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Growth and characterization of LiF single-crystal fibers by the micro-pulling-down method

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Abstract

Good optical quality LiF single-crystalline fibers ranging from 0.5 to 0.8 mm in diameter and 100 mm in length were successfully grown by the micro-pulling-down technique in the resistive mode. A commercial equipment was modified in order to achieve suitable conditions to grow fluoride single-crystalline fibers.

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1. Introduction

There is an increasing interest in the production of single-crystalline fibers. Their unique properties indicate their use for production of a variety of optical and electronic devices [1]. The final shape of the single-crystalline fiber is already in a form suitable for optical testing and applications, reducing the time and cost of preparation. Several fiber materials, as eutectics, semiconductors and oxide single-crystals have already been grown by the laser heated pedestal growth (LHPG) [2] and by the micro-pulling-down (μ-PD) [3] methods. However, the growth and hence the possible applications of fluoride single-crystalline fibers has not yet been investigated.

As it is already known from other methods of fluoride growth, these materials are very sensitive to oxygen and water contamination. Even traces of humidity present in the growth chamber will react with the melt resulting in the production of hydroxide ions in the grown crystals [4]. The moisture contamination diminishes the quality of

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the fluoride crystal leading to significant losses in optical devices. The limitation to pull long and homogeneous fluoride fibers by LHPG technique was attributed to the inadequate purity of the feed-rod and to the technological difficulties to control the growth atmosphere in order to avoid this moisture contamination [5]. We have successfully grown high-quality lithium fluoride (LiF) single-crystalline fibers from 0.5 to 0.8 mm in diameter and 100 mm in length by the μ-PD technique using ohmic crucible heating, reported here for the first time. Commercial μ-PD equipment was adapted in order to allow the pulling process under a carefully controlled atmosphere. Fig. 1(a) shows the μ-PD system and the Pt crucible in detail during a LiF fiber growth experiment.

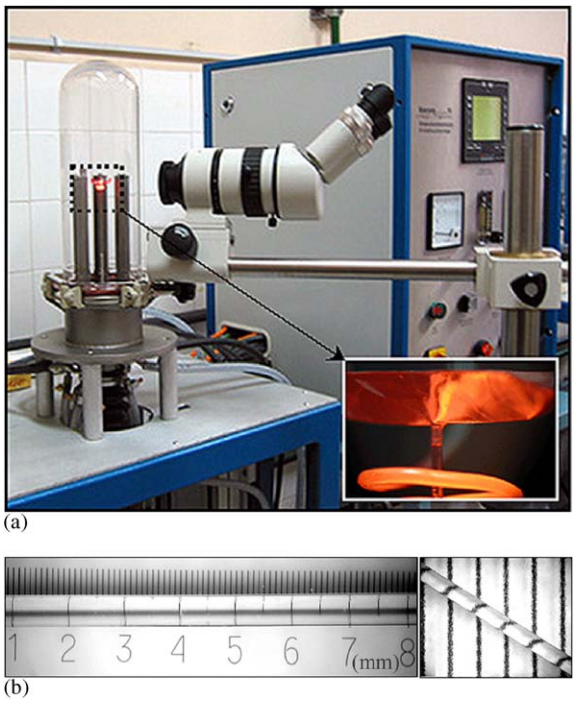

Fig. 1. μ-PD equipment during LiF fiber pulling with Pt crucible in detail (a), and as-grown LiF fibers with a diameter of 600 μm (b).

In order to start the growth with high-purity material, commercial LiF powder (99.99%) was first purified by the zone melting technique under HF atmosphere. The LiF zone melting conditions were previously published [6]. Platinum crucibles were designed to pull fiber crystals with 0.5, 0.6 and 0.8 mm in diameter. The crucibles were made in our laboratory in appropriated shape and dimensions. The quartz growth chamber was thermally treated under vacuum (10^{-3} Torr) and finally back-filled with ultra-pure Argon gas previously dried in molecular sieve traps. The growth was carried out under a gas flow of $20\,l\,h^{-1}$. Crystal fibers with lengths up to 100 mm were pulled from the nozzle with pulling rates in the range of 0.6–0.8 mm min^{-1}. The first seeding was obtained by using an [1 0 0] oriented LiF single crystal seed of approximately 1.5 mm × 1.5 mm, cut from a Czochralski-grown single crystal. Thereafter, seeds were selected from previously pulled fiber crystals.

The obtained LiF fibers are transparent, colorless, uniform in diameter and free of cracks along their length, as shown in the Fig. 1(b). The electronic microscopy image, Fig. 2(a), shows a

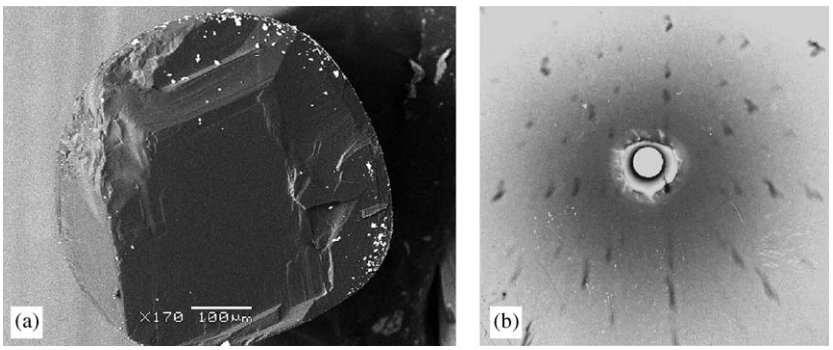

Fig. 2. Scanning electron microscopy image of a LiF fiber cleaved surface (a), and Laue diffraction pattern of the same fiber (b).

nearly cylindrical shape of the fiber with indication of a 4-fold symmetry typical for the $\langle 1\,0\,0 \rangle$ cubic growth direction, which was confirmed by Laue method obtained by back-scattered X-ray diffraction shown in Fig. 2(b). Moisture contamination was evaluated by infrared spectroscopy. The first LiF single-crystalline fibers grown under inert gas flow resulted always in fibers with spurious OH^- contamination. However, a preliminary thermal treatment of the growth chamber under vacuum was very efficient to minimize such contamination.

In summary, the μ-PD method has shown to be a fast and effective technique for the preparation of fluoride single-crystalline fibers. Highly transparent and homogeneous LiF fibers with 100 mm in length and up to 0.8 mm in diameter were successfully grown by μ-PD technique under well-controlled growth atmosphere. We expect that the procedure developed for the preparation of LiF single-crystalline fibers is also appropriated for the growth of other fluoride fiber crystals. Improvements to obtain this class of materials with high crystal perfection could drive the miniaturization of fluoride laser systems. Moreover, μ-PD can be an interesting and low cost technique to evaluate the growth possibilities of new fluoride materials.

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