Microcrystallography, Quetzalcoatlite: A New Octahedral-Tetrahedral Structure from a 2 x 2 x 40 μm^3 Crystal

P. C. Burns,[1] J. J. Pluth,[2] P. J. Eng,[2] M. L. Rivers,[2] J. V. Smith[2]
[1] *University of Notre Dame, Notre Dame, IN, U.S.A.*
[2] *GSECARS, The University of Chicago, Chicago, IL, U.S.A.*

Introduction

Development of new techniques always opens up new opportunities in science. Synchrotron x-ray sources are opening new vistas in crystallography and, at the simplest level, should permit the structure determination for crystals smaller than one micrometer.[1-3] Complex assemblages of fine-grained low-temperature minerals challenge our understanding of mineralogy, both chemically and geologically. Crystal structures of minerals in complex geochemical environments should be related to the paragenetic sequences of the minerals.[4,5] Such an approach is becoming possible, with the determination of many crystal structures in recent years. However, it is only very recently that the introduction of synchrotron radiation[1] and CCD-based detectors[6] to mineralogy has made it possible to solve the structures of crystals with effective volume < 20 μm^3. Determination of the elegant structure of quetzalcoatlite[7] at GeoSoilEnviroCARS (GSECARS) beamline 13-ID-C at the Advanced Photon Source using a 2 x 2 x 40 μm^3 (effective volume 5 μm^3) crystal illustrates the fruitful advances that can be achieved by the combination of scientific and engineering development.

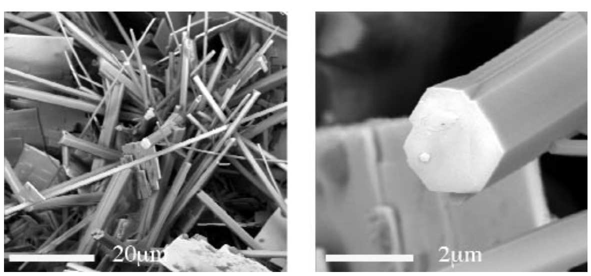

FIG. 1. SEM of quetzalcoatlite crystals.

Methods and Materials

Quetzalcoatlite, $Zn_6Cu_3(TeO_6)_2O_6(OH)_6(Ag_xPb_y)\ Cl_{x+2y}$, $x+y \le 2$, Z = 1, was solved and refined using data collected at beamline 13-ID-C using a 2 x 2 x 40 μm^3 single crystal (Fig. 1).

Results

The structure is trigonal, space group P-31m, a = 10.145(1), c = 4.9925(9) Å, V = 445.0(1) Å^3. Data from the quetzalcoatlite crystal was obtained using the Bruker CCD detector by rotating 0.3 degree in phi in 10 seconds. The data had R(merge) = 0.13 and the structure refined to an R = 0.051.

Discussion

$Te^{6+}O_6$ octahedra and Jahn-Teller distorted $Cu^{2+}O_4(OH)_2$ octahedra share edges to form layers parallel to (001) (Fig. 2) and $ZnO_2(OH)_2$ tetrahedra share vertices to form six rings parallel to (001) (Fig. 3). Layers of octahedra and tetrahedra alternate along c, and form a new framework structure by vertex sharing (Fig. 4). Channels through the framework parallel to c are occupied by alternating Ag or Pb and Cl ions. Electron microprobe analysis revealed Ag and Cl overlooked in the original microchemical analysis. Up to one-third of the Ag was substituted by Pb, and a Pb-rich analog may exist.

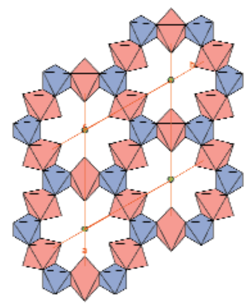

FIG. 2. Quetzalcoatlite layer looking down c axis. Six TeO_6 (blue) and six $CuO_4(OH)_2$ (red) octahedra alternate sharing edges to form 12 rings. Green circles at the center of the rings are Cl atoms.

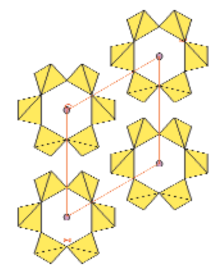

FIG. 3. Quetzalcoatlite layer looking down c axis. Six $ZnO_2(OH)_2$ tetrahedra (yellow) are linked by shared vertices to form 6 rings. Pink circles in the center of the rings are Ag or Pb atoms.

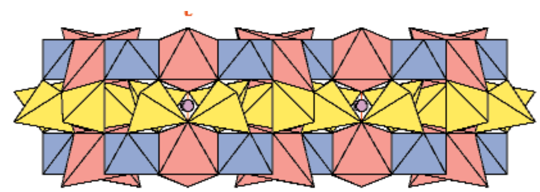

FIG. 4. Quetzalcoatlite layers as viewed perpendicular to c: $ZnO_2(OH)_2$ (yellow), TeO_6 (blue), and $CuO_4(OH)_2$ (red). Tetrahedral layers share vertices with identical octahedral layers above and below.

Acknowledgments

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