Characterization of Defects in Waveguides Formed by Electron Irradiation of Silica-on-Silicon

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***Abstract*—Absorption spectroscopy and electron spin resonance are used to characterize optical waveguides formed by electron irradiation of plasma-enhanced chemical vapor deposition (PECVD) silica-on-silicon. Nonbridging oxygen hole centers and E'_γ defect centers are positively identified in undoped films. Evidence for peroxy radical and phosphorus oxygen hole centers is also found in phosphorus-doped samples. This is the first time that defects have been unambiguously identified in such guides. The charge-dose dependence of the E' center density follows a saturating exponential curve well correlated with refractive index changes previously reported, implying that a single first order process is responsible for both effects.**

***Index Terms*—Defect centers, integrated optics, optical waveguides, silica-on-silicon.**

I. Introduction

BURIED channel waveguides may be directly formed in silica-on-silicon layers by ion implantation and laser or electron irradiation [1], [2]. These processes offer the advantages of reduced fabrication complexity and a flat final surface, which may allow further vertical integration [3]–[5]. So far, electron beam irradiation has attracted little attention, apparently due to the difficulty of characterizing the refractive index changes involved. This is surprising, because the changes are similar to those obtained in photo-induced Bragg gratings in silica, which have been widely researched [6]. However, electron beam irradiation of plasma-enhanced chemical vapor deposition (PECVD) silica has already been successfully used to form a wide range of planar lightwave circuit (PLC) components, such as guides operating at 1.5 μm with propagation losses of 0.1 dB/cm, Mach–Zehnder interferometers, 1×16 Y-junction splitters and thermooptic switches [4], [5].

This paper aims to increase the information available on radiation induced effects and hence improve prospects for this guide fabrication technique. The major cause of the index change is assumed to be the creation of paramagnetic defects from precursors already present in the silica. However, the magnitude and sign of the change has been found to depend on many factors, including the glass deposition method and the presence of dopants and impurities. The radiation source and irradiation temperature are also important. Further complexity arises from the amorphous nature of silica, which allows irradiation to induce variations in bond angle distribution, as well as changes in paramagnetic and diamagnetic defect concentration, refractive index, volume and intrinsic stress. Depending on the use of any heat treatment, annealing effects may also occur. In this work, the process by which electron irradiation induces an index change in PECVD silica is investigated, using optical absorption and electron spin resonance spectroscopy. By comparing results from the two techniques, a consistent picture of the defect types and concentrations causing the index change is obtained.

Previous studies have shown, by electron spin resonance (ESR), that electron-beam irradiation of thermal silica induces oxygen vacancy (E') centers [7] and traps positive charge [8]. Results indicate that these E' centers are E'_γ-like ($O_3{\equiv}Si^\bullet$ $^+Si{\equiv}O_3$). The defect center concentration reported after electron injection into PECVD silica-on-silicon (of about 3×10^{13} electrons/cm^2) is enhanced by a factor of 10^6 compared to the concentration induced in thermal oxide films receiving the same number of electrons [9]. This effect has been attributed to PECVD films containing more precursors for E' centers than those prepared by thermal oxidation.

Since E' centers have been found in both electron irradiated thermal silica [7] and PECVD silica films into which photoemitted electrons have been injected [9], it is likely that they are also found in waveguides formed by electron irradiation of PECVD layers. This possibility, together with that of finding other defect centers such as non-bridging oxygen hole center (NBOHC) and peroxy radical defects, is investigated here.

The defects most commonly induced by irradiation of bulk silica and silica fiber samples exhibit optical absorption peaks in the ultraviolet (UV)-visible wavelength range. The E' defect absorbs most strongly at 215 nm, the NBOHC at 650 nm, and the peroxy radical at 163 nm [10]–[12]. As a preliminary investigation, the absorption spectra of channel waveguides formed by electron beam irradiation are studied in the visible region. This allows any peak at 650 nm to be observed at the same time as the tail from any intense UV peak, such as the expected E' peak. Electron spin resonance experiments are then performed on planar waveguides to clarify the source of the features found in the visible spectra. The charge-dose dependence of the defect concentrations is then examined and comparisons are made with the dose dependences of the previously reported refractive index changes. Conclusions are then drawn about the relationship between these two processes. By comparing the absorption and ESR results, observations are made about oxygen stoichiometry and precursor structures.

Manuscript received April 23, 1999; revised December 1, 1999. This work was supported in part by ESPRC.

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Publisher Item Identifier S 0733-8724(00)03032-2.

TABLE I
PARAMETERS OF SILICA FILMS INVESTIGATED

Source	Thickness (μm)	PECVD reactor	Precursors	Doping
BT	20	tube	SiH_4, NO, N_2	undoped
LETI	16	unknown	TEOS and others	10 mol % P_2O_5
BNR	32	parallel plate	SiH_4, N_2O	undoped

II. EXPERIMENTAL DETAILS

The silica-on-silicon materials investigated are all thick single layer films, and were supplied by BT Labs (England), LETI (France), and BNR Europe, Ltd., now known as Nortel (England). The LETI material was unique in being doped with 10 mol % P_2O_5. The properties of the layers are summarized in Table I. Because a thermal annealing step was known to be required to form waveguides in BNR material [4], these films were annealed at 1000 °C for 20 min prior to irradiation.

The samples used for absorption spectroscopy were straight channel guides formed by flood irradiation through an electroplated Au surface mask [4]. Irradiation was carried out at room temperature using a modified CamScan SEM, with an accelerating voltage of 25 kV and beam currents from 0.04 to 0.06 mA. The charge doses were measured using a coulomb meter. The guides were 7 μm wide, extended to a depth of around 6 μm and were arranged in groups of 8 on 300-μm centers. After irradiation, the wafers were cleaved into chips of approximately 20×35 mm. For the ESR experiments, additional planar waveguide samples 3×25 mm were prepared by flood irradiation of unmasked PECVD silica. The smaller sample size was chosen to minimise the microwave cavity loading in the spectrometer, and planar, rather than channel, guides were examined to maximize the absolute number of defects induced.

Optical absorption spectra were calculated from the ratio of the transmission measured with and without a waveguide in the measurement system. This method was preferred to cutback, since limited samples were available and the effects of variations in cleave quality of the end faces were unclear. The disadvantage of this method was that the spectra recorded included insertion losses. Transmission spectra were measured using a computer-controlled monochromator, and TE and TM mode spectra were obtained using a $\lambda/2$ plate to alter the polarization. An overlay of low-index oil was used to reduce scattering losses. The resolution of the resulting spectra ranges from 10 to 20 nm. It should be noted that normal incidence measurements were not possible because the silica films under test were deposited on opaque silicon substrates.

The absorption spectra were investigated over a visible wavelength region (390–900 nm) rather than a UV range for a number of reasons. These include the preliminary nature of the experiments and the desired investigation of both NBOHC and E' defect peaks using the same grating. Another reason was the difficulty in measuring the light transmitted at around 215 nm given; the low responsivity of the UV enhanced Si photodetector available, the high levels of absorption expected and the sparcity of samples available.

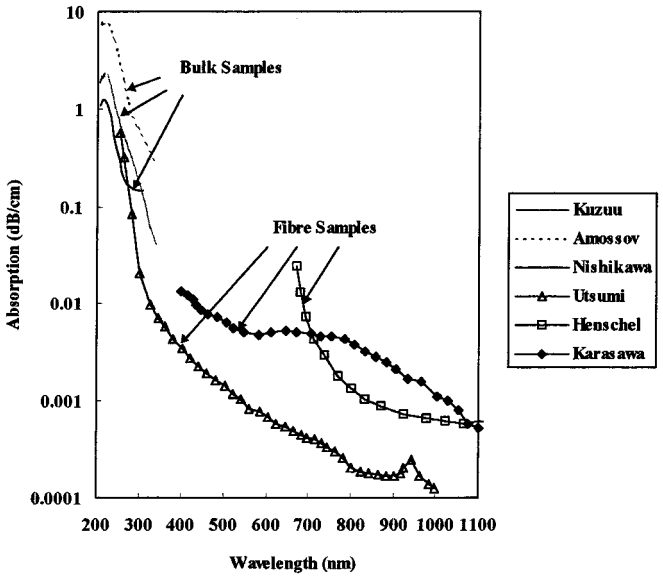

Fig. 1. Reported absorption spectra from irradiated pure silica samples. Bulk samples after [13]–[15] and fiber samples after [16]–[18].

Previous workers dealing with bulk and fiber samples have studied absorption spectra over a variety of wavelength ranges, as shown in Fig. 1 [13]–[18]. Researchers dealing with thin film silica-on-silicon subjected to ionising radiation, have previously avoided absorption spectroscopy and used ESR alone [7], [9], [19], [20]. Other workers investigating doped thin-film silica exposed to UV irradiation have used silica substrates for transverse absorption measurements [21]–[23]. This was not considered a valid approach here since the substrate material may affect the radiation sensitivity of the silica layer under investigation. In this paper, however, it is illustrated that visible absorption spectra, straightforwardly obtained from guided light, can provide a useful comparison to support ESR results.

ESR experiments were performed at room temperature using a Varian E-9 X-band microwave spectrometer with the magnetic field modulated at 100 kHz. The modulation amplitudes were set at 0.5 G for E' spectra and 2.0 G for the NBOHC and peroxy radical measurements, to avoid overmodulation. Nonsaturating microwave powers of 5 μW for the E' signals and 2 mW for the NBOHC and peroxy radical spectra were used. The absolute spin densities were deduced by comparison of a numerical double integration of the signals with that obtained from a weak pitch standard. Absolute densities were considered to be accurate within 50% and relative spin densities within 10%.

III. RESULTS AND DISCUSSION

A. Absorption Spectroscopy

The absorption results presented in this section are all for TE modes. In Fig. 2, averaged absorption spectra obtained from guides formed in BT material at doses ranging from 0.1 C/cm^2 to 0.9 C/cm^2 are shown, together with error data representing the standard deviation of the mean of a set of spectra obtained at a specific dose. It is to be noted that these spectra include losses which are described later in this section.

Three separate contributions to these spectra may be identified. These are illustrated for the guide formed using the lowest

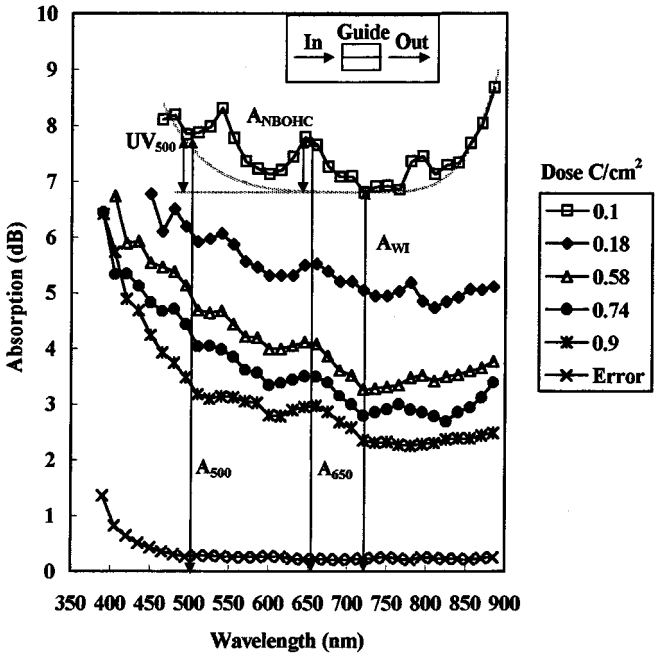

Fig. 2. Absorption spectra (averaged) from waveguides formed in BT silica, for various charge doses and a beam energy of 25 keV. Different components of the spectrum taken from a guide formed at 0.1 C/cm^2 are shown.

dose. First, at low doses, there is a downward trend in absorption with increasing wavelength. This feature is attributed to the tail of an absorption peak in the UV; the most likely cause is the E' center.

Second, there is a pair of absorption peaks at 650- and 540-nm wavelengths. Because absorption at 650 nm has previously been ascribed to NBOHC defects [11], [24] (even though these normally absorb at wavelengths closer to 630 nm), it is also attributed here to NBOHC's. This conclusion is confirmed by ESR results discussed later and by cathodoluminescence data described elsewhere [25]. Other workers have also reported that long wavelength NBOHC peaks are associated with peroxy linkage (Si-O-O-Si) or strained bond (Si-O-Si) precursors in fibers. These precursors are therefore also indicated here. The source of the small feature at 540 nm has not yet been identified, since it has only rarely been reported. Recently, however, it has been linked with absorption at 630 nm in irradiated bulk silica and is therefore also likely to be caused by NBOHC defects [26]. In phosphorus doped silica, it has also been linked with the phosphorus oxygen center, or POHC, defect [27].

Third, there is a component of loss which is wavelength-independent below 800 nm and which increases above this value. It is ascribed to losses suffered by portions of the mode travelling in unirradiated regions and to coupling and substrate losses. These have been reported to decrease with dose, due to increasing confinement [3]. They also increase with wavelength above 800 nm because of the decreasing number and confinement of the modes propagating [25]. The rapid increase in absorption with wavelength above 800 nm for the sample irradiated to 0.74 C/cm^2 was attributed to a reduced coupling efficiency.

LETI films irradiated at similar dose levels provided absorption spectra with similar characteristics. The magnitude of the UV tail was comparable with that in spectra from guides formed in BT material. However, the magnitudes of the 650- and 540-nm peak heights were larger. An increase by a factor of 3 in the 540 nm peak height was attributed to POHC defects in the P-doped LETI material.

To estimate the variation in UV-absorbing defect concentrations with charge dose, all other significant components must be subtracted from the spectra. This is achieved by making three assumptions. First, that the total absorption at 500 nm (which we term A_{500}) consists only of the tail of the UV-absorbing peak (UV_{500}) plus the wavelength independent loss component (A_{WI}) shown in Fig. 2. Second, that the total absorption at 650 nm (A_{650}) consists only of the absorption peak at 650 nm (A_{NBOHC}) plus the loss element A_{WI} (represented by the total absorption at 725 nm). This assumption is considered reasonable, since the effect of the UV-absorbing defect at 650 nm is small at low doses. Third, the magnitude of the peak at 650 nm at higher doses was taken to be equal to that at 0.1 C/cm^2. (The apparent decrease in 650-nm peak height with dose is attributed to increasing magnitudes of small peaks at 600 and 700 nm.)

The third assumption is based on the view that the NBOHC concentration is probably saturated above 0.1 C/cm^2. Since this assumption was also considered reasonable for the 540-nm peak, the only significant variation in absorption peak height is ascribed to UV-absorbing defects. The source of this peak could be peroxy radical centers (Si-O-O$^\bullet$), which cause absorption centered on 163 nm. However, it is most likely to be E' defects, which cause absorption at 215 nm, as confirmed by ESR results presented later.

Based on these three assumptions, estimates for the contribution of UV-absorbing defects at 500 nm were calculated using

$$\mathrm{UV}_{500}(\mathrm{dB}) = A_{500} - A_{\mathrm{WI}}. \tag{1}$$

Fig. 3 shows the dose dependences of UV_{500} (dB/cm) for waveguides in BT and LETI films. These curves were taken to be representative of the dependence of the absorption peak itself. The absorption at zero dose was assumed to be roughly zero, which was later confirmed by ESR and cathodoluminescence data [25]. The error bars in Fig. 3 were calculated by propagating average errors at different wavelengths through (1).

The dose dependences of the changes in the extraordinary refractive index (Δn) observed in this material are also illustrated for comparison (after Lewandowski [3]). Both data sets can be modeled using a saturating exponential equation of the form

$$Y = Y_{\max}[1 - \exp(-d/D)] \tag{2}$$

where d is the charge dose, D is a constant, Y is the refractive index change (or the absorption due to UV-absorbing defects) and $Y_{\max}$ is the saturation value of Y. For BT material, the best fit value for D obtained from the absorption data was found to be 0.070 C/cm^2, and from the refractive index data, 0.072 C/cm^2. The values of D obtained for LETI were 0.080 C/cm^2 and 0.083 C/cm^2, for absorption and refractive index results, respectively. The similarity between the absorption and index change values, in each material, imply that a single first order process is responsible both for inducing the defects and for the index change observed.

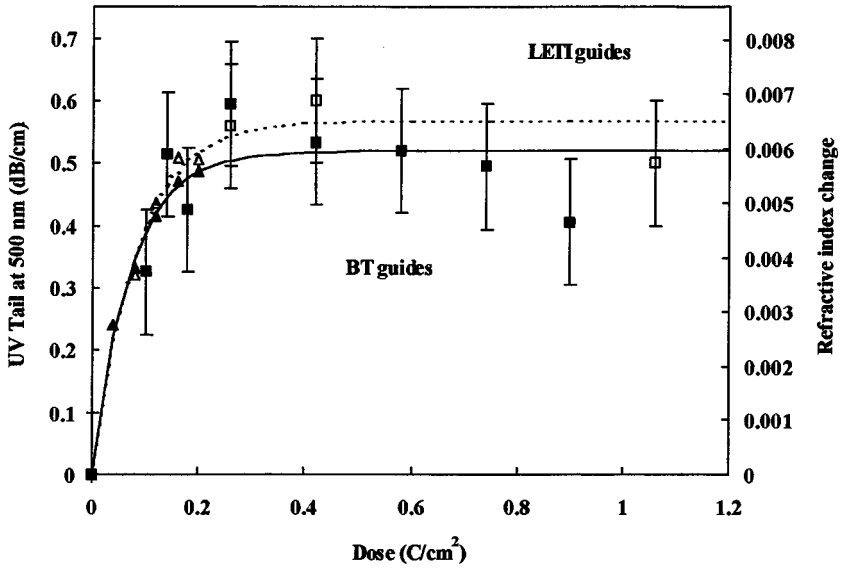

Fig. 3. Tail of UV-absorption peak at 500 nm (squares) and refractive index change (triangles) versus electron dose for BT (filled symbols) and for LETI (open symbols) material. Curves shown (solid for BT, dotted for LETI) were calculated from the refractive index data and (2).

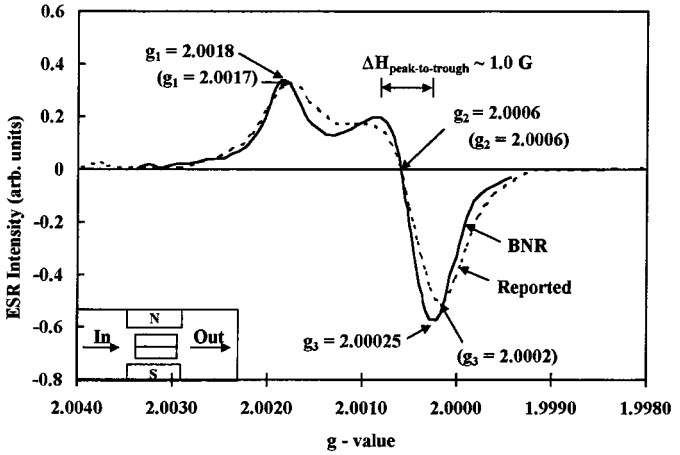

Fig. 4. ESR results for E' signal; experimental (taken from BNR planar waveguide) and reported (taken from an irradiated thermally grown silica film, after Warren [33], shown as dotted line). Reported g values are shown in brackets.

B. Electron Spin Resonance Spectroscopy

In derivative ESR spectrometry, the g-value (or Landé splitting factor) at the zero-crossing point and the shape of the spectrum (including the g-values at peaks or troughs in the spectrum) are used to identify the source of the signal as a particular defect type. To assess further spectral similarities, the difference between magnetic field strengths at particular peaks and troughs (ΔH) may also be compared.

Initial measurements suggested that there are high concentrations of E' centers in the planar waveguides. There was also evidence of a small signal with a g value close to reported values for one of the NBOHC peaks [7], [28]. Finally, small signals were observed in LETI guides with g values close to those attributed to peroxy radical defects [29]–[31]. Fig. 4 shows an E' ESR spectrum from a BNR waveguide, together with a previously reported E' signature [32]. Both have a crossover g value (g_2) of 2.0006, which is within the range of g_2 values reported for E' centers in PECVD films $(2.0000 \leq g \leq 2.0012)$ [9], [19], [20], [32], [33]. The peak-to-trough values of $\Delta H = 1$ G also agree with values reported by others, and the double humped shape of the BNR signature is generally characteristic of E' centers [32].

Of the reported E' defect variants, the E_γ, E'_d and E'_{10} centers have similar cross-over g values [20], [34]. The E'_d center is an unlikely candidate, since its precursor would yield an equal number of NBOHC defects, whereas the results here imply that the NBOHC concentration is much lower than the E' concentration [35]. The E'_{10} center is also unlikely, since no characteristic hyperfine peaks were observed [20]. These observations led to this signal being firmly attributed to the E'_γ center, whose signature also most closely matches the experimental data [9].

When spectra from BNR guides were recorded at 2 mW and 2 G modulation amplitude, signals with a small peak at $g = 2.0100$ were observed. This value is within the reported range for NBOHC signals $(2.0095 \leq g \leq 2.0104)$ [30], [31], [36]. These signals were therefore attributed to NBOHC defects, and this assignment was confirmed by the earlier absorption results.

When spectra from the LETI waveguides were recorded at 2 mW and 2 G modulation amplitude, rather different signals, with a zero crossover value of $g = 2.0076$ and a peak-to-peak linewidth of $\Delta H = 5$ G were observed. An example is shown in Fig. 5. The rapid increase in signal at larger values of magnetic field is caused by the E' center. The cross-over value is within the reported range for peroxy signals in irradiated silica $(2.0070 \leq g \leq 2.0079)$ [7], [30], [36]. In spite of the peak signal being at $g = 2.0091$, which is close to the reported NBOHC peak, the signal was assumed to be caused by peroxy defects because the 5-G peak-to-trough width and the presence of the trough itself are both consistent with reported peroxy signatures. The precursors to peroxy defects are peroxy linkages (Si-O-O-Si), which suggests that there are localized areas of oxygen excess in these phosphorus-doped films.

During a search for NBOHC peaks in a LETI guide, evidence of the phosphorus related POHC defect was found. A resonance, which increased in intensity with charge dose and

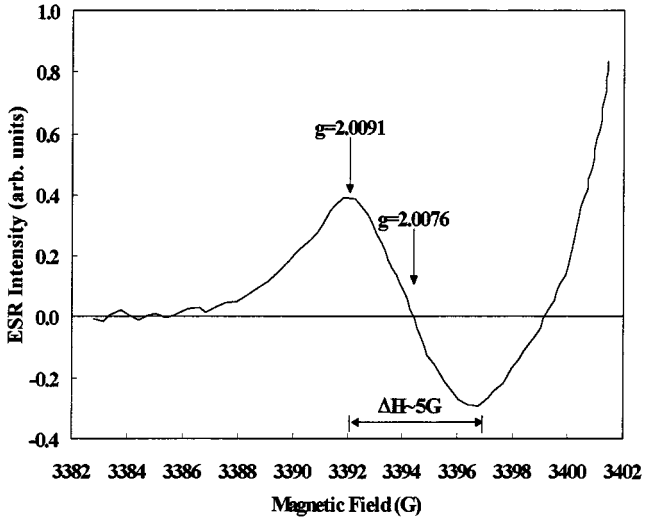

Fig. 5. Electron-spin resonance spectrum optimised for peroxy radical signals taken from LETI waveguide irradiated to a dose of 1.2 C/cm^2.

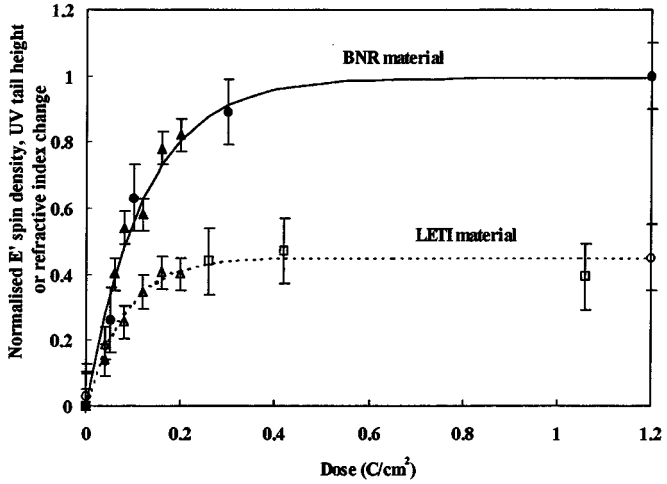

Fig. 6. Normalized relative spin density, absorption intensity and refractive index change versus dose. Circles: E' relative spin density. Squares: UV-absorption tail. Triangles: index change (Lewandowski [3]). Lines: saturated exponential curves.

was observed at 29.0 G from the reported central g value at 2.0097, was recorded [25]. This signal was attributed to a hyperfine peak reported near 26.1 G, due to interaction between 31P atoms (100% abundance) and POHC defects [27]. The mismatch between the observed and published separations may be due to lineshape distortion caused by overmodulation. While this attribution is tentative, these data support the earlier absorption results, which also imply the presence of POHC defects in LETI guides.

C. Comparison Between Absorption and ESR Spectroscopy

In this section, the dependence of the E' center concentration on charge dose is compared with the variations of the UV-absorbing defect concentration and the refractive index. The E' spin densities detected in BNR and LETI films were all normalized to the maximum BNR value and are shown in Fig. 6. Previous results for the UV-absorption tail in LETI waveguides and refractive index measurements from similar samples are also shown [3]. These dose dependencies are similar to those reported for other types of irradiated silica, and the absolute spin density values are also similar [37], [38]. The E' measurements are again matched to saturating exponential curves, which also fit this data well.

These results show that significant concentrations of E' centers are induced in PECVD silica films which exhibit a positive refractive index change on irradiation. The E' density was found to increase by a factor of at least 18 in BNR samples, as the dose increased from 0 C/cm^2 to 1.2 C/cm^2, and by a factor of at least 9 in the LETI guides.

The charge dose dependence of the E' spin density was again found to follow a single saturating exponential function, providing further evidence that the creation of E' centers is a first order process, resulting from the conversion of a limited supply of precursors during irradiation. The similarities between the E' and UV tail variations, together with the absence of a significant peroxy radical signal in the BNR films, imply that the UV-absorption tail described earlier is due mainly to E' defects. The similarities between the E' and refractive index dose responses, and the general dominance of the E' signal, are also consistent with the hypothesis that the most important defects affecting the refractive index in this type of silica are E'.

The spin density of E' centers in BNR samples irradiated to a dose of 1.2 C/cm^2, was found to be 5×10^{19} spins/cm^3. Similarly, the spin density of NBOHC defects in such samples was found to be of the order 6×10^{15} spins/cm^3. It can be seen by inspection of Fig. 6 that the saturation density of E' defects in LETI guides is around 2.5×10^{19} spins/cm^3. This 50% drop in E' center concentration in LETI samples compared with BNR samples can be explained if differences in impurity contents (e.g., higher Si-H content) and the presence of P doping, lead to a decrease in the concentration of the E' precursors. The concentration of peroxy radical defects in such samples irradiated to a dose of 1.2 C/cm^2 was around 2×10^{16} spins/cm^3 and the density of POHC defects was around 3×10^{16} spins/cm^3. These absolute results are considered to be accurate to within 50%.

The E' result agrees with the $\sim 10^{19}$ spins/cm^3 saturation spin density reported for laser or gamma irradiation of oxygen deficient PECVD films, but not with values reported for oxygen surplus films [36], [37]. This implies that, prior to irradiation, these films are most likely oxygen deficient (as do the g values and shape of the E' signals in Fig. 4 [33]). The NBOHC result agrees with saturation levels reported for bulk and thin film silicas with high OH contents [36], [38], [39].

The maximum peroxy radical concentration measured is between the values reported for uncompacted bulk samples ($\sim 7 \times 10^{15}$ spins/cm^3) and those compacted by 16% (5×10^{17} spins/cm^3) [29], [30]. It is also in very close agreement with the concentration previously published for laser irradiation of a PECVD sample containing excess oxygen (3×10^{16} spins/cm^3) [36]. The maximum POHC signal level obtained, although 5 times higher than NBOHC saturation densities in BNR guides, does not provide the two orders of magnitude increase in overall defect concentration in LETI waveguides observed on addition of phosphorus to bulk silica [10]. This can be explained if other differences in the characteristics of LETI

TABLE II
SUMMARY OF DEFECTS FOUND

Film source	Defects	Defect evidence
BT	UV absorbing	Absorption tail
LETI	E_γ'	Absorption tail and ESR signal at g=2.0006
BNR	E_γ'	ESR signal at g =2.0006
BT	NBOHC	650 nm Absorption
LETI	NBOHC	650 nm Absorption
BNR	NBOHC	ESR signal at g = 2.0100
LETI	peroxy radical	ESR signal at g = 2.0076
LETI	POHC	540 nm Absorption and ESR signal at g = 2.0097- 29G

and BNR films, such as the OH content and H content, lead to a reduced concentration of phosphorus related precursors.

IV. CONCLUSION

We have positively identified defect structures in waveguides formed by electron irradiation of PECVD silica. The conclusions reached for each of the materials examined are summarised in Table II, together with the supporting evidence. Absorption and ESR measurements have been used to identify E'_γ and NBOHC defects, and show that E' centers are the dominant species. Evidence of peroxy and POHC defects has also been found in phosphorus doped guides. ESR and absorption results together strongly support the hypothesis that the refractive index change and E' defect density are governed by the same first order process. The spin density (up to 5×10^{19} spins/cm^3) and variant detected (E'_γ with $g_2 = 2.0006$) both imply that these films are oxygen deficient.

By comparing the absorption wavelength with previously reported results it was concluded that NBOHC defects are likely to have peroxy linkage or strained Si-O-Si (rather than Si-OH) precursors. The presence of peroxy defects in phosphorus doped samples implies that there are at least localised areas of oxygen excess in the LETI samples.

The improvement in understanding of radiation effects offered by this work also provides new information for device fabrication. For example, an increased sensitivity to electron beam irradiation, i.e., an increase in index change (and hence a reduction in losses) might be obtained by increasing the oxygen deficiency (and hence the Si-Si content) of these films. This could be achieved by implantation of Si^+ ions or deposition under oxygen deficient conditions.

Previous results have showed that LETI guides are more stable (under heat treatment) than BNR guides [5]. This was attributed to an increased H content in the LETI films. The results obtained here reveal other differences in LETI guides such as a reduced E' concentration and the presence of peroxy and POHC, defects. These characteristics may also affect waveguide stability and it may therefore be desirable to increase the H and P content of electron beam guides in order to induce more stable E'_γ centers and hence more stable guides. It is interesting to note that hydrogen loading (using a pressurised furnace) has been found to improve the index change achieved in UV written waveguides formed in silica-on-silicon films [40].

Guides with losses as low as 0.1 dB/cm, operating at 1.5 μm, have previously been reported [5]. This work offers the prospect of a new level of control over the characteristics of the waveguides fabricated and should allow improvements to be made in the reliability with which suitable samples can be irradiated to form high performance devices. It therefore significantly improves the prospects for electron irradiation as a mainstream PLC fabrication process.

ACKNOWLEDGMENT

The authors gratefully acknowledge the assistance of Prof. D. Goodgame and Dr. T. Tate with the experiments, and BT Labs and Nortel for the provision of PECVD films.

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