

# Uniform refractive index cladding for LiNbO<sub>3</sub> single-crystal fibers

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A uniform refractive index cladding for LiNbO<sub>3</sub> single-crystal fibers has been achieved using a Mg ion indiffusion process. This cladding has a uniform MgO concentration of ~20 mol %, resulting in a uniform refractive index. The refractive indices of the cladding material have been evaluated to be  $n_o = 2.08$  and  $n_e = 2.09$  at  $0.6328 \mu\text{m}$  by using the light reflection technique.

Guided-wave nonlinear optical devices are attractive because of their potential for high conversion efficiency at relatively modest input powers. One of the most promising candidates for such guided-wave nonlinear optical devices is the LiNbO<sub>3</sub> single-crystal fiber. First, LiNbO<sub>3</sub> is a highly nonlinear optical material. Second, a long interaction length is readily available in the fibers. Third, high optical intensity can be achieved in a small-diameter fiber core. To date, there have been many investigations mainly concerned with LiNbO<sub>3</sub> single-crystal fiber preparation.<sup>1-5</sup> Little investigation, however, has been conducted on the formation of waveguide structures in LiNbO<sub>3</sub> fibers.

There are several methods of lowering the refractive index of LiNbO<sub>3</sub> such as Mg ion indiffusion<sup>6</sup> and proton exchange.<sup>7</sup> Another possible approach is to coat a suitable material onto the fiber surface.<sup>8</sup> We believe, however, that the Mg ion indiffusion is the most promising cladding method for LiNbO<sub>3</sub> fibers. First, Mg ion indiffusion lowers both the ordinary and the extraordinary refractive indices. Second, it reduces the effective core size. Third, the diffusion process naturally increases the interface smoothness as the depth of diffusion is increased. In a previous paper,<sup>9</sup> we reported on the MgO:LiNbO<sub>3</sub> single-crystal fiber with Mg ion indiffused cladding which had a parabolic refractive index profile and yielded quasi-single-mode beam propagation. However, such parabolic refractive index profile could not reduce the actual core size because the refractive index changed gradually in the radial direction of the fiber.

To date, uniform refractive index cladding as used in glass fibers has not yet been achieved for a LiNbO<sub>3</sub> fiber. Such uniform refractive index cladding is of vital importance to the realization of high optical intensity in the fiber core and the construction of efficient nonlinear devices, which result from the reduction of the actual core size due to the uniform refractive index cladding formed by the Mg ion indiffusion process.

This letter reports on the formation of a uniform refractive index cladding for LiNbO<sub>3</sub> single-crystal fibers by using a Mg ion indiffusion process. The refractive index of this cladding is measured with a light reflection technique.

LiNbO<sub>3</sub> single-crystal fibers were fabricated using the laser-heated miniature pedestal growth method.<sup>1</sup> In this crucible-less method, a CO<sub>2</sub> laser melts the end of a rod of feed

material in a 360° axially symmetric irradiance. An oriented seed crystal is dipped into the molten zone thus formed, and the fiber is grown by pulling the seed crystal away from the melt while fresh feed material is simultaneously fed into the molten zone.

The as-grown LiNbO<sub>3</sub> fiber prepared for the cladding experiments was a  $90 \mu\text{m} \times 65 \mu\text{m}$  *a*-axis-oriented fiber. This fiber had a MgO concentration of ~5 mol %. The cladding was achieved by the following steps. First, we annealed the fiber at a temperature of 1050 °C for 2 h. The fiber was placed on a platinum boat in an electric furnace and suspended above lithium-rich powder to prevent excessive Li outdiffusion. This first step is called vapor phase equilibration. Second, a thin MgO layer was deposited onto the fiber surface in an evaporator. The MgO layer thickness was 6000 Å. Third, the Mg ion indiffusion process was carried out using the same setup as for the annealing step. The diffusion time was 40 h at a processing temperature of 1050 °C.

It should be mentioned here that the fabrication process differences between the parabolic index profile<sup>9</sup> and uniform refractive index cladding described in this letter are in both the MgO deposition thickness and the diffusion time. After this cladding process, the clad fiber was mounted using UV-cured epoxy in a glass capillary tube to allow both end surfaces of the fiber to be polished.

Figure 1 shows a scanning electron microscope (SEM) photograph of the end face of the *a*-axis  $90 \mu\text{m}$  LiNbO<sub>3</sub> fiber after the cladding process. This photograph was taken with a microprobe (JEOL model 733). This is the standard shape of *a*-axis LiNbO<sub>3</sub> single-crystal fibers. From Fig. 1 it is apparent that the periphery of the fiber consists of a thin layer of different contrast.

Figure 2 shows the MgO concentration profile of this fiber measured to within an accuracy of 0.1 mol % using the above microprobe. The fiber end face was scanned with a  $1 \mu\text{m}$  spot size microprobe electron beam along both the *c* axis and the *y* axis. The MgO concentration was analyzed at  $2 \mu\text{m}$  intervals. The measurement results shown in Fig. 2 have several noteworthy features. First, the layer at the periphery has a high and uniform mol percentage of MgO (~20 mol %), resulting in a uniform refractive index. Second, a graded profile of MgO concentration, which seems to correspond to the standard diffusion profile of Mg ions, follows such a MgO-rich layer in the central part. The initial MgO concentration of this fiber is ~4.2 mol %. Third, the MgO concentration

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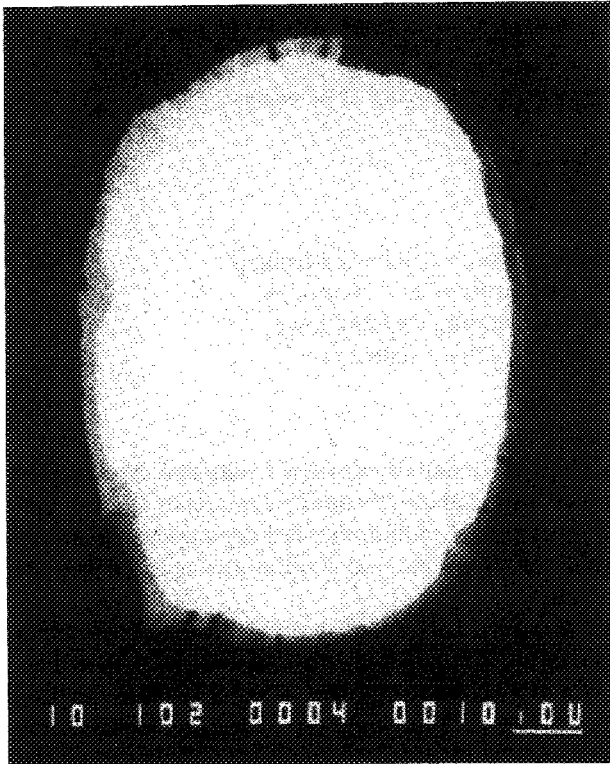


FIG. 1. SEM photograph of the end face of the  $90\ \mu\text{m} \times 65\ \mu\text{m}$   $a$ -axis  $\text{LiNbO}_3$  fiber after the cladding process. The periphery of the fiber consists of a thin layer of different contrast.

rapidly drops from 20 to 7 mol % immediately inside the MgO-rich layer. This MgO concentration of 7 mol % roughly corresponds to the MgO solubility limit in  $\text{LiNbO}_3$ , without a large change in a  $\text{LiNbO}_3$  crystal structure.<sup>10</sup>

The refractive index profile of this fiber with the MgO-rich layer was characterized using the reflection technique described in Refs. 11 and 12. Light from a polarized He-Ne laser at  $0.6328\ \mu\text{m}$  was focused to a small spot by a  $60\times$  microscope objective. The spot size should be only a few microns in order to sample small regions of the fiber end face. The light reflected by the sample end face was collected by the same  $60\times$  objective and directed to a detector by a beamsplitter. The detected optical power was a direct indica-

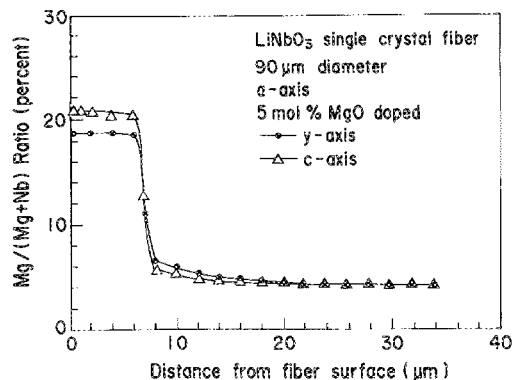


FIG. 2. MgO concentration profile of the fiber measured with the microprobe.

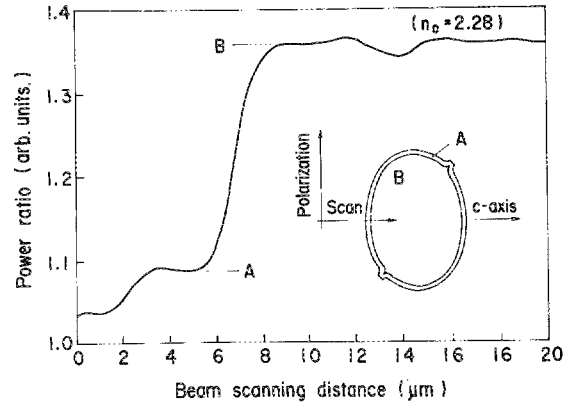


FIG. 3. Scan of the reflected power ratio in the ordinary polarization for an  $a$ -axis  $\text{LiNbO}_3$  fiber.

tion of the refractive index. Details on the experimental results will be published elsewhere.

Figure 3 depicts a scan of the reflected power ratio in the ordinary polarization for the  $a$ -axis  $\text{LiNbO}_3$  fiber shown in Fig. 1. The initial increase in the reflected power is due to the finite dimension of the focused laser beam. The MgO-rich layer extends from the fiber edge to a depth of about  $7\ \mu\text{m}$ . The boundary between the MgO-rich layer and the fiber central region is clearly indicated as a refractive index step ( $\Delta n$ ). The cladding core refractive index difference  $\Delta n$  can be related to the reflected power ratio shown in Fig. 3. Using the results of Ref. 11, we calculated that the core cladding index differences were

$$\Delta n_o = 0.19, \quad (1)$$

$$\Delta n_c = 0.12. \quad (2)$$

The refractive indices of 4.2 mol % MgO-doped  $\text{LiNbO}_3$  at  $0.6328\ \mu\text{m}$  are  $n_o = 2.28$  and  $n_c = 2.20$ .<sup>7</sup> Therefore, according to Eqs. (1) and (2) the refractive indices for the MgO-rich layer are

$$n_o = 2.09, \quad (3)$$

$$n_c = 2.08. \quad (4)$$

In order to identify the nature of the MgO-rich layer, x-ray diffraction analyses were carried out for this fiber and

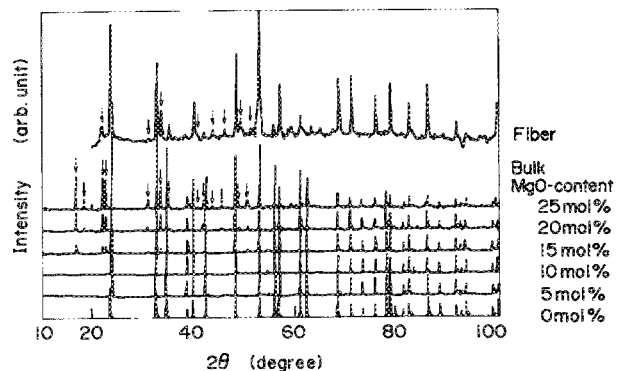


FIG. 4. X-ray diffraction patterns for the fiber and the samples which were prepared through the sintering process using powder mixtures of MgO and congruent  $\text{LiNbO}_3$ . Diffraction lines designated by arrows indicate unknown lines.

several MgO-rich LiNbO<sub>3</sub> bulk samples. The bulk samples were prepared by sintering powder mixtures of MgO and congruent LiNbO<sub>3</sub>. Figure 4 shows x-ray diffraction patterns for both the fiber and the bulk samples. Figure 4 shows that the x-ray diffraction patterns for the fiber and for three bulk samples whose MgO concentration ranges from 15 to 25 mol % include many diffraction lines (designated by arrows) which could not be identified with LiNbO<sub>3</sub>, or with any other known compound such as Mg<sub>4</sub>Nb<sub>2</sub>O<sub>9</sub>, or MgNbO<sub>4</sub>. These eight lines should enable characterization of the crystallographic structure of this new compound, although further investigation on such MgO-rich layers is necessary to elucidate the properties of the new compound.

It seems worthwhile to mention finally that the experimental results shown in Ref. 10 suggest the possibility of a new MgO-LiNbO<sub>3</sub> compound for MgO doping concentrations greater than 15 mol %.

We achieved a uniform refractive index cladding for LiNbO<sub>3</sub> fibers using Mg ion indiffusion. In addition, experimental results suggest that the MgO-rich layer could be a new compound of the Mg-Li-Nb-O ternary system. This uniform cladding technique should be applicable to the construction of highly efficient, nonlinear optical devices such as second-harmonic generators (SHGs) as well as lasers.

SHG experimental results using the cladded LiNbO<sub>3</sub> fibers are described in Ref. 13.

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