16pA4 Crystal growth of fluorides for Laser applications

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Abstract. In this work we review the research done in the crystal growth laboratories of IPEN for the development of synthesis, purification and growth of fluorides crystals for laser applications.

Optical properties of laser hosts are very important in determining laser performance. Even small crystals, as those required by diode pumping systems must exhibit a good quality, mainly for applications of high power devices. The successful operation of a fluoride material as an optical component is dependent on a number of factors directly related to the crystal preparation, but the most important one is the initial use of high purity chemicals. The choice of commercial high purity chemicals do not always assure the purity of the final crystal. Manipulation of the products without the correct procedure can introduce spurious contamination, mainly in the case of more complex compounds, as for example $LiCaAlF_6$ or $LiSrAlF_6$ used as laser matrix hosts; these materials need to be previously synthesized before the crystal growth process.

To improve the purity of the materials utilized at Czochralski laser crystal growth, we studied methods for the fluoride purification. This work included the utilization of reactive atmosphere treatments, synthesis process and zone melting technique. The study and development of these purification process were essential to achieve the quality needed in crystals for laser applications. The crystals growth laboratory of IPEN have worked in the last 15 years in the growth of fluorides laser crystal, such as, $LiYF_4$ (YLF) doped with Nd, Er, Ho, Tm; $BaLiF_3$ doped with Ni, Co and Pb and more recently in $LiSrAlF_6$:Cr and $GdLiF_4$:Nd crystals. In this work we review the research done in the crystal growth laboratories of IPEN for the development of synthesis, purification and growth of fluorides crystals for laser applications.

Although it is possible today to find commercially a great variety of basic compounds with relative high purity, we normally begin the preparation of the laser crystals by synthesizing the basic fluorides. A standard method of fluoride preparation is by the use of gas-solid reaction synthesis methods, as the use of streaming gaseous HF over metals, halides, or oxides heated under appropriated temperatures. The final compounds are obtained by the fusion in controled conditions of two or more previously synthesized fluorides. To convert the appropriate mixtures of components fluorides into polycrystalline bars of the required compound we use the zone melting technique, that also purify the compound to be finally use in the Czochralski crystal growth.

Assuming that high purity materials were synthesized, the optical and crystalline quality of the fluoride laser materials will depend basically on the growth parameters, such as growth rate, crystallographic orientation, interface shape and dopant distribution. The determination of the influence of these parameters in the final quality of the crystal is obtained by their characterization with different techniques, as for example: IR and UV-VIS absorption spectroscopy (to check the presence of spurious impurity absorption bands, such due to OH^- absorption); interferometry (to detect strain and refractive index variations); neutron diffraction (to study the influence of the interface shape and of the crystallographic orientation in quality of the grown crystals); X-ray diffraction, thermal analysis and neutron activation analysis (to determine the dopant incorporation in the crystals).

The fluorides crystals grown using the described methods were sucessfully used to spectroscopic studies and to the preparation of laser rods used in tests of new solid state lasers prototypes.

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