TEM AND SEM CHARACTERIZATION OF THE FAST IONIC CONDUCTOR $La_{2/3-X}Li_{3X}TiO_3$ FIBERS GROWN BY THE LASER FLOATING ZONE (LFZ) METHOD

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Fibers of $La_{2/3-X}Li_{3X}TiO_3$ with perovskite structure obtained from $La_{0.5}Li_{0.5}TiO_3$ as precursor by the laser floating zone (LFZ) method was characterized by mean of X ray diffraction (XRD), Scanning Electron microscopy (SEM), transmission electron microscopy (TEM) and image simulation of high resolution TEM imaging . A Philips XL 30 scanning electron microscope equipped with a backscattered and dispersive energy X Ray detectors (SUTW) was employed. TEM examinations were performed with Philips Tecnai 20 FEG (Cs=1.2mm) microscope working at 200kV with a resolution of 0.2nm. Image calculation was carried out with the programs Mac Tempas, crystal kit and digital micrograph. Specimens suitable for TEM were prepared by standard procedures: mechanical grinding, dimpling and argon ion milling at 5kV in a liquid-nitrogen-cooled holder.

Fibers were analyzed in transverse and longitudinal sections. SEM showed the presence of three different phases: The major phase, is a La-Li-Ti-O perovskite with less Li content that the raw material and presented large single crystal grains. Other small precipitates surrounding to the main phase correspond to TiO_2 rutile. Finally eutectic mixtures of main phase and Ramsdellite ($Li_2Ti_3O_7$) also appeared. X-ray and electron diffraction patterns agree with a tetragonal unit cell a_p x a_p x $2a_p$ (a_p~3.8Å). Figure 1 shows a backscattered electron (BSE) micrograph of a longitudinal section of a grown fiber. Large grains with white contrast oriented along the grown direction can be appreciated. Dark contrast regions shown a second phase formed.

Transmission electron microscopy observations indicated that the major phase present a microdomains structure.(Figure 2) In some crystals the coexistence of two phases, La-LiTi-O perovskite and $Li_2Ti_3O_7$-ramsdellite is observed (figure 3). Locally antiphase domains and Moire frames are also observed. These frames can be interpreted as a consequence of the separation of the two phases of the eutectic (perovskite and ramsdellite).

References

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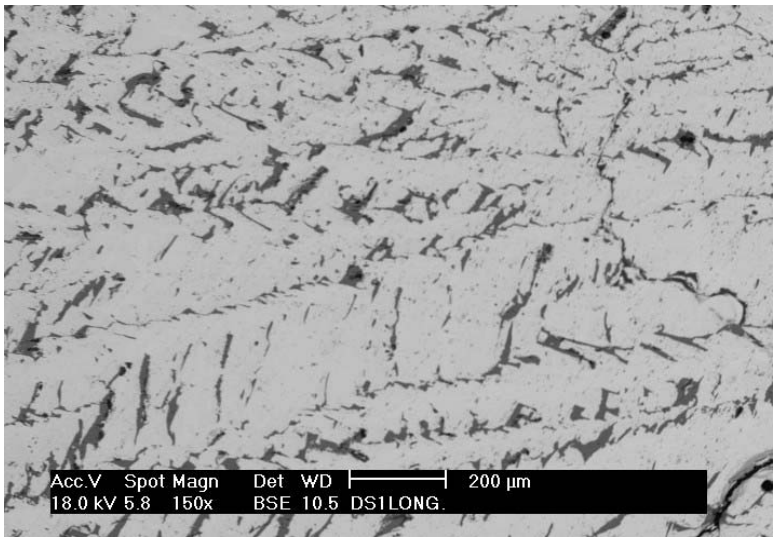

Fig 1

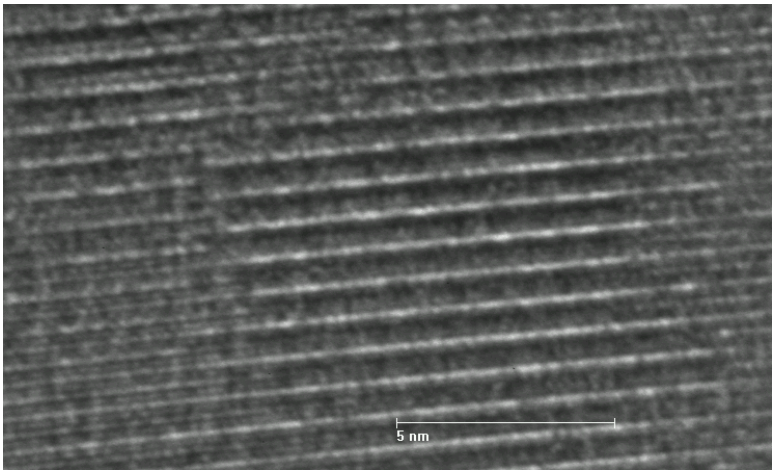

Fig. 2

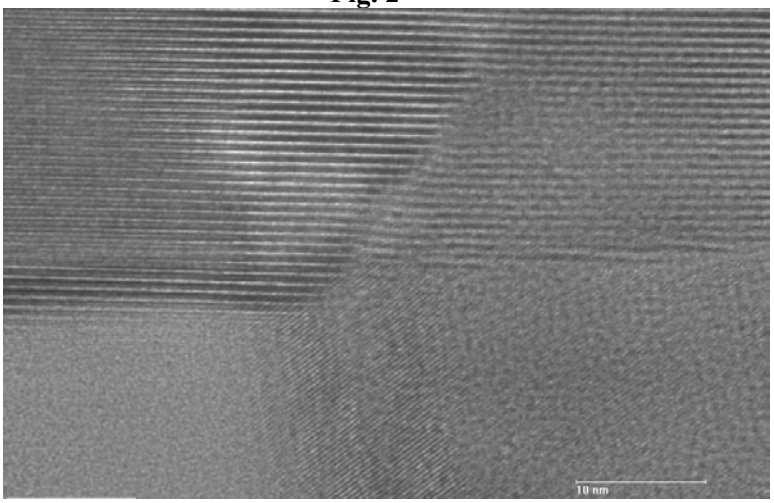

Fig 3