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Structural phase transitions during annealing of proton-exchanged layers on X-cut and Z-cut lithium niobate

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Isothermal phase transitions during annealing of proton-exchanged (PE) layers on X-cut and Z-cut lithium niobate (LN) crystals were studied. Congruent lithium niobate crystals (Crystal Technology, Sipat) were used. Proton exchange was carried out in the molten benzoic acid at temperatures ranging from 175°C to 190°C during 2-6 hours. Samples were annealed consequently with a step of 1 hour at 330°C. Samples were investigated by means of polarized light optical microscopy, mode spectroscopy, IR-spectroscopy and XRD.

The following sequence of phase transitions during annealing of PE-layers on X-cut was proposed and successfully confirmed: β1, β2-phases→κ2-phase→κ1-phase→α-phase. Main feature of κ2-phase→κ1-phase transition in X-cut LN is the formation of particles of κ1-phase (which create a modulated structure) but not a layer.

Since κ2-phase formed on X-cut LN is characterized by high strain perpendicular to the substrate surface while strain of κ_1-phase formed on X-cut LN are one order of magnitude lower, a relaxation through incoherent boundaries formation between κ_2-phase and κ_1-phase takes place during annealing. New interphase boundary leads to the formation of additional structural defects. After interphase boundary vanishing during subsequent annealing, some part of aforementioned defects may persist. These structural defects may attract charges (e.g. during temperature changes) which can adversely affect the operation of integrated optical devices.

Phase transitions during annealing of PE-layers on Z-cut are significantly different. The sequence of phase transitions during annealing is the same: β_1, β_2-phases→κ_2-phase→κ_1-phase→α-phase. However, in this case PE-waveguide consists of coherent layers until their full transformation into α-phase. Particles of κ_1-phase are not formed because of lower strains of κ_2-phase and κ_1-phase in Z-cut crystal and smaller difference in their values compared to X-cut.

Increasing temperature and duration of proton exchange does not change characteristic features of structural phase transitions during waveguide annealing. However thicker waveguides need greater annealing time to complete phase transition.

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