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Investigation of gamma radiation induced changes in local structure of borosilicate glass by TDPAC and EXAFS

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Abstract Gamma radiation induced changes in local structure around the probe atom (Hafnium) were investigated in sodium barium borosilicate (NBS) glass, used for immobilization of high level liquid waste generated from the reprocessing plant at Trombay, Mumbai. The (NBS) glass was doped with ^{181}Hf as a probe for time differential perturbed angular correlation (TDPAC) spectroscopy studies, while for studies using extended X-ray absorption fine structure (EXAFS) spectroscopy, the same was doped with 0.5 and 2 % (mole %) hafnium oxide. The irradiated as well as un-irradiated glass samples were studied by TDPAC and EXAFS techniques to obtain information about the changes (if any) around the probe atom due to gamma irradiation. TDPAC spectra of unirradiated and irradiated glasses were similar and reminescent of amorphous materials, indicating negligible effect of gamma radiation on the microstructure around Hafnium probe atom, though the quaqdrupole interaction frequency (ω_Q) and asymmetry parameter (η) did show a marginal decrease in the irradiated glass compared to that in the unirradiated glass. EXAFS measurements showed a slight decrease in the Hf-O bond distance upon gamma irradiation of Hf doped NBS glass indicating densification of the glass matrix, while the cordination number around hafnium remains unchanged.

Keywords TDPAC · EXAFS · Borosilicate glass · Hafnium · Gamma radiation effects

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1 Introduction

Borosilicate glass has been considered as one of the matrices for vitrification of High Level Waste (HLW) generated during reprocessing of spent nuclear fuel owing to its chemical durability, thermal stability and ability to accommodate large quantities of the waste [1]. One of the important aspects of the vitrified waste forms is radiation damage from radioactive decay of immobilized actinides and long lived fission products. Comprehensive review of the literature on the radiation effects on borosilicate glasses used for immobilization of HLW till mid 1990s have been published by Ewing et al. [2] and later by Weber et al. [3]. Simulation of the temporal profile of the activity of the radionuclides in the waste form suggests that up to 500 years, beta radiation from fission products, such as ^{137}Cs and ^{90}Sr, will dominate the radiation effects, while subsequently alpha radiation from actinides will dominate the same. In the case of gamma radiation from ^{137}Cs, photoelectric absorption of the gamma ray photons will generate photoelectrons, which will have similar effect as beta particles, though the region affected will be wider than that affected by beta particles. Hence, the effect of gamma radiation on the glass matrix is of considerable interest. Further, though the alpha particles and their recoil heavy nuclei will have the major contribution to the creation of atomic displacements in the glass matrix, due to higher nuclear stopping, the higher dose rates in the initial period due to the beta/gamma decay results in radiolytic effects, which causes molecular oxygen formation, decomposition of O-X-O-X linkages (X=Si, B) and thereby alter the microstructure.

It has been reported that gamma radiation of glasses may lead to compaction and changes in bond angles. However, in the alkali borosilicate glasses, the volume change has been found to depend upon the alkali metal ion content, with the alkali rich glasses showing expansion and alkali depleted glases showing compaction due to the irradiation by ions [3].

Recently there have been a large number of studies on the effect of radiations on the borosilicate glasses used for immobilization of HLW. Bonfils et al. studied the effect of ^{197}Au ion iradiation on the borosilicate glass and studied the effects by Raman spectroscopy, which shows that the vibrational Raman band shifted to higher wave number with increasing ion dose, indicating the decrease in the mean Si-O-Si bond angle [4]. EPR study of gamma irradiated alkali borate glasses showed formation of hole centers associated with three fold coordinated boron [5]. FTIR studies of gamma irradiated sodium borosilicate (NBS) glasses up to 6 kGy showed increase in the BO_3 groups and non bridging oxygen (NBO) thereby loosening the glass network [6]. Mohapatra et al. carried out extensive studies on effect of gamma rays, electron beams and heavy ions on borosilicate glass using spectroscopic techniques, such as, electron paramagnetic resonance (EPR) spectroscopy, Raman spectroscopy, positron annihilation spectroscopy (PAS) and photoluminescence (PL) spectroscopy [7–10]. It has also been reported that doping of certain metal ions (viz., Co, Fe) in the glass matrix decreases the radiation effects [11]. It was concluded that by and large the glass matrix is not affected by the gamma radiation, while heavy ions create significant defects.

One of the characteristic features of radiation damage in glasses is devitrification, which is reflected in the form of narrow peaks in the X-ray diffraction pattern. However, the devitrification in the form of nano-crystalline structures might not be observed in XRD pattern. On the other hand, techniques such as time differential perturbed angular correlation (TDPAC) and extended X-ray absorption fine structure (EXAFS) spectroscopy are sensitive to changes in the local structure around the probe atom and hence can provide information about the radiation damage in the glass matrix even if there are no changes in the XRD pattern.

Time Differential Perturbed Angular Correlation (TDPAC) spectroscopy is a technique based on the interaction between the electric quadrupole moment of the intermediate nuclear level and the surrounding electric-field gradient (EFG) which results in the perturbation of the angular correlation between two cascade gamma rays emitted from the probe atom [12]. The EFG, in turn, is dependent upon the lattice structure around the probe atom and hence can be used as a measure of the local structure. The technique has been used to study atomic scale defects and diffusion in condensed matter [13]. Though the technique has been used for investigating nanostructures, and phase transitions in solids [14, 15], it has not been explored to reveal the changes in local structure in amorphous glass matrices, wherein even short range ordering due to devitrification may be reflected in the TDPAC spectra. Hence, it will be interesting to use TPDAC technique for structural characterization of glass.

Extended X-ray absorption fine structure spectroscopy (EXAFS) is another powerful technique for elucidation of local structure around an atom in solid as well as liquid systems. EXAFS is sensitive to any changes in the short range order like coordination number, type of atoms present and even to change in geometry [16]. EXAFS provide the advantage of tuning to absorption edge of particular atom in obtaining chemically selective local atomic structure out of multi component glass. It is the currently preferable technique for studying the structure of glass and its properties. For example, EXAFS of aged Plutonium doped borosilicate glasses revealed different degree of disorder around the probe atoms due to alpha dose with the long M-O bonds showing greater degree of disorder than that with shorter M-O bonds [17]. EXAFS has been used to study the structural environment around Zirconium in borosilicate glasses which revealed the distortion in coordination sphere around the metal ion with increasing metal ion content [18]. In fact, PAC and EXAFS of semiconductors has been carried out to obtain the information about radiation damage in a comparative manner [19].

In present work, sodium barium borosilicate glass used for immobilization of HLW from the reprocessing plant at Trombay, Mumbai, has been synthesized and doped with ^{181}Hf for TDPAC and 0.5 %/2 % Hafnium oxide for EXAFS measurements. The samples were irradiated with gamma rays from a ^{60}Co source and were investigated by TDPAC and EXAFS for the local structure around hafnium in the unirradiated as well as irradiated glass samples so as to reveal the change in local structure (if any) around the probe atom. ^{181}Hf was used as a probe atom for TDPAC studies owing to its favorable decay characteristics (half life 42 days and 133–482 keV $\gamma - \gamma$ cascade with intermediate level life time of 10.8 ns and spin state 5/2+). Hafnium can also be used as a chemical analogue of tetravalent actinides, viz., Pu^{4+} and stable fission products Zr^{4+} and Ce^{4+}. Hafnium doping for EXAFS measurements was followed so enable comparison with the information obtained from the TDPAC study.

2 Experimental

2.1 Sample preparation

The five component (Na_2O-BaO-B_2O_3-TiO_2-SiO_2) glass was synthesized using melt quench technique using Analytical Reagent (AR) grade oxides of sodium, barium, silicon and boron. The details of the synthesis are given elsewhere [20]. The composition is given in Table 1. For the TDPAC Study, the glass was doped with ^{181}Hf radioactive probe during the synthesis. 10 mg of Hafnium Oxychloride ($HfOCl_2.6H_2O$), sealed in quartz tube was irradiated in neutron flux of 5×10^{13} n/(sec.cm^2) in Dhruva Reactor at Trombay for 7 days. After cooling for 2 days, the sample was dissolved in 10mL of 6 M HCL. 2.5 ml

Table 1 Composition of glass used for TDPAC and EXAFS study

Sample	By weight % composition of glass					
	SiO_2	B_2O_3	Na_2O	BaO	TiO_2	HfO_2
Glass	44.16	25.97	15.58	12.99	1.3	–
0.5 % glass[a]	42.23	24.84	17.76	12.4	1.24	1.53
2 % glass[a]	40.34	23.73	16.97	11.85	1.19	5.92

[a]0.5 % and 2 % glass refer to the mole percent of Hafnium oxide in glass.

of this solution was doped in 10 g of glass matrix, which corresponds to Hafnium concentration of 0.01 wt%. For preparation of Hf-doped glass samples for EXAFS measurements, solid Hafnium Oxychloride was used. The doping level of ^{181}Hf into glasses for the TDPAC study was at trace level (due to high activity of ^{181}Hf) and at this level of non radioactive Hf it is difficult to get X-ray absorption signal even in fluorescence mode. Hence, for EXAFS measurements the Hafnium oxide content was increased to 0.5 % and 2 % in order to obtain better signal in the spectra. After synthesis, the glass samples were crushed to coarse powder. One part of the glass samples of EXAFS and TDPAC study was subjected to ^{60}Co gamma ray irradiation using Gamma Chamber GC-5000 (ISOMED-BRIT, INDIA) for a month. The dose rate was 1.6 kGy/hr measured with Fricky dosimeter. The cumulative gamma dose imparted to both the sets of samples was 1.2 MGy.

2.2 TDPAC measurements

A TDPAC facility was developed in-house using three $LaBr_3$(Ce) detectors having energy resolution of 3 % at 661 keV. The instrument is essentially a fast-slow coincidence set up which provides the coincidence spectra between selected gamma lines of a cascade, viz., 133 and 482 keV gamma lines of ^{181}Hf. The $\gamma - \gamma$ cascade at (133–482) keV, populated by the beta decay of ^{181}Hf, was used to measure the quadrupole interaction of the +5/2 state with the surrounding electric field gradient. The time resolution of the instrument was found to be 1 ns for the 133–482 keV gamma ray pair. The time calibration of the analyser was 125 ps/channel. The four parameter data acquisition system was used to store the event by event coincidence data in list mode, which was analyzed after putting proper energy gates. Due to good energy resolution of $LaBr_3$(Ce) detectors, the energy gating could be done without any interference from the neighboring gamma lines.

TDPAC measurements were performed on unirradiated and irradiated glass samples doped with ^{181}Hf. The coincidence spectra at two angles (90° and 180°) between detectors were used to obtain the TDPAC spectra R(t),

$$R(t) = 2\frac{W(180,t) - W(90,t)}{W(180,t) + 2W(90,t)} = A22G22(t) \quad (1)$$

where A22 is the directional correlation coefficient for the $\gamma - \gamma$ cascade and G22(t) is the time dependent perturbation factor. The TDPAC spectra were analysed by the DEPACK software to obtain the hyperfine interaction parameters [21].

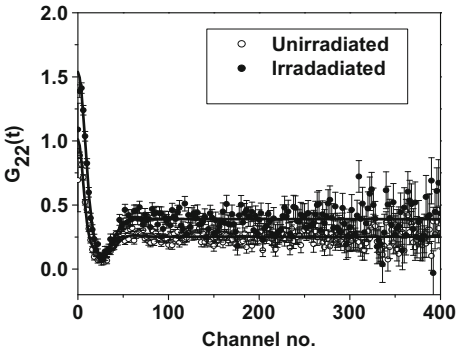

Fig. 1 TDPAC spectrum of ^{181}Hf dopes glass before and after γ-irradiation. *Solid lines* represent the fit of (2) to the experimental data

2.3 EXAFS measurements

EXAFS measurements were performed for the unirradiated and irradiated samples around Hf L_3 edge (9.56 keV) in fluorescence mode using the Scanning EXAFS Beamline (BL-9) of the INDUS-2 Synchrotron Source (2.5 GeV, 100 mA) at the Raja Ramanna Centre for Advanced Technology (RRCAT), Indore, India [22]. For measurements in the fluorescence mode, the sample was placed at 45° to the incident X-ray beam and the fluorescence signal (I_f) was detected using a Si drift detector placed at 90° to the incident X-ray beam. The EXAFS spectra of the samples at Hf L_3 edge were recorded in the energy range 9470–10180 eV with energy steps of 5 eV in the pre-edge region, 0.5 eV in the edge region and with incremental energy steps such that $\Delta k = 0.05$ A$^{\circ -1}$ in the EXAFS region.

The analysis of EXAFS data was carried out using the suite of programmes within the IFEFFIT software package [23], which was used for preparing the χ(k) vs k spectra as well as for fitting of the data to EXAFS equation to obtain the metric parameters, viz., bond distances, co-ordination numbers (including scattering amplitudes) and disorder (Debye-Waller) factors (σ^2), which give the mean-square fluctuations in the distances.

3 Results and discussion

3.1 TDPAC

Figure 1 shows the TDPAC spectrum of the unirradiated and irradiated base glass sample doped with ^{181}Hf. The corresponding Fourier transforms are shown in Fig. 2, which shows a broad frequency distribution reminiscent of amorphous materials without any well defined crystal structure. The following expression for the perturbation factor was fitted to experimental data:

$$G22(t) = \sum_{n=0}^{3} S_{2n} Cos\omega_n t.e^{-\delta^2\omega_n^2 t^2} \tag{2}$$

Where ω_n's are the interaction frequencies and S_{2n} are the corresponding amplitudes. δ is the parameter representing the distribution of frequency around ω_n. The distribution of interaction frequencies was assumed to be Gaussian. ω_n's are related to quadrupole interaction frequency

$$(\omega_Q) \text{ as, } \omega_n = g_n(\eta)\omega_Q$$

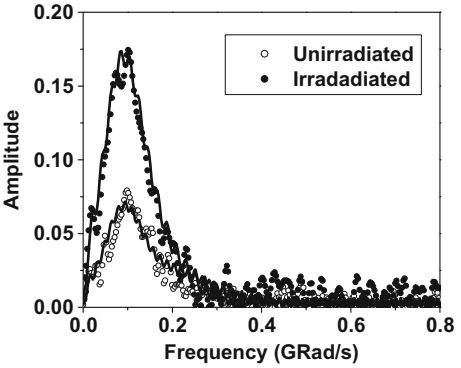

Fig. 2 Fourier Transform of TDPAC spectrum of glass before and after γ-irradiation. *Solid lines* represent fited data

Table 2 TDPAC results of ^{181}Hf-doped NBS glass samples

Sample	ω_Q (Mrad/s)	δ	η	χ^2
Glass sample	94 (1)	6.9 (0.2)	0.61 (0.06)	0.95
Irr. glass sample	83 (1)	7.9 (0.3)	0.57 (0.06)	1.07
Defect free pure HfO_2 [24]	124 (5)	3.3 (3) %	0.35 (0.01)	

$$\text{where } \omega_Q = \frac{eQV_{zz}}{4I(2I-1)} \tag{3}$$

Q is the electric quadrupole moment of the nucleus in the intermediate state and V_{zz} is the electric field gradient (EFG). The coefficient $g_n(\eta)$ is a function of asymmetry parameter η, defined as:

$$\eta = \frac{|V_{xx} - V_{yy}|}{V_{zz}} \tag{4}$$

Where Vkk (k = x,y,z) are the principal component of the diagonalized EFG tensor along respective direction. Table 2 gives the parameters obtained after the fitting. The corresponding data for pure HfO_2 are also given in the table [24]. The ω_Q in the unirradiated glass is much less than that for the defect free HfO_2, indicating the absence of HfO_2 grains in the glass matrix. The Table 2 shows a marginal decrease in ω_Q and η in the irradiated glass compared to that in the unirradiated glass, accompanied by an increase in the width of the frequency distribution (δ). However, in view of the large width of the distribution in ω_Q, it is not possible to derive any conclusion about the effect of gamma radiation in the NBS.

3.2 EXAFS

Figure 3 shows the experimental EXAFS ($\mu(E)$ versus E) spectra of the Hf-doped glass samples. The χ(k) spectra up to k range of 2–8 Å^{-1} were Fourier transformed into R space and fitted using the IFEFFIT suite of software to obtain the metric parameters. The short k range is due to the glassy nature of the samples which in turn reduces the real space resolution. The amplitude reduction factor (S_o^2) is determined to be 0.7 from the fitting of standard HfO_2 standard sample. Figures 4 and 5 show the experimental χ(R) vs R of the

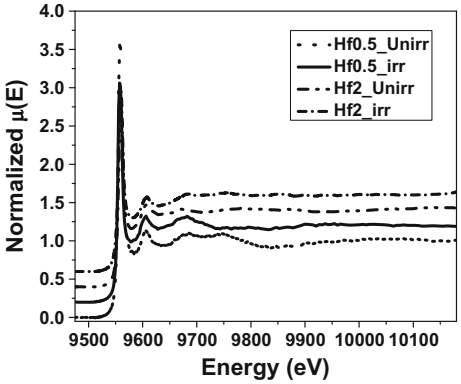

Fig. 3 EXAFS spectrum of unirradiated and irradiated 0.5 % and 2 % HfO_2 glass samples

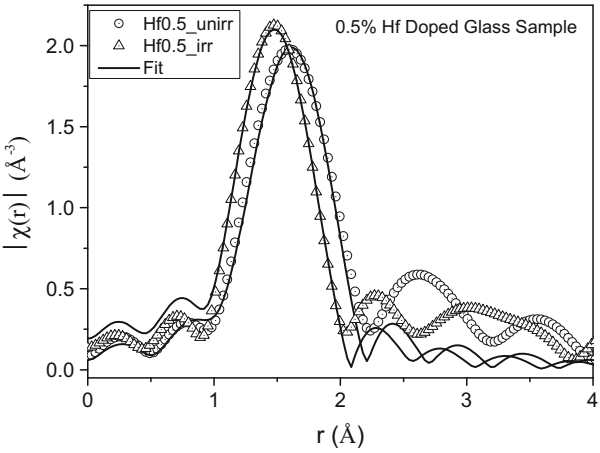

Fig. 4 $\chi(r)$ versus r spectra of the 0.5 % HfO_2-doped unirradiated and irradiated glass sample

0.5 % and 2 % HfO_2-doped glass samples, respectively. The fit parameters for the first coordination shell around Hafnium are given in Table 3.

It can be seen that the Hf-O bond distance in unirradiated NBS glasses is around 2.06 Å which is close to the Zr-O bond distance of 2.09 Å observed in EXAFS of Zirconium doped borosilicate glasses [18] as well as the Hf-O bond distance (2.1 Å) observed in the case of HfO_2 powder [25]. The Hf-O bond distance in irradiated 0.5 % Hf doped NBS glasses has been found to be less than that in unirradiated glass. This suggests slight compaction of the glass network due to the gamma irradiation. This observation is in conformity with the literature reports wherein alkali depleted regions in sodium borate glasses showed compaction in the network [3]. In the case of 2 % Hf-doped NBS glass, the decrease in the Hf-O bond distance upon irradiation is within the uncertainity of the measurement. The absence of compaction in this case could be due to presence of larger clusters of HfO_2 which resist the densification. The coordination number around Hafnium in unirradiated and irradiated NBS glass is close to 7, which is in agreement with the coordination number of Hf in monoclinic HfO_2, the low temperature phase of HfO_2. In the case of 2 % HfO_2 NBS glass

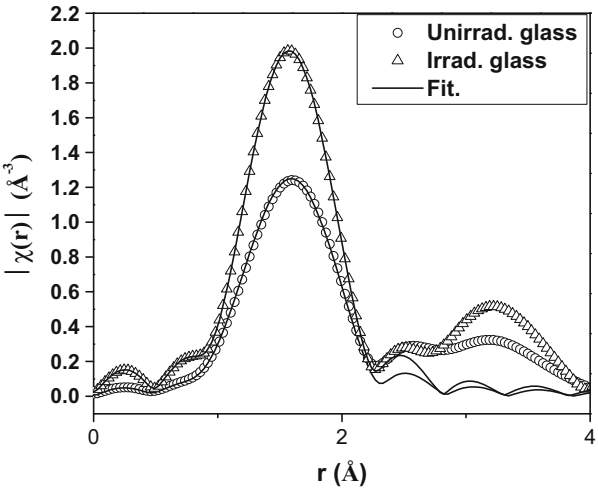

Fig. 5 $\chi(r)$ versus r spectra of the 2 % HfO_2-doped unirradiated and irradiated glass sample

Table 3 EXAFS analysis for 0.5 % and 2 % HfO_2-doped NBS glass samples

Glass sample	Hf-O		
	R (Å)	N	σ^2
0.5 % HfO_2 glass	2.05 ± 0.02	7.3 ± 0.8	0.002 ± 0.002
Irradiated 0.5 % HfO_2 glass	1.97 ± 0.01	7.1 ± 0.5	0.002 ± 0.001
2 % HfO_2 glass	2.07 ± 0.03	5.7 ± 0.8	0.004 ± 0.003
Irradiated 2 % HfO_2 glass	2.05 ± 0.01	8.6 ± 1.0	0.004 ± 0.001

also, the coordination number around Hf in both the cases (irradiated and unirradiated) is close to 7 within the experimental errors. Interestingly the Debye Waller factor in 2 % HfO_2 unirradiated NBS glass is higher than that in the case of 0.5 % HfO_2 NBS glass. This essentially indicates the distortion in the local structure around Hf at higher Hf doping, as reported by Jollivet et al., in the case of Zr-doped glasses [18]. However, the gamma irradiation does not appear to cause any change in the Debye Waller factor.

Gamma irradiaion of NBS glasses is known to produce defects, such as, boron oxygen hole centers, F centers, peroxy radical centers, E' center, etc. [9, 10]. It is also known that sodium plays an important role in reducing the radiation resistance of silica based glasses [26]. Thus it is likely that at the gamma dose of 1.2 MGy, the Trombay NBS glass is not affected by the radiation. However, the microstructural changes in terms of Hf-O bond distances being shortened do take place at these doses. Further, the Hafnium concentration in the case of TDPAC measurements is at <0.1 % which is much less than that in the case of EXAS measurements. Thus it would be interesting to carry out TDPAC measuresements with higher (0.5 and 2 %) concentration of Hafnium and compare the results with that obained from the EXAFS study, so that any distortion in the structure around hafnium could be observed as seen in the case of Zr-doped glasses [18].

4 Conclusions

Gamma irradiation of sodium borosilicate glasses used for immobilization of high level waste at Trombay up to a dose of 1.2 MGy appears to decrease the quadrupole interaction frequency as well as the asymmetry of the electric field gradient around Hafnium as observed from TDPAC measurements. However, the large width of the frequency distribution precludes any definite conclusion about the radiation induced changes in the glass. On the other hand, EXAFS measurements around Hf L_3 edge on 0.5 % Hf-doped glass show shortening of Hf-O bond distance in the irradiated glass compared to the unirradiated glass. In the case of 2 % Hf-doped glass, the change in the Hf-O bond distance after irradiation is within the uncertainity of EXAFS measurements. The coordination number around Hf is not affected by gamma irradiation and is in conformity with seven-fold coordinated Hf in monoclinic HfO_2. The Debye Waller factor also remains nearly constant upon gamma irradiation.

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