Preparation and Dielectric Properties of the Amorphous Basaltic Glass

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Abstract

Due to the continuous basalt fiber is a kind of amorphous material, only the basaltic glass without crystallization can represent its dielectric properties. To explore the dielectric properties of the continuous basalt fiber, amorphous basaltic glass must be prepared. The present research focuses on the influence of chemical component on the preparation process of the amorphous basaltic glass and its dielectric properties. The basaltic rocks from different places of China were melted at 1500℃, then the melt was poured into the mould, at last glass sample was annealed at 650℃ for 2h. Ten groups of basaltic rocks were studied, and the results showed that the melt viscosity of basaltic rocks with 55-58% SiO$_2$ was poor at 1500℃. The high-content of Fe$_2$O$_3$ and TiO$_2$ in basaltic rocks was found to enhance the formation of magnetite (Fe$_3$O$_4$) crystal during the annealing process. The other five groups of basaltic rocks were suit to the amorphous basaltic glass. At 1MHz, the best dielectric constant of amorphous basaltic glass is 6.55, the dielectric loss is 4.034×10$^{-3}$.

1 Introduction

For electronic communication, the substrate of printed circuit board must has excellent dielectric properties: low dielectric constant and low dielectric loss. The lower the dielectric constant is, the faster the signal transfers; The lower the dielectric loss is, the better the integrity of signal transfers in the medium. Generally, the substrate is composed of resin and glass fibers. The resin has excellent dielectric properties, its dielectric constant is about 2.8 ~ 3.0. But the dielectric constant of glass fibers is too higher, such as E glass fiber’s dielectric constant is 6.7 ~ 7.3 at 1 MHz frequency. Therefore, the poor dielectric property of glass fibers is the key factor restricting the improvement of substrate performance. However, the development of low dielectric properties glass fibers has been a bottleneck all along, so it is imperative to find a substitute.

Just like glass fibers, the basalt fiber can also be used as reinforced materials. Compared with glass fibers, the basalt fiber has some better properties such as good modulus, high temperature resistance, high strength and capable of chemical resistance. Basaltic rocks are the only raw materials for basalt fiber production. Basalt is an igneous rock and it is the main component of the earth's oceanic crust, so basaltic rocks are abundant and low cost. However, there are no reports on the dielectric properties of basalt fibers, to the best of my knowledge.

In general, the dielectric properties of glass fibers can be suggested through measuring its amorphous glass blocks without crystallization. Similarly, the dielectric properties of basalt fiber can also be obtained through evaluating the properties of amorphous basaltic glass. So the preparation of amorphous basaltic glass is the key to get the dielectric properties of its basalt fiber.

But basaltic glass is easy to crystallize and form glass-ceramics during the annealing process. Basaltic glass that contained 46 ~ 50% SiO$_2$ and 9% FeO gave monophasic omphacite pyroxene at 950℃. Basaltic glass containing 49% SiO$_2$, 3.81% Fe$_2$O$_3$ and 2.68% FeO formed pyroxene glass-
ceramic with small portions of a glassy phase [16]. Crystallization of basaltic glass from the northern Harrat area in the temperature range of 800 ~ 1000 °C gave augitic pyroxene as the major phase with small amounts of olivine, haematite and magnetite [17]. The magnetite was easy to crystallize from basalt glass at 650°C and augite at 860°C [18]. In view of the fact that basaltic glass is easy to crystallize during high temperature annealing progress, the annealing temperature is set at 650 °C in our experiment to avoid crystallization. Therefore, the major purpose of this paper is to prepare amorphous basalt glass and measure its dielectric properties.

2 Experiments And Methods

2.1 Experimental Material

Ten samples of basaltic rocks selected from different regions of China were classified into three main types. G1: 1 ~ 2 samples with much higher SiO_2; G2: 3 ~ 5 samples with much higher Fe_2O_3 and TiO_2, G3: 6 ~ 10 samples with lower SiO_2 than G1 and lower Fe_2O_3 and TiO_2 than G2. The basaltic rocks were crushed and milled for a while in an agate mill till reaching very fine powder particle size.

The chemical components were carried out of the obtained basalt powder by using X-ray Fluorescence (XRF). Table 1 shows the chemical analyses of the raw materials.

| Types | Sample | SiO_2 | Al_2O_3 | TFe_2O_3 | TiO_2 | MgO | CaO | Na_2O | K_2O |
|-------|--------|-------|---------|----------|-------|-----|-----|-------|------|
| G1    | 1      | 58.43 | 16.88   | 6.52     | 1.18  | 3.56| 5.88| 3.86  | 3.37 | 0.65 |
|       | 2      | 55.16 | 15.46   | 9.59     | 1.89  | 3.33| 5.65| 2.94  | 2.75 | 2.81 |
| G2    | 3      | 52.25 | 12.71   | 16.01    | 2.39  | 3.61| 7.14| 2.68  | 2.04 | 1.69 |
|       | 4      | 48.29 | 13.51   | 14.38    | 4.20  | 4.72| 9.10| 2.25  | 1.01 | 2.86 |
|       | 5      | 48.48 | 12.83   | 14.49    | 4.40  | 4.96| 7.85| 2.00  | 1.34 | 3.44 |
| G3    | 6      | 52.84 | 15.29   | 10.59    | 1.59  | 5.03| 9.04| 2.99  | 0.88 | 1.55 |
|       | 7      | 49.21 | 13.84   | 12.42    | 2.41  | 6.66| 8.95| 3.26  | 1.40 | 1.39 |
|       | 8      | 49.61 | 14.99   | 11.56    | 1.98  | 7.37| 7.93| 3.28  | 1.78 | 0.92 |
|       | 9      | 45.59 | 16.34   | 13.92    | 1.80  | 7.64| 7.87| 3.37  | 0.38 | 3.58 |
|       | 10     | 48.67 | 14.70   | 10.94    | 1.97  | 7.67| 8.00| 2.74  | 1.36 | 3.37 |

2.2 Preparation of Amorphous Basaltic Glass

As shown in Fig. 1, the preparation process of amorphous basaltic glass mainly included melting, pouring, annealing and machining (cutting and polishing). The detailed process was as follows: large
blocks of basaltic rocks were crushed by a crusher and then milled for 5 min. The basaltic powders in crucibles were melted at 1500°C for 2 h. After the melting, the bubble-free melt was poured into the graphite mould. The hot glass samples were then transferred to a preheated electric muffle furnace for annealing at 650 °C. After two hours, the muffle was switched off. The sample was cooled to room temperature in the furnace. At last, the annealed glass was cut into disk with the diameter of 40 mm and thickness of 3 mm, and then the upper and lower surfaces of the disk were polished to be parallel.

2.3 Electrical and Dielectric Properties

The samples were introduced into the test equipment made by Beijing Beiguang fine instrument Co. LTD. The dielectric constant and dielectric loss of the samples were measured at 1 MHz.

3 Results And Discussion

3.1 Melting

No crystals are present after the melting process, this is the primary condition for preparing amorphous basaltic glass. In order to make sure plagioclase, pyroxene and other crystals in basaltic rocks were completely dissolved when basalt was kept at 1500°C for 2 hours. The melt was poured into water for quenching, and the phase of the sample after quenching was equivalent to that of its high temperature melting. The quenched glass block was ground into powder for XRD detection.

The XRD pattern in Fig. 2 shows that there is no obvious crystallization peak in 1～10 samples, but there are obvious peaks of steamed bread in the range of 20°~40°. All the samples have typical amorphous structure and good glass-forming performance. The results show that the 1500°C for 2 h meets the melting requirements.

3.2 Pouring

Basaltic melt has to be poured after the melting, and its melt viscosity is the key factor for pouring. Therefore, the viscosity of basaltic melt was studied in this experiment. The crucible was taken out from the silicon molybdenum furnace, the melt was poured into the graphite mold.

| Whether the viscosity meets the requirements of pouring | G1 | G2 | G3 |
|---------------------------------------------------------|----|----|----|
| 1 2 3 4 5 6 7 8 9 10                                   | ×  | √  | ×  |

As shown in Table 2 (“×” means it cannot be poured, “√” means it cannot be poured), the viscosity of G1 is poor, so the pouring cannot be completed, while the remaining samples can be successfully completed. Compare to G2 and G3, G1 has high content of SiO₂. It causes the melt viscosity to be high at 1500°C. This is consistent with previous studies [19]. In order to further confirm this conclusion, the viscosity of 1
~10 samples was calculated using the model proposed by Daniele Giordano [20]. As shown in Fig. 3, the viscosity of 1 and 2 samples were up to 299 dPa·S; 207 dPa·S, but the viscosity of the other samples were below 100 dPa·S. The data further show that the G1 sample cannot be poured due to its high viscosity at 1500°C.

### 3.3 Annealing and Machining

As shown in Fig. 4, when the basaltic glass without annealing was cut directly, it was extremely easy to crack. This was caused by the large stress in the glass due to the rapid cooling in pouring process. Therefore, annealing should be carried out to eliminate the stress before machining. As shown in Fig. 5, the sample was not cracked after annealing and it meets the machining requirements.

However, the glass-ceramics cannot represent dielectric properties of basalt fiber. So basaltic glass with crystallization caused by annealing cannot meet the test standard of electronic glass. In this experiment, 3–10 groups of samples were annealed. After annealing at 650°C, the glass block was ground into powders. XRD was used to examine whether there was crystal in the annealed glass.

It can be seen from Fig. 6 that magnetite (Fe₃O₄) crystallized from G2 (3, 4 and 5 groups). With the increase of the total content of Fe₂O₃ and TiO₂, the peak of magnetite becomes more sharp, which is consistent with the research results of relevant literature [21]. Compared with the samples of G2 (3 ~ 5 groups), the samples of G3 (6 ~ 10 groups) with lower Fe₂O₃ and TiO₂ did not form Fe₃O₄ crystals. Therefore, the content of Fe₂O₃ and TiO₂ is directly related to the crystallization, so it is necessary to control Fe₂O₃ and TiO₂ in a certain range to prepare amorphous basaltic glass.

### 3.4 Dielectric Properties of Basaltic Glass

The dielectric properties of glass-ceramic cannot truly represent the dielectric properties of amorphous continuous basalt fiber. So G3 (6–10 groups) of non-crystallized basalt glass were cut and polished to test the dielectric properties.

At 1 MHz, the dielectric constant of basalt glass is between 6.55–7.55, and the dielectric loss is between 4.034 × 10⁻³ and 6.493 × 10⁻³. The dielectric property of basalt glass sample is basically the same as that of E glass fiber (dielectric constant is 6.7). The experimental results show that it is feasible to replace electronic grade E glass fiber with basalt fiber, and the natural basalt producing area which have much better dielectric property will be further searched in the future.

### 4 Conclusions

In this paper, amorphous basaltic glass can directly and truly reflect the dielectric properties of continuous basalt fibers. The research shows that the content of SiO₂, Fe₂O₃ and TiO₂ in basaltic rock affects the analysis and test method. High SiO₂ content or high Fe₂O₃ and TiO₂ content of basalt raw materials make it difficult to get amorphous basaltic glass in this experiment. However, the current continuous basalt fiber production does not want the high content of SiO₂, Fe₂O₃ and TiO₂. Therefore,
this method is suitable for the application of continuous basalt fiber. This paper further confirmed that the dielectric properties of continuous basalt fiber can reach the level of electronic grade E glass fiber, and laid a solid foundation for exploring the raw material formula suitable for the production of low dielectric continuous basalt fiber.

**Declarations**

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**Conflict of Interest**

The authors declared that they have no conflicts of interest to this work.

**Author contributions**

Changjiang Liu, Chuncheng Yang and Letao Jiang: Investigation and Research.

Xiaoccong Tong, Zhong Liu and Heyu Huang: Grinding, Pouring and Drawing.

Lei Zhang and Baoming Ding: Rock-collection and Analysis.

Yan Li: Supervision and Methodology.

Hongchao Li: Research, Validation and Writing-review.

**Availability of data and material**

The datasets used or analysed during the current study are available from the corresponding author on reasonable request.

**Compliance with ethical standards**

Yes.

**Consent to participate**
Consent for Publication

Yes.

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