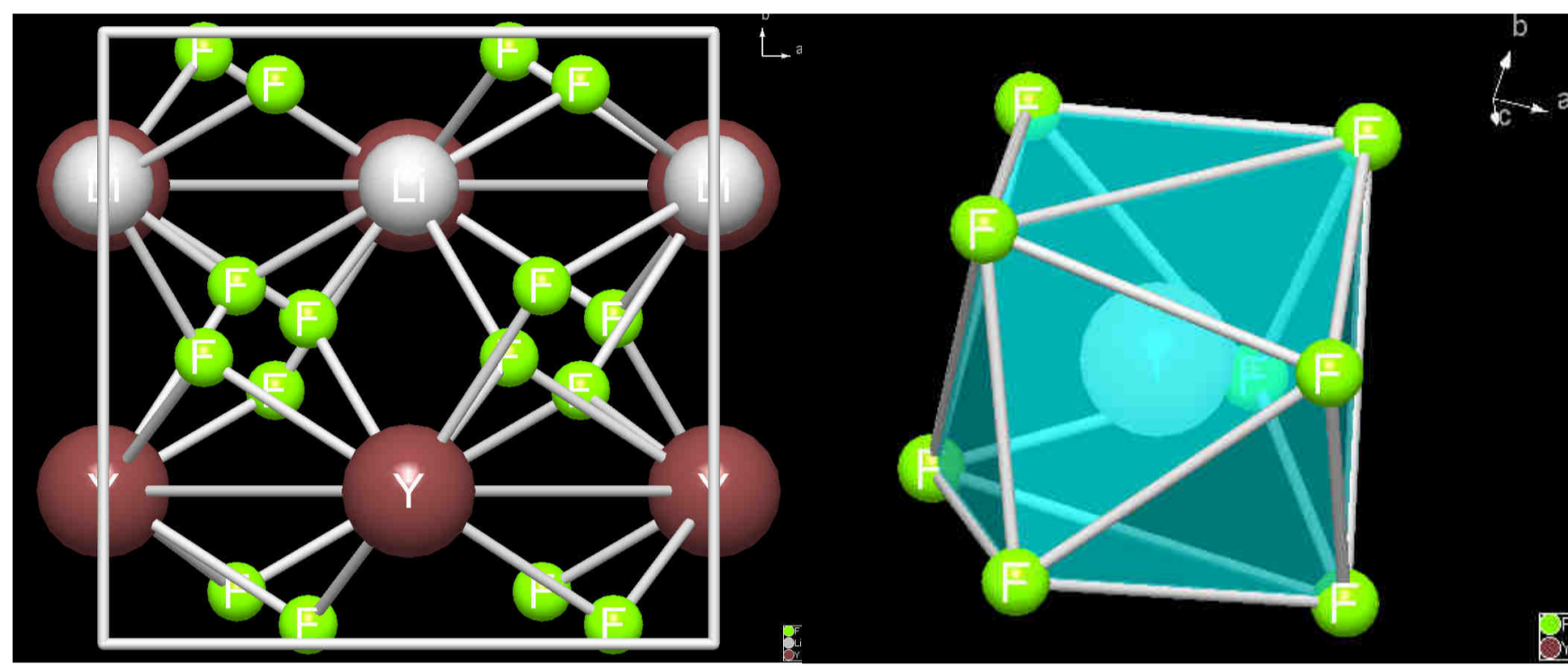


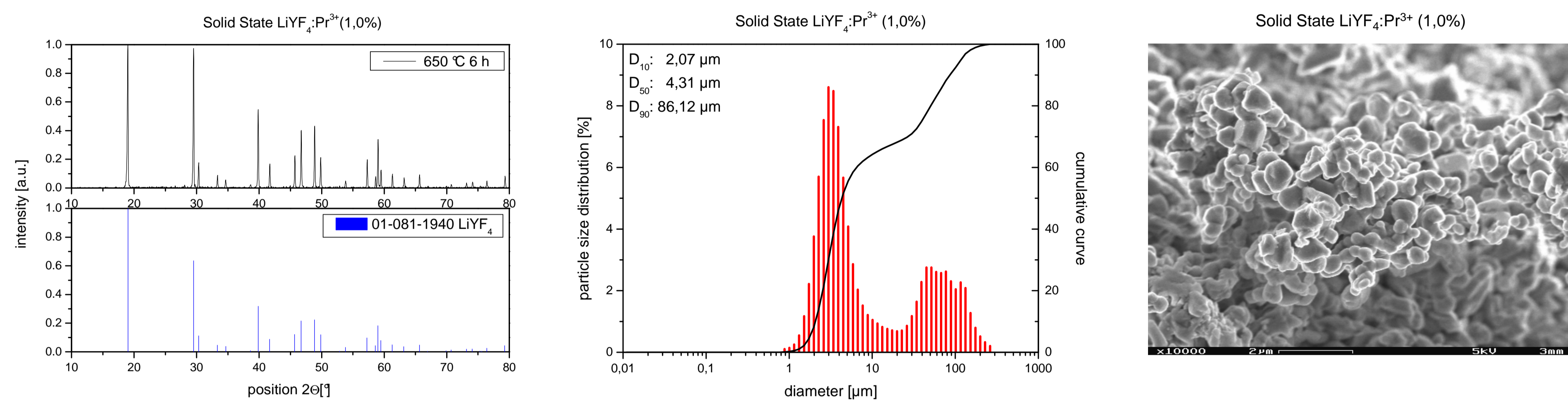
**Background**

During the last decades, a large variety of nanoscale materials have become an essential part of lighting, imaging, and sensing applications. Due to the strongly deviating physical properties of nanoscale materials from bulk materials a closer look into novel systems is always of considerable interest.

**Fig. 1** Crystal structure of LiYF<sub>4</sub>**Synthesis**

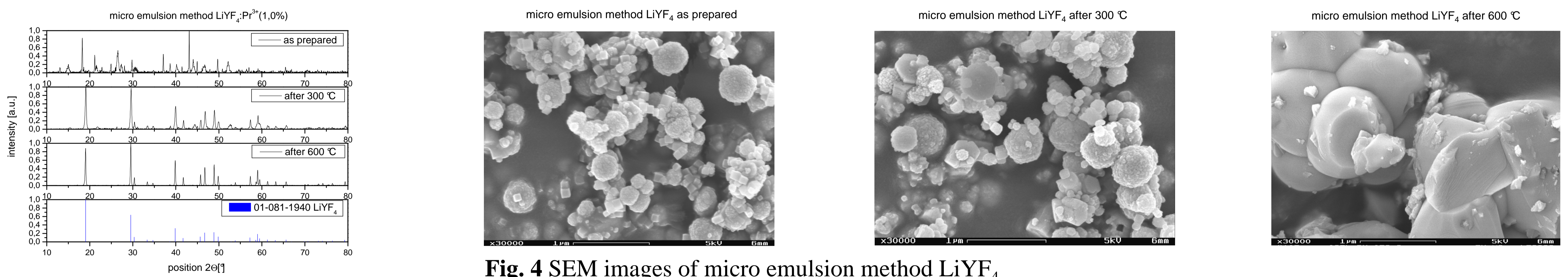
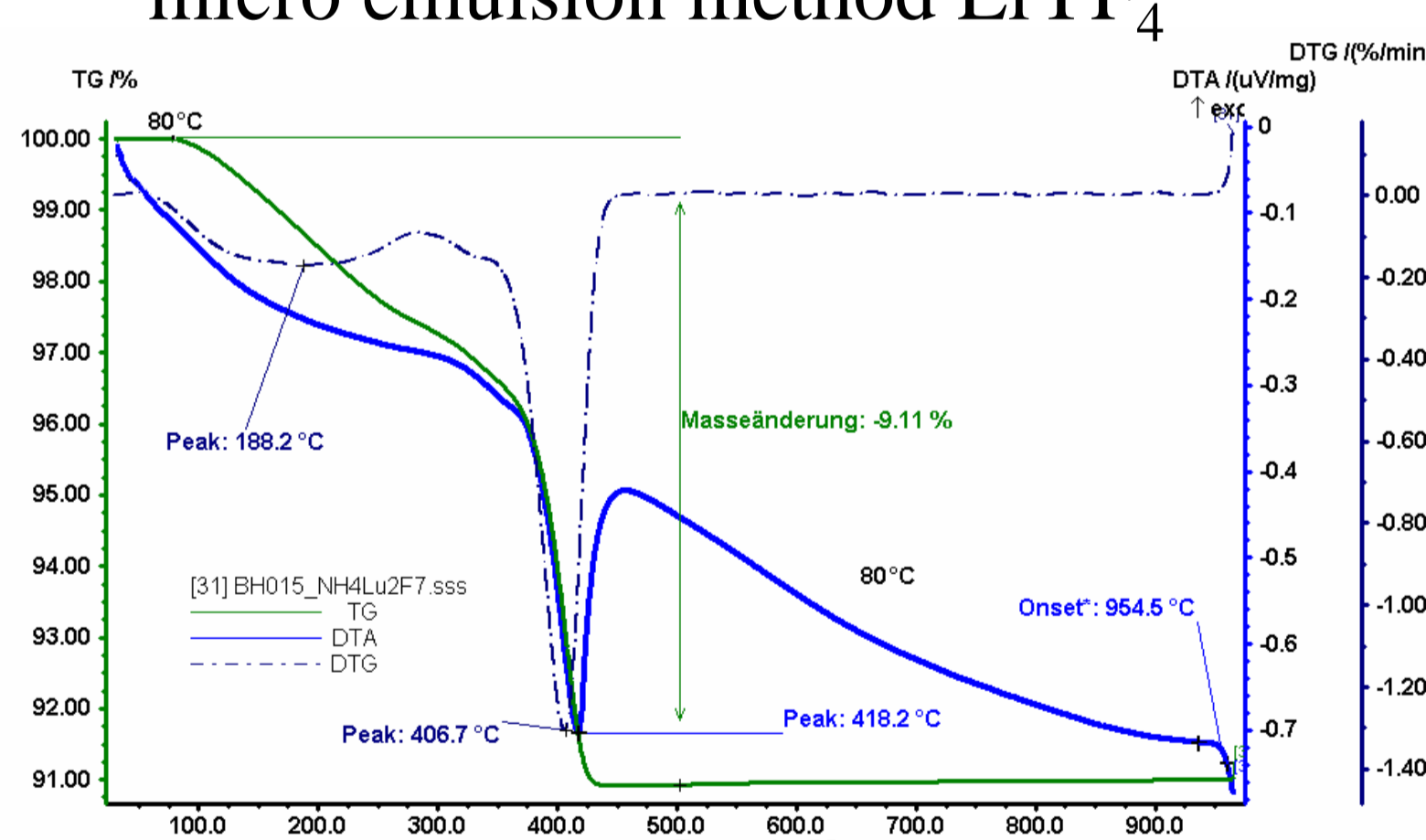
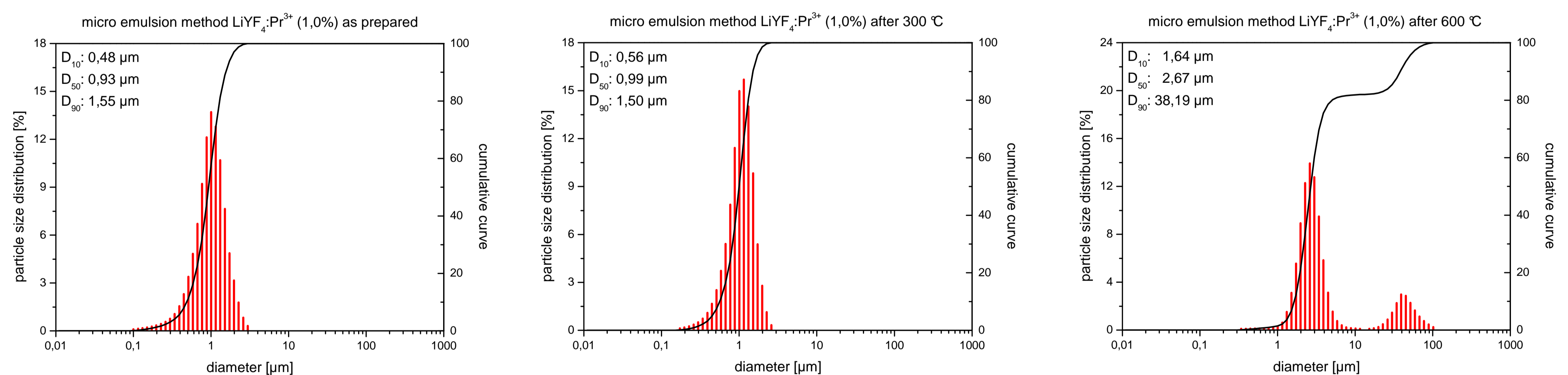
For comparison purposes, the bulk material was prepared via solid state method using a homogeneous mixture of the corresponding fluorides, viz. high purity YF<sub>3</sub>, LiF, and PrF<sub>3</sub>. Stoichiometric blends were sintered at 650 °C for 6 h in a dried Nitrogen stream to obtain single phase material.

In this study, the host lattice LiYF<sub>4</sub> was investigated w.r.t. synthesis of nanoscale batches and optical properties. It exhibits a tetragonal crystal system (I41/a), a wide band gap (10.6 eV), and a low melting point of 819 °C. This host lattice can be easily doped by trivalent lanthanides (viz. Pr<sup>3+</sup>) onto the Y<sup>3+</sup> position, which results in UV and VIS emitting luminescent materials. Such nanoscale LiYF<sub>4</sub> powder might be a suitable precursor for the preparation of transparent ceramic laser materials.

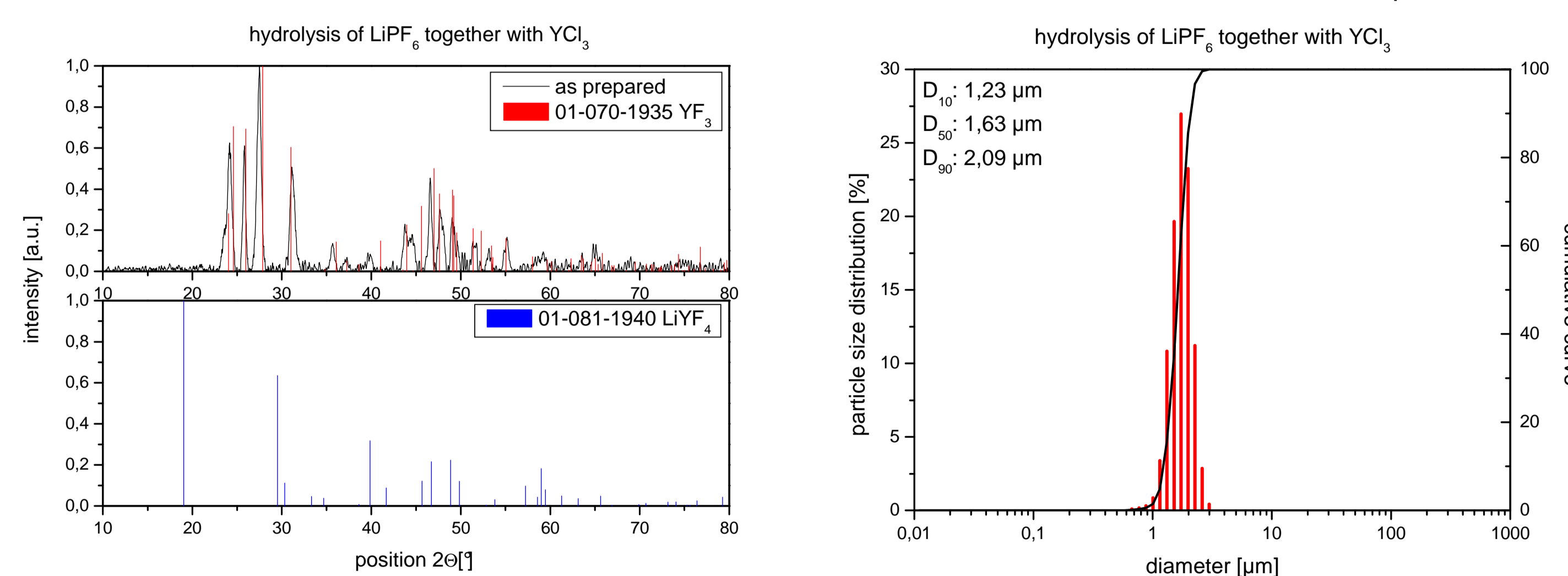
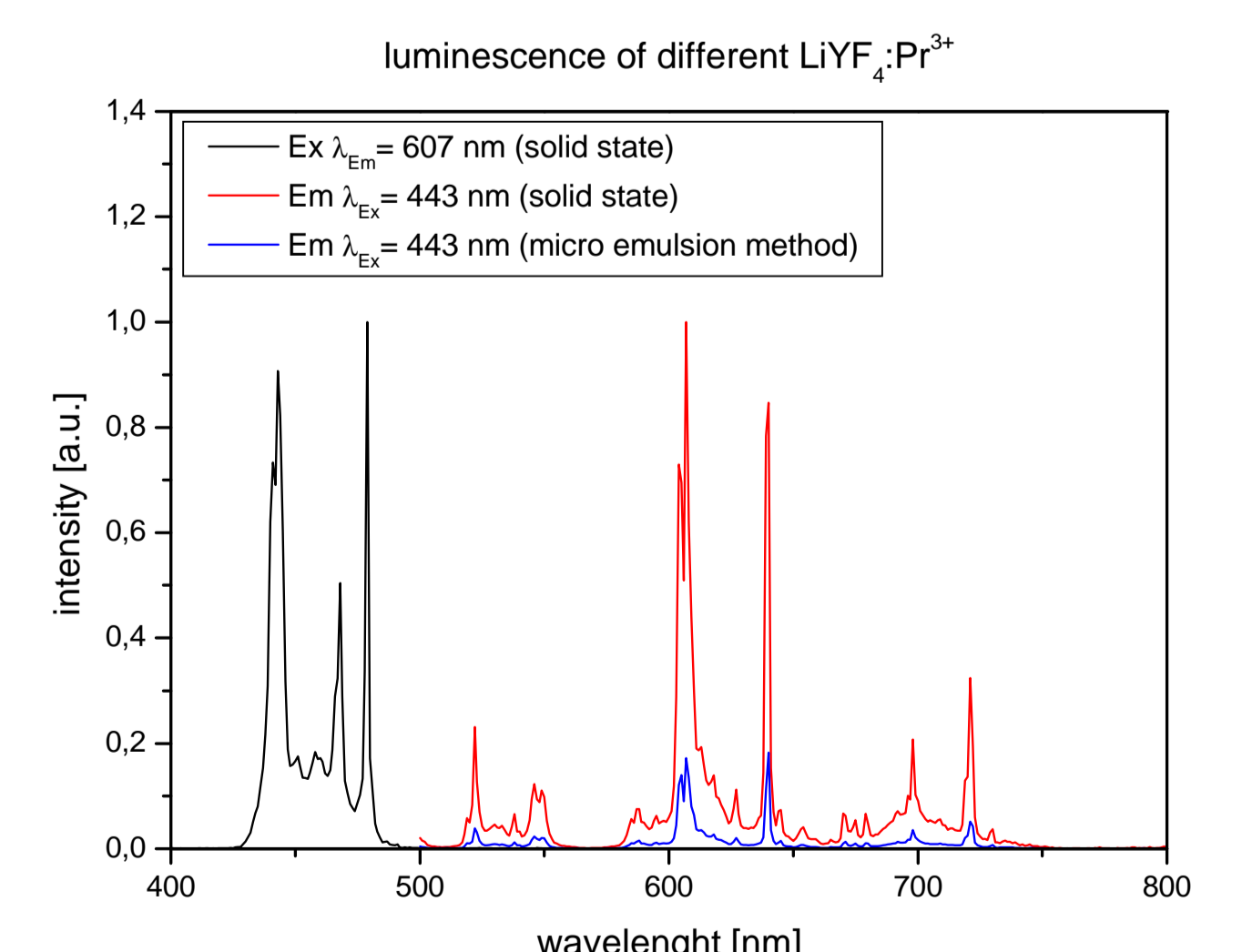
**Fig. 2** Powder diffraction pattern, particle size distribution, and SEM images of solid state LiYF<sub>4</sub>

The so-called micro emulsion method is using cetyltrimethylammonium bromide (CTAB) dissolved in n-octanol. In the resulting micelles a mixture of high purity LiCl, YCl<sub>3</sub> diluted in water was introduced. An important aspect is the proper ratio of Li<sup>+</sup> to Y<sup>3+</sup> due to the different solubility of the corresponding fluorides. Subsequently, a second solution with an excess of NH<sub>4</sub>HF<sub>2</sub> was introduced in the micelles and the precipitation of the fluorides took place.

Resulting powders were sintered at different temperatures to investigate particle agglomeration and to increase the crystallinity of the particles.

**Fig. 3** Powder diffraction pattern of micro emulsion method LiYF<sub>4</sub>**Fig. 4** SEM images of micro emulsion method LiYF<sub>4</sub>**Fig. 5** DTA/TG of micro emulsion method LiYF<sub>4</sub>**Fig. 6** Particle size distribution of micro emulsion method LiYF<sub>4</sub>

Hydrolysis of LiPF<sub>6</sub> forms Fluorine anions (F<sup>-</sup>) and Lithium cations (Li<sup>+</sup>) by heating in aqueous solution. For the precipitation of LiYF<sub>4</sub>, YCl<sub>3</sub>, and LiPF<sub>6</sub> was diluted in water. These solution was heated until cooking and after several minutes the precipitation of the fluorides took place.

**Fig. 7** Powder diffraction pattern and particle size distribution of hydrolysed LiPF<sub>6</sub> together with YCl<sub>3</sub>**Fig. 8** Luminescence spectra of different prepared LiYF<sub>4</sub>:Pr<sup>3+</sup>**Conclusions**

Single phase LiYF<sub>4</sub>:Pr<sup>3+</sup> could be easily prepared by sintering in a Nitrogen stream. Moreover, it was found, that the direct precipitation of LiYF<sub>4</sub> under certain conditions is not possible. The micro emulsion method leads to LiF and NH<sub>4</sub>Y<sub>2</sub>F<sub>7</sub> particles in nanoscale. After sintering at 300 °C NH<sub>4</sub>Y<sub>2</sub>F<sub>7</sub> decomposes to YF<sub>3</sub>, NH<sub>3</sub> and HF. Resulting LiF and YF<sub>3</sub> particles reacting to LiYF<sub>4</sub>. Hydrolysis of LiPF<sub>6</sub> leads to YF<sub>3</sub> particles while the Li<sup>+</sup> ions remain in solution. Comparison of the luminescence properties of the different particles shows that solid state LiYF<sub>4</sub> has the highest luminesc. intensity.