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Micro Structure Analysis Of Glass Samples Using Sem And Edx Characterisation Techniques

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Abstract

By conventional Melt – Quench method Ti doped borosilicate glasses prepared by varying the concentration of Na_2O and K_2O alkalis from 5% - 25% keeping the total concentration of two alkalis as 30% in all 5 samples. Scanning Electron Microscopy (SEM) pictures and Energy Dispersive X-ray Spectrum (EDX) of Titanium doped borosilicate glasses and undoped glasses were recorded and their micro structure was analysed . From glass composition weight % and atomic Concentration % of elements was calculated and are in agreement with experimentally recorded EDX data.

1. Introduction

Glasses are used in many different ways, from transparent cooktops to mirrors for satellites. Changing the composition of glass, such as by adding transition metals, can significantly alter its properties. For example, titanium dioxide in glass is very effective at cleaning dirt when exposed to sunlight. It's also non-toxic, chemically stable and inexpensive [1].

2. Experimental

2.1 Sample preparation

Glasses of composition xNa_2O-$(30-x)K_2O$-$39.5SiO_2$-$30B_2O_3$-$0.5TiO_2$($5 \leq x \leq 25$),where x was varied from 5 to 25 in steps of 5 keeping total alkali concentration remain as 30 mol% in all samples ,were prepared by conventional melt-quench method. Analytical grade SiO_2, B_2O_3, K_2CO_3, Na_2CO_3, TiO_2 were used as starting materials. The weighed chemicals were homogeneously mixed and grinded. The powdered mixture was taken in a silica crucible and melted in high temperature Muffle furnace at 1050^0C temperature up to 25-30 minutes until a bubble free liquid was formed. The melt was then quenched to room temperature in air by pouring it on a brass slab and the obtained glasses were annealed about 3 hours at 350^0C.

The prepared glasses are labelled according to their sodium oxide (Na_2O) molar percentage as follows.

Table 1 Glass code & Composition of prepared Glasses

Glass Code	Na_2O mol%
X_5	5
X_{10}	10
X_{15}	15
X_{20}	20
X_{25}	25

3. Results

3.1 X-Ray Diffraction

X-ray Diffarctometer Philips PW1830 is used to record the X-ray diffraction spectra.

The X-ray powder diffraction graph is drawn between intensity of scattered X-rays at different angles by sample glasses. All these graphs are found to be the same. From the **Fig.**X-ray scattering pattern features broad, poorly defined 'humps'. This informs long range order in particles arrangement is missing and confirms the amorphous nature of studied glasses.

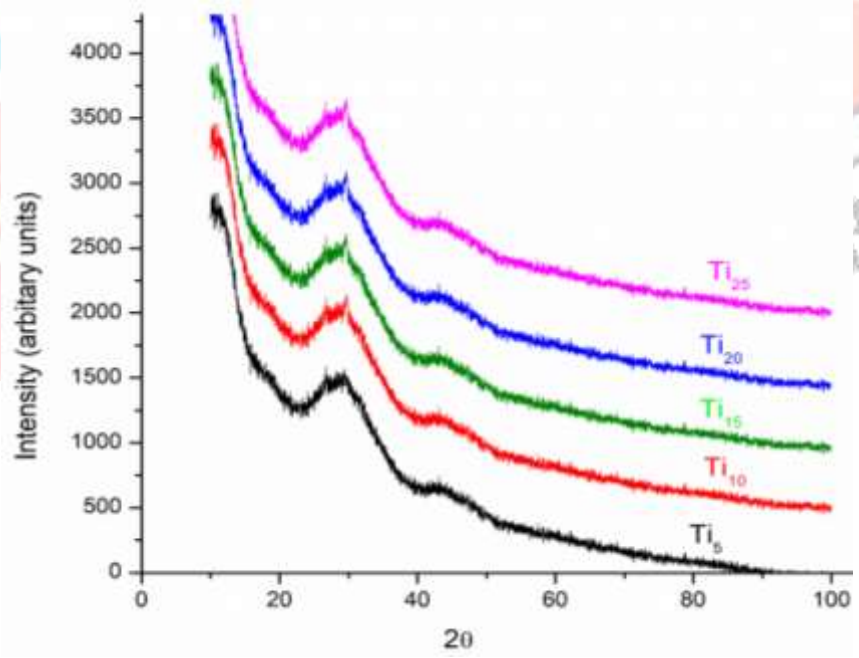

Fig . XRD spectra of TiO_2 doped Borosilicate Glass

3.2 Micro structural analysis

3.2.1 SEM images

The micro structure of undoped glass , Ti_5glass and Ti_{20} glass SEM pictures aretaken with ZEISS Scanning Electron Microscopeand are shown in **Fig.1**,**Fig.2& Fig.3**. It was observed that the texture of the glass was altered by adding TiO_2 and varying the alkali concentration ratio. These changes indicate that the structure of the glass is influenced by compositional variations.

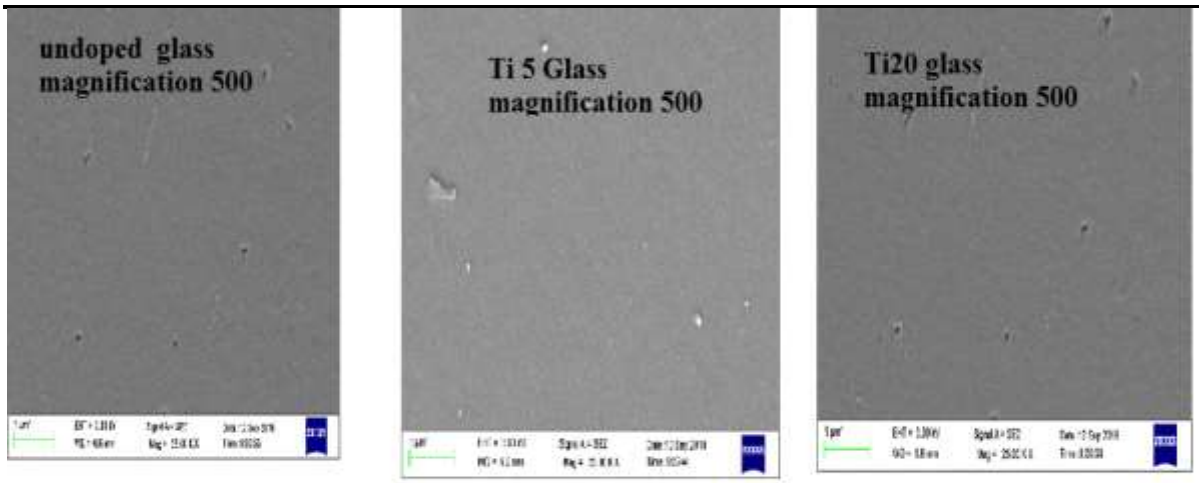

Fig 1 SEM images of 39.5SiO_2-30 B_2O_3-xNa_2O-(30-x)K_2O -0.5TiO_2 Glasses

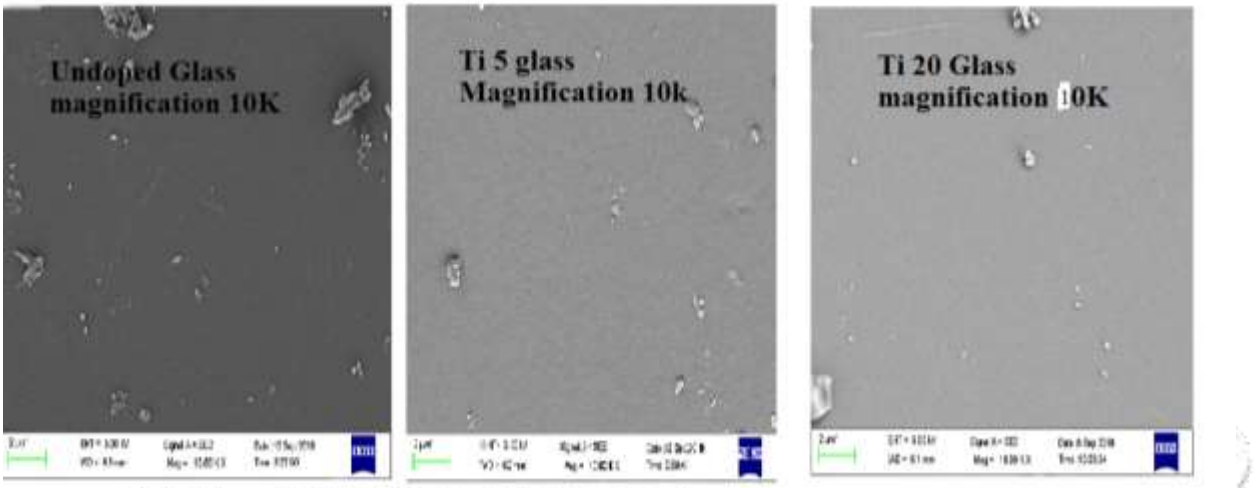

Fig 2 SEM images of 39.5SiO_2-30 B_2O_3-xNa_2O-(30-x)K_2O -0.5TiO_2 Glasses

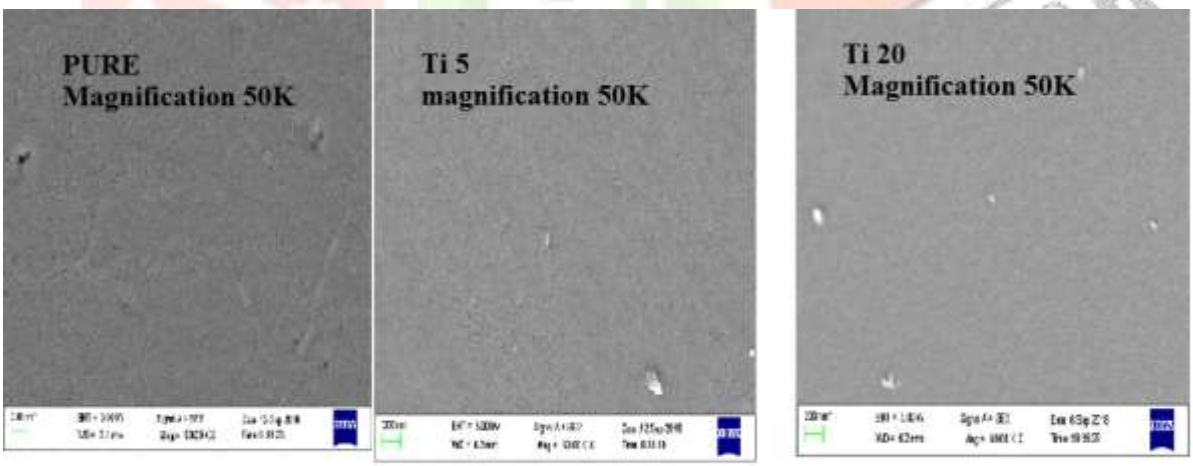

Fig 3 SEM images of 39.5SiO_2-30 B_2O_3-xNa_2O-(30-x)K_2O -0.5TiO_2 Glasses

3.2.2Energy Dispersive X-ray Spectrum Analysis (EDX)

Energy Dispersive X-Ray Spectroscopy (EDS or EDX) is a chemical microanalysis technique frequently employed alongside scanning electron microscopy (SEM). This method involves detecting X-rays emitted from a sample when it is bombarded by an electron beam, enabling the characterization of the elemental

composition of the analysed volume. EDS is capable of analysing features or phases as small as 1 µm or even smaller.

In EDS, the X-ray detector measures the relative abundance of emitted X-rays in relation to their energy. The resulting spectrum, which plots X-ray energy against counts, is then evaluated to determine the elemental composition of the sampled volume[2, 3].

For instance, EDX spectra recorded for undoped, Ti_5, and Ti_{20} glasses reveal the presence of various elements within these glasses and provide insights into their proportional composition.

Table 2 . Percentage of elements in Undoped Glass

Element	weight %	Atomic %
Si	27.36	20.71
B	17.71	13.40
Na	9.95	7.53
K	6.54	4.95
O	70.54513	53.40

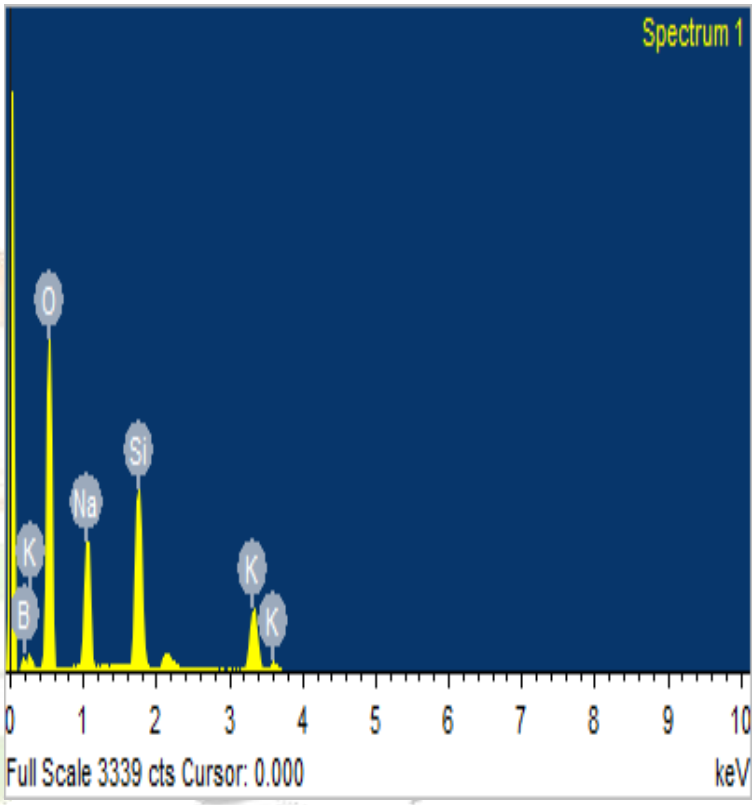

Fig 4. EDX spectrum of Undoped Glass

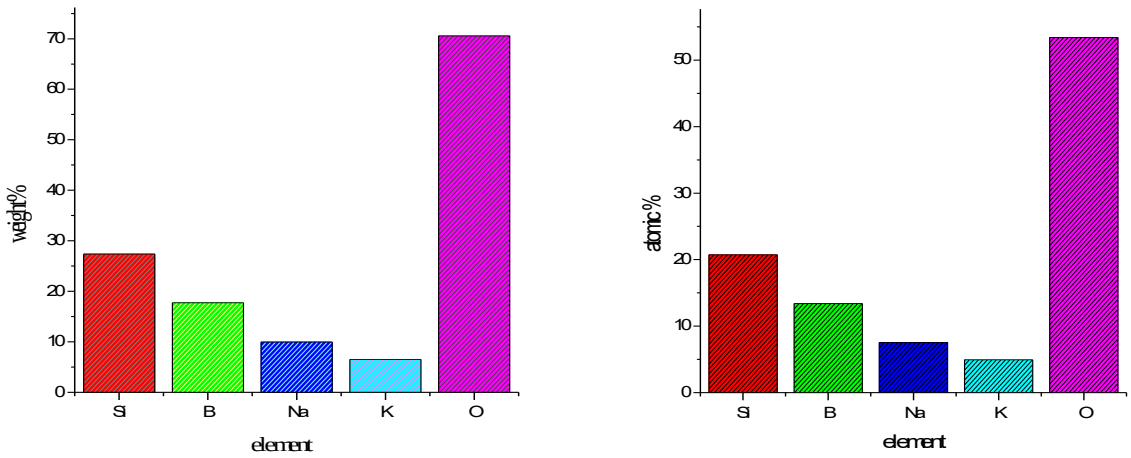

Fig. 5. Elemental analysis of undoped Glass

Table3.

Percentage of elements in

Ti^{4+} ion doped Na_2O -K_2O- B_2O_3 - SiO_2 Glasse

Element	weight %	Atomic %
Si	26.98	18.49
B	13.61	9.33
Na	16.26	7.43
K	18.19	16.62
Ti	0.43	0.30
O	68.18	47.83

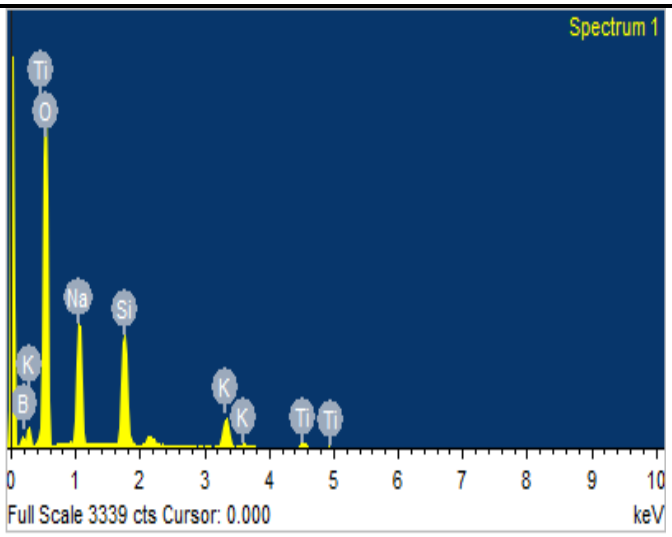

Fig .6. EDX spectrum of X_5Glass

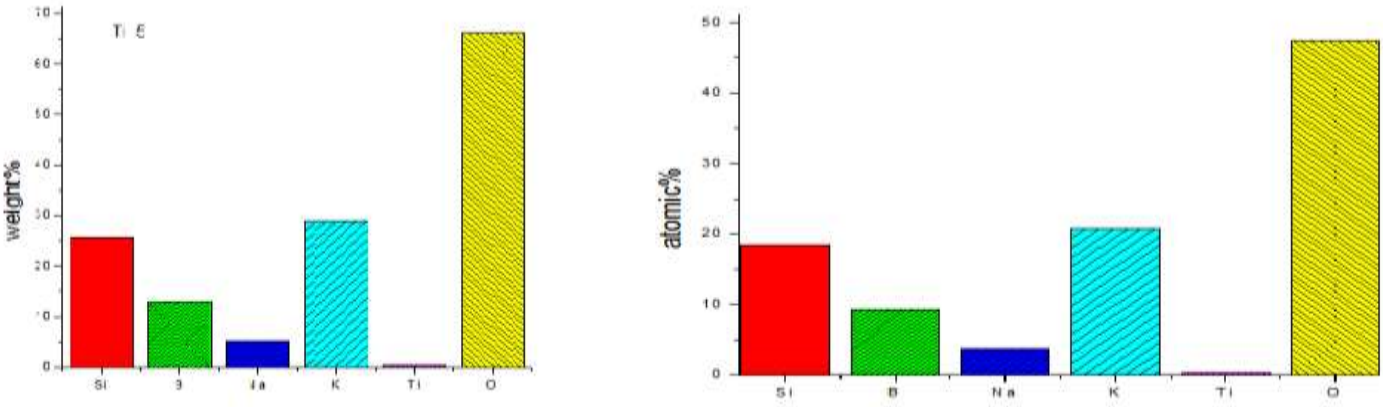

Fig.7. Elemental analysis of undoped Glass

Table 4.

Percentage of elements in Ti^{4+} ion doped X_{20} Gl

Element	weight %	Atomic %
Si	27.63	18.51
B	13.94	9.34
Na	22.20	14.87
K	12.42	8.32
Ti	0.45	0.30
O	76.18544	48.66

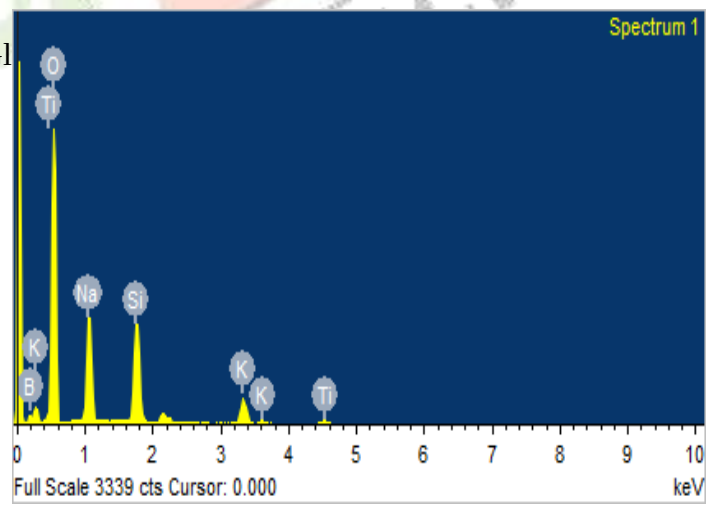

Fig .8. EDX spectrum of X_{20}Glass

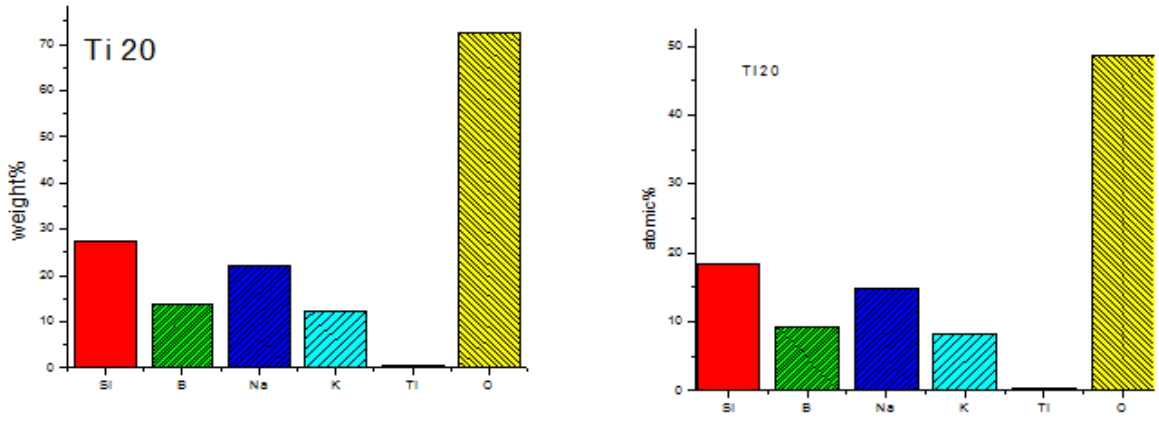

Fig.9. Elemental analysis of X_{20} Glass

4. Conclusions

A series of mixed alkali borosilicate glasses were prepared with titanium dioxide (TiO_2) as a dopant. Scanning Electron Microscopy (SEM) images reveal significant structural changes in the glass, both due to the addition of TiO_2 and variations in the concentration of alkali elements. Energy Dispersive X-Ray Spectroscopy (EDS) confirms that all the elements are present in the glass according to its intended composition.

References

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