sensors [3].

Glaze is a mixture of various fine raw materials and colorants. They are ground into fine powder, mixed with water to make a liquid suspension, and then coated on the surface of bisque porcelain. This mixture melts and vitrifies when exposed to high temperatures during the firing process, forming a smooth glassy surface that fuses to the ceramic surface [4].

The aims of glaze layer are to decorate, waterproof or color an article. Glass coating renders ceramic products vessels suitable for fluid containment, stamping the porosity of fired ceramic. It additionally gives a harder surface [5].

In this work, a new mechanism was adopted to improve the functional properties of ceramic bodies such as electrical insulation. This mechanism relied on adding nano barium titanate powders to the glaze layer mixture characterized by a high dielectric constant.

Barium titanate (BaTiO_3) is a very interesting ceramic material with excellent ferroelectric properties. This makes it very useful in various applications, such as capacitors [6]. In addition, BaTiO_3 is the most promising lead-free material with excellent properties, such as high dielectric constant, low dielectric loss tangent, dielectric reliability, high electromechanical coupling coefficient, good thermal shock resistance and large ductility [7].

Generally electrical ceramic insulators have a wide range of applications in alternating current circuits. A dielectric can be polarized by an applied electric field. When a dielectric is placed in an electric field, the charge deviates slightly from its average equilibrium position, causing dielectric polarization. Due to dielectric polarization, positive charges move toward the electric field, while negative charges move in the opposite direction [8].

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The study of the dielectric properties of the samples as a function of frequency may help to explore their potential applications. The characterization of the dielectric behavior is important not only for the theory of the polarization mechanism, but also from a practical perspective, it is important to understand the frequency dependence of the dielectric constant. The dielectric constant of a material determines its ability to store electrostatic energy [9].

The capacitance with a dielectric (C) of a plane capacitor has thickness (h_0) and area (A) of the plates on each side is directly proportional to the value ϵ_r (dielectric constant), and will be expressed by the formula:

$$C = \epsilon_r \frac{8.854 \times 10^{-12} A}{h_0} \quad (1)$$

When an electric field acts on any substance, the latter dissipates a certain amount of electrical energy, which is converted into thermal energy. This phenomenon is commonly referred to as power loss and refers to the average power dissipated by matter over a certain time interval.

The angle δ is called the dielectric loss angle. Clearly, the tangent of this angle ($\tan \delta$) is equal to the ratio between the active and reactive current. Tangent loss is given by relationship [10]:

$$\tan \delta = \frac{1}{2\pi f \epsilon_r \epsilon_0 \rho_e} \quad (2)$$

Electrical breakdown of the dielectric is the third property that gives an accurate description of electrical insulators, especially in high voltage applications. Puncture test of insulator used to determine the breakdown voltage as shown in figure1a. The insulator to be tested is suspended in insulating oil. Apply voltage and gradually increase voltage until puncture occurs. The voltage at which an insulator starts to break down is called the breakdown voltage. It is possible that the electrical spark passes over the surface of the specimen, and often occurs due to the higher specimen thickness compared to the surface area. This type is called flashover breakdown as shown in figure1b. This dry flashover voltage is usually 50% lower than puncture voltage of the hanging insulator [11].

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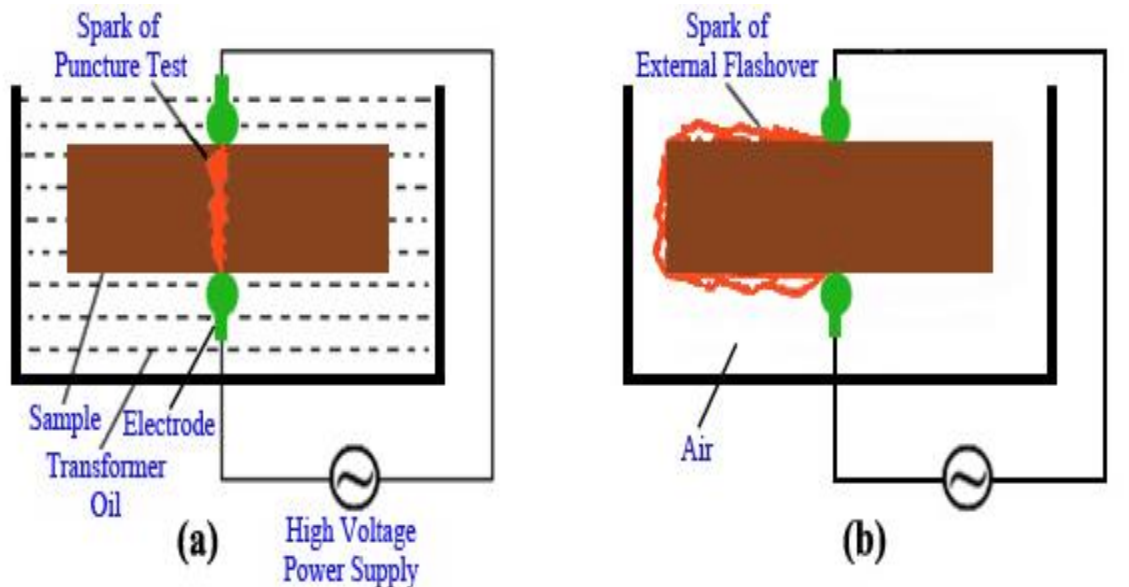


Figure 1: Types of electrical breakdown test, (a) Puncture test (b) Flashover test.

The electrical strength or breakdown (E_{br}) is calculated from the max. voltage (V_{br}) at which an electrical breakdown occurs:

$$E_{br} = \frac{V_{br}}{h_o} \quad (3)$$

The mechanism of failure and the electrical breakdown depends on the time of applicable voltage (or RRV: ramp rate of the voltage kV/s). These mechanisms can be divided into different types, as shown in figure 2 [12].

There are other types of breakdown that do not originate from intrinsic breakdown or from thermal breakdown but occur after a long time, such as breakdown due to tracking and treeing in which dry conducting tracks are formed on the surface of the insulation. Clean, dry, intact surfaces and a clean environment prevent electrical treeing. The conduction path is usually in the shape of a tree branch; therefore, ‘electrical treeing’ is used to describe the growing of the tree-shaped conduction path [13].

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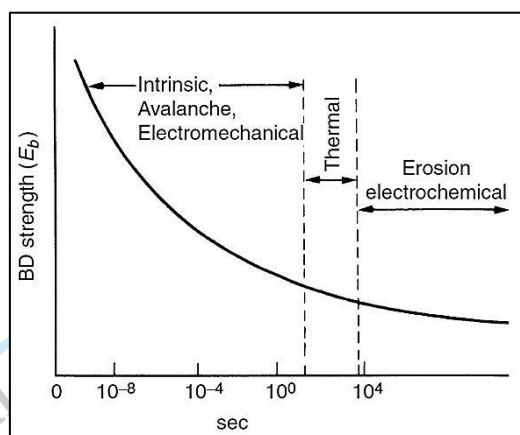


Figure 2: Mechanisms of electrical breakdown in solids and variation of with time after application of voltage [12]

Various types of dielectric materials have been developed for high-temperature capacitors, but each has its own limitations. Generally, ceramics can withstand high temperatures and exhibit a high ϵ_r , but their breakdown strength (E_{br}) is low, and their dielectric properties vary greatly with temperature, limiting their applications. Glass always has higher E_{br} and relatively good thermal stability, but its lower ϵ_r will hinder the increase of energy storage density [14].

The originality in this work is ideally designed to have the optimal characteristics of both a glaze layer, such as high breakdown strength, and those of a ferroelectric ceramic additives, such as high dielectric constant. This procedure aims to obtain high temperature dielectric materials for electrical energy storage devices.

Materials and methods

The specimens of PSZ or 3% mol Ytria-Tetragonal Zirconia Polycrystalline (3Y-TZP) were prepared from catting pre-sintered green zirconia blanks as commercial material supplied by (VITA YZ HT white disk– Germany) by using electric saw with diamond cutting disk. Dimensions of the cut specimens were 10 mm in diameter and of different thicknesses. The sintering process is completed by using an electrically programmable furnace type

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(NABERTHERM-P310-GERMANY) to reach 1450 °C at a heating rate of 15 °C / min within 4 hours of sintering soaking time to complete the process of grain growth and obtain required phase. The surface of specimens was glazed by the following mixture; DENTSPLY Ceramco3 Over Glaze (Low Temperature) powder U.S.A. mixed with different percentage of barium titanate nano powder (0, 20, 40 and 60 wt%). Nano BaTiO₃ powder (99.95%, 60-80 nm) was supplied from Sinopharm Chemical Reagent Co., Ltd, Beijing, China. After adding BaTiO₃ to the glaze powder mixed it with liquid glass (DENTSPLY Ceramco3, stain and Glaze Liquid U. S. A.). After coating on just one side of the disc, the specimens are left to dry then firing up to 800 °C. Glazing layer thickness of the specimens ranged from 130 to 150 µm. The codes and mixing ratios of the coated specimens are listed in the table 1.

Table 1: Specimens codes and weight percentage of coating components.

Specimen code	Substrate	Glaze powder (wt %)	BaTiO ₃ nano powder (wt %)
S0	3Y-TZP	0	0
S1	3Y-TZP	100	0
S2	3Y-TZP	80	20
S3	3Y-TZP	60	40
S4	3Y-TZP	40	60

X-ray profiling is commonly used to measure average crystallite size and lattice strain, these two parameters are determined by the width of the XRD pattern peaks. Lattice strain is a measure of the distribution of lattice constants and it is caused by lattice defects such as lattice dislocations. XRD Data Analysis, such as Williamson-Hall method is used to estimate the crystallite size and lattice strain.

X-ray powder diffraction (XRD) studies were performed for all of the prepared solid specimens using Shimadzu XRD-6000 system with the radiation source of Cu, NF type. The crystallite size of the specimens was calculated using Scherrer's equation:

$$D = \frac{0.9 \lambda}{\beta \cos \theta} \quad (4)$$

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Where D is the average crystallite size of the studied phase, λ is the wavelength of the X-ray beam used ($1.54056 \text{ \AA}$), β is the full width at half max. (FWHM) of the diffraction peak, and θ is the angle of the diffraction. The dislocation density (ρ_D) is a measure of the number of dislocations in a unit area (lines / m^2) of a crystalline material [15]. The presence of low dislocation density is essential for implant applications.

$$\rho_D = \frac{1}{D^2} \quad (5)$$

Addition of Scherrer's formula and the strain (ϵ) induced broadening results in [16]:

$$\epsilon = \frac{\beta}{4 \tan \theta} - \frac{0.9 \lambda}{4 D \cos \theta} \quad (6)$$

Dielectric properties were measured with temperature by using MICROTTEST 6377 impedance analyzer. As well the dielectric strength was tested by using High Voltage supplier (BAUR - PGO - S -3) Germany with a range (0-60 kV) and frequency (50 Hz). Breakdown device has the ability to change the ramp rate of the voltage RRV (0.5, 1, 2, 3 and 5 kV/s). All dielectric strength tests are performed by immersing the sample in transformer oil to prevent external flashover before breakdown [17].

Results and discussion

Phase transformations occurred during the 3Y-TZP sintering process at 1450°C , in which the most important of this transition from monoclinic to tetragonal. Practically, loss of mass due to ignition is 5.1 % and associated with phase transformation. As well, the measured volume shrinkage and bulk density are 47.7 % and 5.93 g/cm^3 , respectively.

XRD patterns are used to diagnose and determine crystal structure parameter of the ceramic base phases and coating layer. Figure 3-S0 shows the diffraction pattern of the zirconia specimen without a glazing layer, in which it is clear that the two main phases are t and m of ZrO_2 . The largest phase is t- ZrO_2 because the sintering temperature has reached the preferred value for stabilizing t- ZrO_2 phase at 1450°C [18]. Lattice parameters are calculated from the

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formulas (4,5 and 6) are listed in table 2. The results indicated that the crystallite size of t-ZrO₂ phase at (101) crystal plane is greater than m-ZrO₂ phase at (111) plane. This result agreed with (Rifki Septawendar et al. 2012) results [19]. Also, the dislocation density of t-ZrO₂ and m-ZrO₂ are 5×10^8 lines/mm² and 9×10^8 lines/mm², respectively. These values are considered relatively low, as well the lattice strain of m-ZrO₂ is higher than that of t-ZrO₂ and its value ranged from 2.2 to 3×10^{-3} .

Glazing ceramic bisque with amorphous glass leading to block the crystal phases of zirconia ceramics base, since the diffraction occurred in the glaze layer. As for the m-ZrO₂ phase shown in the diffraction pattern figure 3-S1. Monoclinic phase grows due to the low glazing temperature of about 800 °C. The crystallite size of m-ZrO₂ phase was decreased to 6.8 nm after glazing, whereas the dislocation density and strain were increased to 216×10^8 lines / mm², 14.7 respectively.

Nano-BaTiO₃ addition to the glass mixture (40 wt%) did not significantly affect the crystal structure parameters of m-ZrO₂ phase, as shown in figure 3-S3. However, the parameters of the crystal structure of the cube BaTiO₃ phase were near to the previous results, except high dislocations density due to the effect of amorphous glaze structures in addition to the different thermal parameters between BaTiO₃ and glaze layer.

Increasing the percentage of BaTiO₃ to 60 wt% in plane (110) led to a decrease in the dislocation density to half and reached 175 lines / mm², in spite of the increase in crystallite size. As well, other phases and crystalline peaks became eminent at that addition, as shown in figure 3-S4.

The dielectric constant is one of the important objectives of this research. 3Y-TZP is one of the dielectric materials and has an energy gap ranging from 3 to 5 eV depending on the zirconia phases [20]. Figure 4 shows that the dielectric constant changes with frequency for all specimens. The value of the dielectric constant decreased from 35 to 29 as a result of coating with glass. Dielectric constant decreased as frequency increases due to the decrease in

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polarization, especially the interfacial or space charge polarization in this range of microwave frequencies.

The high dielectric constant value of BaTiO₃ resulted in an increase in the dielectric constant of the glaze layer containing mixture of glass with BaTiO₃. Therefore, the dielectric constant values were doubled and reached to ($\epsilon=75$) when adding 60 wt% of BaTiO₃.

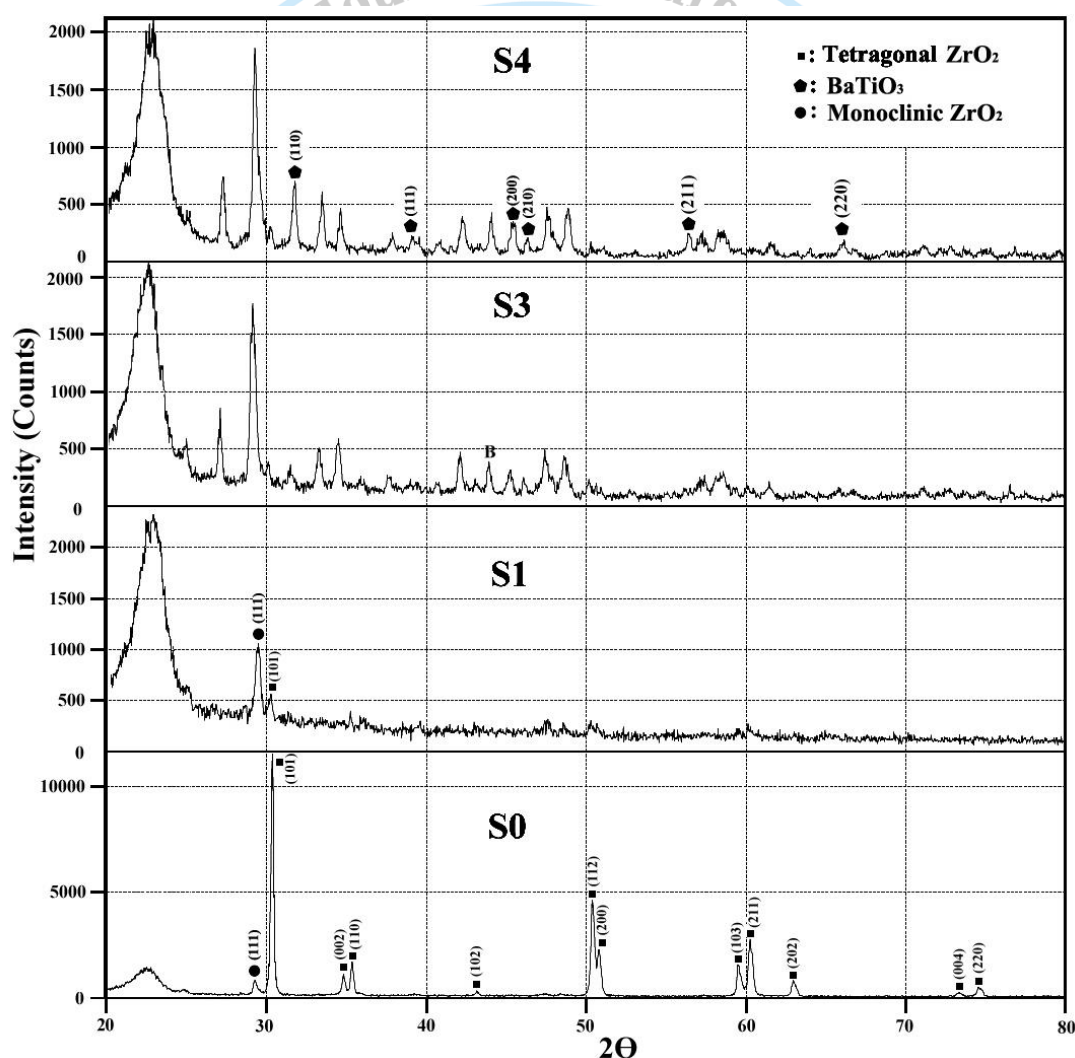


Figure 3: X-Ray diffraction patterns of: (S0) 3Y-TZP without Glaze, (S1) 3Y-TZP with Glaze, (S3) 3Y-TZP with (Glaze + 40 wt% BaTiO₃) and (S4) 3Y-TZP with (Glaze+60 wt% BaTiO₃).

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Table 2: Lattice parameters of different specimens.

Specimen Code	t- ZrO ₂			m- ZrO ₂			BaTiO ₃		
	D nm	$\rho_D \times 10^8$ lines/mm ²	$\epsilon \times 10^{-3}$	D nm	$\rho_D \times 10^8$ lines/mm ²	$\epsilon \times 10^{-3}$	D A°	$\rho_D \times 10^8$ lines/mm ²	$\epsilon \times 10^{-3}$
S0	44.8	5	2.2	33.5	9	3	---	---	---
S1	---	---	---	6.8	216	14.7	---	---	---
S3	---	---	---	6.7	221	15.1	5.3	357	17.4
S4	---	---	---	7.3	190	13.9	7.6	175	12

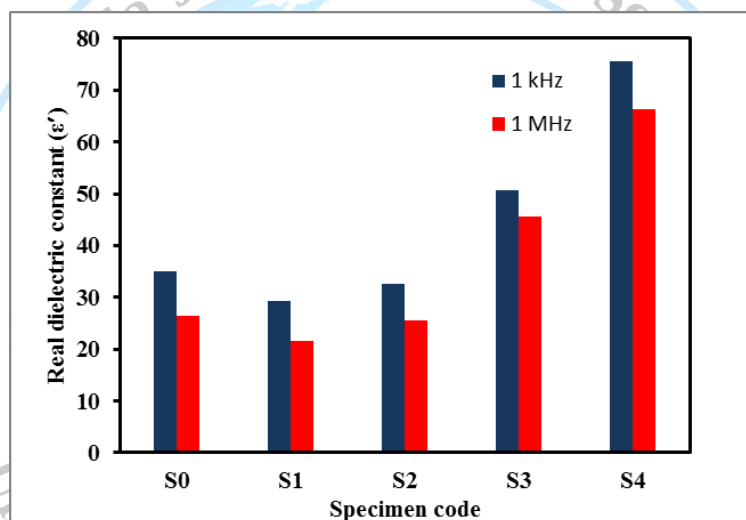


Figure 4: Dielectric constant of the specimens at two frequencies

Generally, the dielectric constant of the nanocomposite glaze layer was increased, due to the dipole interaction between the spherical filler (Nano-BaTiO₃) and glass matrix. The dielectric constant of a ferroelectric material (BaTiO₃) depends on the polarity and the activity of the dipoles in the glass, and the mobility of the macro-molecular segments, which depends on the interaction between the inorganic nano-filler and the glass matrix [21]. Moreover, the increase in the dislocation density leads to an increase in the dielectric constant due to the interfacial polarization increase, especially in microwave frequencies range [22].

Dielectric loss is the amount of power losses in a dielectric material under the action of an alternating voltage. Figure 5 shows a decrease in the dielectric loss tangent when the specimens

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are coated with glaze to a minimum value of $\tan\delta=0.08$. The value of $\tan\delta$ is inversely proportional to frequency, that the relationship is described by the formula (2). Dielectric loss is the combined result of the electrical conductivity and directional polarization of a material. The energy loss that occurs in the dielectric is due to dc conductivity and dipole relaxation. The dissipation factor ($\tan\delta$) of a dielectric material is a useful indicator of energy as thermal energy loss [23].

Although the barium titanate dielectric constant value is high, its dielectric loss is considered high compared to the glass and zirconia [24]. Therefore, high dielectric loss was observed with an increase in the percentage of adding (60 wt%) BaTiO_3 to glass, and the values for different frequencies are 0.26 and 0.23 at 1kHz and 1MHz respectively.

Due to the competitive mechanism between the dielectric constant (ϵ_r) and the dielectric loss ($\tan\delta$), an increase in ϵ_r will cause an increase in $\tan\delta$, especially at high applied electric fields [14].

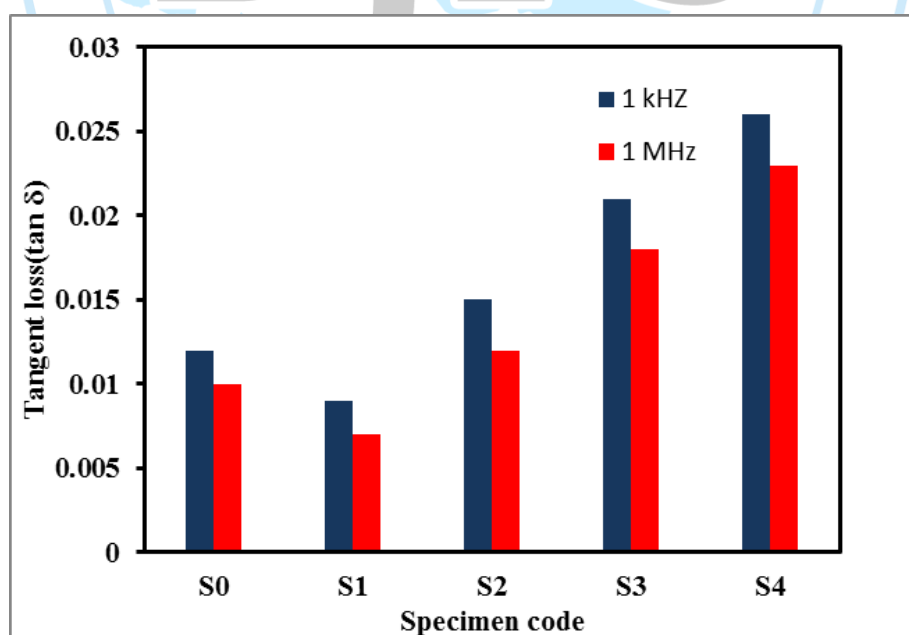


Figure 5: Dielectric losses of the specimens at two frequencies.

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The Electrical breakdown strength test is considered as one of the destructive tests. Practically, the puncture breakdown was measured with number of repeat breakdown and at different ramp rates of the voltage (RRV) as shown in figure 6. It was noticed that a deterioration in the electrical strength had occurred in all specimens. Intrinsic electrical breakdown occurs at high RRV, and it is accompanied by electromechanical effects that lead to the deterioration of mechanical properties as a result of microscopic cracks arising from electrical stress. Sometimes, the flashover can be occur at high thicknesses or if the test is done without immersing the specimen in the oil. The channel (flashover path) is formed due to the breakdown of the dielectric material. For AC tests, this happens at high and low of RRV as shown in Figure 7 [25]. The deterioration was more dangerous in case of lower RRV, due to heating resulting from the long time. In other words, electrothermal breakdown occurs at a low RRV (0.5 kV/s) and this is accompanied by electrochemical changes that include changes in the structure of specimen material, as well as a change in electrical characteristics, i.e. the material becomes more electrical conductivity. The values of breakdown ranged from 35 to 55 kV/s, and retreated to approximately 5 kV/s after five repetitions of breakdown.

Although the glaze coating was necessary to improve the surface properties, it was also useful to increasing the electrical breakdown. Figure 8 shows a change in maximum breakdown values with RRV, a clear increase in breakdown for glazed specimen without adding BaTiO₃. Due to the high dielectric losses associated with electrical conductivity, this caused a decrease in breakdown with an increase in the percentage of BaTiO₃ added to the glaze.

Electrical trees are degradation phenomena that develop in dielectric insulation exposed to high electric fields. Partial discharges can cause the formation of a tree-like structure and cause the tree to propagate and eventually over the entire thickness of the insulating layer, causing it to short circuit [25]. This phenomenon occurred in most of the tested specimens, especially in the case of unglazed specimens. Various tree inscriptions are formed depending on the RRV as shown in figure 9.

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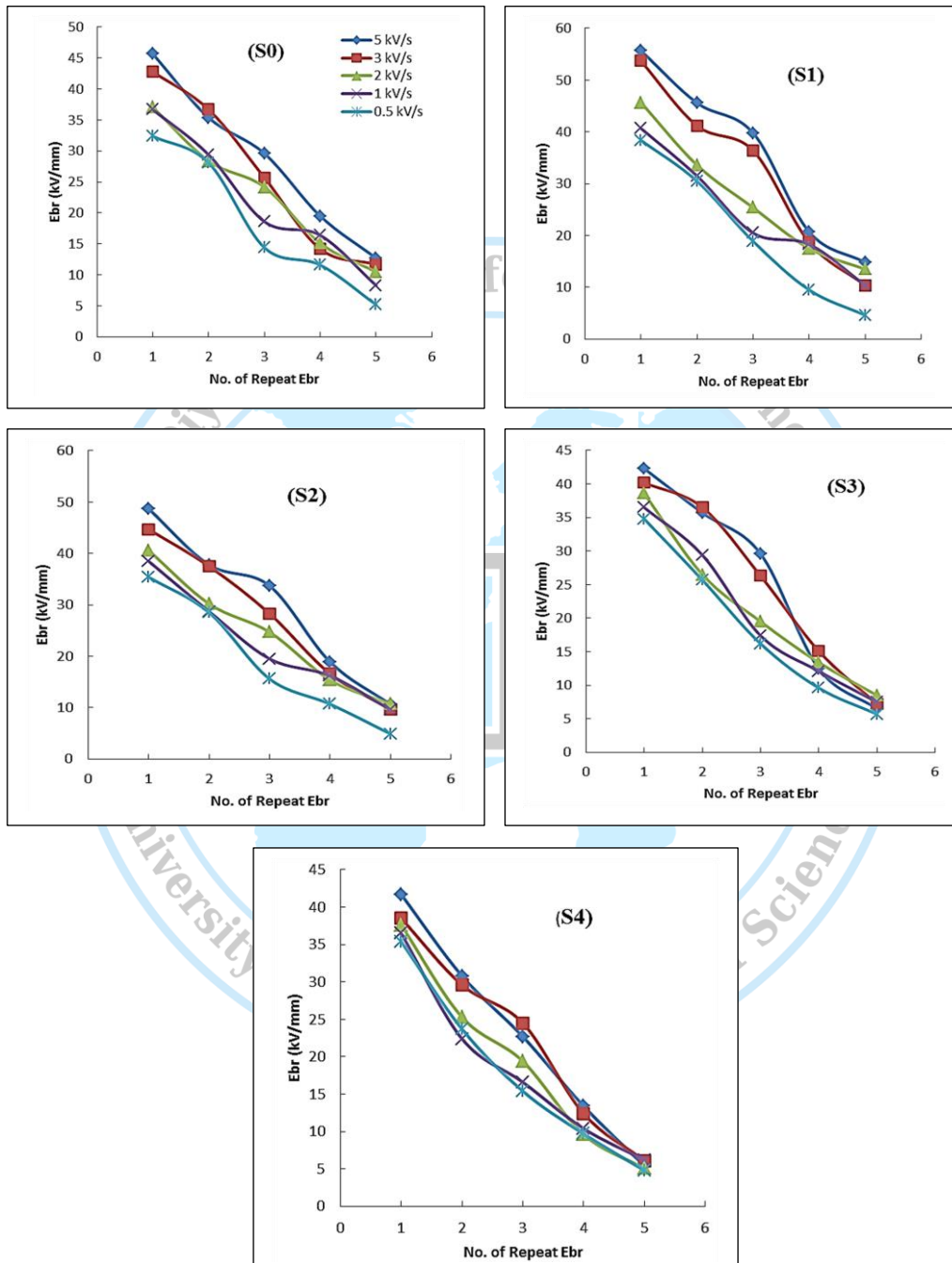


Figure 6: Variation of puncture breakdown with number of repeat breakdown and at different RRV

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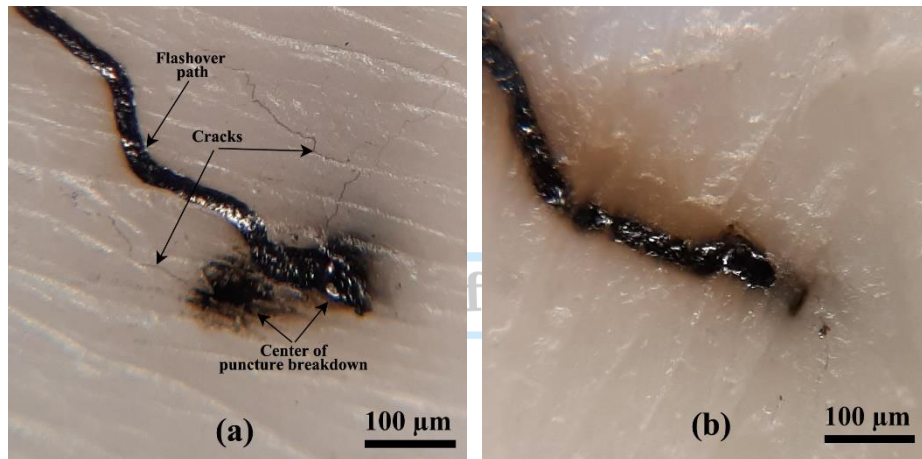


Figure 7: Traces of breakdown at RRV:(a) 0.5 kV/s, (b) 5 kV/s for S0 specimen

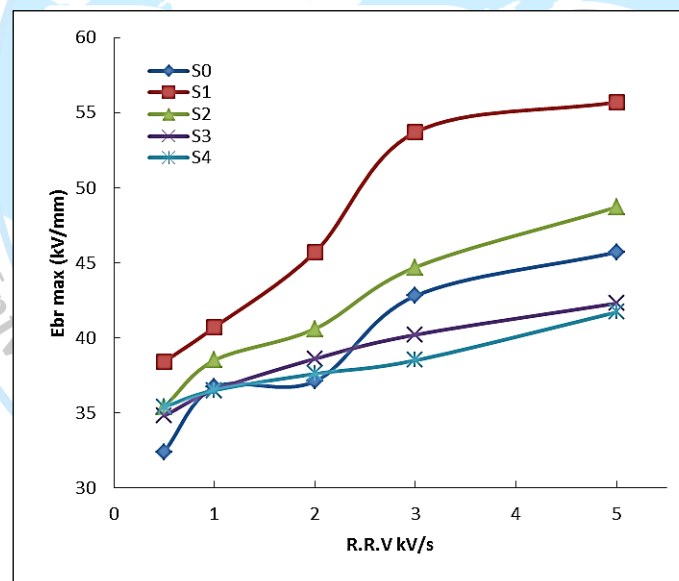


Figure 8: Dependence of maximum electrical breakdown on RRV of specimens

The BaTiO₃ added to the glaze mixture masked the electrical trees on the specimens' surfaces, because the glaze layer dispersed the energy of the applied voltage. Thus, the electric spark destroyed the glass structure and penetrated to the ceramic basis, as shown in the figure 9. The length of the trees depends on the time of applied voltages (RRV), in other words, the increase in RRV leads to increase the length of electrical trees [26].

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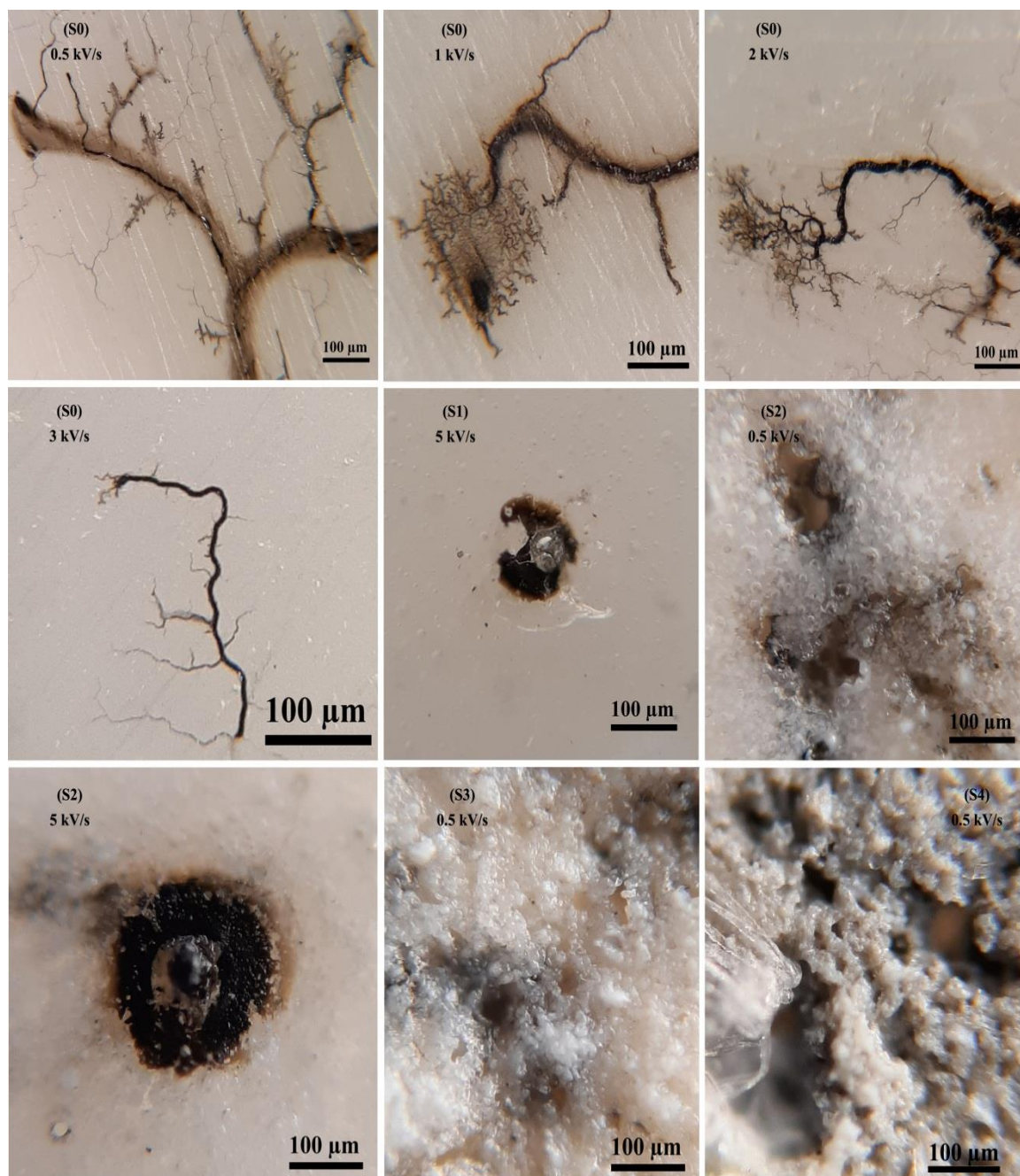


Figure 9: Captured photographs of breakdown traces for specimen's surface at different RRV

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Conclusions

Adding nano barium titanate powder to an amorphous glazing mixture leads to convert the glaze layer to ceramic glass. The glaze coating layer dispersed the electrical field at high voltage, thus protecting the ceramic basis from the phenomena of electrical treeing and early electrical breakdown. Low ramp rate of the voltage leads to electrothermal breakdowns. On the other hand, high ramp rate of the voltage leads to electromechanical breakdowns and has highest breakdown strength. Despite the disadvantages of increasing the dislocation density, it is useful for increasing the dielectric constant. Increasing the dielectric constant values, with permanence loss tangent and electrical breakdown within acceptable limits, confirms the possibility of using these materials in the manufacture of electrical energy storage insulators.

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