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Preparation and Charecterizaion of Glass Materials

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Abstract: *This paper describes preparation of glasses by melt quench method and experimental techniques employed in the characterization of glasses. Characterization techniques such as X-ray Diffraction technique (XRD), Fourier Transform Infrared spectroscopy (FTIR); UV analysis, photo luminescence and dielectric studies described in detail.*

Key words: *XRD, FTIR, Thermal, Fluorescence spectroscopy, dielectric constant.*

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I. INTRODUCTION

Melt quenching technique was the first glass preparation technique used in glass industry as well as in research field, before chemical vapour deposition and sol gel technique. One of the important features of the melt quenching technique is the flexibility of preparing a large number of compositions of glass of silicate, borate, phosphate, oxide or non oxide systems. There are three classes of components for oxide glasses network formers, intermediates and modifiers. The doping or co doping of different types of active ions is quiet easy using this method. The mechanical properties of the doped glass-ceramics were mainly influenced by their micro structures.

1.1.1 Preparation of Glass materials

Most common glass preparation techniques in glass research are melt quench method, chemical vapor deposition and sol gel method. Bulk glasses in this research were prepared using melt quenching method Compared to other glass preparation methods, the disadvantage in this method is that the lack of purity of the prepared glass sample. In order to avoid contamination, the crucibles made of noble metals like Platinum can be used. In his method glass starting materials were weighed using electronic balance after batch calculation. The glass batch was melted in a platinum crucible and cast into a rectangular brass mold at room temperature. All the melting processes were done using an electric furnace as shown in Fig. 1. Each glass sample was immediately transferred to an annealing furnace at 300°C and slowly cooled to room temperature. These samples were then polished using various grades of silicon carbide powder for obtaining parallel, smooth and clear surface for experiment. The thickness of the glass specimens was measured using a digital micrometer gauge.

1.1.2 The glass transition

Glass formation is a matter of bypassing the process of crystallization and almost all materials can be prepared as amorphous solids by quick cooling. The rate of cooling varies enormously from material to material. When glass is made, the material is quickly cooled from a super cooled liquid, an intermediate state between liquid and glass. To become an amorphous solid, the material is cooled, below a critical temperature called the glass transition temperature. The newly formed amorphous structure is not as organized as a crystal, but it is more organized than a liquid. In the process of glass transition, it is important to plot the behavior of amorphous materials, from the super cooled state to glass, in a V-T diagram [Fig. 2] Here, temperature is plotted in the x-axis and the volume/enthalpy occupied by the material is plotted along the y-axis, where temperature Tm is the melting point, and Tg is the glass transition temperature

Fig. 1: Furnace used for melt quenching

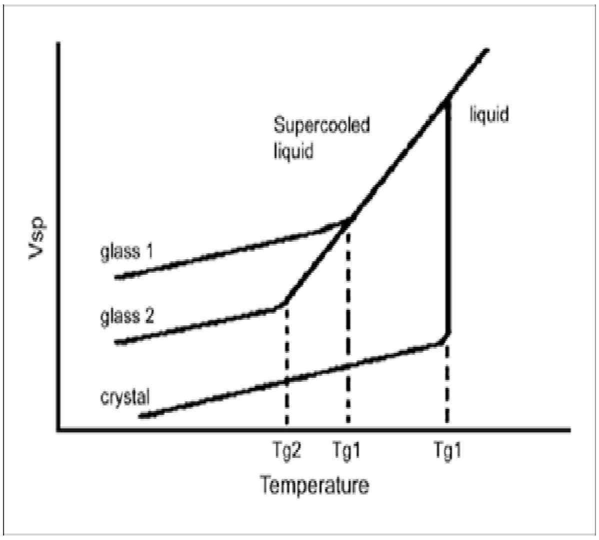

Fig. 2: V-T Diagram showing glass formation

.. The glass transition occurs when the super cooled liquid freezes into an amorphous solid with no abrupt discontinuity in volume, near the glass transition temperature. The glass transition temperature Tg is not as sharply defined as Tm; Tg shifts towards lower temperature when the cooling rate is reduced. The reason for this phenomenon is the steep temperature dependence of the molecular response with time. Tg also varies with the composition of raw materials selected for preparation of glass. When the temperature is lowered below Tg, the response time for molecular rearrangement becomes much larger than experimentally accessible times, so that liquid like mobility disappears and the atomic configuration becomes frozen into a set of fixed positions to which the atoms are tied. Possible range of glass formation is represented by temperatures Tg1 and Tg2. Tg1 is the glass transition temperature for a glass formed when the cooling rate is reduced and Tg2 is that of increased rate of cooling. The slopes of crystal and glass lines in a V-T diagram are small, compared to the high slope of the liquid section, which reflects the fact that the coefficient of thermal expansion of a solid is small in comparison with that of the liquid. An important change, the jump of heat capacity, also happens during liquid-glass transition, which is why physicists thought there is some sort of a phase transition, between the liquid phase and the glass phase.

2.1 Characterization techniques

Characterization techniques are important for researchers as these are the basic tools in identifying the structure and thereby properties of materials synthesized, which help in determining whether the designed materials are suitable for particular applications. We present the preparation and the experimental techniques to find out the structural, thermal, linear and nonlinear optical properties of glasses. The characterization of a material can be defined as a complete description of its physical and chemical properties. A thorough and extensive characterization of a glass is very difficult, because it requires a variety of tests using a number of sophisticated instruments and an accurate analysis of the results of these tests and their confirmations. The use of instrumentation is an exciting and fascinating part of any analysis that interacts with all the areas of chemistry and with many other fields of pure and applied sciences. In most of the cases of chemical analysis, a signal is produced, which reflects the chemical or physical property of a chemical system. The present paper deals with the description of various characterization techniques and methods of preparation of glasses.

2.1.1 Powder X–ray diffraction

X–rays are electromagnetic radiation of wavelength about 1 Å, which is about the same size as an atom. Max von Laue, in 1912, discovered that the crystalline substances act as three-dimensional diffraction gratings for X-ray wavelengths similar to the spacing of planes in a crystal lattice. X-ray diffraction is a most common technique for the study of crystal structures and atomic spacing. Each crystalline solid has its unique characteristic X-ray powder diffraction pattern, which can be used as a "fingerprint" for its identification. Once the material has been identified, X-ray crystallography may be used to determine its structure, average particle size, unit cell dimensions and sample purity.

2.1.2 Principle

X-ray powder diffraction is based on constructive interference of monochromatic X-rays and a crystalline sample. These X-rays are generated in a cathode ray tube by heating a filament to produce electrons, accelerating the electrons toward a target by applying a voltage, and bombarding the target material with electrons. When electrons have sufficient energy to dislodge inner shell electrons of the target material, characteristic X-ray spectra are produced. The interaction of the incident rays with the sample produces constructive interference (and a diffracted ray) when conditions satisfy Bragg's Law ($n\lambda=2d \sin\theta$). Where 'n' is an integer referring to the order of reflection, 'λ' is the wavelength, 'd' is the spacing between the crystal lattice planes responsible for particular diffracted beam and 'θ' is the angle that incident beam makes with lattice planes.The path difference between the incident beam and the reflected beam in the consecutive lattice planes is shown in Fig. 3. The width of the Bragg's reflection in a standard X–ray powder diffraction pattern can provide information on the average grain size. The peak breadth increases as the grain size decreases, because of the reduction in the coherently diffracting domain size, which can be assumed to be equal to the average crystallite size. However, the average particle size can be estimated by using Scherrer's relation (1).

$$D = K\lambda/\beta \cos\theta \ \ldots(1)$$

where 'λ' is the X-ray wavelength, 'β' is the full width at half maximum of a diffraction peak, 'θ' is the diffraction angle, and 'K' is the Scherrer's constant of the order of unity for usual crystal.

3.1 Fourier transform infrared (FT-IR) analysis

Fourier transform spectroscopy is a simple mathematical technique to resolve a complex wave into its frequency components. The conventional IR spectrometers are not of much use for the far IR region, as the sources are weak and the detectors are insensitive. FT-IR made this energy-limited region more accessible. It also made the mid-infrared (4000-400 cm^{-1}) more useful. In the fourier transform spectrometer, a time domain plot is converted into a frequency domain spectrum. The actual calculation of the Fourier transform of such systems is done by means of high-speed computers.

3.1.1 EXPERIMENTAL

The FT-IR spectrometer consists of an infrared source, a sample chamber with a provision for holding glass samples, monochromator, a detector and a recorder, which are integrated with a computer. Interferometric multiplex instruments employing the Fourier transform are now finding more general applications in both qualitative and quantitative infrared measurements. The interference pattern is obtained from a two-beam interferometer, as the path difference between the two beams is altered and then Fourier transformed output gives rise to the spectrum. .This instrument has resolution of 0.1 cm^{-1}. The recording of IR spectra of solid sample is more difficult because the particles reflect and scatter the incident radiation and therefore transmittance is always low. A few milligram of the sample is mixed with about 100 times the quantity of KBr which is transparent to the infrared using mullitemortar.The powder is pelletized in proper form suitable for IR transmission.

3.1.2 UV-Vis-NIR spectroscopy

UV-Vis-NIR spectroscopy is defined as the measurement of the absorption or emission of radiation associated with changes in the spatial distribution of electrons in atoms and molecules. In practice, the electrons involved are usually the outer valence or bonding electrons, which can be excited by the absorption of UV or visible or near IR radiation.

3.1.3 Photoluminescence studies

Absorption of ultraviolet radiation by a molecule leads to electronic excitation among various energy levels within the molecule. The transitions generally occur in between a bonding or lone-pair orbital and an unoccupied non-bonding or anti-bonding orbital. The σ orbitals, involved in forming σ bonds, are the lowest energy occupied molecular orbitals. The π-orbitals lie at somewhat higher energy and the orbitals holding the unshared electron pairs (non-bonding orbitals) lie at even higher energy levels than the π-orbitals. The unoccupied or anti-bonding orbitals (π* and σ*) are the orbitals of highest energy. The effect of the resonant absorption loss is determined by the parameter

$$\boldsymbol{\beta} = \boldsymbol{\sigma}_{abs}/(\boldsymbol{\sigma}_{abs} + \boldsymbol{\sigma}_{em}))$$

The probability for electronic transitions determines the intensity of spectral lines. There must be large overlap between the vibrational states in the initial and the final electronic states to have a large absorption cross-section, or high probability that the molecule will absorb/emit radiation. Electronic transitions are possible for a wide range of vibrational levels within the initial and final electronic states. Saturated hydrocarbons and compounds containing only alkyl groups, alcohol groups and ether groups are transparent in the region 200-1000 nm.Such compounds are useful as solvents for making solutions of the specimen to study in this region. An isolated functional group not in conjugation with any other group is said to be achromophore if it exhibits absorption of a characteristic nature in the ultraviolet or visible region. Thus, it is readily seen that the spectrum of a compound, when correlated with data from the literature for known compounds, can be a very valuable aid in determining the functional groups present in the molecule.

3.1.4 Optical properties of glass materials

For each wavelength of light passing through the spectrometer, the intensity of the light passing through the reference cell is measured (Io). The intensity of the light passing through the sample cell is also measured for that wavelength (I). If I is less than Io, then obviously the sample has absorbed some of the light. For reasons to do with the form of the Beer- Lambert Law. Generally, the absorption edge of these glasses is determined by the oxygen bond strength in the glass forming network. Any change of oxygen bonding in the glass network, for instance, changes the formation of non-bridging oxygen, thereby changing the characteristic absorption edge. Additionally, all glasses regardless of high purity of the chemicals used may contain impurity to small extent. In luminescent glasses the sensitizer may transfer the excitations. All these factors may account for the observed increase of luminescence associated with the dopant materials .

4.1 Di electric studies

The temperature and frequency dispersions of dielectric constant as well as dielectric loss have been analyzed using space charge polarization model. The ac and dc conductivities have exhibited in the dopant concentration range. Both quantum mechanical tunneling (QMT) and correlated barrier hopping models (CBH) were used for clarification of ac conductivity origin and the corresponding analysis has indicated that CBH model is more appropriate for the glass system. For the better understanding of relaxation dynamics of the electrical properties we have drawn the scaling plots for ac conductivity and also electric moduli. It is observed that the relaxation dynamics is independent on temperature but depends on concentration of the dopant. The dc conductivity is analyzed using small polaron hoping model. The increase of conductivity with the concentration is explained in terms of variations in the ratio dopant ions in the glass network.

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