MS25 P08

Contribution of microchemical surface analysis of Archaeological artefacts

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Many analytical methods have been used to identify the chemical composition of archaeological artefacts [1-4].

Museum CIRTA of the town of Constantine has a collection of more than 35000 coins and statuettes going back to Numide, Roman, Republican, Vandal and Byzantine times and is struck in the name of the cities, of the kingdoms and the empires.

Surface analysis of these coins gives informations about the chemical composition and leads to recommendations for restoration and presevation.

This work is a contribution of microchemical surface study of three coins with the effigy of the *Numide King Massinissa* (between 3rd and 2nd century before Jesus Christ).

Scanning electron microscopy coupled with energy dispersive spectrometry (SEM + EDS) and energy dispersive fluorescence spectrometry (XRF) were used. The bulk of three coins is massif homogeneous metallic. Two coins are manufactured with lead (99.896% and 61.56%) and were probably conserved differently. The third one is from copper (92.28%).

The optic microscopy (OMP) and SEM pictures of coins show heterogeneous surface with typical lead alteration products white patina of plumbonacrite, cerussite and hydrocerussite and red patina of cupric oxide on the cupric artefact.

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MS25 P09

Effects of Er^{3+} and Yb^{3+} doping on phase transitions of $LiNH_4SO_4$.

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Keywords: $LiNH_4SO_4$, phase transition, rare earth.

We are interested in the study of crystalline structure, dielectric and optical properties of different matrices, especially ferroic tungstates and molybdates because they have possible applications as laser crystals when they are doped by active ions [1]. It is known that the molybdates and tungstates often have very close structural relation with sulfates. Moreover, there are phase transitions in sulfates which remind of the transitions in molybdates and tungstates (or vice versa). It would be interesting to compare these phenomena in both compound classes [2].

This work is concerned with the structural, dielectric, and optical study of the sequence of phase transition in $LiNH_4SO_4$ (LAS), doped by Er^{3+} and Yb^{3+} at different concentrations. Single crystals were obtained from slow evaporation of aqueous solutions. ICP and optical emission experiments have confirmed the presence of the rare earths in the prepared samples. We have solved the structure, at room temperature, of several single crystals by X-ray diffraction of different concentrations and dopes, and we have observed some changes with respect to the expected polar orthorhombic space group ($P2_1nb$). For example, a monoclinic centrosymmetric space group ($P2_1/c$) is observed in a single crystal doped by Yb^{3+} at 1% mol.

In order to detect changes in the sequence of phase transition, specially for crystals with different structures at room temperature, we have carried out different experiments in the same temperature range in a cyclic process. From thermal analysis DSC, dielectric spectroscopy and SHG, we have observed the influence of the rare earth with respect to the pure compound [3]: 1) the ferroelectric-paraelectric transition temperature (460K) increases and 2) the ferroelastic-ferroelectric transition (285K) vanishes. Also, we have characterized the crystal phases at different temperatures in the temperature range 200 to 550K, for several single crystals with different structures, at room temperature, by X-ray diffraction and luminescence experiments.

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MS25 P10

New experimental results to clarify the sequence of phases of $LiNH_4SO_4$.

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Keywords: $LiNH_4SO_4$, phase transition, ferroic.

$LiNH_4SO_4$ (LAS) has been the object of many experimental researches, because it exhibit a sequence of phase transitions in the temperature range 10 to 615 K and a variety of structural phase, some of them still controversial [1,2]. The most recent discussion is focused on a new sequence of phases reported when the β-LAS