

MS13-P13**High-Accurate Measurement of Cell Dimensions of Niobium α -phase in Niobium-Oxygen System**Maksim Reylyanu¹, Boris Kodess¹, Igor Komme¹, Aleksandr Alpatov²

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We observe traceability of high-purity niobium properties in the oxides and chemical bonds of niobium-content chemical compounds minerals and related materials [1-3]. Two approaches for the determining of the structural characteristics of niobium are used. The first variant compares our and different results of X-ray studies of “oxygen-free” niobium using conventional and synchrotron X-Ray methods. After additional annealing of samples in ultra-high vacuum and at high temperatures, the oxygen content in the samples does not exceed 0.03 % mass. The nitrogen concentration is very small 9 ± 1 ppm w, the average content oxygen in these samples is $0.015 \pm 0.003\%$ (mass fraction).

The second variant of approach use extrapolates dependence of the unit cell dimension from the oxygen content obtained for samples of the Nb-O system. The impurity content has been estimated by X-ray fluorescence spectrometry on an M1 Mistral analyzer (of Bruker firm). The niobium content is estimated from the measurement results as 99.99% for possible 34 elements. A sample of the alpha-phase of niobium with an oxygen content of about 0.3 wt. % is investigated by single crystal X-ray diffraction. Two complete experiments were performed in the for spherical single, using a detector CCDs, MoK α 1-radiation and a wavelength of 0.070932 nm. At room temperature, from 2200 to 2800, the Bragg reflections are collected for the samples in order to fill to maximum the entire Ewald sphere. The spatial group of the unit cell is Im3m (No. 229). The average value of the two experiments of the unit cell dimension (lattice constant) is 0.33062(5) nm. This value is in good agreement with the results of six experiments given in the ICCD PDF-database.

Based on the results of the experiments of the increased accuracy and data from the references, the dependence of the value of the unit cell dimension of niobium on the oxygen content has been plotted. Linear extrapolation to zero value results in a value of $a=0.32988(5)$ nm. This value is in good agreement with the data on unit cell dimension for “oxygen-free” niobium $a=0.33005(2)$ nm. Thus, this dependence can also be used to estimate the oxygen content in various samples of the alpha-phase in the Nb-O system. Such samples with low content of oxygen are used also to create more efficient thermal converters. Thin protective layers of coatings of niobium oxides are also widely used in various spheres of the industry.

References:

- [1] Vousden, P. (1951). *Acta Crystallographica*, 4(4), 373-376.
- [2] Kodess, B. N. (1979). *Physics Letters A*, 73(1), 53-54.
- [3] Bojko et al. (1972). *Kristallografiya*, 17(3), 683-685, (in russian).

Keywords: high-purity Niobium, system Nb-O, unit cell dimension

MS13-P14**Structural studies of crystalline phases occurring when temperature cycling a stabilised system of sodium sulfate decahydrate**Emily Goddard¹, David Oliver², Colin Pulham¹

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Salt hydrates represent a class of compound with a high latent heat of energy; they absorb a large amount of heat upon melting and release it upon re-crystallisation. They form part of a group of materials known as phase-change materials (PCMs), which are of great interest in the field of heat storage. Each PCM has a defined melting point and energy density and can therefore be tailored for different applications. The materials should show a stable performance over thousands of charge–store–discharge cycles, with no degradation in performance. They should also nucleate reliably and crystallise at a suitable rate. However, few systems fit this profile.

Sodium sulfate decahydrate ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$) is a good candidate for storing low-grade solar energy. However, it melts incongruently, decomposing into a mixture of the anhydrous Na_2SO_4 and a solution of salt in water. Upon formation, the dense anhydrous salt precipitates out to the bottom of the container, decreasing the concentration of the active PCM in the system, so the amount of heat stored decreases with each cycle. Various approaches have been trialled to suppress the incongruent melting and allow the system to be used as a PCM.

This poster describes the development of a method that suppresses the formation of the anhydrous salt, allowing the system to be cycled reliably, reversibly and reproducibly. Crystallographic techniques were used to identify and characterise the various crystalline phases that appeared during temperature cycling of the system.

Keywords: heat storage, phase-change materials, incongruent melting