Microwave Preparation of SiO₂-B₂O₃-Na₂O-K₂O-CaO-Fe₂O₃-TiO₂ Glass System

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Abstract: This study reports preparation of glass composition (54.50 wt.%) SiO₂, (10.80 wt.%) B₂O₃, (14.20 wt.%) Na₂O, (1.20 wt.%) K₂O, (6.00 wt.%) CaO, (4.00 wt.%) Fe₂O₃ and (9.30 wt.%) TiO₂ by melt quenching method using direct microwave heating and conventional resistive heating. Study of dielectric loss factor of the glass as function of temperature illustrated increasing loss factor above 370 °C, 550 °C, 650 °C and 900 °C, indicating enhanced microwave absorption by the glass at above these temperatures. Chemical analysis results of both the glasses depicted more volatilization loss of volatile ingredients in conventional heating. The study of chemical durability was performed from leachate analysis describing less leaching of Na₂O, K₂O and other constituents from glass melted in microwave furnace. Glass transition temperatures (T_g) were found to be 576.3 °C and 569.5 °C for glass melted in conventional and microwave heating route, respectively. Laboratory experiment of glass melting utilizing microwave energy as an alternate heating source demonstrated 70%-75% electrical power saving.

Key words: Microwave heating, energy efficient glass melting, dielectric constant, loss factor, chemical durability.

1. Introduction

Microwave heating has found its application in material processing mainly because of rapid volumetric heating, flexibility in furnace operation (i.e., switching off at maximum temperature) and comfortable working environment owing to only sample heating keeping the cavity cold, substantial reduction in energy consumption, improved mechanical properties and many others [1, 2]. The difficulty of glass melting using microwave energy is mainly due to low dielectric loss factor of major raw material at room temperature making the material as microwave transparent material. However, the loss factor of glass increases with temperature enabling microwave coupling with materials above “critical temperature” and thereby generating heat within it [3].

Hence, glass batches are heated conventionally above ~ 500 °C before direct microwave heating for melting [4]. Feasibility study of glass melting by microwave heating has been reported in different glass systems such as, niobium phosphate [5], alumino-phosphate [6], molybdo-phosphate and tungsto-phosphate [7], iron phosphate [8] and inorganic glasses [9] etc.. However, most of researches were carried out in kitchen microwave oven in which proper control of process parameter could not be done.

Present study reports preparation of glass composition (54.47 wt.%) SiO₂, (10.81 wt.%) B₂O₃, (14.16 wt.%) Na₂O, (1.19 wt.%) K₂O, (6.02 wt.%) CaO, (4.05 wt.%) Fe₂O₃ and (9.30 wt.%) TiO₂ using microwave heating. Some of the physical and optical properties were measured and compared with the glass melted from identical batch in resistive heating. The study of power consumption in microwave heating and in conventional resistive heating were presented and discussed.

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2. Experiments

2.1 Conventional Melting of Glass

The glass was prepared using high purity raw materials like quartz, H_3BO_3 , Na_2CO_3 , K_2CO_3 , CaCO_3 , Fe_2O_3 and TiO_2 . Properly mixed raw materials for 60 g glass was uniaxially pressed to form pellet in a hydraulically operated pellet press and the pellet was placed in a 50 mL alumina crucible which was subjected to heating in electrical resistive furnace (raising hearth furnace from Bysakh & Co., India; heating zone: 230 m $\times$ 230 m $\times$ 300 m). B-type thermocouple was fixed with the furnace to monitor the temperature. Power consumption, temperature and time were recorded to generate T-t-P (temperature-time-power) profile in resistive heating. Melting was carried out at 1,300 °C for 2 h with a heat up rate 4 °C/min and manual stirring was adopted twice with 30 min interval using silica rod to yield homogeneous glass prior to pouring into a preheated steel mold. The hot glass block was annealed in a muffle furnace at 560 °C, below glass transition temperatures T_g , for 2 h followed by controlled cooling to room temperature. The annealed glass was termed as SBNRH (glass prepared in resistive heating).

2.2 Microwave Melting of Glass

Identical pellet of glass batch for 60 g glass was placed inside an alumina crucible insulated by microwave transparent cage insulating box and placed inside a 3 kW, 2.45 GHz, multimode microwave furnace (from M/S Enerzi Microwave Systems Pvt. Ltd., India, microwave cavity size 367 mm $\times$ 349 mm $\times$ 386 mm). An opening of 20 mm diameter was made at top cover of insulation box to allow temperature monitoring through a non contact IR (Infrared) pyrometer (260-1,800 °C). The temperature measurement accuracy and repeatability was $\pm$ 0.3% of the measured value $\pm$ 1 °C with adjustable emissivity (ϵ : 0.1-1.0). MW power (Microwave output power) and

temperature were recorded by DAQSOFT software in a separate computer. A separate energy meter (kW-h meter) was also connected to the input of the electrical circuit to monitor mainly consumption of instant electrical power and total power consumption for overall system including utilities. An integrated closed loop water cooling system was used to cool the microwave cavity, magnetron housing (model: 2M265-M12WJ-Panasonic) and others. The melting of the studied glass was carried out in air atmosphere. Fumes, hot gas generated during melting of batch was exhausted out intermittently by a diaphragm pump. Glass melting was carried out at 1,350 °C for 1 h. Molten glass was cast into a preheated mold and annealed as explained earlier section. The annealed glass was termed as SBNM (glass melted in microwave furnace).

2.3 Characterization

XRD (X-ray diffraction) analysis was performed on 94 X'Pert, PANalytical using Ni-filtered $\text{CuK}\alpha$ radiation with wavelength of 1.5406 Å to confirm the sample amorphous characteristic. The scanning was carried out in the range of 10°-85° with a step size of 0.05 °/min.

ICP-AES (inductively coupled plasma atomic emission spectroscopy) (model: Spectro Ciros Vision, Germany) was used for chemical analysis of Fe_2O_3 , TiO_2 , Na_2O and K_2O ; volumetric titrimetry for SiO_2 and B_2O_3 and compleximetry for Al_2O_3 and CaO . Chemical durability was evaluated from leachate analysis by ICP-AES. The bulk glasses were pulverized and sheaved (within mesh No. 40-60). Then, the glasses were washed in acetone and dried at 110 °C for 2 h. The washed and dried glasses were cooled down inside a desiccator. Each sample of weight 5 g was taken into a Teflon beaker with 100 mL deionised water and kept at 90 °C for 14 days. During the treatment in the above mentioned condition, some of the constituents of the glasses were leached out into the water and these were measured by ICP-AES

(model: Spectro Ciros Vision, Germany). The relative standard deviation of three successive measurements for all the constituents was in the range from 0.59%-0.74%.

Dielectric loss factor and dielectric constant were measured at 2.45 GHz as a function of temperature (up to 1,000 °C). A cylindrical cavity, a vector network analyzer, a microwave amplifier, an infrared pyrometer and a computer were employed to carry out the measurement. The cylindrical rod shape sample was placed inside a quartz tube containers and microwave are applied for heating and measuring simultaneously inside a microwave cavity [10]. Typical uncertainty of these measurements is around 2% in real part and 5% in imaginary part. For high-temperatures (around 1,000 °C), the uncertainty can increase to 10% because the dimensions of the sample change when the materials are melting. The dimension of the sample is a variable used in the dielectric calculations.

The T_g was identified by DSC (differential scanning calorimeter) (NETZSCH STA 449F3) at a heating rate of 10 K/min in flowing nitrogen with an accuracy ± 1 °C. The refractive indices of glass samples prepared by both methods were measured at five different wavelengths (473 nm, 532 nm, 633 nm, 1,064 nm and 1,552 nm) on Prism Coupler (Metricon Model-2010, NJ, USA) fitted with five different lasers as illuminating sources. Three measurements were recorded for each sample and the standard deviation was reported as error of measurement (± 0.0004).

FTIR (Fourier transform infrared spectroscopy) measurements of both the glasses were performed on a FTIR spectrophotometer (model: 1615, Perkin Elmer Corporation, USA) with a spectral resolution of 4 cm⁻¹ over a spectral range of 400-2,000 cm⁻¹ to see the structural changes, if any.

Longitudinal velocity (V_L) and shear velocity (V_T) were measured by the ultrasonic pulse-echo method, using Ultrasonic Flaw Detector (model No. EPOCH 1000, Blue Star). An ultrasonic pulse of 20 MHz

frequency employed through longitudinal and shear transducers. The Poisson's ratio (ν), Young's (E), shear (G) and bulk (K) modulus were calculated applying the measured bulk density (ρ), V_L , and V_T in Eqs. (1)-(4) [11, 12].

$$\nu = 0.5 \frac{(V_L^2 - 2V_T^2)}{(V_L^2 - V_T^2)} \quad (1)$$

$$E = \rho \cdot \left(\frac{3V_T^2 V_L^2 - 4V_T^4}{V_L^2 - V_T^2} \right) \quad (2)$$

$$G = \rho \cdot V_T^2 \quad (3)$$

$$K = \frac{\rho}{3} \cdot (3V_L^2 - 4V_T^2) \quad (4)$$

2.4 T-t-P Profile

Temperature and variation of MW power with time for melting of 60 g studied glass using direct microwave heating was presented in Fig. 1. MW power was manually controlled to see the microwave interaction with sample. A rapid temperature increase was observed after 30 min with slow increase of MW power at 0.6 kW. A sharp increase of T-t profile at 900 °C was seen in the Fig. 1, predicting higher dielectric loss factor of the glass above this temperature. Fig. 1 also depicted maximum MW power ~ 0.60 kW during the melting till 1,350 °C. However, efficiency of microwave generator was within the range 55%-65% of input electrical power. Thus maximum input electrical power would be ~ 1 kW during the melting of studied glass in microwave furnace. Fig. 1 further illustrated that total time was within 3 h during melting including 30 min initial start up time prior to initiation of heating. Further, operation flexibility in microwave furnace enables easy switch off at high temperature reducing further time and energy.

Temperature and variation of input power with time for the studied glass melted in the resistive heating furnace was displayed in Fig. 2. Heating rate in conventional furnace was restricted to 4 °C/min to protect the life of heating element and refractory lining inside the furnace. Soaking time at maximum temperature in conventional melting was almost

Fig. 1 Temperature and variation of microwave output power with time during microwave melting of glass.

Fig. 2 Temperature and electrical power profile with time for conventional heating in resistance heating furnace.

double the time needed for microwave processing resulting total melting time was around ~ 7 h. Fig. 2 also illustrated that maximum instant electrical power in electrical raising hearth furnace was ~ 4.00 kW almost four times than that of microwave heating.

A comparison of total power consumption for both the heating method using above furnaces for the equal amount of studied glass was illustrated in Fig. 3. It was clearly seen from the Fig. 4 that the total power consumption was ~ 26 kWh in the conventional laboratory glass melting process using above raising hearth furnace. Overall melting time was found to be 7 h^1 in conventional glass melting process. In microwave melting, the total power consumption was recorded ~ 6 kWh incorporating additional power load for

utilities, i.e., chilled water system, fume exhaust system. However, total power requirement could be further minimised by changing the design of the microwave furnace using air-cooled magnetron and by placing a small amount of microwave susceptor like SiC around the crucible, which preheats the raw material before directly absorbing microwave radiation. At higher temperature, microwaves interact with the entire volume of glass at the same rate causing volumetric heating, which further reduces holding time at maximum temperature in glass melting compared to conventional glass melting. Since glass melting is a huge energy consuming process, it would be of great relief if alternate method of producing glass reduces energy requirement substantially. In this context, microwave glass melting could be a potential energy efficient technology saving a significant amount of electrical energy and time, needed for present glass making. Further, microwave melting of glass would eventually contribute to the cleaner environment. However, the difficulty remains in microwave glass melting with proper melting crucible and design of suitable microwave equipment for larger scale glass melting.

3. Results and Discussion

3.1 Microwave Absorption of Material

Microwave heating is the result of absorption of electromagnetic energy by the material exposed to the microwave field distributed within a reflective cavity. It is based on the power absorbed per unit volume (Eq. (5) [13, 14]).

$$P = \sigma |E|^2 = 2\pi f \epsilon_0 \epsilon'_r \tan \delta E^2 \quad (5)$$

where, E is the magnitude of the internal electric field, ϵ_0 is the permittivity of free space, f is the microwave frequency, σ is the total effective conductivity, ϵ'_r is the relative dielectric constant, and $\tan \delta$ is the energy loss required to store a given quantity of energy, ϵ''_{eff}

¹Furnace cooling down to room temperature was not considered.

Fig. 3 Total power consumption profile with temperature; for resistance heating and for microwave heating.

Fig. 4 Dielectric constant and loss factor for the studied glass was measured around 2.45 GHz as a function of temperature up to 1,000 °C.

($= \epsilon'_r \cdot \tan \delta$) is the relative effective dielectric loss factor. It is clearly seen from the above Eq. (5) that the dielectric properties (ϵ_o , ϵ'_r and $\tan \delta$) assume a significant role in determining the extent of power absorbed by a material.

Fig. 4 portrayed the dielectric constant and dielectric loss factor as function of temperature (up to 1,000 °C) for this glass. Dielectric loss factor, one of the important parameters, indicates the degree of microwave absorption by the material to generate heating within it for a fixed frequency (2.45 GHz) and a given electrical field within the material. The Fig. 4 depicted that loss factor increased at ~ 370 °C, ~ 560 °C, ~ 650 °C and ~ 900 °C suggesting enhance microwave absorption and heating of glass at these temperatures. Sharp increase of loss factor 0 at around 900 °C corroborated the rapid heating observed from the T-t

profile in microwave heating in Fig. 1. However, microwave interaction with batch material at lower temperature was found different due to the presence of different raw material (i.e., carbonate, H_3BO_3).

3.2 Comparison of Properties

Fig. 5 displayed photograph of 1mm thick polished glass ($15 \text{ mm} \times 20 \text{ mm}$) melted in a conventional resistance-heating (SBNRH) and in microwave heating (SBNM). In Fig. 5, improved homogeneity of SBNM was seen, apparently compared to SBNRH. Though the SBNM was prepared without employing any mechanical stirring, improved homogeneity was due to the result of microwave induced “self-stirring” effect [15].

The XRD patterns for the SBNRH and SBNM samples, shown in Fig. 6, depicting identical broad humps confirmed the amorphous nature of the samples. The absence of sharp Bragg peak in the XRD profile of SBNM, similar to that of SBNRH, confirmed no unmelted crystalline phase left within the glass melted using microwave.

Chemical analysis results for SBNM and SBNRH samples were listed in Table 1. B_2O_3 and Na_2O were found less in SBNRH glass illustrating higher evaporation loss of these ingredients in conventional melting. Al_2O_3 was found 6.47% in SBNRH and 1.56% in SBNM sample. In conventional heating, heat travels from the heating element to the material by means of

Fig. 5 Photographs of polished SBNRH and SBNM glass samples.

Fig. 6 XRD curves for SBNM and SBNRH samples.

radiation, conduction, convection mechanism and thus temperature of the crucible wall remains higher than the molten glass. This higher wall temperature together with higher melting time contributed higher leaching of Al_2O_3 into the molten glass in conventional melting. In microwave heating, heat generates within the material internally resulting in volumetric heating within the glass without heating the alumina crucible owing to its low dielectric loss factor. Due to this rapid internal heating, temperature gradient in microwave heating exists in the reverse direction than that of conventional heating resulting less temperature in crucible wall and in the top surface of molten glass. Thus, less crucible wall temperature along with less melting time favour low contamination of alumina into the glass melt in microwave heating. This reverse temperature gradient also contributes less evaporation of volatile ingredients of glass in microwave heating. However, slightly higher melting temperature is needed in case of microwave

melting due to less surface temperature of molten glass.

The chemical durability of both the glasses was evaluated from leachate analysis by Inductively Coupled Plasma-Atomic Emission Spectroscopy. The results, as shown in the Table 2, illustrated higher concentration of Al_2O_3 , CaO , Fe_2O_3 , TiO_2 , Na_2O and K_2O , leached out into water from the SBNRH sample signifying higher leaching rate of above constituents from SBNRH sample. This indicated improved chemical durability of glass was prepared in microwave heating. This may be explained that more evaporation of glass former, i.e., SiO_2 and B_2O_3 the main building block in glass, weaken the glass structure in conventional heating method.

Fig. 7 exemplified DSC thermograph for both the glasses SBNRH and SBNM. T_g was identified 569.5°C and 576.3°C for SBNM and SBNRH samples, respectively. T_g value for SBNM was observed less due to difference in the composition of the glasses resulting from low loss of volatile constituents of glass. Higher presence of Al_2O_3 in SBNRH owing to leaching from crucible wall, also contributed the higher T_g in SBNRH.

The some of the physical, optical and elastic properties of SBNM and SBNRH were listed in Table 3. Optical properties for SBNRH and SBNM samples were calculated from their measured refractive indices using relevant expressions and were presented in Table 3.

Fig. 8 portrayed dispersion curves, obtained by fitting the measured refractive indices with the

Table 1 Chemical analysis results for SBNRH and SBNM samples.

Composition (wt.%)	SBNRH	SBNM
SiO_2	56.36 (± 0.08)	58.88 (± 0.08)
B_2O_3	7.92 (± 0.04)	9.13 (± 0.04)
Na_2O	10.92 (± 0.04)	12.02 (± 0.04)
K_2O	0.68 (± 0.03)	0.82 (± 0.03)
CaO	4.82 (± 0.04)	5.03 (± 0.04)
Fe_2O_3	3.65 (± 0.03)	3.65 (± 0.03)
TiO_2	8.91 (± 0.04)	8.57 (± 0.04)
Al_2O_3	6.47 (± 0.03)	1.56 (± 0.03)

Table 2 The concentration (in ppm) of different constituents present in deionised water after 14 days insertion of SBNRH and SBNM glasses at 90 °C.

Leachate from samples	Al ₂ O ₃	CaO	Fe ₂ O ₃	TiO ₂	Na ₂ O	K ₂ O
SBNM	31	19	10	15	131	21
SBNRH	59	68	21	35	377	74

Table 3 Glass transition temperature, optical properties and elastic properties for SBNRH and SBNM are presented.

Property	SBNRH	SBNM
Measured Refractive index (<i>n</i>) at		
473 nm (± 0.0004)	1.5969	1.6006
532 nm (± 0.0004)	1.5904	1.594
633 nm (± 0.0004)	1.5827	1.586
1,064 nm (± 0.0004)	1.5691	1.573
1,552 nm (± 0.0004)	1.5624	1.5656
Estimated Refractive indices at		
<i>n_{d-587.6}</i> (± 0.0005)	1.5860	1.5895
<i>n_{F-486.1}</i> (± 0.0005)	1.5953	1.5988
<i>n_{C-656.3}</i> (± 0.0005)	1.5814	1.5848
<i>n_{d-1}</i>	0.586	0.59
Mean dispersion (<i>n_F-n_C</i>)	0.014	0.014
Abbe No.	42.16	42.10
Reflection loss (%)	5.17	5.13
Elastic properties		
Longitudinal velocity (m/s)	5,776	5,813
Shear velocity (m/s)	3,448	3,505
Young's modulus (<i>E</i> , GPa)	78.90	80.20
The Poisson's ratio (<i>ν</i>)	0.219	0.235
Shear modulus (<i>G</i> , GPa)	90.07	93.76
Bulk modulus (<i>K</i> , GPa)	46.90	50.46
Glass transition temperature, <i>T_g</i> (± 1 °C)	576.3	569.5

Fig. 7 DSC thermographs for SBNRH and SBNM sample.

Cauchy Eq. (6). The refractive indices at standard wavelengths *n_d* (at 587.6 nm), *n_F* (at 486.1 nm) and *n_C* (at 656.3 nm) were estimated from the curve.

$$n(\lambda) = A + \frac{B}{\lambda^2} + \frac{C}{\lambda^4} + \dots \quad (6)$$

where, *n* is refractive index at wavelength *λ*. The Abbe number *V* of a material is defined as Eq. (7):

$$V = \frac{(n_d - 1)}{(n_F - n_C)} \quad (7)$$

where, *n_d*, *n_F* and *n_C* are the refractive indices of the samples at the wavelengths 587.6 nm, 486.1 nm and 656.3 nm, respectively. Abbe number was calculated and found very similar for both SBNRH and SBNM as 42.16 and 42.10, respectively. Mean diffraction and reflection loss for SBNRH and SBNM samples were also found similar.

Elastic properties like *ν*, Young's (*E*)-shear (*G*)-bulk (*K*) modulus were calculated and listed in Table 3. The identical or slightly higher value of these properties in the microwave prepared glass confirmed

Fig. 8 RI vs. wavelength fitted by Cauchy curve fitting equation.

Fig. 9 FTIR reflectance spectra of studied samples in the wave number range 400-2,000 cm^{-1} .

that similar glass or slightly improved property of glass could be prepared in microwave heating.

The FTIR reflectance spectra of samples SBNRH and SBNM were presented in Fig. 9. Both the spectra of these glasses consisted of similar broad and sharp bands in different regions ($400\text{-}2,000\text{ cm}^{-1}$) confirming identical structure. FTIR spectra for both glasses showed the presence of silicate peak centered at $1,022\text{ cm}^{-1}$ and Si-O-Si bend at around 443 cm^{-1} [16]. The bands at around $1,020\text{ cm}^{-1}$ peak were attributed to a stretching vibration of B-O-Si linkage [17]. The peak $\sim 700\text{ cm}^{-1}$ was due to the bending of B-O-B linkages in the borate glassy network [18]. The

boroxol ring appeared at 780 cm^{-1} [19]. The band around $1,360\text{ cm}^{-1}$ was assigned to B-O stretching vibration of trigonal $[\text{BO}_3]$ unites [20, 21].

4. Conclusions

A glass containing $\text{SiO}_2\text{-B}_2\text{O}_3\text{-Na}_2\text{O-K}_2\text{O-CaO-Fe}_2\text{O}_3\text{-TiO}_2$ was successfully melted using direct microwave heating. Dielectric constant and loss factor as function temperature depicted increase of these properties at $370\text{ }^\circ\text{C}$, $550\text{ }^\circ\text{C}$, $650\text{ }^\circ\text{C}$ and $900\text{ }^\circ\text{C}$, indicating enhanced microwave absorption with this glass composition above these temperatures. Similar glass was also prepared from identical batch in conventional resistive heating. Physical properties and structure of both the glasses were found similar. Glass melted in microwave heating was found closer chemical composition as prescribed in batch, improved chemical durability and lower glass transition temperature. Substantial less consumption of energy and shorter processing time was demonstrated in laboratory scale glass melting in microwave furnace. Thus, microwave processing of glass could be a potential method for reduction of energy consumption and time.

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References

- [1] Sutton, W. H. Microwave Processing of Ceramic

- Materials. *Cer. Bull.* **1987**, *68*, 376-386.
- [2] Thostenson, E. T.; Chou, T. W. Microwave Processing: Fundamentals and Applications. *Composites: Part A* **1999**, *30*, 1055-1071.
- [3] Siligardi, C. D.; Arrigo, M. C.; Leonelli, C.; Pellacani, G. C.; Cross, T. E. Bulk Crystallization of Glasses Belonging to the Calcia-Zirconia-Silica System by Microwave Energy. *J. Am. Ceram. Soc.* **2000**, *83*, 1001-1003.
- [4] Knox, M. P.; Copley, G. J. Use of Microwave Radiation for the Processing of Glass. *Glass Technol.* **1997**, *38*, 91-96.
- [5] Ghussn, L.; Martinelli, J. L. A Novel Method to Produce Niobium Phosphate Glasses by Microwave Heating. *J Mater Sci.* **2004**, *39*, 1371-1376.
- [6] Mandal, A. K.; Biswas, K.; Annapurna, K.; Guha, C.; Sen, R. Preparation of Alumino-Phosphate Glass by Microwave Radiation. *J. Mater. Res.* **2013**, *28(14)*, 1955-1961.
- [7] Chenu, S.; Rocherullè, J.; Lebullenger, R.; Merdignac, O.; Chevire, F.; Tessier, F.; et al. Synthesis and Characterization of Tin Containing Molybdophosphate and Tungstophosphate Glasses. *J. Non-Cryst. Solids* **2010**, *356*, 87-92.
- [8] Almeida, F. J. M.; Martinelli, J. R.; Partiti, C. S. M. Characterization of Iron Phosphate Glasses Prepared by Microwave Heating. *J. Non-Cryst. Solids* **2007**, *353*, 4783-4791.
- [9] Vaidhyanathan, B.; Ganguli, M.; Rao, K. J. A Nobel Method of Preparation of Inorganic Glasses by Microwave Irradiation. *J. Solid State Chem.* **1994**, *113*, 448-450.
- [10] Peñaranda-Foix, F. L.; Janezic, M. D.; Civera, J. M. C.; Canós, A. J. Full-Wave Analysis of Dielectric-Loaded Cylindrical Waveguides and Cavities Using a New Four-Port Ring Network. *IEEE T Microw. Theory* **2012**, *60*, 2730-2740.
- [11] Varshneya, A. *Fundamentals of Inorganic Glasses*; Academic Press: New York, 1994.
- [12] Afifi, H.; Marzouk, S. Ultrasonic Velocity and Elastic Moduli of Heavy Metal Tellurite Glasses. *Mater. Chem. Phys.* **2003**, *80*, 517-523.
- [13] Clark, D. E.; Folz, D. C.; West, J. K. Processing Materials with Microwave Energy. *Mat. Sci. Eng. A-Struct.* **2000**, *287*, 153-158.
- [14] Rao, K. J.; Vaidhyanathan, B.; Ganguli, M.; Ramakrishnan, R. K. Synthesis of Inorganic Solids Using Microwaves. *Chem. Mater.* **1999**, *11*, 882-895.
- [15] Mandal, A. K.; Agrawal, D.; Sen, R. Preparation of Homogeneous Barium Borosilicate Glass Using Microwave Energy. *J. Non-Cryst. Solids* **2013**, *371*, 41-46.
- [16] Abiraman, S.; Varma, H. K.; Kumari, T. V.; Umashankar, P. R.; John, A. Preliminary *in vitro* and *in vivo* Characterizations of a Sol-Gel Derived Bioactive Glass-Ceramic System. *Bull. Mater. Sci.* **2002**, *25*, 419-429.
- [17] Tenny, A. S.; Wong, J. Vibrational Spectra of Vapor-Deposited Binary Borosilicate Glasses. *J. Chem. Phys.* **1972**, *56*, 5516-5523.
- [18] Dwoidari, H.; Abou, Z.; Damraway, E. I. Effect of Gamma Radiation and Thermal Treatment on Some Physical Properties of $\text{ZnO-PbO-B}_2\text{O}_3$ Glasses. *J. Phys. D: Appl. Phys.* **1991**, *24*, 2222-2228.
- [19] Silim, H. A. Structure and Properties of $\text{BaO-B}_2\text{O}_3\text{-Al}_2\text{O}_3\text{-NaCl}$ Glass System Egypt. *J. Sol.* **2003**, *26*, 15-24.
- [20] Nolan, M.; Perova, T. S.; Moore, R. A.; Beitia, C. E.; McGilp, J. F.; Gamble, H. S. Spectroscopic Investigations of Borosilicate Glass and Its Application as a Dopant Source for Shallow Junctions. *J. Electro Chem. Soc.* **2000**, *147*, 3100-3105.
- [21] Lal, R.; Sharma, N. D.; Chandra, K. Mössbauer and Infrared Studies of Some Glasses and Glass-Ceramics from the $\text{MnO}_{(40-X)}\text{YFe}_2\text{O}_{3.10}\text{Al}_2\text{O}_{3.50}\text{B}_2\text{O}_3$ System. *Indian J. Pure Appl. Phys.* **2004**, *42*, 498-505.