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Ion implantation in silicate glasses

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This review examines the effects of ion implantation on the physical properties of silicate glasses, the compositional modifications that can be brought about, and the use of metal implants to form colloidal nanosize particles for increasing the nonlinear refractive index.

1. INTRODUCTION

Ion implantation allows the experimentalist a means of gaining new degrees of freedom with respect to the effects of radiation on glasses (as in other materials). The slowing-down of energetic ions in materials results in the deposition of energy into electronic processes (excitation and ionization) and into collisional events (Coulomb and hard-sphere interactions). The percentage of the ion energy going into these two processes depends upon the magnitude of the ion velocity relative to the velocity of the target electrons. These processes have been well characterized and described [1,2]. Unlike metals and semiconductors, electronic energy deposition may be important in modifying the physical properties of glasses (and polymers); the efficiency of utilization is dependent upon the particular glass system.

The basic radiation-induced (ions, e^- , n , γ -rays) defect of importance (E' -center) in high-purity fused silica glasses has been well described beginning primarily through a combination of optical absorption measurements and electron paramagnetic resonance (EPR) studies on irradiated crystalline quartz and fused silica by R.A. Weeks [3] and Weeks and Nelson [4-6]. These studies have been greatly extended by the work of D. L. Griscom and E.J. Friebele (see, e.g., [7, 8]). The B_2 -center, variously described as a

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neutral oxygen vacancy [9] or an unrelaxed oxygen vacancy [10], is also a primary radiation-induced defect. These defects and their modifications imposed by glass structure variations suffice to explain most of the ion-implantation induced effects to be discussed in the following sections.

2. ION IMPLANTATION EFFECTS

2.1 Absorption, Reflectance, and Thermoluminescence

Figure 1 shows both the E'-center and the B₂-center development induced by ion implantation, as well as the dependence of the B₂-center concentration on collisional processes. The optical density induced by H, He, Ar, Kr, and Xe at various energies and fluences (curves displaced for clarity). The collisional energy deposited increases with ion mass and it is clear that the B₂-center concentration (negligible for H and He) is dependent on energy into displacement encounters. More detailed discussions of the B₂-center can be found in the references previously cited [9, 10].

The vibrational modes of silica glass and their modification by ion implantation can be usefully observed using infrared reflection spectroscopy. Figure 2 shows the reflectance over the wavenumber region from 1300-400 cm⁻¹. The unimplanted side of the sample (CFS 7940) has a reflectance maximum at 1128 cm⁻¹ which corresponds to the Si-O-Si stretching vibration. Implantation of 3.52×10^{16} 200 keV Ar⁺⁺/cm² shifts this maximum to lower wavenumber and decreases the intensity. The shift is due to changes in Si-O bond angle and to the creation of dangling Si-O bonds. Si-O, Si-OH, and Si-OD modes at 1015 cm⁻¹, 985 cm⁻¹, and 960 cm⁻¹ have been identified for these modes in implanted porous silica glass [11]. More recent studies of the effects of ion implantation on lattice modes should be consulted [12, 13].

Irradiated glasses are known to luminesce when heated and have been studied for many years [14]. Fused silica Types I-IV contain varying amounts of OH and impurities dependent upon the processing technique. Figure 3 shows the variation in thermoluminescence (TL) spectra for the 4 types room-temperature implanted with 150 keV H at the indicated fluences [15]. The common dominant TL feature

is the peak near 150 °C. Figure 4 shows the integrated TL intensity of this peak (Suprasil W1) for various ions and energies plotted against energy into collisional processes (keV/cm^2). The data coincide over the initial linear rise portion of the curves. At high fluences ($\approx 10^{19} \text{ keV}/\text{cm}^2$) the TL intensity decreases which is characteristic of many physical properties of fused silica at high collisional energies [16]. The 300 °C peak was tentatively associated with the excitation of a center involving Al.

2.2 Volume Changes

As in most materials, glasses undergo volumetric changes as a result of ion implantation. These changes in density alter the refractive index, surface stress, and hardness, as well as the chemical reactivity. These changes occur within the shallow implanted layer which is typically on the order of a micron or less in depth. The undamaged substrate acts to oppose the volumetric change; compaction in the layer results in a tensile surface stress while expansion generates a compressive stress. Changes in density result in a dilatation normal to the surface which can be measured with a profilometer. These instruments can register changes as small as $5\text{\AA}$. An alternative method for measuring surface stress and volumetric change is the cantilever-beam technique. Thin samples which are fixed at one end will bend toward or away from the ion beam direction according to the dilatation sign. The movement of the free end of the beam can be sensitively measured by a method developed by EerNisse [17,18]; the displacement can be converted into lateral stress and to the $\Delta V/V$ dilatation through a knowledge of the elastic constants of the material.

Although conventional wisdom asserts that fused silica glass compacts under ion implantation, the sign of the volume dilatation may depend on the fluence level and the impurity concentration. Shelby has shown [19] that hydrogen-loaded silica expands under ^{60}Co γ -ray irradiation. The determining factor influencing volume expansion or compaction is the ability for network atoms to move into the interstices. When this movement is not hindered, compaction results when bonds are broken either by ionization or by direct displacement events. If, however, the void space is occupied by H, the H can react with the broken bonds to form Si-OH or Si-H. For this case, densification is prevented, the network relaxes instead, and a net expansion occurs. Figure 5 shows cantilever beam measurements for Pb, Xe, and He implanted into

Suprasil 1 [20]. For the Pb implants, the stress is tensile (compaction) over the fluence range utilized. For the He and Xe implants, however, the induced stress is compressive (expansion) at low fluences, becoming tensile for increasing fluence levels. In Fig. 6, the data is plotted as a function of the energy deposited into collisional processes (keV/cm^3). The good correspondence of the data sets when plotted in this manner is evident and is in agreement with the observation of EerNisse [18] that the collisional component of the ion energy, for fused silica, is more important than the ionizing component (by a factor of ≈ 200); this was reinforced by work of Presby and Brown [21]. The stress relief seen in Fig. 6 at about $10^{21} \text{ keV}/\text{cm}^3$ occurs at fluences for which ion track overlap may be expected; other physical property changes show a similar behavior [16]. The dominance of the collisional component is not found in borosilicate glasses; Fig. 7 presents surface stress data for the same implants as Fig. 6 into the borosilicate glass Pyrex [20]. The data are seen to have excellent agreement when plotted as a function of energy deposition into electronic processes. The same functional dependence was found for the borosilicate nuclear waste glass PNL 76-68 [20] and for the borosilicate glasses $(1-x) \text{Na}_2\text{O} \cdot x \text{B}_2\text{O}_3 \cdot 3 \text{SiO}_2$ [22]. In the latter work, the fluence regime over which expansion occurred was found to be proportional to the alkali concentration; this observation is understandable as an extension of the effects of interstitial H on the volumetric changes in fused silica given above.

2.2.1 Refractive Index Changes

Changes in refractive index occur as a result of the changes in density. For fused silica, the maximum change in index is an increase of about 1-2%. Such changes are quite enough to provide waveguiding regions as first demonstrated by Schineller et al. [23] for 1.5 MeV He implanted in fused silica; the collisional damage at the end of the track formed a high-index region surrounded by undamaged low-index silica and formed a guide for $6238\text{\AA}$ He-Ne light. Larger changes in index are possible when the implanted ion interacts chemically with the network atoms to form a different phase. Webb and Townsend [24] were the first to show that implanted N increased the index by about 6% for fluences on the order of $2 \times 10^{16} \text{ ions}/\text{cm}^2$ and suggested that the formation of silicon oxynitride or silicon nitride might be responsible. Subsequent work by Wang et al. [25] and Naik [26,27] reinforced

this speculation and has been completely verified by a number of experimental techniques and investigators [28-32] which established the formation of SiO_xN_y for N implants and also B_2O_3 for B implantation.

2.2.2 Hardness

Because of the induced surface stress, it is to be expected that there will also be changes in hardness. These changes were noted in early work by Jensen et al. [33] for soda-lime glass implanted with 480 keV H. Their diamond indenter (Vickers; 60 g load) measurements showed a softening with fluence which reached a maximum softness at about $5 \times 10^{20} \text{ keV/cm}^3$ (collisional energy deposition) followed by increased hardness with increasing fluence. This progression was reflected in the median-vent surface cracking; longer cracks accompanying increasing tensile stress (compaction) and shorter track traces at higher fluences. Figure 8 compares Knoop (shallow indenter; 15 g load) measurements [34] with surface stress data [18] for fused silica implanted with 500 keV Ar. The comparison shows a close correspondence between increasing tensile stress and softening with maxima occurring again at about $5 \times 10^{20} \text{ keV/cm}^3$. Note that at still higher deposition energies, the hardness increases to values greater than that of the original glass (collisional energy deposition of $\approx 3 \times 10^{22} \text{ keV/cm}^3$) and then decreases again. This reversal in hardness is also found in the fracture toughness data of Battaglin [35] for 100 keV Ar on soda-lime glass at about the same energy deposition value.

The changes in hardness at a deposition value on the order of 10^{23} keV/cm^3 may be indicative of a fairly abrupt change in the damaged depth region at this level. In fact, a variety of different experiments have shown [12] that the depth of the damaged region in silicate glasses can extend to twice the theoretical range (TRIM [36]: $R_p + \Delta R_p$) for collisional energy deposition values greater than $\approx 10^{23} \text{ keV/cm}^3$. A compilation of such data is shown in Fig. 9. The reason for the extended damage range indicated by the data of Fig. 9 has been speculated [12] to be associated with the high defect concentration ($\approx 6 \text{ dpa}$) and enhanced defect transport due to the large strain gradient between the damaged layer and the undamaged substrate, but this matter is still under investigation.

It is important to note that Weber and Matzke [37] have measured the hardness of a borosilicate nuclear waste glass (PNL 76-68) resulting from the 5 MeV α -particles emanating from a surface source of $^{238}\text{PuO}_2$. They found a decrease in hardness with increasing energy deposition (electronic processes; dominating as discussed above) and over the deposition range, equivalent to $\cong 10^{22}$ keV/cm³, the data is consistent with the stress measurements made on this glass with He, Xe, and Pb implants and indicates the usefulness of ion implantation in simulating α -decay processes.

2.3 Compositional Changes

The chemical incorporation of the implanted species into the glass network can occur and has already been touched upon in the discussion of refractive index changes (B and N implants in silica glass). Preferential sputtering can also alter the near-surface composition but will not be discussed here. In the following we will present the modifications in composition which can be brought about by implantation-induced alkali depletion and by crystallization.

2.3.1 Alkali Depletion

Alkali depletion in an ion-implanted soda-lime glass was probably the cause for the observation of a decreased refractive index found by Hines [38] in early work. This effect was noted and related to Na depletion by later work of Geotti-Bianchini et al. [39]. It is now known to be a common occurrence in such glasses when subjected to electron- or ion-bombardment. The experimental observations [40,41] were that the depletion for charged-particle irradiation resulted in a movement of alkali from a depth corresponding approximately to the range of the particles. Such effects make Auger electron spectroscopy (AES) difficult because of the rapid disappearance of the alkali signal with time due to the electron analysis beam. Difficulties are also encountered in secondary-ion mass spectrometry (SIMS) which employs ion-milling. For electron irradiation, the alkali from the surface accumulates at a depth near the electron range while for MeV protons the movement is toward the surface. The alkali migration in both of these cases has been explained in terms of an ordinary and field assisted diffusion [42-45]. For heavy ions incident on alkali-containing glass, the alkali is first removed from a depth corresponding to the ion

range and at progressively higher fluences the entire near-surface region is depleted. This is shown by the RBS K depth profiles in Fig. 10 for a 250 keV Xe implant into a 12 K₂O • 88 SiO₂ glass as a function of ion fluence [46]. The results for heavy-ion depletion have been explained as due to radiation enhanced diffusion within the damaged region with a migration to the surface due to a surface layer depletion of alkali [46, 47]. A practical application of this effect is the production of an anti-reflection layer due to the decreased refractive index in the depleted region [48].

2.3.2 Crystallization Phenomena

The controlled conversion of a lithia-alumina-silica (LAS) glass surface into a polycrystalline glass ceramic was accomplished through the implantation of Au [49] or Ag [50] ions followed by suitable heat treatment. Conventional bulk crystallization of such glasses was carried out by the introduction of photoreducible ions (Au, Ag, Pt, Cu) into the LAS glass melt; the metal ions were reduced by incident absorbed light [51]. For both cases, aggregation of the metal atoms was accelerated by elevated temperatures and for certain critical dimensions the catalyzed precipitation of crystalline material occurs in accordance with the phase diagram for the glass. The crystallization can be monitored by infrared reflection as shown in Fig. 11 for a 1×10^{16} 285 keV Au/cm² implant followed by annealing at 550 °C for the indicated times. The major IR reflection feature is the Si-O-Si stretch vibration near 1100 cm⁻¹. The appearance of crystalline features occurs after about 35 min and the peaks can be associated with a lithium metasilicate phase. It is important to note that no crystallization occurs on the reverse unimplanted sample surface. The critical metal colloid particle radius for crystallization was determined to be about 35 Å. Annealing metal doped LAS bulk glass at higher temperatures was shown by Stookey [52] to result in the further precipitation of β-spodumene and quartz (Corning Pyroceram). This was also found to occur in the glass of Fig. 11 after 90 min at 750 °C. The base LAS glass had a Knoop hardness of 522. After the 750 °C anneal the hardness was 626. The Corning Pyroceram glass has a hardness of 696. The high mobility of implanted Ag makes it more difficult to achieve crystallization. It was found [50] that small colloids were formed directly upon implantation and that the critical radius for crystalline precipitation was exceeded at temperatures which were too low for such phase separation (minimum

temperature $\cong 550$ °C). Crystallization could be achieved by implanting at low temperature (77 °K) or at low beam current (200-300 nA/cm²) in order to prevent the dispersion of Ag.

It has also been shown [53] that a fine-grained (1-5 μ m) polycrystalline α -quartz surface can be formed on fused silica by 250 keV Li-ion implantation at fluences from 5×10^{16} - 1×10^{17} Li/cm² followed by annealing at 750 °C for 30 minutes. The conversion to α -quartz was verified by x-ray diffraction and IR reflection spectroscopy. Knoop hardness measurements for various loads allowed a depth profile of the hardness to be determined; the hardness attained the value expected for crystalline quartz at a depth approximating the layer thickness.

The results obtained for Li-ion implantation into fused silica led to investigations into the effects of inert gas implantation into a $\text{Li}_2\text{O} \cdot 2 \text{SiO}_2$ glass [54]. Elastic recoil detection (ERD) measurements, shown in Fig. 12, illustrate the depletion of Li from a depth corresponding to the range of the incident 5×10^{16} 250 keV Xe/cm² and their replacement by H which diffuses in from the surface. Annealing to 550 °C results in the formation of crystalline lithium disilicate in the implanted region; no changes were seen in the unimplanted regions. This result can be understood from a consideration of the phase diagram [55] for the $\text{Li}_2\text{O} - \text{SiO}_2$ system (Fig. 13). From the position of the $\text{Li}_2\text{O} \cdot 2 \text{SiO}_2$ composition it is clear that a loss of ≥ 2.2 mol. % Li_2O , easily exceeded by the Li loss shown in Fig. 12, would result in a composition which could phase-separate at temperatures ≤ 500 °C and result in the crystallization phenomena noted.

Some of the possible advantages of generating surface crystallization by ion implantation are, (1) precision control of the number of implanted species and therefore good reproducibility of results, (2) variation of fluence levels and the use of multiple-energy implants allows control of the depth distribution and concentration of implanted ions, (3) selected area crystallization by suitable masking, and (4) the change in index of refraction of the crystallized layer might be utilized to serve as an anti-reflection layer.

2.4 Increased Nonlinear Optical Properties

There is presently intense interest in the possibility of developing a computer based on an all-optical photonic switch which could replace electronic transistor switching. Such a switch would be competitive if switching times of less than 100 ps could be demonstrated at an energy consumption less than 1 pJ [56]. The suggestion has been made [57] that the inherent optical nonlinearities of glass could be utilized for switches of this type. An all-optical switch is one where the switching is accomplished through changes in the refractive index that are proportional to the light intensity; the third-order susceptibility, $\chi^{(3)}$, is the non-linearity which provides this feature. The incorporation of semiconductor microcrystallites (e.g., $\text{CdS}_x\text{Se}_{1-x}$) enhances the third-order response. However, it would be desirable to use a method in which the non-linearity could be confined to specific patterned regions so as to provide effective design of optical circuitry. This clearly calls for the use of ion implantation and, serendipitously from the point of view of Section 2.3.2 of this paper, it happens that metal colloidal particles can also introduce the desired third-order enhancement. Ion-implanted metals (Au, Ag, Cu) a few nm in size exhibit an electron plasmon surface resonance which depends on the optical constants of the metal and the refractive index of the host glass. The measured optical absorption of the particles and the peak position can be handled within the Mie formalism and with suitable corrections for the mean free path and additional magnetic dipole terms (for larger radii particles) the colloidal radii can be determined [50]. Figure 14 shows the calculated Mie extinction coefficient for Ag colloids of various radii as a function of wavelength while Fig. 15 shows the experimental optical absorption induced by 270 keV Ag implanted into CFS 7940 at the indicated fluences. The beam current was minimized to prevent Ag migration. Figure 15 shows that the colloid size for fluences above about 1×10^{16} Ag/cm² is on the order of a few nanometers [58]

Ricard et al. [59] were the first to measure the non-linear properties of such colloids (Ag, Au) in bulk silica and other papers from the same group [60, 61] extended these measurements. The optical Kerr susceptibility was found to be 2-3 orders of magnitude greater than that of the bulk metals. Fukumi et al. [62] implanted 1×10^{17} 1.7 MeV Au/cm² into silica glass and found that the measured third-order susceptibility was of the same order as that for Au added to the melt [61]. Recent work on implanted Cu [63] and Pb [64] have shown that these implants are useful in achieving enhanced values of $\chi^{(3)}$. These

implantation results seem to open the way for advances in all-optical switching devices, e.g., in implanting metal colloids into coupled waveguides acting as a directional coupler.

3. SUMMARY REMARKS

This brief review does not do justice to the large body of work that has formed the field of ion beam modification of glasses over the past 30 years. The author apologizes for many omissions. Rapid advances in this field are being made at Padova University, Vanderbilt University, Oak Ridge National Laboratory, Nagoya and Tokyo Institutes of Technology and, on a more modest scale, at Sandia National Laboratories. The biennial proceedings of the Radiation Effects in Insulators Conferences (inaugurated in 1961) should be consulted for details. A review was published on the general field of ion beam modification of insulating materials in 1983 [65].

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FIGURE CAPTIONS

Fig. 1 Optical density vs wavelength (nm) for CFS 7940 silica implanted with H, He, Ar, Kr and Xe ions at various energies and fluences (after Ref. 9).

Fig. 2 Infrared reflectance vs wavenumber for CFS 7940 silica implanted with 200 keV Ar⁺⁺ as compared with the unimplanted reference side of the sample.

Fig. 3 Thermoluminescence (TL) intensity vs temperature for fused silica types I-IV as a function of 150 keV implanted H at various fluences (after Ref. 15).

Fig. 4 Integrated 150 °C TL intensity vs energy into atomic processes for Suprasil W1 silica implanted with the indicated ions at various energies and fluences (after Ref. 15).

Fig. 5 Integrated stress vs ion fluence for Suprasil 1 silica implanted with He, Xe and Pb at the indicated energies (after Ref. 20).

Fig. 6 The data of Fig. 5 plotted as stress vs energy deposition into atomic (collisional) processes (after Ref. 20).

Fig. 7 Integrated stress vs energy into electronic processes for Pyrex implanted with He, Xe and Pb at the indicated energies (after Ref. 20).

Fig. 8 Lateral stress and square of Knoop indenter diagonal vs collisional energy deposition for 500 keV Ar on CFS 7940 silica (after Refs. 18, 34).

Fig. 9 Ratio of damage range to $(R_p + \Delta R_p)$ vs collisional energy deposition for various experiments on implanted silica glasses (after Ref. 12).

Fig. 10 Rutherford backscattering (RBS) K depth profiles for 250 keV Xe implanted into a 12 K₂O • 88 SiO₂ glass at the indicated fluence levels. The spectra are displaced for clarity (after Ref. 46).

Fig. 11 IR reflectance vs wavenumber for LAS glass implanted with 1×10^{16} 285 keV Au/cm² and annealed at 550 °C for the indicated times (after Ref. 49).

Fig. 12 Comparison of the Li and H elastic recoil detection (ERD) depth profiles in the near-surface region of Li₂O • SiO₂ glass implanted with 5×10^{16} 250 keV Xe/cm² (after Ref. 54).

Fig. 13 Phase diagram of the lithia-silica system showing the region of phase separation (after Ref. 55).

Fig. 14 Calculated Ag extinction coefficient (Mie theory) vs wavelength for Ag colloids in silica glass for the indicated radii (after Ref. 58).

Fig. 15 Optical absorption vs wavelength for CFS 7940 silica after implantation with 270 keV Ag at the indicated fluences (after Ref. 58).

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