Chapter 2

Materials and Methods

2.1 Materials used for melt annealing methods

- Quartz (SiO_2)
- Phosphorus pentoxide (P_2O_5)
- Sodium carbonate (Na_2CO_3)
- Ammonium dihydrogen ortho phosphate ($(NH_4)H_2PO_4$)
- Potassium carbonate (K_2CO_3)
- Calcium fluoride (CaF_2)
- Tin oxide (SnO_2)
- Iron oxide (Fe_2O_3)
- Cuprous oxide (Cu_2O)
- Cupric oxide (CuO)
- Zinc oxide (ZnO)
- Lead monoxide (PbO)
- Samarium oxide (Sm_2O_3)
- Praseodymium oxide (Pr_2O_3)

(Lobachemie, Avarice industries, India; Tested 98-100%)

2.2 Details of the conventional melt annealing methods

Utilizing an electronic microanalytical balancing machine, a suitable quantity of AR-graded dry ingredients was measured and well-mixed using a mortar and pestle before melting in the melt-annealing process. Following initial processing, the batches were melted in crucibles made of high-quality alumina at a temperature of 1300 ± 100 °C in an electric furnace. To cast

and air-quench the glasses, the melted glass was poured over an alumina plate that had been heated. The glass samples were then allowed to gradually cool to room temperature inside the furnace after being annealed at around 450 °C to remove thermal strains and stresses. The samples were then mechanically cut to the desired shape using a glass cutter, and polished using silicon carbide (SiC) and cerium oxide (CeO_2), for physical, optical, fluorescence, and mechanical characterization. The rectangular glass samples with irregular dimensions that were made shortly after casting were cut with a water-cooled low-speed diamond saw. The edges of the samples were then gradually ground and polished employing water-cooled SiC grits of 400/800/1200 and diamond pastes of 6/3/1/.025 µm to guarantee that both edges of the prepared samples are parallel to one another with a deviation of less than.05 cm. The samples were then thoroughly cleaned in water, dried with a warm air blower, and re-annealed using the time-temperature profile, ramping, and cooling rate indicated in **Fig. 3.1** This was done to eliminate any remaining stresses created during the cutting, grinding, and polishing processes; the samples' lack of residual stresses was later verified with a polariscope.

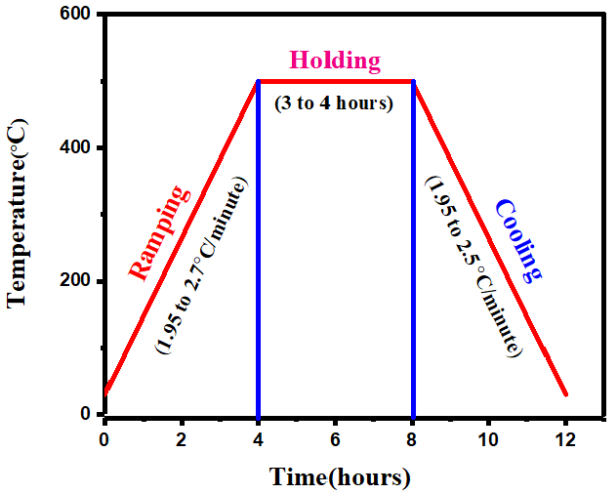

Fig. 2.1. This figure shows the annealing process's time-temperature profile.

2.3 Instrumental details of the characterization techniques, and measurement process

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All the produced glasses were ground into fine powders using a mortar and pestle to perform room-temperature XRD, FTIR, and Raman spectroscopic analyses to investigate the structural details of the examined samples. Using a sample container, the fine powder of the glass samples was placed in a 9 KW powder-type X-ray diffraction machine from RIGAKU Corporation (Japan). This machine includes a Ni filter tube and a graphite monochromatic copper Kα radiation source (= 1.540 Å), and it works at 40 kV and 20 mA current. All XRD data has been collected within the range of 2θ spanning 10º to 80º with a scan rate of 3º min^{-1}. PANalytical XPert High Score plus matching with conventional ICDD (International Centre for Diffraction Data) cards were used for analyzing each set of data. All the glass powder samples' infrared spectra were examined in transmittance mode at 4 cm^{-1} resolution throughout a range of wavenumbers (4000-450 cm^{-1}) to highlight any functional groups found in the glasses. All the FTIR data of the powder of the all examined glass samples were acquired at room temperature by using a single-beam Nicolet iS5 FTIR spectrometer owned by THERMO Electron Scientific Instruments LLC Company (USA), employing the KBr disc approach. The Raman data of selected powder of the glass samples doped with rare earth elements were collected by using UHTS -300 (WITec GmBH, Germany) confocal-Raman microscope employing a 532 nm laser excitation with a power of 1.5 mW included a grating of 600g/mm to evaluate the selected analyzed samples' phonon energy. The ultraviolet and visible spectra of the optically polished glass samples were captured applying a Jasco V-770 double-beam UV-VIS spectrophotometer (Japan) connected to PC UV-Win lab software. The visible along solar optical characteristics of the tint glasses were calculated via a weighted average of the experimental data throughout the range of 360 nm to 830 nm. The normal incidence of light in the ultraviolet and visible spectral zones was utilized to collect the measurements at the spacing

of 5 nm, which includes reflection in the diffuse reflection mode, transmission in the specular transmission mode, and absorption in the spectrum mode. The US standard environment, which typically has a highly clean atmosphere with 3.4 mm of ozone and 20 mm of precipitable H_2O vapor, was applied to collect the weighted factors for measuring solar optical properties [37]. The materials absorption coefficient $\alpha(\omega)$ for each glass sample at various photon energies $\hbar(\omega)$ was determined using the Lambert-Bear equation,$\left(I_t = I_0 e^{-\alpha(\omega)d}\right)$, where d is the sample thickness and I_0 and I_t are the incident and transmitted photon intensities, correspondingly. The fluorescence spectrum of the specified rare earth-doped glass samples was recorded at room temperature using a WI Tec α-300 (Germany) spectrophotometer equipped with a 75W xenon light source in the visible frequency range.

Applying the Archimedes principle and using water as the immersion liquid, the density of all the created glass bars of all compositions was measured at room temperature. The following formula was used to determine each glass sample's density (D), where D stands for the sample's density, W_D for the dry weight of the glass sample, W_W For wet weight of the glass sample, and ρ for water density. The calculated variance of the total density values was around ± 0.004 g cm^{-3}.

$$D = \frac{W_D \times \rho}{(W_D - W_W)}$$

The mechanical properties of the discussed iron and copper-based stannic soda phosphate silicate tint glass samples having the desired dimensions by using ASTM D695 standards protocol with the absence of residual stresses later checked by using a polariscope were evaluated using the Tinius Olsen H10KL universal tensile machine (UTM) with the prearranged setup of .05 mm/min crosshead speed under 10 MN load cell. The young modulus was then calculated using the stress-strain diagram and the following equation.

$$\sigma_C = \frac{P}{A}$$

$$\varepsilon = \frac{D}{d}$$

$$E = \frac{\sigma_C}{\varepsilon}$$

In the present scenario, σ_C Stands for compressive strength, P for the load (Newton), A for the surface area of load (mm^2), ε for compressive strain, D for displacement measured by the instrument along the sample thickness prior to breakdown, d for sample thickness, and E for the young module.

Implementing an ultrasonic flaw detector machine with two quartz X- and Y-cut transducers operating at a 5 MHz resonant frequency and an error of ± 10 m/s ambiguity, the longitudinal (V_L) and shear (V_S) ultrasonic wave velocities of PbO-added sodium silicate glass samples, as well as Cu_2O, CuO, SnO_2, and PbO-added sodium silicate glass samples, were calculated at room temperature. Later these measured longitudinal ultrasonic velocity (V_L), and shear ultrasonic velocity (V_S) were used to compute the longitudinal (L_m), shear (G_m), bulk (k_m), Young's(E_m)modulus, hardness(H), and Poisson's ratio (υ) of the respective glass samples.

The weight loss of the tested sample of the mentioned iron and copper-based stannic soda phosphate silicate glass in a corrosive environment was evaluated following the initial weight measurements of the glass samples to assess the chemical durability, which is an indicator of its ability to withstand in opposition to chemical attacks [**38**]. The corresponding samples were then placed in a fixed concentration of 1(N) of HNO_3, H_2SO_4, and HCl for a week at roughly 25 °C, and the weight loss of the respective samples was then measured which was utilized to compute the chemical durability. The chemical durability and the weight loss of the respective samples were connected by using the following equation,

$$\text{Chemical durability (gram/ (cm}^2\text{.hour)} = \frac{W_i - W_f}{st}$$

The variables W_i and W_f represents the initial and final weights of the glass samples, respectively, before and after they were dipped in the corresponding corrosive liquid, surface area (s) for the glass piece, and time (t).

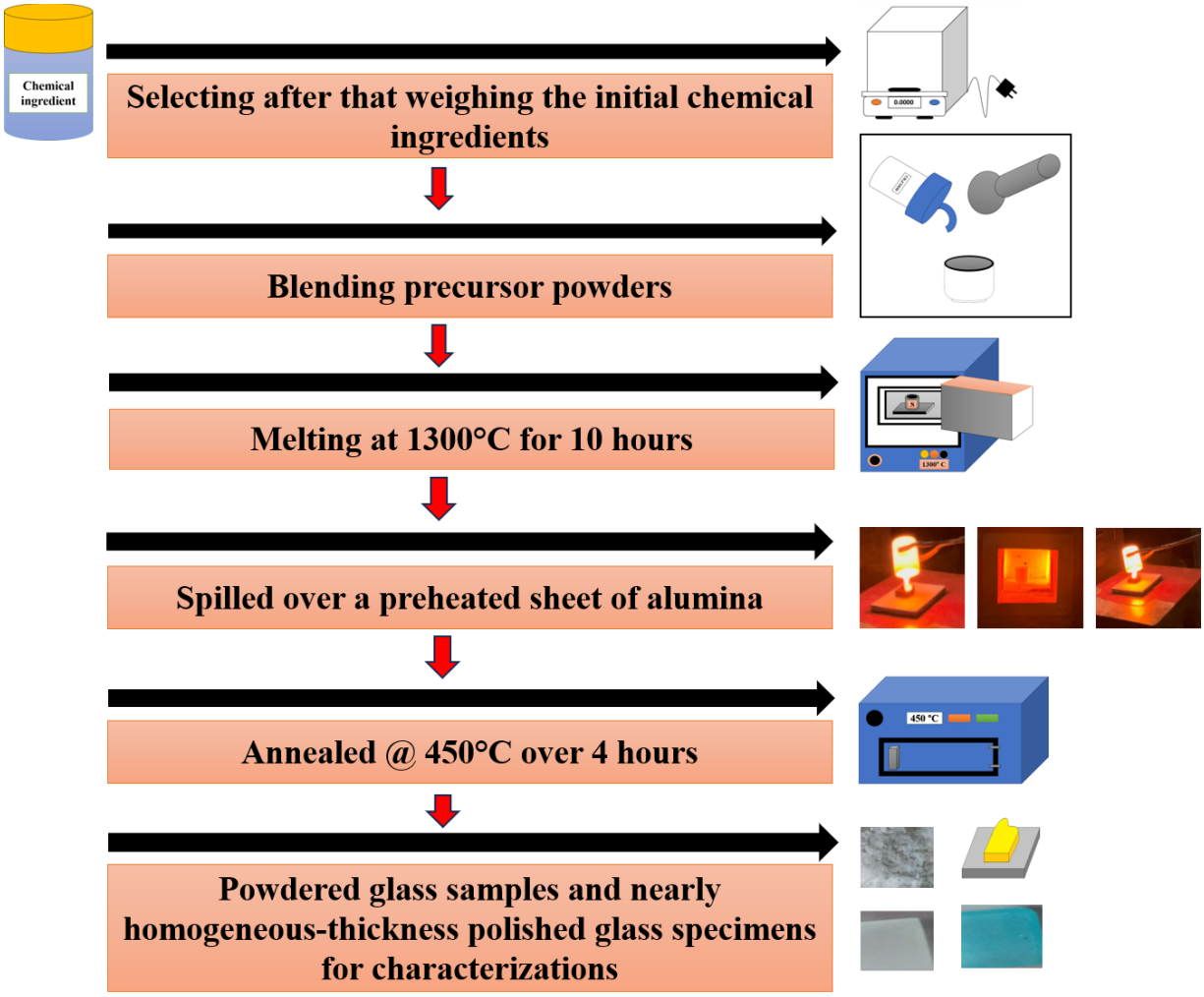

Fig. 2.2. The schematic diagram of the conventional melt-quench method for glass samples characterizations, was used to make all the prepared glass samples.

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