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Microwave insertion loss measurements in phosphate glasses containing transition metal oxides

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Abstract
The scope of this work is to report the results of microwave insertion loss measurements as a function of frequency and composition of some selected phosphate glasses containing transition metal oxide. Borosilicate glasses are commonly used in microwave travelling wave tubes, microwave windows, etc. They present very high microwave transmittance (low microwave insertion loss) in a certain frequency domain, which must be a very important feature for microwave power applications. The works regarding microwave insertion loss studies in glass systems are found to be very scarce at present. The results show that there is a possibility of our glasses being used in windows for high-power microwave tubes, once the microwave signal insertion loss at the frequencies of 8.3 and 11.8 GHz attains values nearly 0 dB. The results show that it is possible to control the 9.7 GHz absorption peak, by increasing the transition metal oxide concentration in the glass, above a certain value. The transmittance technique can also be used for the study of a greatest variety of materials.

Keywords: microwave windows, microwave glasses, insertion loss measurements

1. Introduction

Microwave radars operate with power peak levels beyond some megawatts in most cases. In order to achieve such high RF power, they employ in their final stage, devices such as klystron amplifiers and travelling-wave tubes (TWT). Such devices are complex with respect to its construction, demanding a set of knowledge, such as ultra-high vacuum technology, sealing techniques, study of physical properties of metallic, ceramic and glass materials, construction of the electronic gun and magnetic focusing systems, interaction of the electron beam with the electromagnetic field and microwave measurement techniques.

Since these devices operate in the microwave range, the knowledge and measurements of the electrical properties of the materials employed in the construction of microwave devices become very important. Several microwave techniques and measurement procedures are reported in the literature [1–4], however, few works describing measurements of dielectric properties in glasses at microwave frequencies could be found [5–7]. The development of special glasses of low insertion loss is an important step in the construction of microwave tube windows. In most cases, borosilicate glasses containing some specific elements such as sodium oxide, for example, are used as microwave windows and travelling-wave tubes, since they present very low microwave insertion loss in a given microwave frequency domain. However, these glasses are commercial and their technical references are not easily available. In view of these facts, the need for equivalent glasses of high microwave transmittance and lower cost for a possible alternative glass for microwave windows led to the study of the technique of microwave insertion loss measurements. Results of insertion loss measurements in some phosphate glasses are reported in this work as a function of frequency and composition of these materials. Dielectric properties, as well as other physical properties of these glasses, are the subject of current research in our group.

Microwave transmittance was found to be a powerful and sensitive technique for the study of the greatest variety of materials, as a function of their composition and temperature, and can be used in addition to other spectroscopic techniques, such as optical absorption (OA) and Fourier-transform infrared (FTIR).

This work is organized as follows: in section 2, the glass preparation method and a microwave measurement technique are described. In section 3, the microwave response as a function of frequency for different glass compositions is presented and discussed. Finally, conclusions are presented in section 4.

2. Experimental procedure

The glasses used in this investigation were prepared from the fusion of reagent grade materials $NH_4H_2PO_4$, $BaCO_3$, NaOH, LiOH, CoO and Fe_2O_3. These quantities were weighed and mixed in an alumina crucible. Then, the batch was melted in an electric furnace at 1000 °C for about 2 h in order to achieve homogenization. Then, the melt was poured inside a brass mould and the resulting glass annealed at 490 °C for 1 h in order to eliminate the mechanical stresses resulting from a fast cooling rate. The shape of each glass sample was in the form of a parallelepiped of dimensions 1.0 × 1.5 × 2.2 cm^3, in order to fit inside a conventional WR-90 waveguide. The glasses prepared for this work, which are extensively investigated by our research group, have the following compositions:

- $(1 - x)\cdot(60P_2O_5\cdot 40BaO)\cdot x$CoO, where $x = 0\%$, 4%, 8% and 12% (glass samples A_0–A_3);
- $(1 - x)\cdot(25Na_2O\cdot 25Li_2O\cdot 50P_2O_5)\cdot x$BaO, where $x = 0\%$, 15% (glass samples B_0, B_1) and
- $(1 - x)\cdot(25Na_2O\cdot 25Li_2O\cdot 50P_2O_5)\cdot xFe_2O_3$, where $x = 3\%$, 6% and 9% (glass samples C_0–C_2),

where the parameter x is expressed in mol%.

The microwave measurements were performed by using a microwave sweeper generator mod. HP-3050B and a network scalar analyser mod. HP-8756A. The frequency ranged from 8 to 12 GHz. Also, a microwave sweeper generator with an incorporated network scalar analyser (mod. HP-8757D) was used for the experiments, in the range from 7 to 13 GHz. Both pieces of equipment are equal in terms of operation. The only difference is that in the first case, only one scan was made instead of more than one scan made in the second case, which gives a better resolution in the spectra.

The schematic diagram of the setup can be seen in figure 1, showing the sample placed inside a section of waveguide.

The point B was used to measure the microwave signal insertion loss. The point A can be used to measure the microwave signal return loss, with the aid of a short circuit to calibrate the return loss level, as shown in figure 1. In this experiment, only the microwave signal insertion loss measurement is relevant. Return loss measurements were found to be less than −2 dB attenuation for all glasses investigated in this work, in such a way that we found it unnecessary to show the results. The calibration of the signal was made as follows: first, with the empty waveguide, one measures the transmitted spectra, in order to see the air effects. The amplitude of this signal was found to be less than −2 dB at all frequencies. Then, one sets this signal as a baseline, i.e. to 0 dB of attenuation. The signal decreases only in the presence of the sample. The pos Z blank was used to erase the trace in each sweep.

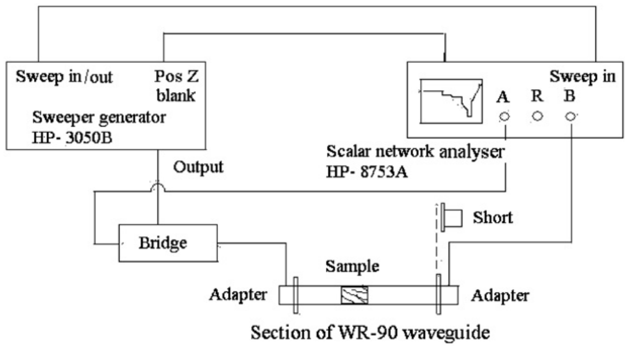

Figure 1. Setup used in the microwave response signal measurements. The glass sample is placed inside a section of WR-90 waveguide.

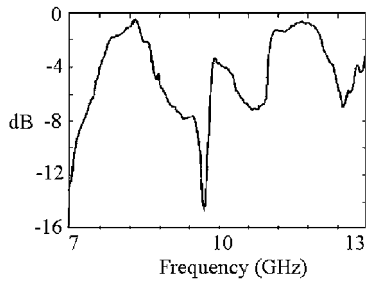

Figure 2. The microwave response as a function of frequency for glass sample A_0 (0% CoO).

3. Results and discussions

The microwave response as a function of frequency, measured in the range from 7 to 13 GHz (HP-8757D), for the glass sample A_0 (0% CoO) can be seen in figure 2.

The microwave response obtained as a function of frequency shows a strong attenuation peak of −14 dB at 9.6 GHz, probably due to the presence of barium in the glass. At frequencies near 8.3 and 11.8 GHz, the attenuation is near 0 dB. For the glass sample A_1 (4% CoO), the microwave response as a function of frequency can be seen in figure 3.

The microwave response for sample A_1 also presents an intense attenuation peak at 9.7 GHz, greater than −16 dB, and an attenuation peak of −12 dB, near 12.6 GHz. Also, the signal presents very low attenuation at 8.3 and 11.8 GHz. Figure 4 shows the spectra of the microwave response for the glass sample A_2 (8% CoO).

The attenuation peak observed in the glass sample A_2 has an approximate value of −8 dB, at the frequency of 9.7 GHz, which is smaller than in the case of samples A_0 and A_1. Figure 5 shows the microwave response for the glass sample A_3 (12% CoO).

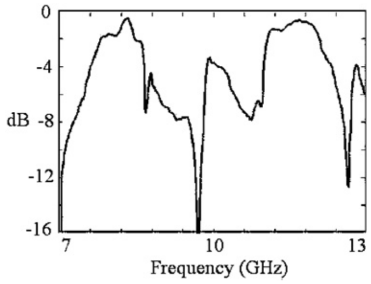

Figure 3. The microwave response as a function of frequency for glass sample A_1 (4% CoO).

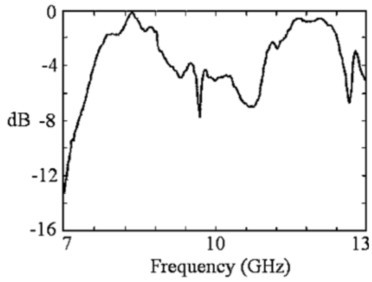

Figure 4. The microwave response as a function of frequency for glass sample A_2 (8% CoO).

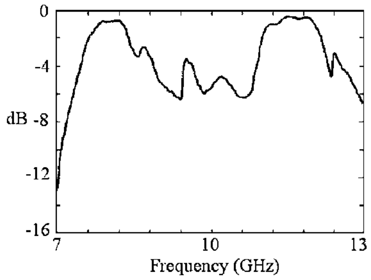

Figure 5. The microwave response as a function of frequency for glass sample A_3 (12% CoO).

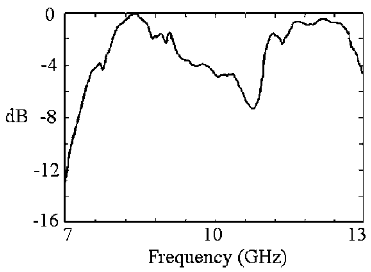

Figure 6. The microwave response as a function of frequency for glass sample B_0 (0% BaO).

In the case of sample A_3, the attenuation peak, at 9.7 GHz, is less than −6 dB. This means that for concentrations of cobalt beyond 8%, the maximum attenuation of the microwave signal begins to decrease. This behaviour also occurs for glasses containing barium and iron, as seen further. Figure 6 shows the microwave response for a glass sample (B_0—0% BaO) with high return loss in relation to the glasses A_0, A_1 and A_2.

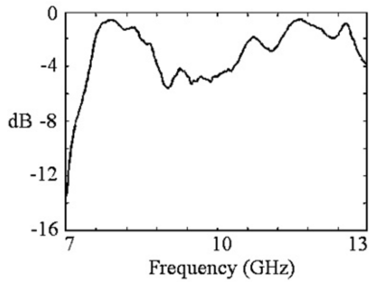

Figure 7. The microwave response as a function of frequency for glass sample B_3 (15% BaO).

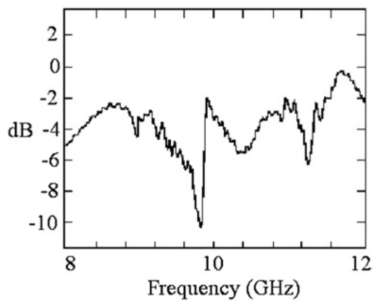

Figure 8. The microwave response as a function of frequency for glass sample C_0 (3% Fe_2O_3).

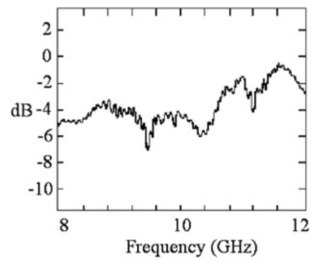

Figure 9. The microwave response as a function of frequency for glass sample C_1 (6% Fe_2O_3).

Figure 6 shows that for frequencies near 8.3 GHz and 11.8 GHz, the signal presents attenuation near 0 dB. The microwave response is very similar to that of glass sample A_3. Figure 7 shows the microwave response for another glass sample containing barium (B_1—15% BaO), which presents lower insertion loss in relation to the samples containing less than 8% cobalt.

In figures 6 and 7, the presence of sodium and lithium in the glass does not cause attenuation peaks in the microwave signal. Figure 8 shows the results of the microwave response for the glass sample C_0 (3% Fe_2O_3) and the measurements were performed from 8 to 12 GHz, by using a microwave sweeper generator HP-3050B and a network scalar analyser HP-8756A.

The microwave response of figure 8 shows an intense attenuation peak of about −10 dB, at a frequency near 9.7 GHz and another attenuation peak of about −6 dB at the frequency 11.2 GHz. Figure 9 shows the microwave response for the glass sample C_1 (6% Fe_2O_3).

In figure 9, the microwave response shows a small attenuation peak of about −7 dB, at the frequency 9.5 GHz.

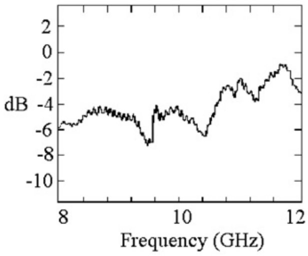

Figure 10. The microwave response as a function of frequency for glass sample C_2 (9% Fe_2O_3).

Finally, figure 10 shows the microwave response for the glass sample C_2 (9% Fe_2O_3).

Figure 10 shows that the signal presents attenuations less than −8 dB, in relation to the glass samples C_0 and C_1.

The results also show that in all cases, there are regions in which the microwave signal presents very low attenuation, mainly, near 8.3 GHz and 11.8 GHz. Also, below 8 GHz, the microwave signal is highly attenuated, probably due to some unknown resonance, and near 10.5 GHz, for all glass samples, there is an attenuation of about −7 dB. In the spectra of air (empty waveguide), the absorption at 8 GHz is not observed; the absorption is less than −2 dB in all measured frequency regions.

The glasses A_3 (12% CoO), B_0 (0% BaO), B_1 (15% BaO), C_1 (6% Fe_2O_3) and C_2 (9% Fe_2O_3) present lower insertion losses than the other glasses investigated in this work. Therefore, these glasses are more suitable for microwave window development, in which the insertion losses must be as low as possible in a given frequency region, in the present case, in the X-band.

The physical reasons for the intense attenuations observed at some specific frequencies, are not very well known in the present work, which demands further studies and measurements in other glass systems in order to try to elucidate these effects. These attenuations can be caused by rotational, stretching, bending forces or some other possible vibrational mode of specific structural groups in the glass, which responds in the measured frequency interval. The nature of these attenuations can probably be similar to that observed in Fourier transform infra-red (FTIR) and OA spectroscopy.

Finally, the differences in quality in the spectra in figures 2–7 and 8–10 are due to the number of scans made in both measurements. In figures 2–7, more than one scan was made, whereas in figures 8–10, only one scan was made.

4. Conclusions

The glasses A_3 (12% CoO), B_0 (0% BaO), B_1 (15% BaO), C_1 (6% Fe_2O_3) and C_2 (9% Fe_2O_3) present lower microwave insertion loss in relation to other glasses, meaning that they are more suitable for use in microwave windows, like the borosilicate glasses used in microwave windows. There are some regions in which the signal presents relatively lower attenuation, and for concentrations of cobalt, barium and iron greater than a certain value, the attenuation peaks seem to vanish, contributing to the decrease in the insertion loss. The origin of these intense absorption peaks is not very well known at present, demanding further studies in order to try to elucidate the physical behaviour of these effects. Probably, the observed absorptions can be related to rotational, vibrational, stretching or bending forces, as occur in infrared spectroscopy and optical absorption.

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