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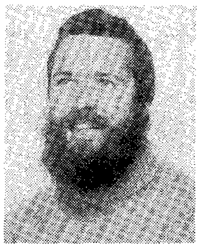

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CO_2 Laser Annealing of Si_3N_4, Nb_2O_5, and Ta_2O_5 Thin-Film Optical Waveguides to Achieve Scattering Loss Reduction

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Abstract—**Significant reductions in the optical scattering losses of Si_3N_4, Nb_2O_5, and Ta_2O_5 waveguides fabricated on SiO_2/Si substrates have been measured following CO_2 laser annealing. The largest improvements were observed for Si_3N_4 waveguides, where waveguide attenuation values of about 6.0 dB/cm before laser annealing were reduced to as low as 0.1 dB/cm afterwards. An improvement of more than an order of magnitude was obtained for a Nb_2O_5 waveguide upon laser annealing, the attenuation coefficient decreasing from 7.4 to 0.6 dB/cm. In the case of one Nb_2O_5 waveguide no improvement was obtained upon laser annealing. The attenuation coefficient of a reactively sputtered Ta_2O_5 waveguide was found to decrease from 1.3 dB/cm before laser annealing to 0.4 dB/cm afterwards. In the case of a thermally oxidized Ta_2O_5 waveguide a small initial improvement in waveguide attenuation was followed by degradation upon further laser annealing.**

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I. Introduction

Scattering of light propagating in thin-film optical waveguides represents a loss mechanism and, concommittantly, a limitation on the dynamic range obtainable in integrated optical signal processing devices [1]. An order of magnitude reduction in waveguide scattering loss, for instance, leads to an approximately 10 dB increase in dynamic range [1]. In addition, a lower loss waveguide requires a lower power light source, an advantage in some applications. Reduction of scattering in thin-film optical waveguides is thus desirable, as it would make integrated optical signal processing devices more competitive with other approaches.

We have previously reported CO_2 laser annealing that dramatically reduces scattering losses in Corning 7059 glass [2], [3] and ZnO [4] thin-film waveguides fabricated on thermally oxidized silicon substrates. Losses as low as 0.05 dB/cm for Corning 7059 glass waveguides and 0.01 dB/cm for ZnO waveguides have been achieved by this technique, a factor of 50–100 times lower than the best results previously reported for these two waveguide materials [5]–[10]. In this paper we wish to report the success of CO_2 laser annealing in reducing scattering losses in Si_3N_4, Nb_2O_5, and Ta_2O_5 waveguides, all fabricated on thermally oxidized silicon substrates.

The lowest loss reported for any thin-film optical waveguide

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prior to our recently reported laser annealing results [3], [4] had been 0.1 dB/cm for a Si_3N_4 waveguide, [11]. This makes Si_3N_4 a promising waveguide material despite difficulties experienced in fabricating good quality films [11], [12]. We have reduced the waveguide attenuation of Si_3N_4 waveguides from values of about 6.0 dB/cm before laser annealing to as low as 0.1 dB/cm after laser annealing. Our success in reducing the waveguide loss by laser annealing implies that the film deposition conditions are no longer as critical to the fabrication of low-loss Si_3N_4 waveguides. Nb_2O_5 has been used as a low-loss waveguide material for several years [5], [13], and its high refractive index makes it potentially useful for waveguide lens applications as well [14]. We have reduced the scattering loss of a Nb_2O_5 waveguide from 7.4 to 0.6 dB/cm by laser annealing. Ta_2O_5 has long been considered an attractive material for integrated optical device applications, and it is widely used for thin-film waveguide lenses because of its high refractive index [15]-[17]. We have reduced the waveguide attenuation of an RF sputtered Ta_2O_5 waveguide from 1.2 dB/cm before laser annealing to 0.4 dB/cm after laser annealing.

In what follows we first describe, in Section II, the fabrication of the Si_3N_4 films by low pressure chemical vapor deposition, the Nb_2O_5 films by RF sputtering, and the Ta_2O_5 films by both reactive sputtering and thermal oxidation of sputtered tantalum films. We then detail, in Section III, the laser annealing conditions for each type of waveguide and our technique for the measurement of low values of waveguide loss. Here we include a discussion of the errors introduced by the statistical fluctuations inherent in this type of measurement. In Section IV we present and discuss the optical waveguide attenuation data obtained before and after laser annealing, and the refractive index changes induced by laser annealing. Finally, in Section V, we summarize our conclusions for the three different types of waveguides.

II. Waveguide Fabrication

The Si_3N_4 waveguides were deposited on 3 in diameter silicon substrates, each of which had a ~1.0 μm thick surface layer of SiO_2 formed by thermal oxidation at 1100°C for 2.5 h in a steam environment. The Si_3N_4 was deposited using a low pressure chemical vapor deposition (LPCVD) furnace at a temperature of 800°C. The LPCVD process used was based on the thermal reaction of dichlorosilane (H_2SiCl_2) and anhydrous ammonia (NH_3) in a hot wall diffusion furnace at a pressure of approximately 350 mtorr. The deposition rate was 0.32 Å/s. Film thicknesses of 0.280 μm for sample Si_3N_4-1 and 0.175 μm for sample Si_3N_4-2 were obtained. The films were uniform in thickness to within ±1 percent as determined by optical measurements. There were no microfractures evident in the films, as seen by prism-coupling He-Ne laser light into the waveguides.

The Nb_2O_5 waveguides, samples Nb_2O_5-1 and Nb_2O_5-2, were formed by reactively sputtering niobium metal in an argon-oxygen atmosphere. The optical properties of these films are stongly dependent upon the conditions under which the films are grown [18]. This is illustrated by the two samples used in this study. Two-inch diameter silicon wafers with ~1.0 μm thick layers of SiO_2 formed on their surfaces were used as substrates for the optical waveguides. Both Nb_2O_5 films were deposited in an RF diode sputtering machine with bias sputtering capability. Both samples were bias sputtered with a total chamber gas pressure of 4.0 mtorr and a ratio of argon to oxygen of 3:1. The total RF power of sample Nb_2O_5-1 was 150 W, with the power divided approximately 4:1 between sputtering target and substrate, resulting in a deposition rate of 6.0 Å/min. This led to bias voltages of −810 V on the target and −190 V on the substrate. The film thickness was measured to be about 0.742 μm. For sample Nb_2O_5-2, the total power was 50 W, with a 6:1 power ratio. The bias voltages were −450 and −75 V for target and substrate, respectively. This resulted in a deposition rate of 4.3 Å/min and a film thickness of 0.518 μm.

Of the two Ta_2O_5 waveguides, the first, sample Ta_2O_5-1, was formed by the oxidation of sputtered tantalum metal, and the second, sample Ta_2O_5-2, was formed directly by reactive sputtering. In both cases the films were deposited on thermally oxidized silicon substrates in an RF magnetron system using a high purity tantalum target. The tantalum metal was deposited at a background pressure of 10.0 mtorr with an argon:oxygen ratio of 97:3 using 500 W applied power. The accumulation rate was 1.5 Å/s. The directly sputtered Ta_2O_5 was also deposited with a background gas pressure of 10.0 mtorr but with oxygen increased to a 90:10 ratio and applied power of 700 W. The accumulation rate was 0.3 Å/s. The relative amount of oxygen in the ionized gas accounts for the formation of the metal or the oxide, with the transition occurring at an argon:oxygen ratio of approximately 95:5. The addition of slight amounts of oxygen in the metal deposition result in a better and less process sensitive Ta_2O_5 optical waveguide after oxidation [19]. The resistivities of the tantalum metal film and the reactively sputtered Ta_2O_5 film were 4.5×10^{-4} $\Omega \cdot$ cm and $<10^{13}$ $\Omega \cdot$ cm, respectively. The tantalum metal film was oxidized at 550°C for 24 h in an open tube furnace with an oxygen flow rate of approximately 2500 cm^3/min. This brought the resistivity to above 10^{12} $\Omega \cdot$ cm. The dielectric properties of the thermally oxidized and reactively sputtered films were measured as capacitors at 1.0 kHz. They had dielectric constants of 25.0 and 19.0, and dissipation factors of 0.0008 and 0.0002, respectively. The variations in the dielectric properties are attributed to stoichiometry and density differences of the films. The thermally oxidized film was 0.510 μm thick, and the reactively sputtered film was 0.445 μm thick.

III. Laser Annealing and Waveguide Characterization Techniques

Laser annealing was carried out using a 50 W, CW CO_2 laser, a 6.4 cm focusing lens, and a variable frequency beam scanner. The beam was scanned horizontally across a distance of 1 cm at a rate of 1.0 cm/s, and the sample, which was mounted vertically on a motorized translation stage, was translated at the end of each horizontal beam scan in vertical increments ranging from 150-200 μm. A laser power density of 1.6×10^5 W/cm^2 associated with a spot size of ~150 μm was used to anneal the Si_3N_4 waveguides. Laser annealing was carried out at power densities varying from 3.0×10^3 W/cm^2 to

1.0×10^5 W/cm^2, associated with spot sizes varying from 900 to ~150 μm, for the Nb_2O_5 waveguides. The Ta_2O_5 waveguides were laser annealed at power densities varying from 9.3×10^2 W/cm^2 to 2.0×10^5 W/cm^2, associated with spot sizes varying from 1.5 mm–150 μm.

Careful measurements of the out-of-plane scattering loss of each of these waveguides were made both before and after laser annealing. He-Ne laser light was prism-coupled into each waveguide and the intensity of the light scattered out of the waveguide was measured as a function of distance along the axis of propagation to determine the attenuation coefficient. The measurement was carried out with a scanning photometric microscope incorporating a 50 μm aperture optical fiber probe within the microscope eyepiece and a 10 X microscope objective. This allowed precise positioning of the probe with respect to the waveguide surface. For each measurement along the axis of propagation, the microscope was defocused by 3 mm, the fiber probe was scanned a distance of 1 mm transverse to the axis at a rate of 1 mm/min, and an integrating digital voltmeter was used to integrate the signal over the scan [3]. Defocusing the microscope resulted in a spatial smoothing of the light scattered from isolated waveguide defects, thus smoothing the fluctuations in the data caused by the uneven distribution of scattering centers. A schematic of the experimental arrangement is given in Fig. 1. Defocusing, scanning, and integrating in this manner provided an appropriate average of the scattered light intensity. In addition, for the low-loss waveguides the laser power was monitored with a precision of ±0.05 percent during the measurement, and the readings were normalized accordingly. Using these averaging techniques has allowed consistent measurement of waveguide attenuation values as low as 0.01 dB/cm [3].

Although we have reduced considerably the errors due to statistical fluctuations in the density of scattering centers in each waveguide by our careful averaging procedure, we have not eliminated them completely. In the case of waveguides which have losses greater than 1.0 dB/cm this statistical error representing the deviation of the data from the least squares fit never exceeds 10 percent. However, for lower-loss waveguides this uncertainty becomes considerably larger because the same deviation of a data point from the best straight line on a semilog plot of the scattered intensity versus distance will have a greater effect on a shallow slope than it will on a steep slope. For example, in Fig. 3, curve (a), omitting the fifth data point, which deviates significantly from the best straight line, changes the slope by no more than 0.6 percent. In Fig. 3, curve (c), however, omitting the second data point, whose deviation from the best straight line is a third of the deviation considered in curve (a), changes the slope from 0.6 to 0.1 dB/cm. The statistical uncertainty for each best fit is tabulated with the optical attenuation data in Tables I, II, and III. We have calculated the uncertainty in each best fit by taking an arithmetic average of the deviations of the data points from the best fit and using this to calculate a maximum and a minimum slope. The maximum slope is calculated from the straight line formed by adding the average deviation to the left-hand extreme of the best fit straight line and subtracting it from

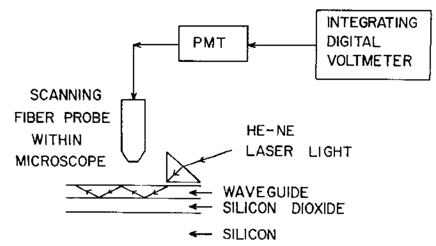

Fig. 1. Schematic of the experimental arrangement for measurement of out-of-plane waveguide scattering loss.

the right-hand extreme. The minimum slope is calculated by the reverse procedure. The uncertainty, then, is given by

$$\frac{\text{maximum slope} - \text{minimum slope}}{2\sqrt{n}}$$

where n is the number of data points. This method yields a slightly smaller uncertainty than that calculated using a similar formula for a least-squares fit error [20]. We wish to emphasize that this uncertainty is a result of the statistical distribution of scattering centers and not due to a lack of measurement precision. If we were to measure the waveguide attenuation along several different propagation axes across the entire width of the waveguide, we would expect that the differences in attenuation would be less than the statistical error in each best fit. This indicates that the fluctuation in the density of scattering centers is not a random error, so the error in the attenuation should be smaller than the statistical uncertainty tabulated, which is calculated assuming a random Gaussian distribution.

IV. Results and Discussion

A. Si_3N_4 Waveguides

The optical attenuation data for the two Si_3N_4 waveguides, samples Si_3N_4-1 and Si_3N_4-2, are listed in Table I. Both samples were laser annealed at a power density of 1.6×10^5 W/cm^2. In sample Si_3N_4-1, the waveguide attenuation decreased dramatically from 6.0 dB/cm before laser annealing to 0.1 dB/cm afterwards, a value equal to the lowest value previously reported for Si_3N_4 waveguides [11]. Scattering loss measurements for sample Si_3N_4-1 before and after laser annealing are presented in Fig. 2(a). The waveguide attenuation in sample Si_3N_4-2 also showed a significant decrease, from 3.1 dB/cm before laser annealing to 0.2 dB/cm afterwards, as shown in Fig. 2(b).

The refractive index of these Si_3N_4 films as determined by the measurement of TE and TM waveguide modes before laser annealing was 2.017 at the He-Ne laser wavelength ($\lambda = 6328$ Å). This value of index compares favorably with sputtered Si_3N_4 films formed by reactive sputtering [12]. The optical loss of LPCVD films was lower than that which had been achieved with the sputtered films. After laser annealing, the angles at which prism coupling occurred remained the same as before, indicating that the refractive index and thickness of neither of the films had changed upon laser annealing [21].

At the CO_2 laser wavelength, $\lambda = 10.6$ μm, Si_3N_4 has a very

TABLE I
EFFECT OF LASER ANNEALING ON Si_3N_4 WAVEGUIDE ATTENUATION

Sample Number	Si_3N_4 Film Thickness (μm)	Loss Before Laser Annealing (dB/cm)	Loss After Laser Annealing (dB/cm)	Annealing Power Density (W/cm^2)
Si-1	0.280	6.0 ± 0.52	0.1 ± 0.5	1.6 x 10^5
Si-2	0.175	3.1 ± 0.32	0.2 ± 0.8	1.6 x 10^5

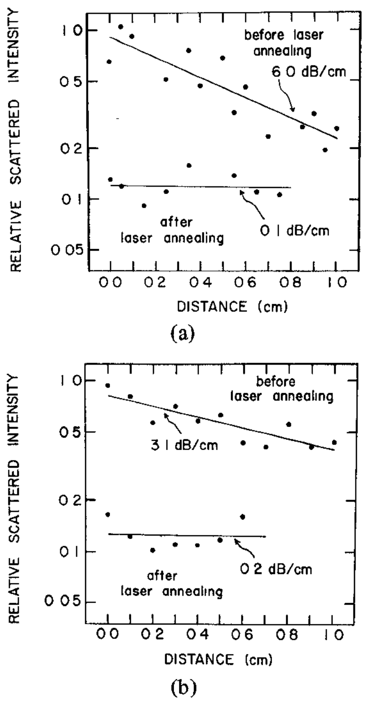

Fig. 2. Si_3N_4. Logarithm of the relative scattered intensity is plotted versus distance along the waveguide. (a) The upper and lower straight lines represent best fits to the data obtained from sample Si_3N_4-1 before and after laser annealing, respectively. (b) The upper and lower straight lines represent best fits to the data obtained from sample Si_3N_4-2 before and after laser annealing, respectively.

large absorption coefficient of 4.51×10^4 cm^{-1} [22]. This implies that although the Si_3N_4 film thicknesses were small (0.280 and 0.175 μm) the radiation absorbed generated sufficient heat to anneal out defects in these films. Thus, laser annealing resulted in significantly improved optical waveguides.

B. Nb_2O_5 Waveguides

Optical waveguide attenuation data for the two Nb_2O_5 waveguides, samples Nb_2O_5-1 and Nb_2O_5-2, are collected in Table II. For sample Nb_2O_5-1, a waveguide attenuation of 7.4 dB/cm was measured before laser annealing, as shown in Fig. 3, curve (a). This sample was initially laser annealed at a power density of 3.0×10^3 W/cm^2, resulting in a lower attenuation of 3.6 dB/cm, as shown in Fig. 3, curve (b). A second laser anneal was carried out on sample Nb_2O_5-1 at a power density of 1.0×10^5 W/cm^2. Using this higher power density for the second laser anneal, we observed a dramatic reduction of the attenuation coefficient to 0.6 dB/cm, as shown in Fig. 2, curve (c). This value of loss is comparable to 0.5 dB/cm, the best result previously obtained for RF sputtered Nb_2O_5 waveguides [18].

The experiment was repeated on sample Nb_2O_5-2, which was a very high-loss waveguide. The laser light prism-coupled into this waveguide was attenuated so strongly that the waveguiding light streak was too short to allow any measurements of the attenuation coefficient to be made. This sample was laser annealed at 0.65×10^5 W/cm^2. This did not result in any noticeable improvement of the waveguide, however. Visible damage occurred when a slightly higher laser power density of 1.0×10^5 W/cm^2 was used.

The refractive indexes of the Nb_2O_5 films were measured before and after laser annealing and were found to remain essentially unchanged. At $\lambda = 6328$ Å the refractive indexes were measured to be 2.186 for sample Nb_2O_5-1 and 2.286 for sample Nb_2O_5-2.

Nb_2O_5 has a fairly broad absorption band peaking at $\lambda = 11.8$ μm [23], so that there is significant absorption at $\lambda = 10.6$ μm. Sample Nb_2O_5-1, which was a reasonably good waveguide to start with, showed moderate improvement after laser annealing at a power density of 3.0×10^3 W/cm^2. When it was reannealed at the much higher power density of 1.0×10^5 W/cm^2, the improvement was much more dramatic. A slightly higher power density of 1.3×10^5 W/cm^2 caused visible damage, indicating that 1.0×10^5 W/cm^2 was the optimum annealing power density for this waveguide. Sample Nb_2O_5-2, however, could not be improved by laser annealing.

C. Ta_2O_5 Waveguides

Table III displays optical attenuation data for the two Ta_2O_5 waveguides, samples Ta_2O_5-1 and Ta_2O_5-2, the thermally oxidized and reactively sputtered waveguides, respectively. A waveguide attenuation of 7.4 dB/cm was measured for sample Ta_2O_5-1 before laser annealing. Initially, laser annealing was tried at a power density of 3.0×10^3 W/cm^2, resulting in a lower attenuation coefficient of 5.8 dB/cm. A second laser anneal was carried out at 1.3×10^5 W/cm^2. The waveguide attenuation was left unchanged by this second anneal, a value of 5.7 dB/cm being measured afterwards. A higher power density of 2.0×10^5 W/cm^2 was used for a third laser anneal. This caused a degradation of the waveguide attenuation to 9.5 dB/cm.

The reactively sputtered waveguide, sample Ta_2O_5-2, was of better optical quality than sample Ta_2O_5-1, and an attenuation of 1.3 dB/cm was measured before laser annealing, as shown

TABLE II
EFFECT OF LASER ANNEALING ON Nb_2O_5 WAVEGUIDE ATTENUATION

Sample Number	Nb_2O_5 Film Thickness (μm)	Loss Before Laser Annealing (dB/cm)	Power Density of First Anneal (W/cm^2)	Loss After First Anneal (dB/cm)	Power Density Of Second Anneal (W/cm^2)	Loss After Second Anneal (dB/cm)
Nb-1	0.742	7.4 ± 0.63	3.0×10^3	3.6 ± 0.55	1.0×10^5	0.6 ± 0.26
Nb-2	0.518	100	0.65×10^5	100		

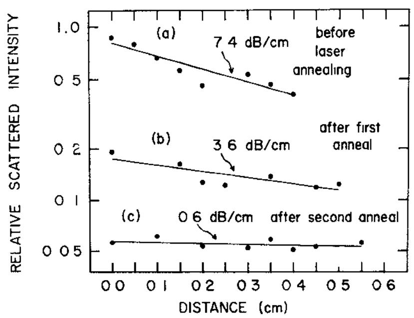

Fig. 3. Nb_2O_5. Logarithm of the relative scattered intensity is plotted versus distance along the waveguide. The straight lines (a), (b), and (c) represent best fits to the data obtained from sample Nb_2O_5-1 before laser annealing, after laser annealing at a power density of 3.0×10^5 W/cm^2, and after a second laser anneal at a higher power density of 1.0×10^5 W/cm^2, respectively.

TABLE III
EFFECT OF LASER ANNEALING ON Ta_2O_5 WAVEGUIDE ATTENUATION

Sample Number	Ta_2O_5 Film Thickness (μm)	Loss Before Laser Annealing (dB/cm)	Power Density of First Anneal (W/cm^2)	Loss After First Anneal (dB/cm)	Power Density Of Second Anneal (W/cm^2)	Loss After Second Anneal (dB/cm)	Power Density of Third Anneal (W/cm^2)	Loss After Third Anneal (dB/cm)
Ta-1	0.45	7.4 ± 0.63	3.0×10^3	5.8 ± 0.50	1.3×10^5	5.7 ± 0.77	2.0×10^5	9.5 ± 0.58
Ta-2	0.45	1.3 ± 0.18	9.3×10^2	0.4 ± 0.28				

in Fig. 4, curve (a). Sample Ta_2O_5-2 was laser annealed at a power density of 9.3×10^2 W/cm^2. The waveguide attenuation decreased to 0.4 dB/cm after laser annealing, as shown in Fig. 4, curve (b). This compares favorably with the best result previously measured for Ta_2O_5 waveguides [15].

The refractive indexes of the two Ta_2O_5 films were measured using TE and TM waveguide modes both before and after laser annealing. At $\lambda = 6328$ Å, sample Ta_2O_5-1 had an average index of 2.214 before laser annealing, which decreased to 2.057 after laser annealing. The refractive index of sample Ta_2O_5-2 decreased from 2.120 before laser annealing to 2.106 after laser annealing. A decrease in index has also been observed after conventional thermal annealing in the 500-600°C region. Above 600°C transformation of the film from an amorphous to a crystalline state takes place with a further decrease in index and high optical waveguide losses due to scattering from microcrystallites [19].

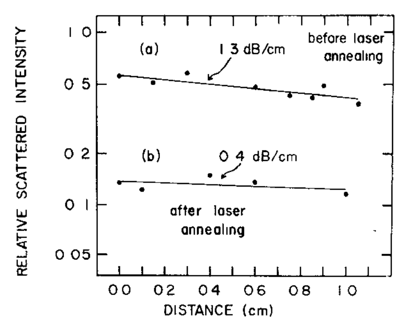

Fig. 4. Ta_2O_5. Logarithm of the relative scattered intensity is plotted versus distance along the waveguide. The straight lines (a) and (b) represent best fits to the data obtained from sample Ta_2O_5-2 before and after laser annealing, respectively.

The fact that laser annealing proved more effective on the directly sputtered Ta_2O_5 film than the thermally oxidized film might be accounted for by the differences in film formation. The reactively sputtered oxide film, which was formed on an unheated substrate, should have had a more uniform amorphous composition than the thermally oxidized film. Laser annealing at a lower power density may have reduced bulk defects throughout the sputtered film without promoting the growth of microcrystals. In the case of the thermally oxidized film the higher power density used may have promoted microcrystalline growth while simultaneously annealing bulk defects.

To summarize the discussion of our results for the three waveguide materials, the Si_3N_4 waveguides were improved most effectively by CO_2 laser annealing. This is a reasonable result considering the strong absorption of Si_3N_4 at $\lambda = 10.6$ μm. A large improvement was noted after laser annealing a fair quality Nb_2O_5 waveguide, as was anticipated from the broad absorption band peaking at $\lambda = 11.8$ μm for Nb_2O_5. Distinctly different results were obtained for the directly sputtered Ta_2O_5 waveguide and the thermally oxidized Ta_2O_5 waveguide. The infrared spectrum for Ta_2O_5 shows an absorption band peaking at $\lambda = 16.7$ μm [23], suggesting that the absorption at 10.6 μm may not be very high. This may explain the smaller improvements in the Ta_2O_5 waveguides induced by laser annealing as compared with the improvements in the Si_3N_4 and Nb_2O_5 waveguides.

V. Conclusions

We have extended the technique of CO_2 laser annealing, which we have used previously to improve Corning 7059 glass and ZnO thin-film waveguides, to reduce optical scattering losses in Si_3N_4, Nb_2O_5, and Ta_2O_5 waveguides, all fabricated on thermally oxidized silicon substrates. Values of waveguide attenuation comparable with or better than the lowest losses previously reported for each type of waveguide were obtained for all three waveguide materials. The optimum power density for laser annealing was found to differ widely for the two Ta_2O_5 films which had been formed by different methods, with a larger improvement being obtained for the reactively sputtered film. These results have helped to establish CO_2 laser annealing as a powerful technique for reduction of optical waveguide scattering loss, representing a major advance in silicon-based integrated optics technology.

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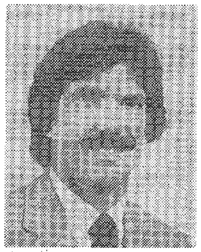

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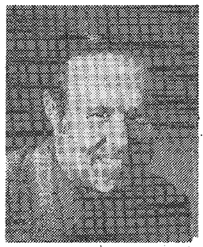

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