

# Testing media influences on the dielectric properties of lead sodium niobate glass-ceramics

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## Abstract

The influences of testing media on the breakdown strength (BDS) and dielectric properties of glass-ceramics in the  $\text{Na}_2\text{O}-\text{PbO}-\text{Nb}_2\text{O}_5-\text{SiO}_2$  system were investigated. This work was brought out by consideration of the electric conductance, dielectric constant and breakdown strength of different testing media, which are the main reasons for the different dielectric properties and BDS values of the identical dielectric sample. Leakage current (LC),  $P-E$  hysteresis loops,  $C-V$  curves and breakdown tests show that the BDS and the dielectric properties of the glass-ceramics could be optimized through using appropriate testing medium. It turns out that three favorable characteristics of the dielectric composites could be optimized in silicon/castor oil mixture: the lowest LC ( $\text{LC} = 6.72 \times 10^{-6}$  A, at  $E = 25$  kV/mm), thin  $P-E$  hysteresis loops and low hysteresis. Furthermore, the highest BDS of the glass-ceramic was obtained in glycerin ( $\text{BDS} = 105.6$  kV/mm with sample thickness of 0.108 mm) compared to other media.

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## 1. Introduction

Test condition plays a very important role in dielectric properties measurement. The measured results of some specific properties of dielectric materials, such as breakdown strength, leakage current and  $P-E$  hysteresis loops, are usually sensitive to the testing environment and also the electrode geometry. To date, the main focus of improvement has been on the influences of the electrodes [1–3]. However, the testing media, especially the testing liquids, also have major impacts on the measured results of the dielectric performance.

Liquid testing media have been widely used to prevent flashover and corona discharge in dielectric properties measurement at high voltage. Traditionally, silicon oil has been mainly used as the testing medium in breakdown strength (BDS) tests [4–7] and  $P-E$  hysteresis loops measurements [8]. In addition, some work was about the application of transformer oil [9]. Both of the above mentioned oils are low dielectric constant liquids. Also, these are some other work

concerning the use of high dielectric constant liquid media. Luo et al. [3] performed dielectric failure measurement in ethylene glycol/water mixture, and observed high breakdown strength of dielectrics. However, it appears highly necessary to establish a detailed relationship between the liquid media characteristics and the measured dielectric properties, especially when it concerns about dielectric materials with high dielectric constant and high breakdown strength, such as in the case of glass-ceramics.

In the present paper, we investigated the relationship between different testing media and the measured properties of the dielectrics. A modeled glass-ceramic based on lead sodium niobates was used as the dielectric material to approach to the optimal value of its dielectric properties. The relationship between the measured BDS of the dielectric material and the dielectric constant of the liquid media has been discussed.

## 2. Experimental procedure

Lead sodium niobates based glass-ceramic dielectrics consisting of high BDS glass phase and high dielectric constant ceramic phases,  $\text{Pb}_2\text{Nb}_2\text{O}_7$  and  $\text{NaNbO}_3$  [3], were provided for the samples. The dielectric constant and dielectric loss of this

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Table 1  
Characteristics of some testing liquid media.

Medium	Dielectric constant	BDS (kV, 2.5 mm)	Leakage current (2 kV/1 cm)	Thermal conductivity (W/m K)
Silicon oil	2.2–2.6	13.7–17.7	$8.56 \times 10 \text{ PP}^{-9} \text{ PPA/cmPP}^2$	0.1
Transformer oil	2.2	35–50	$3.97 \times 10 \text{ PP}^{-9} \text{ PPA/cmPP}^2$	0.128
Silicon/castor oil	3.5–3.9	19.9–21.9	$2.40 \times 10 \text{ PP}^{-9} \text{ PPA/cmPP}^2$	0.14
Glycerin	37	~20	$3.45 \times 10 \text{ PP}^{-3} \text{ PPA/cmPP}^2$	0.276
Glycol	42.5	~20	$4.23 \times 10 \text{ PP}^{-3} \text{ PPA/cmPP}^2$	0.252

glass-ceramic are 170 and 0.0098 at 1 kHz, respectively. Five different testing liquids were selected and studied in this work according to their typical characteristics, as summarized in Table 1. The glass-ceramics were cut and polished into sheets of 0.05–0.40 mm in thickness. Then, Au metal films, as contact electrodes of the plane dielectric capacitor, were coated on both surfaces of the glass-ceramic samples by DC magnetron sputtering (Model JK-200B, Instrument Company, Ltd., Beijing, China).

The morphology of glass-ceramic samples and perforation after breakdown test were observed by field-emission scanning electron microscopy (FE-SEM; Model S-4800, HITACHI, Japan). The breakdown strengths were measured by a Tektronix AFG 3021 Arbitrary/Function Generator (Beaverton, OR) and a Trek Model 30/20A high-voltage amplifier (Beaverton, OR) under 1 Hz AC triangle wave with a rate of increase of 300 V/step in silicon oil (SO), silicon/castor oil mixture (SCO), transformer oil (TO), glycol (GO) and glycerin (GE), respectively. Furthermore, leakage currents,  $P$ – $E$  hysteresis loops and capacitance–voltage ( $C$ – $V$ ) curves were measured using a ferroelectric tester (RT6000HVA, Radiant Technology, Albuquerque, NM).

### 3. Results and discussion

#### 3.1. Dielectric properties

As shown in Fig. 2, prior to 20 kV/mm, there is no significant difference in leakage currents of the glass-ceramics tested in SO, TO and SCO, while  $j_{LC}(\text{SO}) > j_{LC}(\text{TO}) > j_{LC}(\text{SCO})$  is evidenced after 20 kV/mm. Also, the leakage current densities tested in glycol and glycerin are  $\sim 10^{-3} \text{ A/cm}^2$ , which are five orders of magnitude higher than those tested in the other three liquids. This is due to the high electric conductivity of GO and GE ( $10^{-3} \text{ A/cm}^2$ , shown in Table 1). Thus, in what follows, only transformer oil (TO), silicon oil (SO) and silicon/castor oil mixture (SCO) testing liquids were used to study the leakage current density–electric field ( $j$ – $E$ ) behavior of the dielectric glass-ceramic samples in order to establish the relationship between the measured leakage current of the dielectric glass-ceramic and the electric conductivity of the liquid media.

In Fig. 3(a) are shown the current–voltage ( $I$ – $V$ ) results of the dielectric glass-ceramic measured in these three testing media. Obviously, the leakage current increased with the electric conductivity of the liquid media. The conduction mechanism of the dielectric glass-ceramic can be analyzed by

three regions as seen in Fig. 3(a), i.e. Ohmic conductivity region, region of exponential current increase and region of vertical current enhancement. The resistivity values ( $\approx 1 \times 10^{10} \Omega \text{ m}$ ) of the dielectrics with nano-sized ceramic phases embedded in glass matrix (as shown by the FE-SEM image in Fig. 1) via controlled crystallization process [10] were determined for Ohmic part. Since the  $\ln j$  versus  $E^{1/2}$  plot is the best liner fitted line which is shown in Fig. 3(b), the second part on the  $I$ – $V$  curves might be governed by Schottky emission [11,12]. A similar phenomenon was also observed in (Ba,Sr)TiO<sub>3</sub> ferroelectric ceramics on copper by Dedyk et al. [13]. The third part of the vertical current increase may be related to the regime of the complete filling of deep traps [13,14]. The practically vertical  $I$ – $V$  dependence could be interpreted as dielectric breakdown; however, the electrical properties of the sample were restored after a voltage decrease [13] (Figs. 2 and 3).

Due to the large leakage current, curves tested in glycol (GO) and glycerin (GE) cannot exhibit  $P$ – $E$  hysteresis loops [15]. The  $P$ – $E$  hysteresis loops tested in SO, TO and SCO are plotted in Fig. 4. Obviously, it can be seen that the  $P$ – $E$  loops get thick accompanied by the increase of dielectric leakage current. Due to the largest electric conductivity of SO, the dielectric glass-ceramic shows the pronounced broadening  $P$ – $E$  loops (indicating an increases in dielectric loss) [16], while the thin  $P$ – $E$  hysteresis loops tested in SCO is possibly induced by the lowest leakage current of the SCO. In Fig. 5 are illustrated the DC bias voltage dependence of dielectric constant of the glass-ceramic tested in transformer oil (TO), silicon oil (SO) and silicon/castor oil mixture (SCO). The glass-ceramic

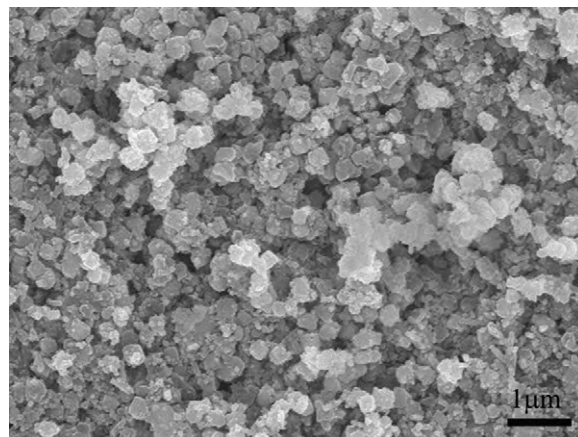


Fig. 1. FE-SEM micrograph of glass-ceramics.

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