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Structural and functional characterisation of slab waveguides written in Er^{3+} - doped tellurite glass, CaF_2, $Bi_4(GeO_4)_3$ and $Bi_{12}GeO_{20}$ crystals via implantation of MeV N^+ ions

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Abstract. Ion implantation proved to be a universal technique for producing waveguides in most optical materials. Tellurite glasses are used as hosts of rare-earth elements for the development of fibre and integrated optic amplifiers and lasers covering all the main telecommunication bands. Er^{3+}- doped tellurite glasses are very attractive materials for the fabrication of broadband amplifiers in wavelength division multiplexing (WDM) around 1.55 μm, as they exhibit large stimulated cross sections and broad emission bandwidth. First objective of the present research was to optimise parameters of waveguide fabrication in the Er: tellurite glass via implantation of MeV energy N^+ ions in a wide range of implanted doses. Besides of glasses, slab optical waveguides were designed and fabricated in CaF_2, $Bi_4Ge_3O_{12}$ and $Bi_{12}GeO_{20}$ crystals, also using MeV energy N^+ ions. Waveguides were characterised using UV/VIS and NIR absorption spectroscopy, spectroscopic ellipsometry and m-line spectroscopy. Part of the implanted samples was annealed to improve waveguide properties. We report on first working slab waveguides fabricated in CaF_2 crystals using implantation of MeV-energy medium-mass ions.

1. Introduction

Ion implantation, compared with other waveguide fabrication methods, has some unique advantages. It proved to be a universal technique for producing waveguides in most optical materials [1]. It has better controllability and reproducibility than other techniques. The first articles reporting fabrication of waveguides by ion implantation appeared between the end of 1960's and early 1980's [2 – 5]. The first

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ion implanted waveguides were produced in 1968 by proton implantation into fused silica glass [2], and the index changes for H^+, He^+ and N^+ ions have been characterized by several other groups [3-5]. A detailed review on ion-implanted optical waveguides has been published recently [6].

Tellurite glasses have gained a widespread attention because of their potential as hosts of rare-earth elements for the development of fiber and integrated optic amplifiers and lasers covering all the main telecommunication bands [7, 8]. Er^{3+} doped tellurite glasses in particular are very attractive materials for the fabrication of broadband amplifiers in wavelength division multiplexing (WDM) around 1.55 μm, as they exhibit large stimulated cross sections and broad emission bandwidth [9]. Furthermore, tellurite glasses have low process temperature, good chemical durability and nonlinear properties [10-12].

We have recently reported fabrication of channel waveguides in an Er-doped tungsten-tellurite glass [13]. We decided to fabricate 1D structures, as well, in order to optimize the fabrication processes by ion-implantation in this material. The composition of the glass we developed for our experiments was 60 TeO_2-25 WO_3-15 Na_2O-0.5 Er_2O_3 (mol. %) and its fabrication procedure has been reported elsewhere [8].

Calcium fluoride is an excellent optical material, due to its perfect optical characteristics from UV wavelengths up to near IR. Recently it has become a promising host material, doped with rare earth elements [14-16]. Successful waveguide fabrication in alkali fluoride and alkali earth halide crystals using only light ions (H^+ and He^+) has been reported so far [17,18]. Based on our successful previous experiments with N^+ - implanted waveguides in both amorphous and crystalline materials, we decided to try to fabricate optical waveguides in CaF_2 single crystal samples via implantation of MeV energy N^+ ions.

Bismuth germanate is a well known scintillator material. It has high electro-optic coefficients (3.3 pm/V for $Bi_{12}GeO_{20}$), making it useful in nonlinear optics for building Pockels cells, and can also be used for photorefractive devices. Formation of planar waveguides in $Bi_4Ge_3O_{12}$ (eulytine) crystals by implantation of He+ ions of the 1 - 2 MeV energy range was first reported by Mahdavi et al. [19]. Here we present our preliminary results in fabricating slab waveguides in both eulytine and sillenite type bismuth germanate crystals using a medium-sized ion, N^+, instead of the light ions used in the above mentioned works.

2. Waveguide fabrication and SRIM simulations

We fabricated five types of slab waveguides using the following ion - target combinations: 1.5 MeV N^+ ions implanted into Er: Te glass, using a wide range of implanted doses, 3.5 MeV N^+ ions implanted into Er: Te glass, 3.5 MeV N^+ ions implanted into CaF_2 single crystal, 3.5 MeV N^+ ions implanted into $Bi_4Ge_3O_{12}$ (eulytine) single crystals and 3.5 MeV N^+ ions implanted into $Bi_{12}GeO_{20}$ (sillenite) single crystals.

Irradiations were carried out with a 1.5 MeV and 3.5 MeV N^+ collimated beam from a Van de Graaff accelerator (available at the Research Institute for Particle and Nuclear Physics, Budapest), with normal incidence on the glass samples and at 7° incidence on the crystal samples, to avoid channeling. Lateral homogeneity of the irradiation was ensured by defocusing the ion beam with a magnetic quadrupole and by scanning the sample under a 2 mm x 2 mm beam. Useful size of the implanted waveguides was 6 mm x 6 mm.

Names and implanted doses of the slab waveguides implanted in Er: tellurite glass by 1.5 MeV and 3.5 MeV N^+ ions, in CaF_2, $Bi_4Ge_3O_{12}$ and $Bi_{12}GeO_{20}$ crystals, all by 3.5 MeV N^+ ions, are shown in Table 1. Structure of the ion implanted slab waveguides is determined mainly by the energy and dose of the implanted ions. Distribution of the implanted ions or that of the collision events along the depth of the implanted sample can serve as rough estimation of the refractive index profile of the implanted waveguide. We used SRIM 2008 [20] code (Stopping and Range of Ions in Matter) to simulate the fabrication of the ion-implanted slab waveguides. We performed SRIM calculations for each experiment. Maxima of the distributions of the implanted N^+ ions are at 1.6 μm and 2.5 μm below the surface of the Er: Te glass sample in case of 1.5 MeV and 3.5 MeV ion energies. Maxima of the

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collision events (vacancy production) distributions roughly coincide with those of the ion distributions, but they extend considerably towards sample surface.

Table 1. Summary of the implantation energies and doses for all the waveguides

		Names of the waveguides and implanted doses ($x10^{15}$ ions/cm^2)							
Name of the target	N+ energy (MeV)	A	B	C	D	E	F	G	H
Er: Te glass	1,5	0.005	0.05	0.5	5	10	20	40	80
Er: Te glass	3.5	10	20	40	80	-	-	-	-
CaF_2	3.5	5	10	20	40	-	-	-	-
$Bi_4Ge_3O_{12}$	3.5	2	4	8	16	-	-	-	-
$Bi_{12}GeO_{20}$	3.5	2	4	8	16	-	-	-	-

In case of the CaF_2 sample and 3.5 MeV energy of the implanted N^+ ions maxima of the range and the vacancy distributions are around 3.0 μm. In case of elulytine bismuth germanate crystals the maximum of the range distribution is at approximately 2.5 μm, while for the sillenite bismuth germanate the place of this maximum is at 2.2 μm. One can expect waveguide operation even at infrared wavelength in case of the 3.5 MeV energy N^+ implantation since in all the three materials it produces a guiding layer of over 2.0 μm width.

3. Optical absorption measurements

UV/VIS spectra of all the nonimplanted samples and the implanted waveguides have been measured using a JASCO Corp. V-550 spectrophotometer to check the effects of ion implantation on absorption of the samples (in the 190 nm - 900 nm wavelength range). NIR spectra of the non implanted Er:Te glass and of the waveguides implanted in it have also been measured, using a Brukker spectrophotometer, in the 15000 cm^{-1} - 5000 cm^{-1} wavenumber range. As for the slab waveguides implanted in the Er: Te glass, overall absorption increased monotonically with the implanted dose both in the visible and NIR wavelength ranges, but no new absorption peaks appeared. Implantation of the CaF_2 crystal with high doses of N^+ ions resulted in the appearance of new absorption bands in the visible - UV spectrum, due to the colour centres created by the irradiation. There are three new absorption bands in the UV/VIS absorption spectrum of the N^+ - implanted waveguides in CaF_2 crystal (Figure 1), at 344, 404 and 550 nm.

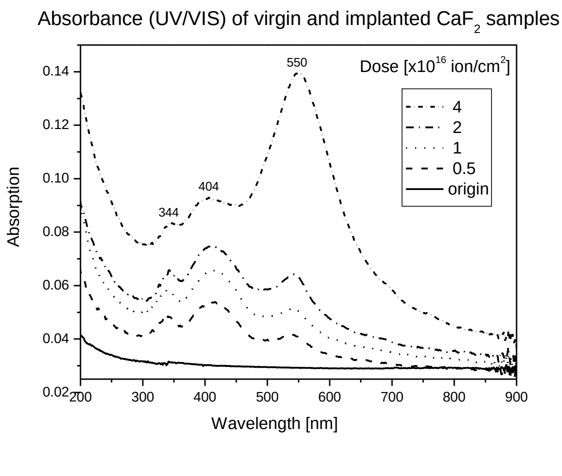

Figure 1. Absorbance (UV/VIS) of virgin and N^+ -implanted CaF_2 samples.

They can be attributed to F1, F2 and F3 colour centres. UV/VIS absorption spectra of the N+ - implanted $Bi_4Ge_3O_{12}$ and $Bi_{12}GeO_{20}$ waveguides have also been measured. The most evident effect of ion implantation on the absorption spectra is an abrupt shift of the UV absorption edge, even at the lowest dose applied (2 x 10^{15} ions/cm^2). The absorption edge shifted from 300 nm to 350 nm in the eulytine-type crystal, and from 390 nm to 415 nm in the sillenite BGO.

4. Spectroscopic ellipsometric measurements

Spectroscopic ellipsometry is a powerful tool for characterisation of stacks of thin transparent layers. All the ion implanted slab waveguides were measured by spectroscopic ellipsometers at the Department of Photonics of Research Institute for Technical Physics and Materials Science, Budapest. Er:Te glass samples, implanted with 1.5 MeV N^+ ions, were measured using a SOPRA ES4G spectroscopic ellipsometer (SE) with microspot facility (about 0.15 x 0.35 mm) [21], and all the other samples were measured with the recently acquired WOOLLAM M-2000DI spectroscopic ellipsometer.

4.1. Results for Er-doped tungsten-tellurite glass

The wavelength range of the measurements of the 1.5 MeV N^+ - implanted Er: Te glass waveguides of the highest four implanted doses, carried out with the SOPRA ES4G spectroscopic ellipsometer, was 300 – 750 nm.

Evaluation of the ellipsometric spectra of the nonimplanted glass surface was performed using a three-phase optical model consisting of ambient (air), a surface roughness layer and the substrate (glass). The roughness layer was taken into account on basis of effective medium approximation [22], the roughness layer consists of 50% of glass and 50% of void. For the analysis of the spectroellipsometric data we applied the evaluation software WVASE32 created by the J. A. Woollam C., Inc [23]. The evaluation yielded 7.90 ± 0.06 nm for the thickness of the surface roughness layer and 2.081 for the refractive index of the non-implanted glass substrate for the wavelength of 635nm.

Table 2. Results of spectroscopic ellipsometric measurements of Er: tellurite glass waveguides

N+ energy (MeV)	1.5				3.5			
Names of the waveguides	E	F	G	H	A	B	C	D
Doses (x10^{16} ions/cm^2)	1	2	4	8	1	2	4	8
Thickness of layer$_2$ [nm]	1781 ± 19	1785 ± 28	1779 ± 16	-	2615.07 ± 1.608	2643.60 ± 14.511	2403.83 ± 5.108	2384.71 ± 4.644
Refractive index of layer$_2$ at 635 nm	2.052	2.048	2.052	-	2.070	2.042	2.040	2.015
Thickness of layer$_1$ [nm]	67 ± 22	83 ± 35	97 ± 19	-	183.38 ± 9.771	195.82 ± 46.248	457.33 ± 4.724	489.76 ± 8.606
Refractive index of layer$_1$ at 635 nm	2.071	2.068	2.071	-	2.015	2.025	2.070	2.10
Refractive index of the non-implanted glass at 635 nm	2.081	2.081	2.081	-	2.019	2.019	2.019	2.019

The optical model applied in the evaluation of the SE data for different ion-implanted areas consisted of three layers on top of the glass substrate. The layer adjacent to substrate (layer$_1$) represents the stopping region. The second layer (layer$_2$) is the region which the implanted ions traverse before they stop in layer$_1$. The third layer was a surface roughness film. Dielectric functions of layer$_1$ and layer$_2$ were described by Cauchy dispersion relation. Parameters of the Cauchy dispersion relations and layer thicknesses were considered as free parameters.

There was a good agreement between measured and simulated spectroscopic ellipsometric data in most cases, thanks to a relatively high refractive index contrast of the guiding and barrier layers. Results of spectroscopic ellipsometric measurements of Er: tellurite glass waveguides are shown in Table 2. Data for waveguide "H" of the 1.5 MeV series are missing due to some problems at the measurements. Width of the guiding layers (approximated with a rectangular refractive index distribution) is at both implantation energies somewhat over the projected range, while width of the barrier layer increases with the implanted dose, as expected. In case of implantation with 3.5 MeV N+ ions width of the barrier layer is close to longitudinal straggling calculated by SRIM.

4.2. Results for CaF_2 crystals

Ellipsometric measurements of this sample were difficult to perform because of its low thickness (about 0.5 mm). Only ellipsometric spectra obtained for the highest implanted dose, 4 x 10^{16} ions/cm^2, could be simulated successfully. Thickness of the guiding layer is 2.56 μm, well below the projected range (3.0 μm) obtained by SRIM. Thickness of the barrier layer (0.38 μm) is close the lateral straggling. Refractive indices of the guiding and barrier layers were 1.467 and 1.451.

4.3. Results for BGO crystals

Table 3. Results of spectroscopic ellipsometric measurements of BGO waveguides

BGP crystal type	$Bi_4Ge_3O_{12}$				$Bi_{12}GeO_{20}$			
Names of the waveguides	A	B	C	D	A	B	C	D
Doses (x10^{16} ions/cm^2)	0.2	0.4	0.8	1.6	0.2	0.4	0.8	1.6
Thickness of layer$_2$ [nm]	2552.70 ± 0.878	2575.80 ± 1.075	2632.96 ± 0.854	2628.09 ± 0.825	2500.16 ± 21.633	2571.61 ± 3.122	2588.20 ± 3.105	2685.67 ± 1.680
Refractive index of layer$_2$ at 635 nm	2.115	2.121	2.202	2.350	2.380	2.371	2.375	2.362
Thickness of layer$_1$ [nm]	337.90 ± 10.594	287.91 ± 2.897	346.38 ± 4.243	412.89 ± 4.592	294.86 ± 42.469	517.90 ± 19.553	472.81 ± 27.424	348.24 ± 9.171
Refractive index of layer$_1$ at 635 nm	2.116	2.131	2.110	2.125	2.346	2.348	2.353	2.335
Refractive index of the non-implanted glass at 635 nm	2.086	2.086	2.086	2.086	2.50	2.50	2.50	2.50

Results of ellipsometric measurements of waveguides written in both $Bi_4Ge_3O_{12}$ (eulytine) $Bi_{12}GeO_{20}$ (sillenite) crystals and are presented in Table 3. Thickness of the guiding layer is around 2.6 μm in both crystals, slightly changing with dose. This is somewhat higher than ranges of 3.5 MeV N^+ ion in eulytine (2.5 μm) and considerable higher than that in sillenite (2.2 μm). Note that rectangular refractive index profiles were assumed in spectroscopic ellipsometric simulations. Thickness of the barrier layer (layer$_1$) slowly changes with the implanted dose. The most important result is the very high refractive index modulation obtained in the sillenite sample, refractive index of the barrier layer is about 0.15 lower than that of the nonimplanted sample. Thus good confinement in the barrier-type waveguides can be expected.

5. M-line spectroscopic measurements

In order to characterize the effect of N^+ ion implantation in the tungsten-tellurite glass and CaF_2 and BGO single crystals, we used a semi-automatic instrument developed at IFAC ("COMPASSO"), based on m-line spectroscopy. The accuracy of the instrument is around ± 1x 10^{-4} and ± 5 x 10^{-4} on the effective refractive index and bulk refractive index, respectively.

5.1. Results for Er-doped tungsten-tellurite glass

In this case, due to the lower contrast in the measurement, the accuracy was lower, about ± 5 x 10^{-4} and ± 1 x 10^{-3} respectively. Due to the high refractive index of the bulk (around 2.0 at 635 nm), we used a rutile prism to couple the light in the irradiated regions.

5.1.1. Waveguides implanted with 1.5 MeV N^+ ions. Examples of the measurements performed at 635 nm and 980 nm for the region exposed to 4 x 10^{16} ions/cm^2 (waveguide G) are shown in Figures 2 and 3. Figure 4 shows effective refractive index of the single guiding mode at 980 nm as a function of the implanted dose, and Table 4 summarises the values obtained from the measurements at 635 nm. Each value is an average of three measurements. No modes were found at higher wavelengths (i.e.: at 1300 nm and at 1550 nm). There were no observable guiding modes at 635 nm in the sample implanted at the lowest dose, 5 x 10^{12} ions/cm^2. Because all the effective refractive indices measured at different wavelengths (635 nm and 980 nm, respectively) are below the corresponding value of the bulk refractive index, we expect that the refractive index change is negative and the guiding structure is an optical barrier waveguide. Thus light can be confined in the layer between the optical barrier and the surface.

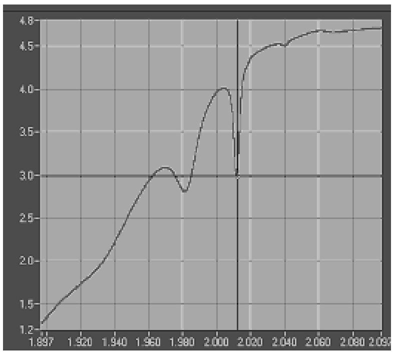

Figure 2. M-line spectrum of zone G (dose of 4 x 10^{16} ions/cm^2) at 635 nm.

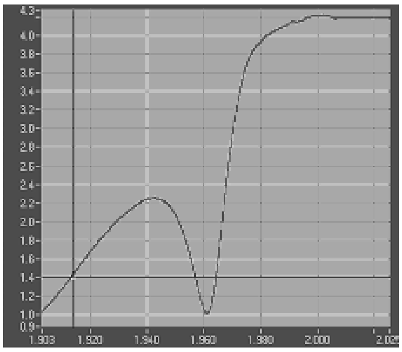

Figure 3. M-line spectrum of zone G at 980 nm.

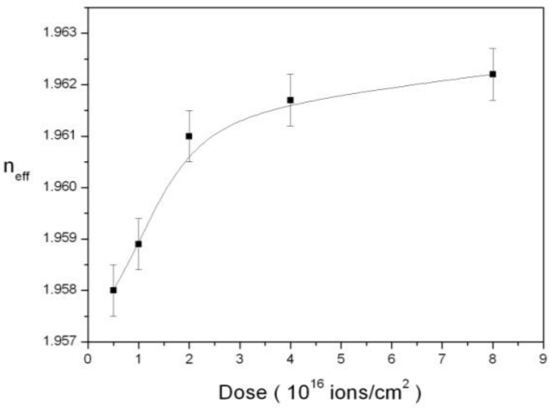

Figure 4. TE single mode effective refractive index measured at 980 nm as a function of ion dose.

Table 4. Effective refractive indices vs. ion dose measured at 635 nm for Tellurite glass.

Dose (x10^{15} ions/cm^2)	Effective Refractive Index (average value)	Bulk Refractive Index
0.005	no modes	2.041 (± 1 x 10^{-3})
0.05	$N_{eff,0}$ = 2.0059 (± 5 x 10^{-4}) $N_{eff,1}$ = $N_{eff,2}$ =	2.041 (± 1 x 10^{-3})
0.5	$N_{eff,0}$ = 2.0097 (± 5 x 10^{-4}) $N_{eff,1}$ = $N_{eff,2}$ =	2.041 (± 1 x 10^{-3})
5	$N_{eff,0}$ = 2.0111 (± 5 x 10^{-4}) $N_{eff,1}$ = $N_{eff,2}$ =	2.041 (± 1 x 10^{-3})
10	$N_{eff,0}$ = 2.0404 (± 5 · 10^{-4}) $N_{eff,1}$ = 2.0112 (± 5 · 10^{-4}) $N_{eff,2}$ = 1.9817 (± 5 · 10^{-4})	2.041 (± 1 x 10^{-3})
20	$N_{eff,0}$ = 2.0400 (± 5 · 10^{-4}) $N_{eff,1}$ = 2.0124 (± 5 · 10^{-4}) $N_{eff,2}$ = 1.9823 (± 5 · 10^{-4})	2.041 (± 1 x 10^{-3})
40	$N_{eff,0}$ = 2.0400 (± 5 · 10^{-4}) $N_{eff,1}$ = 2.0124 (± 5 · 10^{-4}) $N_{eff,2}$ = 1.9816 (± 5 · 10^{-4})	2.041 (± 1 x 10^{-3})
80	$N_{eff,0}$ = 2.0391 (± 5 · 10^{-4}) $N_{eff,1}$ = 2.0137 (± 5 · 10^{-4}) $N_{eff,2}$ = 1.9814 (± 5 · 10^{-4})	2.041 (± 1 x 10^{-3})

Moreover, even with an increase of the ion dose, it is possible to detect a shift of the effective refractive indices towards the bulk refractive index at various wavelengths, with particular reference to the leaky mode $N_{eff,1}$ at 635 nm – see Table 4 – and to the single mode at 980 nm.

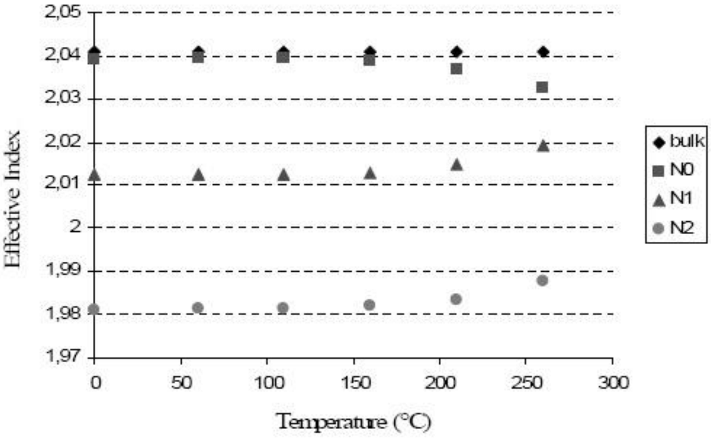

Figure 5. Annealing effect on the TE effective indices measured at the wavelength of 635 nm for zone G ($4{\times}10^{16}$ ions/cm^2).

Stepwise annealing process was performed in air on the slab waveguides irradiated with higher N^+ ion doses ($\geq 10^{16}$ ions/cm^2). Duration of each annealing step was 20 minutes. Effective indices measured with m-line spectroscopy for zone G are plotted as a function of the post-annealing temperature after each annealing step in Figure 5.

5.1.2. Waveguides implanted with 3.5 MeV N^+ ions. As expected, fabrication of the Er: Te glass waveguides with 3.5 MeV energy N^+ ions resulted in better working waveguides. Even guiding at a wavelength of 1550 nm has been demonstrated. Here only two examples are presented. M-line spectrum of TE modes for the highest implanted dose, 8 x 10^{16} ions/cm^2, measured at 635 nm, is presented in Figure 6. Note the presence of five modes. Proof of guiding of the same waveguide at 1550 nm is shown in Figure 7.

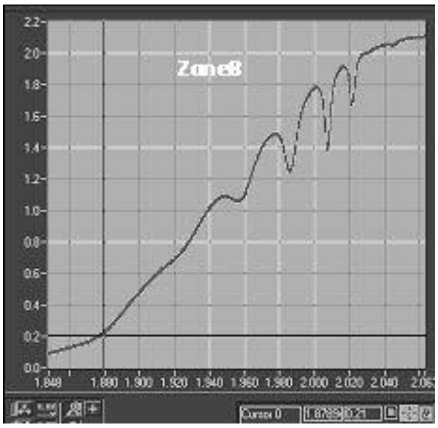

Figure 6. M-line spectrum of waveguide D, D = 8 x 10^{16} ions/cm^2, at 635 nm

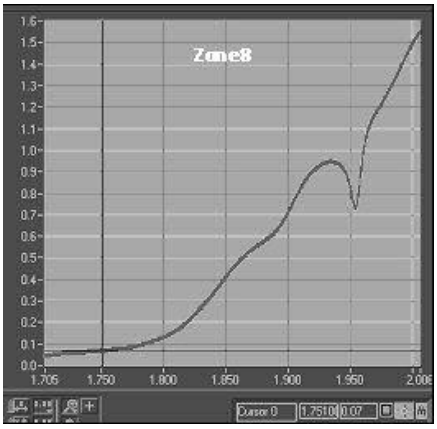

Figure 7. M-line spectrum of waveguide D at 1550 nm

5.2. Results for CaF_2 crystals

In spite of the fact that ion implantation induced high - concentration colour centres in the CaF_2 sample, evidenced by the violet colour of the two waveguides implanted at the higher doses, the N^+ - implanted slab waveguides proved to work both at 635 nm and 980 nm. Measured effective refractive indices are shown in Table 5. Effective refractive index of the fundamental mode increases monotonically with the implanted dose at 635 nm. We expect that an annealing process would improve waveguide properties by partial elimination of the colour centres.

5.3. Results for BGO
Because of the lack of appropriate prisms for the COMPASSO m-line spectroscopic device, no m-line spectra of the two BGO samples could be measured so far. However, the excellent results for index contrast of the waveguides obtained by spectroscopic ellipsometry indicate that BGO waveguides should work, too at visible and NIR wavelengths.

Table 5. Effective refractive indices of CaF_2 waveguides

Dose (x 10^{15} ions/cm^2)	Effective Refractive Index at 635 nm	Effective Refractive Index at 869 nm	Bulk Refractive Index
5	$N_{eff,0}$ = 1.4290 (± 1 x 10^{-4})	no modes	1.4332 (± 5 × 10^{-4})
10	$N_{eff,0}$ = 1.4293 (± 1 × 10^{-4})	no modes	1.4332 (± 5 × 10^{-4})
20	$N_{eff,0}$ = 1.4296 (± 1 × 10^{-4})	$N_{eff,0}$ = 1.4203 (± 1 × 10^{-4})	1.4332 (± 5 × 10^{-4})
40	$N_{eff,0}$ = 1.4303 (± 1 × 10^{-4})	$N_{eff,0}$ = 1.4205 (± 1 × 10^{-4})	1.4332 (± 5 × 10^{-4})

6. Conclusion
Slab waveguides were fabricated in a rare-earth element - doped glass and in three optical single crystals, using irradiation with MeV energy. Energy of the implanted N^+ ions was 1.5 MeV and 3.5 MeV. Doses of the implanted ions ranged from 5 x 10^{12} to 8 x 10^{16} ions/cm^2. M-line spectroscopic study of the samples revealed that the waveguides in these materials were of optical barrier type, where the implanted layer had a reduced index of refraction with respect to the nonimplanted bulk material, and the layer between the sample surface and the implanted ion range acted as a waveguide. Beginning from an implanted dose of 5 x 10^{14} ions/cm^2, all waveguides fabricated in a tellurite glass did work. Saturation of the refractive index change occurred in the 10^{16} – 10^{17} ions/cm^2 range of implanted doses. Waveguides implanted with N^+ ions of 3.5 MeV energy worked at 1550 nm, too. In spite of the high concentration of the colour centres generated by the ion implantation, m-line spectroscopic measurements revealed that the majority of the slab waveguides implanted in a CaF_2 sample were also fully functional at wavelengths up to 980 nm. Spectroscopic ellipsometric measurements essentially confirmed the results obtained in the m-line tests. Fitting the ellipsometric data with a three-layer model yielded a thin buried layer, centred at the range of the implanted ions. Thickness of the buried layer increased with increasing dose, thus corroborating the results obtained with the m-line technique. Moreover, from these latter measurements, one noted how the shape of the lowest leaky mode, with effective refractive index $N_{eff,2}$, became more defined and peaked with increasing ion doses, and the same thing happened with the single mode at 980 nm. However, refinement of the ellipsometric model and processing of the m-line measured data are needed to obtain a better assessment of the refractive index profile. Thermal annealing of the waveguides fabricated in the tellurite glass up to 260 °C resulted in improvement of guiding properties. Very high modulation, up to 0.15 of the refractive index of the implanted waveguides in the sillenite type BGO crystal was found.

Acknowledgment
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