**MS15-P9 Cell Dimensions of Titanium from 10 K to 290 K**

Håkon Hope¹, Boris Kodess²

1. Department of Chemistry, University of California, Davis  
2. ICS&E-VNIIMS, Crystals Metrology Laboratoy, Moscow 119361, Russian Federation

email: hhope@ucdavis.edu

We have measured the unit cell of Ti from 10 K to 290 K, using a Bruker area detector diffratometer. The three lowest temperatures were 10, 28 and 45 K. From 90 K to 290 K we used a Cryoindustries of America nitrogen cooler. Below 90 K we used a helium cooler from the same company. The sus for our measurements are about 0.00007 Å for a and 0.00017 Å for c. 290 K values are 2.95151 Å for a and 4.68483 Å for c. Corresponding 10 K values are 2.94650 and 4.67977 Å.

From 290 K to 45 K we see no unusual behavior, but at 28 K we measure the c axis to be longer than at 10 and 45 K. The a axis did not show any unusual behavior. This is at variance with earlier published measured and computed values, which showed negative thermal expansion for c below 170 K. Our crystal was of high purity, and the temperatures are reliable to better than 1 K. The measurements we have for comparison were performed with a capacitance dilatometer.

**Keywords:** Titanium, thermal expansion, 10 K to 290 K

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**MS15-P10 Structure Determination of Synthetic Shlykovite by Using Rotation Electron Diffraction**

Zhehao Huang¹, Jiho Shin¹, Xiaodong Zou¹


email: zhehao.huang@mmk.su.se

Electron crystallography has attracted rapidly attention in recent years for structure solution of unknown crystals that are too small to be studied by single crystal X-ray diffraction. We developed rotation electron diffraction (RED) for collection and processing of 3-dimensional (3D) electron diffraction (ED) data. It combines fine electron beam tilt and coarse goniometer tilt to collect ED patterns semi-automatically with a large tilt range (~±70º) [1, 2]. The RED method has been used for structural analysis of polycrystalline materials including complex zeolites [3], and would be useful for studying structures of rare and new minerals. For TEM investigations, the crystals are observed in high vacuum. It was found that crystal structures could be changed under such conditions due to the removal of water and organic molecules in the crystal. This problem could be solved if the samples are studied under cryogenic conditions, i.e. at liquid nitrogen temperature.

Recently, we are succeeded for the first time in synthesis of a mineral analogue to shlykovite (KCaSi₄O₉(OH)•3H₂O) [4], which is one type of phyllosilicates and belongs to the montainite family. The synthetic shlykovite crystals show a 2D nanosheet-like morphology. Therefore, the water molecule and organic molecules in the interlayer space are easily removed in high vacuum, which results in a different unit cell with that in the air. Hence, the structure of shlykovite was determined by using cryo-TEM, in which water molecules remained in the structure. By analyzing the reconstructed 3D reciprocal lattice, the space group and unit cell parameters of the synthetic shlykovite were determined as P2₁/c and a=6.59 Å, b=7.03 Å, c=26.99 Å, β=95.1°. The structure could be solved from the RED data. The combination of cryo-TEM and RED technique shows the capability for the analysis of crystals with flexible structures. It is complement to X-ray crystallography for studying crystals which have small sizes and unique morphology. The synthesis of shlykovite reveals a new route to synthesize natural minerals and can improve strategies toward the synthesis of new crystal structures.