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Pulsed glow discharge enables direct mass spectrometric measurement of fluorine in crystal materials – fluorine quantification and depth profiling in fluorine doped potassium titanyl phosphate

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Abstract

A pulsed direct current glow discharge time-of-flight mass spectrometry (GD TOF MS) method for the quantification of fluorine in insoluble crystal materials with fluorine doped potassium titanyl phosphate (KTP) $KTiOPO_4$:KF as an example has been proposed. The following parameters were optimized: repelling pulse delay, discharge duration, discharge voltage, and pressure in the discharge cell. Effective ionization of fluorine in the space between sampler and skimmer under short repelling pulse delay, related to the high-energy electron impact at the discharge front, has been demonstrated. A combination of instrumental and mathematical correction approaches was used to cope for the interferences of $^{38}Ar^{2+}$ and $^{1}H_3{}^{16}O^{+}$ on $^{19}F^{+}$. To maintain surface conductivity in the dielectric KTP crystals and insure its effective sputtering in combined hollow cathode cell, silver suspension applied by the dip-coating method was employed. Fluorine quantification was performed using relative sensitivity factors. The analysis of a reference material and scanning electron microscope-energy dispersive X-ray spectroscopy were used for validation. Fluorine limit of detection by pulsed direct current GD TOF MS was 0.01 mass %. Real sample analysis showed that fluorine seems to be inhomogeneously distributed in the crystals. That is why depth profiling of F, K, O, and P was performed to evaluate the crystals' non-stoichiometry. The approaches designed allow for fluorine quantification in insoluble dielectric materials with minimal sample preparation and destructivity as well as performing depth profiling to assess crystal non-stoichiometry.

Keywords: fluorine; pulsed glow discharge; time-of-flight mass spectrometry; potassium titanyl phosphate (KTP); nonlinear optical materials

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