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Determination of microwave electrical characteristics of boron nitride at high temperature

Thoria A. Baeraky[*]

Faculty of Science, Physics Department, King Abdulaziz University, Jeddah, Saudi Arabia

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The microwave electrical characteristics of a powder Boron Nitride, BN, are determined at high temperature in term of the complex permittivity. The real and the imaginary parts of the complex permittivity are measured using the shift on frequency of the full computerized cavity perturbation technique working in microwave frequency ranges, 615 MHz, 1412 MHz, 2214 MHz, and 3017 MHz, and at temperature from 25 °C to 2000 °C in step of 50 °C. The temperature and frequency-dependent electrical conductivity, σ, of BN is calculated using the complex permittivity imaginary part, . ε'' measured in the microwave frequency and the temperature ranges of the above technique. The frequency exponent n, and the activation energy, E_A, is determined using the calculated electrical conductivity, σ, in the same microwave frequency and the temperature ranges.

1 Introduction

Advanced ceramics, carbides, pure oxides, and nitrides offer many advantages compared to other materials. Ceramics display a wide range of properties which facilitate their use in many different product areas. They are harder and stiffer than steel; more heat and corrosion resistant than metals or polymers; less dense than most metals and their alloys [1]. Microwave processing of ceramics has advantages in reducing the time and temperature of processing as well as improving the homogeneity of heating. Microwave energy interact with the material at the molecular level, it rises inside the material itself, and depends on the dielectric properties of the material and on the incident microwave frequency. Microwave measurements the dielectric properties of materials at different frequencies and different temperature can be helpful in understanding the microwave mechanism heating [2].

Boron nitride is a unique material. It offers outstanding thermal conductivity, excellent dielectric strength, very good thermal shock resistance and is easily Machinable. This material is an advanced synthetic ceramic available in powder, solid, liquid and aerosol spray forms. In an oxidizing atmosphere it can be used up to 900 °C. However, in an inert atmosphere some grades can be used as high as 3000 °C. Grades are available with a very low porosity and ultra high strength for use in semiconductor processing applications. The technological importance of boron nitride is evident from the increasing use of this material in tribological and electronic device applications. [3–5]. In this work the dielectric properties of BN in microwave frequency range and high temperature will be measured by using the technique that is based on cavity perturbation theory.

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[*] Corresponding author: e-mail: Tbaerky@kaau.edu.sa, Phone: +966 269 300 67, Fax: +9662214496

2 Experimental

Cavity perturbation technique was used to measure the complex permittivity of *BN* in microwave frequencies, 615 MHz, 1412 MHz, 2214 MHz, 3017 MHz, and 3820 MHz and at temperature from 25 °C to 2000 °C making measurements every 50 °C. The details of this technique have been reported in [6] and will not be discussed further. Measurements are performed by measuring the resonant frequencies of the cavity with and without the sample, f and f_o respectively, and the quality factors of the cavity with and without the sample, Q and Q_o, respectively. The simple perturbation formula derived by Nakamura and Furuichi [7] is based on the shift of frequency, Δf, and on the shift of the reciprocal quality factor, $\Delta(1/Q)$. The real, ε,' and the imaginary, ε', parts of the complex permittivity can be calculated by using Eqs. (1), (2).

$$\varepsilon' = 2 j^2 (\chi_{on}) \frac{a^2}{b^2} \frac{\Delta f}{f} + 1 \tag{1}$$

$$\varepsilon'' = j^2 (\chi_{on}) \frac{a^2}{b^2} \Delta\left(\frac{1}{Q}\right) \tag{2}$$

where χ_{on} is the root of the zero order Bessel function, j, of the first kind. a and b are the sample and the cavity volumes, respectively. The electrical conductivity, σ, the activation energy, E_A, and the exponent frequency, *n*, can be calculated by substituting the measured imaginary part, ε'', of the complex permittivity in Eq. (3) and using the slope of Eqs. (4), (5) respectively [8].

$$\sigma(\omega) = \varepsilon_o \omega \varepsilon''(\omega) \tag{3}$$

$$\sigma = A \exp\left(-E_A / k T\right) \tag{4}$$

$$\sigma(\omega) = A \omega^n \tag{5}$$

where k is Boltzmann constant and T is the absolute temperature (in K).

3 Results and discussion

White powder Boron Nitride of density 2.25 $g\ cm^{-3}$ was selected to study its electrical characteristics in microwave frequency range and at high temperature. Holder sample is a special silica tube, stand to temperature up to 2000 °C. The tube of 5 mm in diameter was filled by BN to hold it during the measurements between the cavity and the furnace. Figure 1 shows the variation of the two parts of the complex permittivity ε' and ε'', with the variation of temperature. Changing of ε' and ε'' with the changing in temperature appeared when temperature reached 1000 °C. The real and the imaginary parts, ε' and ε'', respectively, varied from 1.5 to 2.5 and from zero to 0.05 in temperature range from 1000 to 1800 °C at the frequencies 615 MHz and 1412 MHz. The variation of ε' and ε'' with frequency at the (2214 MHz, 3017 MHz, and 3820 MHz) is small as shown in Fig. 1. The change in the electrical conductivity with increasing temperature is shown in Fig. 2. Figure 3 shows the unknown results for the activation energy that all the points of measurements are not increasing in regular manner. The temperature dependence of the frequency exponent, *n*, in Eq. (5) illustrated in Fig. 4. The results show that *n* varied at low temperature but at high temperature the veration is very small.

crowave frequencies, 2214 MHz, 3017 MHz, and 3820 MHz. At frequencies 615 MHz and 1412 MHz, *BN* absorbs microwave energy and its electrical resistivity decreases with increasing temperature.

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Microwave Measurements of the Dielectric Properties of Zinc Oxide at High Temperature

Thoria A. Baeraky,

Faculty of Science, Physics Department, King Abdulaziz University, Jeddah, Saudi Arabia.

*The dielectric properties of zinc oxide material are measured at high temperature and in microwave frequency ranges. The real and the imaginary parts of the complex permittivity are calculated using the measured shift on frequency of the full computerized cavity perturbation technique working in microwave frequency ranges, 615 MHz, 1412 MHz, 2214 MHz, 3017 MHz, and 3820 MHz at temperature from 25 °C to 2000 °C in step of 50 °C. The temperature and frequency-dependent electrical conductivity, σ, of zinc oxide is calculated using the complex permittivity imaginary part, ε'', measured in the microwave frequency and the temperature ranges of the above technique. The frequency exponent **n**, and the activation energy, E_A, are determined using the calculated electrical conductivity, σ, in the same microwave frequency and the same temperature ranges.*

1. Introduction:

Ceramics are inorganic nonmetallic materials which consist of metallic/nonmetallic compounds that are bonded primarily by ionic bonds, sometimes with covalent character. Microwave dielectric ceramics, which have high permittivity, are used as materials of microwave components such as resonator, band pass filter and duplexer. These materials received attention due to the rapid progress in microwave telecommunications and satellite broadcasting etc. [1,2]. Microwave processing of ceramics has advantages in reducing the time and temperature of processing as well as improving the homogeneity of heating. Microwave energy interact with the material at the molecular level, it rises inside the material itself, and depends on the dielectric properties of the material and on the incident microwave frequency. Microwave measurements for the dielectric properties of materials at different frequencies and different temperature can be helpful in understanding the microwave mechanism of heating [3].

Zinc oxide (ZnO) is of great interest as a suitable material for high temperature, high power electronic devices either as the active material or as a suitable substrate for epitaxial growth of group III-nitride compounds With its large, direct band gap (3.4 eV at 300 K) and high excitonic binding energy (60 meV). With appropriate dopants such as aluminum and galum, it is both transparent in the visible region and electrically conductive.[4,5]. In this work the dielectric properties of ZnO in microwave frequency range and high temperature will be measured by using the technique that is based on cavity perturbation theory.

2. Experimental:

Cavity perturbation technique was used to measure the complex permittivity of ZnO in microwave frequencies, 615 MHz, 1412 MHz, 2214 MHz, 3017 MHz, and 3820 MHz and at temperature from 25 *°C* to 2000 *°C*. The details of this technique have been reported in [6] and will not be discussed further. Measurements are performed by measuring the resonant frequencies of the cavity with and without the sample, ***f*** and ***fo***, respectively, and the quality factors of the cavity with and without the sample, ***Q*** and ***Qo***, respectively. The simple perturbation formula derived by Nakamura and Furuichi [7] is based on the shift of frequency, Δf, and on the shift of the reciprocal quality factor, $\Delta\ (1/Q)$. The real, ε', and the imaginary, ε'', parts of the complex permittivity can be calculated by using Eqs. (1), (2).

$$\varepsilon' = 2\, j^2 (\chi_{on}) \frac{a^2}{b^2} \frac{\Delta f}{f} + 1 \tag{1}$$

$$\varepsilon'' = j^2 (\chi_{on}) \frac{a^2}{b^2} \Delta\left(\frac{1}{Q}\right) \tag{2}$$

Where χ_{on} is the root of the zero order Bessel function, ***j***, of the first kind. ***a*** and ***b*** are the sample and the cavity volumes respectively. The electrical conductivity σ, the activation energy, E_A, and the exponent frequency, ***n***, can be calculated by substituting the measured imaginary part, ε'', of the complex permittivity in Eq. (3) and using the slope of Eq. (4, 5) respectively [8].

$$\sigma(\omega) = \varepsilon_o \omega\, \varepsilon''(\omega) \tag{3}$$

$$\sigma = A \exp(-E_A / kT) \tag{4}$$

$$\sigma(\omega) = A \omega^n \tag{5}$$

Where k is Boltzmann's constant and T is the absolute temperature (K).

3. Results and Discussion :

White powder Zinc Oxide of density 1.1 $g\ cm^{-3}$ was selected to study its electrical characteristics in microwave frequency range and at high temperature. Holder sample is a special silica tube, stand to temperature up to 2000 °C. The tube of 3 mm in diameter was filled by ZnO to hold it during the measurements between the cavity and the furnace. The variation of the complex permittivity real part with the changing of frequency and temperature is shown in Fig. (1).

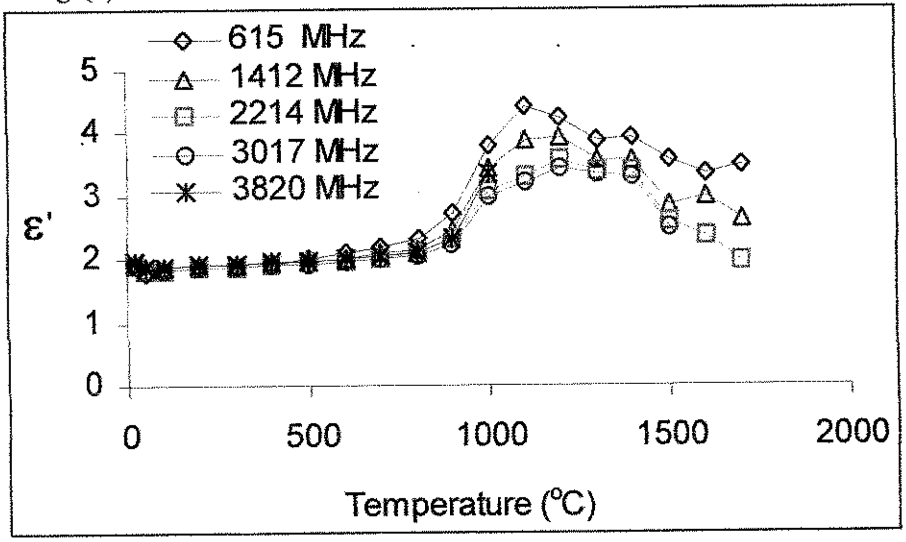

Fig. (1): The complex permittivity real part

The variation of the complex permittivity real part, ε', with the changing in frequency and temperature appeared when the temperature reached 600 °C. The complex permittivity real part, ε', varied from 1.9 to 2.1 values in temperature range from 25 to 600 °C at all the frequency ranges, 615 MHz, 1412 MHz, 2214 MHz, 3017 MHz and 3820 MHz.. However, at 615 MHz, ε' increased to the

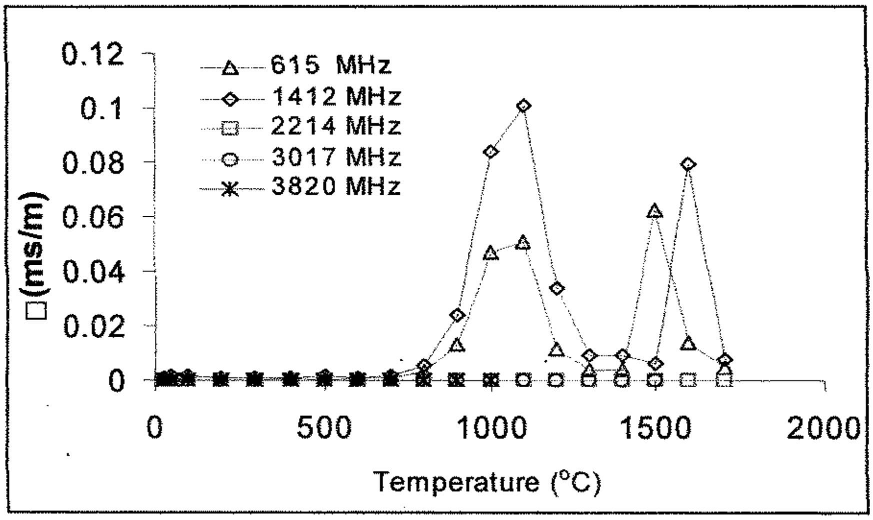

Fig. (3): The electrical conductivity of ZnO

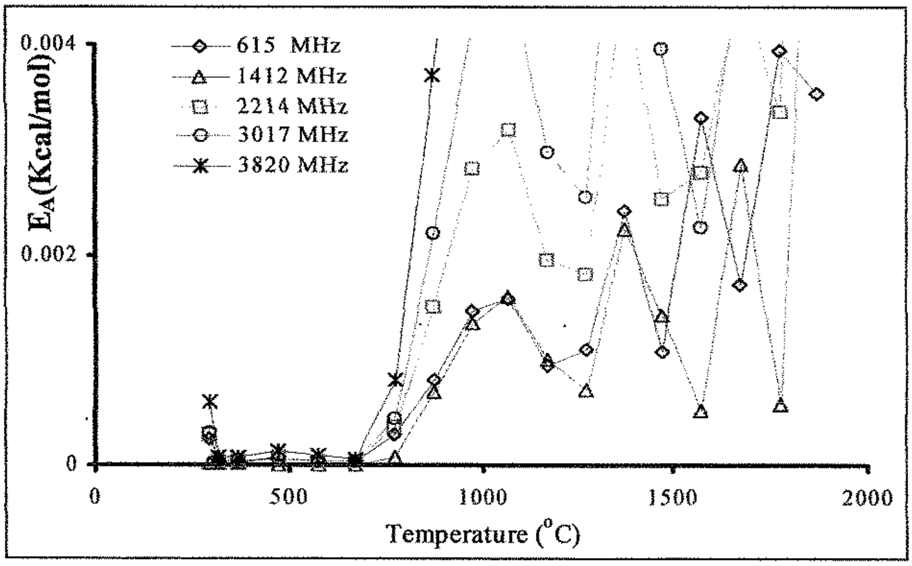

Fig. (4): The activation energy of ZnO

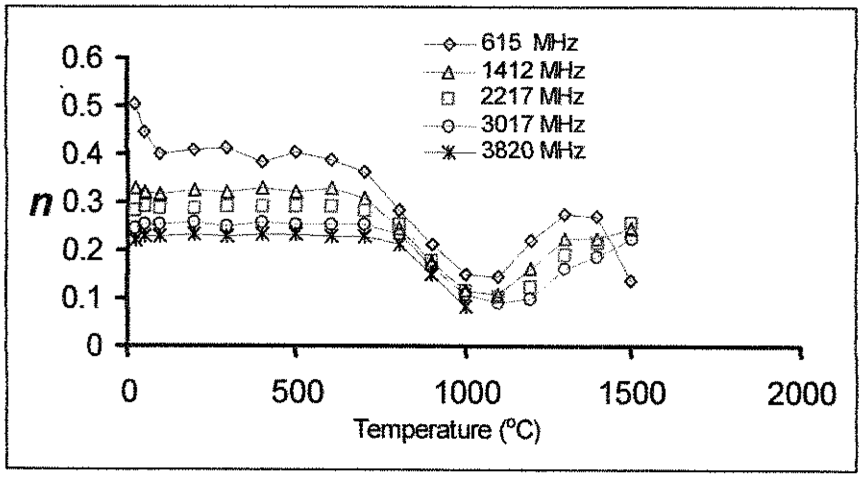

Fig. (5): The temperature dependence of the frequency exponent, *n*, of ZnO

4. Conclusion:

The complex permittivity measurements of ZnO in microwave frequency ranges, *615 MHz, 1412 MHz, 2214 MHz, 3017 MHz, and 3820* MHz, and high temperature up to 2000 °C, show that the microwave transparent and the electrical resistivity of this material are very high such that they have a relatively small change during the measurements when the temperature reached 500 °C. At the temperature 1100 °C and 1500 °C, ZnO seams to be microwave absorpting material.

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Microwaves Absorption in Lead Molybdenum Phosphate Glasses at Low Temperature

Thoria A. Baeraky

Faculty of Science. Physics Department. King Abdulaziz University.
Jeddah. Saudi Arabia

Seven samples of lead molybdenum phosphate glasses are prepared due to mol % composition $PbD_x\ MoO_{3\,(80-x)}\ P_2O_{3(20)}$ *over the compositional ranges from* $x = 0$ *to 60. The microwave behavior of this kind of glasses is investigated in term of the microwave dielectric properties measurements at low temperature due to lead addition. The full computerized cavity perturbation technique working in microwave frequencies 615 MHz, 1412 MHz, 2214 MHz, 3017 MHz, and 3820 MHz are used for these measurements. The real* ε' *and imaginary* ε'' *parts of the complex permittivity are measured at room temperature and the frequency-dependent electrical conductivity* σ *is calculated. The frequency exponent* n *and the activation energy* E_A *are determined. Sample containing molybdenum and phosphate showed microwave absorption rather than the other samples with lead addition.*

1. Introduction:

Advanced specialty glasses play important roles in several industries. In the last several years, these materials have continued to find new applications in the areas of telecommunications, electronics, and biomedical uses. Phosphate glasses offer several advantages over silicate and borate glasses. Phosphate glasses are important materials in technologically comparing with the conventional oxide glasses. They possess some superior physical properties such as high thermal expansion coefficients, low melting temperature, low softening temperature, low transition temperature and high electrical conductivity [1&2]. Lead phosphate glasses with metal oxide materials such as iron, molybdenum and zinc are very attractive for the

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E-mail: Tbaerky@kaau.edu.sa

structural study from both the academic and technological points of view [3-5]. Applications of lead phosphate glasses, containing metal oxide materials, in industries such as optical, electronics, and radiation shielding are immense due to their good physical and electrical characteristics [6–8].

Microwaves can be reflected, absorbed and/or transmitted by materials. Reflection and absorption require interaction of microwaves with material; transmission is the result of partial reflection and incomplete absorption. Materials reflect and absorb microwave to various degrees depending on their composition, structure, temperature, and frequency of the microwaves [9]. Complex permittivity is the parameter which characterises the electrical properties of a material and hence describes the interaction between the microwave radiation and a material. The real part ε' describing the energy stored within the material whilst the imaginary part ε'' describes the energy dissipated [10]. Observing the microwave absorption on the prepared molybdenum phosphate glasses containing different amounts of lead and molybdenum in terms of complex permittivity is the aim of this work. Cavity perturbation technique is used in this work to measure the complex permittivity ε' & ε'' of the glass material in microwave frequency ranges 615 MHz, 1412 MHz, 2214 MHz, 3017 MHz, and 3820 MHz. This technique has higher measuring precision and it does not need a special requirement for the geometry and size of the measured sample [11].

2. Experimental:

Seven samples of glasses were prepared by melting dry mixtures of $x\text{PbO}-(80-x)\text{MoO}_3-20\text{P}_2\text{O}_5$ where $x = 0 - 60$ mol% PbO in ceramic crucibles for three hours at 1300 °C. The glass samples were formed by pouring the melt on a brass plate held at room temperature. The density of the glass was measured by the Archimedes method using distilled water as the immersion liquid and the results are shown in Table (1) for different compositions.

Table (1): The seven samples compositional and densities

Samples no.	Composition (mole%)			Density
	PbO	MoO_3	P_2O_3	(g cm^{-3})
Sample 1	00	80	20	2.230
Sample 2	10	70	20	2.901
Sample 3	20	60	20	3.674
Sample 4	30	50	20	4.232
Sample 5	40	40	20	4.539
Sample 6	50	30	20	5.405
Sample 7	60	20	20	5.418

Cavity perturbation technique was used to measure the complex permittivity of the seven samples of glasses in microwave frequencies 615 MHz, 1412 MHz, 2214 MHz, 3017 MHz, and 3820 MHz. The details of this technique have been reported in [12] and will not be discussed further. Measurements were performed by measuring the resonant frequencies of the cavity with and without the sample f and f_o, respectively, and the quality factors of the cavity with and without the sample Q and Q_o, respectively. The complex perturbation equations based on the shift of frequency Δf and on the shift of the reciprocal quality factor $\Delta(1/Q)$ are given below [13]:

$$\varepsilon' - 1 = 2J_1^2(\chi_{0n})\frac{\Delta f}{f_o}R_0^2 / R_s^2 \qquad (1)$$

$$\varepsilon'' = J_1^2(\chi_{0n})\Delta(1/Q_L)R_o^2 / R_s^2 \qquad (2)$$

where χ_{on} is the root of the zero order Bessel function J, of the first kind. R_s and R_o are the sample and the cavity radii respectively.

The real ε' and imaginary ε'' parts of the complex permittivity were calculated by substituting the shift in frequency Δf into eqs. (1) and (2).

The electrical conductivity σ, the activation energy E_A, and the exponent frequency n, were calculated by substituting previously measured imaginary part ε'',

$$\sigma(\omega) = \varepsilon_o \omega \varepsilon''(\omega) \qquad (3)$$

$$\sigma = A \exp(-E_A / kT) \qquad (4)$$

$$\sigma(\omega) = A\omega^n \qquad (5)$$

Where k is Boltzman's constant and T is the absolute temperature (K).

3. Results and Discussion:

The compositions of the seven samples of powder lead molybdenum phosphate glasses with their corresponding densities are shown in Table 1. The density shown in this table increases with the increase in lead.

The variation of ε' & ε'' with different frequency ranges for the seven samples are shown in Figs. (1 & 2). Figure (1) shows that there is no variation on the values of the real part ε' with the variation of frequency. These values increase slightly with the increase decrease in the amount of lead and molybdenum, respectively. The variation of the permittivity imaginary part ε'' with frequency shown in Fig. 2 indicate that there is microwave absorption on sample one, doesn't contain lead, that it decreases with increasing frequency from 615 MHz to 1412 MHz, seems to be constant for the other frequencies ranges 2214 MHz, 3017 MHz, and 3820 MHz . Microwave absorption on the other samples, containing lead, is very small that their values are almost the same at a value less than 0.1.

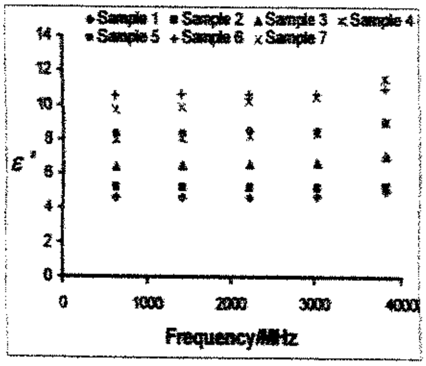

Fig. (1): The permittivity real part vs. Frequency.

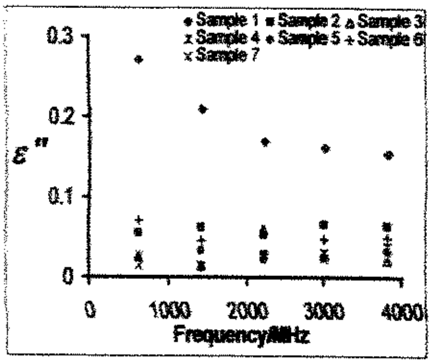

Fig. (2): The permittivity imaginary part vs. Frequency.

The microwave dependence of the electrical conductivity σ for the seven samples with different microwave frequency ranges are shown in 3. In this figure the microwave absorption is observed on the electrical conductivity σ for sample one. It increases with increasing frequency, this is in agreement with Eqn. (3). While the increasing of the electrical conductivities σ of the other samples, containing lead, is noticed to be very small with the increasing of frequency.

The relationship between the activation energy E_A and the frequency exponent n, for different microwave frequency ranges are shown in Fig. (4&5). These two parameters are found to decrease by increasing frequency. The results shown in Fig. 4 and 5 indicate that the values of the activation energy E_A and the frequency exponent n of sample one doesn't contain lead are very small comparing with those for the other samples containing lead. This means that the increase in the values of E_A and n could be attributed to the increase in the amount of lead.

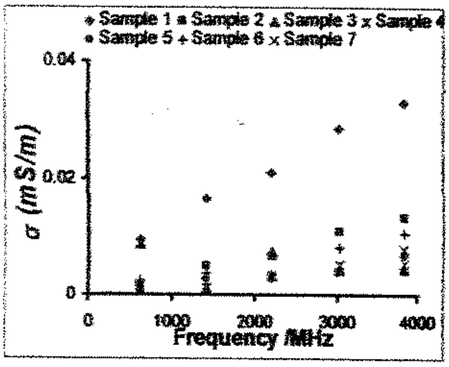

Fig. (3): The electrical conductivity vs. Frequencies.

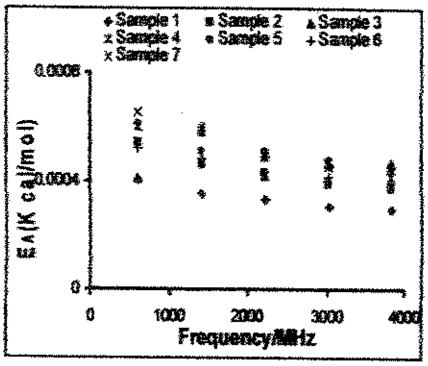

Fig. (4): The activation energ vs. Frequency.

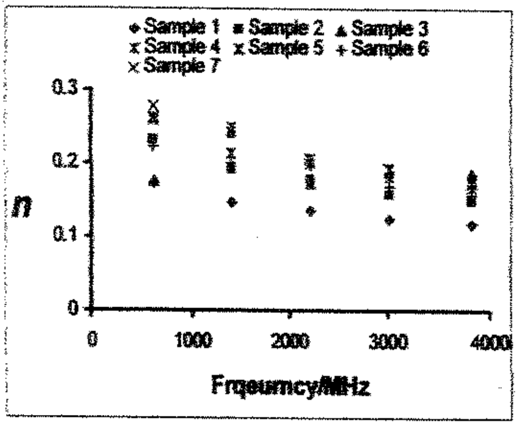

Fig. (5): The frequency exponent *n* vs. frequency.

4. Conclusion:

The results of the dielectric properties of the lead molybdenum phosphate glasses indicated that no microwave absorption on this kind of glass was observed with more addition of lead. This means that glass contains lead has a very small tendency to microwave absorption

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