MS16-P21

Synthesis and properties of puninite-type $A_2Cu_3O(SO_4)_3$ (A = Na, K, Rb, Cs) sulfate materials

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Mineral puninite, $Na_2Cu_3O(SO_4)_3$ was described recently [1] in fumaroles of Tolbachik volcano.

Crystal structure of puninite is based on oxocentered $[O_2Cu_6]^{8+}$ dimers. Four sulphate tetrahedra are 'face-to-face' attached to the dimers, whereas the other sulphate tetrahedral groups provide their linkage in two dimensions. Structural architecture of puninite can be described as being organized via a "*host-guest*" principle.

Five new compounds with the general formula $A_2Cu_3O(SO_4)_3$ (A = Na, K, Na/K, Rb, Cs) were obtained by solid-state reactions using anhydrous reagents A_2SO4 (A = Na, K, Rb, Cs), $CuSO_4$ and CuO mixed in the stoichiometric ratios. The reaction mixture was loaded into platinum crucibles, and kept at 600 °C for 3 h in air, followed by cooling to 25 °C at a cooling rate of 10 °C/min. The products consisted of green crystals of different shape. Pure powder sample of $Na_2Cu_3O(SO_4)_3$ was prepared for the investigation of magnetic properties, electrochemistry, IR spectroscopy and thermal behavior by the means of HT powder X-ray diffraction and DSC-TGA.

The $Na_2Cu_3O(SO_4)_3$ phase as a positive electrode was tested using Swagelok-type cells cycled at room temperature. The difficulties of electrochemical Na^+ extraction have been encountered, and the removal of significant amount of sodium ions from the structure of the title compound was failed. Note, however, that our tests revealed an occurring of a probable redox process corresponding to the $Cu^{2+/3+}$ transition at high voltage of around 4.8 vs Na^+/Na.

This work was financially supported by the Russian Science Foundation through the grant 16-17-10085. Ph.D. of D.O.N. is carried out within the framework of the international French–Russian co-tutorial Ph.D. program. D.O.N. also acknowledges support of the Saint-Petersburg State University through its internal grant. Technical support by the SPbSU X-ray Diffraction Resource Centre is gratefully acknowledged.

References:

[1] Siidra O.I. et al. (2017) Copper oxosulphates from fumaroles of Tolbachik volcano: Puninite, Na2Cu3O(SO4)3 - a new mineral species and structure refinements of kamchatkite and alumoklyuchevskite, European Journal of Mineralogy, 29, 499-510.

Keywords: sulfate, X-ray diffraction

MS16-P22

How heating and surface finishing affects the crystalline and mechanical properties of CAD-CAM dental lithium disilicate glass-ceramic

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The crystalline characteristics of a dental lithium disilicate glass-ceramic (LDGC) determine the esthetics, strength, and applicability of this material in clinical procedures. The computer-aided design and computer-aided manufacturing (CAD-CAM) technique and heat treatments (temperature, pressure and time) precisely controls the material properties of a LDGC during the manufacturing of dental restorations. During clinical manipulation, finishing and polishing procedures have to be performed on dental restorations. These procedures may affect the topography and modify the microstructural properties of dental ceramics. In the present study, the effect of final crystallization and different finishing and polishing procedures on crystalline and mechanical properties of LDGC were analyzed. For this purpose, the crystalline properties of the LDGC (IPS e.max CAD, Ivoclar Vivadent) were evaluated using 1D and 2D X-ray diffraction (XRD) analyses. The 1D-XRD analyses were performed with an X'Pert Pro (PANalytical) powder diffractometer while 2D-XRD patterns were obtained using an X-ray single crystal diffractometer (Bruker D8 VENTURE) equipped with an area detector (PHOTON100). The crystallization mechanism after heat treatment resulted in a conducted transformation process of the material microstructure. Thus, lithium metasilicate (LM) crystals are transformed into lithium disilicate (LD) crystals. The crystallite size, representing the average coherent crystal diffraction domains, were determined as a crystallinity index for LM and LD crystals after final crystallization. This parameter can be also influenced by the chemical composition of the material aimed for specific clinical purposes. The degree of crystallinity (i.e. crystalline to amorphous) was also obtained by 1D-RXD analyses using the ratio of the intensity of the crystalline peaks to the total intensity in the diffraction pattern, including the signal corresponding to the glass component from the ceramic. The 2DXRD patterns showed a randomly orientation of crystals in both pre and post crystallization during heating. These XRD analyses were also complementary with the results obtained by SEM observations. The increment on the crystallite size and crystal orientation during heat treatment could allow the lower presence and size of voids (densification of the material) that could be acted as potential locations for cracks or affect the optical properties of the material surface. Furthermore, finishing and polishing protocols of ceramic dental restorations differently affect the texture of the material surface, resulting in changes on the flexural strength of the restoration. Overall, translucency and mechanical properties of the lithium disilicate glass-ceramic may be influenced by the crystalline properties of the glass-ceramic during final crystallization and finishing and polishing procedures.

Keywords: dental ceramics, x-ray diffraction, silicate crystals