Application of Electron Diffraction Tomography: Donwilhelmsite - a New Mineral from the Moon

Mariana Klementova[1,a)], Lukas Palatinus[1], Jörg Fritz[2], Ansgar Greshake[3], Richard Wirth[4], Vera Assis Fernandes[3,5], and Ludovic Ferrière[6]

[1]*Institute of Physics of the Czech Academy of Sciences, v.v.i., Na Slovance 2, Prague, Czechia*
2Saalbau Weltraum Projekt, Liebigstraße 6, D-64646 Heppenheim, Germany
3Museum für Naturkunde Berlin, Invalidenstraße 43, D-10115 Berlin, Germany
4Helmholtz-Zentrum Potsdam - Deutsche GeoForschungsZentrum, D-14473 Potsdam, Germany
5University of Manchester, Williamson Building, Oxford Road M13 9PL Manchester, UK
6Natural History Museum, Burgring 7, A-1010 Vienna, Austria

[a)]Corresponding author: klemari@fzu.cz

Abstract. Electron diffraction tomography is a technique of choice for the structure determination of nanocrystals. It can be performed in any transmission electron microscope (TEM) allowing for high sample tilts and equipped with a precession device. The technique nowadays approaches the accuracy of X-ray diffraction due to the application of data treatment using the dynamical diffraction theory [1].

Calcium aluminium silicon oxide (CAS) phase, the new mineral "Donwilhelmsite" [2], formed in shock melt pockets in the lunar meteorite Oued Awlitis 001. The meteorite is classified as an anorthositic lunar melt rock [3]. Shock melt pockets 100 μm in size of roughly anorthitic chemical composition contain bundles of up to 20 μm long and less than 1 μm wide needle-shaped crystals.

A lamella was cut out of the thick section NHMV-O104 with a focused ion beam (FIB) to study the structure of the needle-like crystals. It was investigated by Precession Electron Diffraction Tomography (PEDT) on a Philips CM 120 (LaB6, 120kV) equipped with a NanoMEGAS precession unit DigiStar and an Olympus SIS CCD camera Veleta (2048x2048px). The data were collected from -50 to +50 deg. with a step of 1 deg. using a precession angle of 1 deg. The data were processed in the PETS software. Structure solution and refinement were performed in the computing system Jana2006. The structure was solved by the charge flipping algorithm using the program Superflip, and refined using dynamical approach [1].

The structure of the needle-shaped crystals was investigated by precession electron diffraction tomography. Eight datasets (Fig. 1) were collected with average lattice parameters a = 5.44(1) Å, c = 12.76(3) Å, space group $P6_3/mmc$. The structure is identical to the one of synthetic crystals experimentally produced at pressures of >15 GPa and temperatures of >1550 K by Gautron et al. [4]. The structure is composed of M1 octahedral sheets that contain Al and Si (Fig. 2). These are intercalated with two M2-octahedra occupied by Al, one larger site occupied by Ca coordinated by 12 oxygen atoms, and two Al-tetrahedra with 50% occupancy. The structure was refined dynamically to *R1(obs)* = 8.98%. The chemical composition derived from the structure model is $CaAl_4Si_2O_{11}$, which is in good agreement with chemical composition of $Ca_{1.02}Al_{3.92}Si_{2.06}O_{11}$ obtained experimentally.

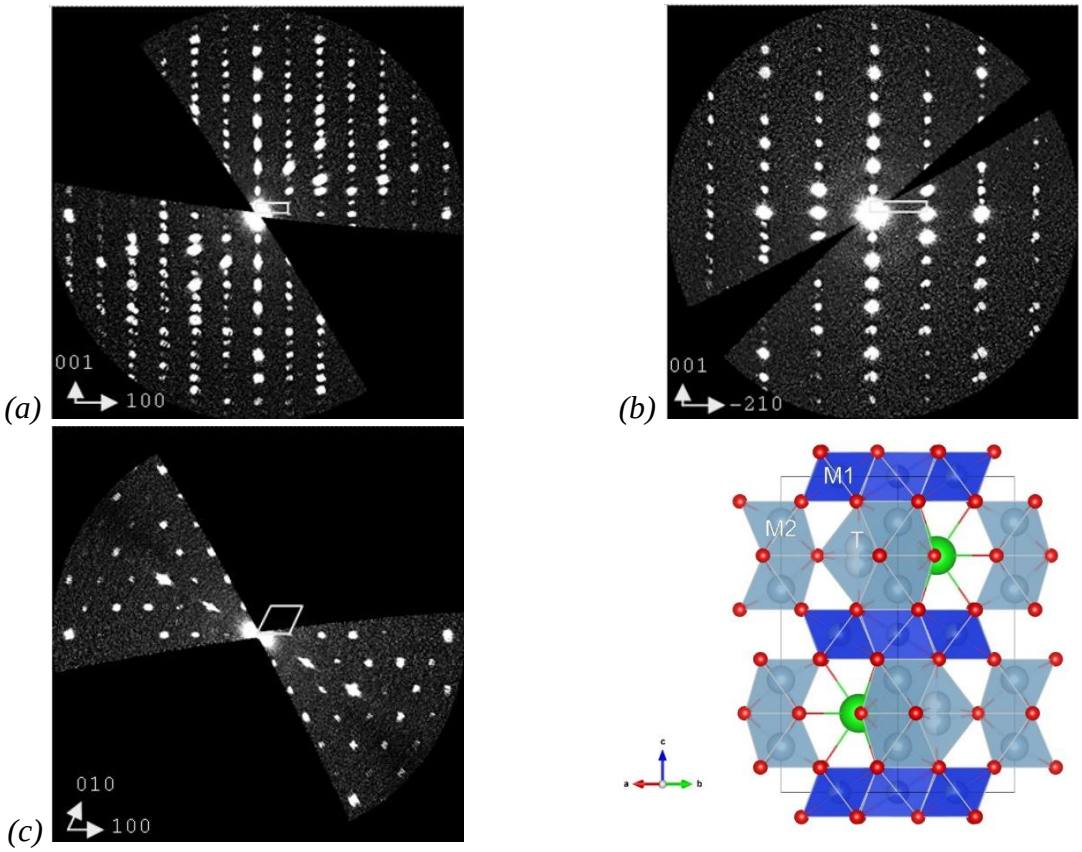

Figure 1. PEDT experiment. Reciprocal space sections (a,b,c). Structure of Donwilhelmsite (d). Structure is composed of octahedral (M1) layers (dark blue) occupied by Al and Si in 1:2 ratio and interlayer containing octahedral position M2 (grey) fully occupied by Al, tetrahedral position T (grey) half occupied by Al, and cavity occupied by Ca (green).

Acknowledgements

The work was performed using instruments of the ASTRA laboratory established within the Operation program Prague Competitiveness (project CZ.2.16/3.1.00/24510), and the infrastructure LNSM under project LM2015087 of the Czech Ministry of Education, Youth and Sports.

References

1. L. Palatinus et al., Acta Crystallogr. A **71** (2015) 235.
2. J. Fritz et al., European Journal of Mineralogy **31** (2019) 201.
3. A. Ruzicka et al., Meteoritics & Planetary Science **52** (2017) 1014.
4. L. Gautron et al., Physics and Chemistry of Minerals **27** (1999) 47.