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THE CRYSTAL STRUCTURE OF SKIPPENITE, Bi_2Se_2Te, FROM THE KOCHKAR DEPOSIT, SOUTHERN URALS, RUSSIAN FEDERATION

LUCA BINDI[§] AND CURZIO CIPRIANI

Museo di Storia Naturale – sez. di Mineralogia e Litologia, Università degli Studi di Firenze,
via La Pira 4, I–50121 Firenze, Italy

ABSTRACT

Skippenite, Bi_2Se_2Te, was found in a sample from the Kochkar deposit, southern Urals, Russian Federation. The sample forms part of the mineralogical collection of the Natural History Museum of the University of Florence. Electron-microprobe analyses gave the chemical formula $(Bi_{2.06}Cu_{0.05}Sb_{0.03})_{\Sigma 2.14}Se_{2.00}(Te_{0.89}S_{0.11})$. The mineral occurs as anhedral grains up to 0.3 mm across. Skippenite is steel-grey in color, shows a black streak, and has a perfect basal cleavage. In reflected light, the mineral is greyish white, weakly bireflectant, nonpleochroic, moderately anisotropic in yellowish tints. Reflectivity values ($R_{\min}$ and $R_{\max}$, in %) are 47.8, 48.1 (471.1 nm), 49.0, 50.2 (548.3 nm), 49.1, 50.3 (586.6 nm), and 49.3, 50.7 (652.3 nm), respectively. Skippenite is trigonal, space group $R\bar{3}m$, with the following unit-cell parameters: a 4.183(1), c 29.137(3) Å, V 441.5(2) Å^3 and $Z = 3$. The crystal structure has been refined to $R = 2.93\%$. It consists of hexagonal close-packed sheets of Bi, Se and Te, respectively. The unit-cell of skippenite contains 15 layers, and the stacking sequence is: A'cBAc – B'aCBa – C'bACb, where A', B', C' = Te and S, A, B, C = Se, and a, b, c, = Bi.

Keywords: skippenite, Bi–Se telluride, chemical composition, physical properties, crystal-structure refinement, Kochkar, Russia.

SOMMAIRE

Nous avons trouvé la skippenite, Bi_2Se_2Te, dans un échantillon provenant du gisement de Kochkar, dans le secteur sud des Ourales, Fédération Russe. Cet échantillon fait partie de la collection minéralogique du Musée d'Histoire Naturelle de l'Université de Florence. Les analyses effectuées avec une microsonde électronique ont mené à la formule chimique $(Bi_{2.06}Cu_{0.05}Sb_{0.03})_{\Sigma 2.14}Se_{2.00}(Te_{0.89}S_{0.11})$. Le minéral se présente en grains xénomorphes atteignant 0.3 mm. La skippenite, de couleur gris acier, possède une rayure noire et un clivage parfait le long des feuillets. En lumière réfléchie, elle est blanc grisâtre, faiblement biréflectante, non pléochroïque, et modérément anisotrope en teintes jaunâtres. Les valeurs de réflectivité ($R_{\min}$ et $R_{\max}$, en %) sont 47.8, 48.1 (471.1 nm), 49.0, 50.2 (548.3 nm), 49.1, 50.3 (586.6 nm), et 49.3, 50.7 (652.3 nm), respectivement. La skippenite est trigonale, groupe spatial $R\bar{3}m$, avec les paramètres réticulaires a 4.183(1), c 29.137(3) Å, V 441.5(2) Å^3 et $Z = 3$. Nous avons affiné la structure cristalline jusqu'à un résidu R de 2.93%. Elle est faite de feuillets à empilement hexagonal compact d'atomes Bi, Se et Te. La maille élémentaire contient quinze couches, et la séquence d'empilement serait A'cBAc – B'aCBa – C'bACb, avec A', B', C' = Te et S, A, B, C = Se, et a, b, c, = Bi.

(Traduit par la Rédaction)

Mots-clés: skippenite, tellurure de Bi et Se, composition chimique, propriétés physiques, affinement de la structure cristalline, Kochkar, Russie.

INTRODUCTION

In order to evaluate the extent of solid solution between the synthetic end-members Bi_2Te_3 and Bi_2Se_3, Nakajima (1963) synthesized compounds having the chemical composition $Bi_2Te_{3-x}Se_x$, with $x = 0$, 1, 2 and 3. This author established the crystal structure of these synthetic compounds (*i.e.*, Bi_2Te_3, Bi_2SeTe_2, Bi_2Se_2Te and Bi_2Se_3) in the space group $R\bar{3}m$ and reported a detailed study of the relations between the crystallographic data and physical properties. More recently, Dumas *et al.* (1987) showed that the phase relations along the join Bi_2Te_3–Bi_2Se_3 have a more complex subsolidus pattern than previously recognized, and that the range of solid solution for end-member Bi_2Te_3 only partially extended toward Bi_2Te_2Se. Moreover, Sokolov *et al.* (2004) have presented data for the Bi_2Te_3–Bi_2Te_2Se segment of the phase system showing that solid solution in tellurobismuthite, ideally Bi_2Te_3, extends only to 14 mol.% of Bi_2Se_3.

[§] *E-mail address*: lbindi@steno.geo.unifi.it

The natural analogue of the synthetic Bi_2Se_2Te, was found by Johan *et al.* (1987) during a study of selenide and telluride minerals at the Otish Mountains basin, Quebec, Canada. The mineral, named skippenite, occurs as massive aggregates of lamellar crystals intimately associated with watkinsonite, $Cu_2PbBi_4(Se,S)_8$, and with unnamed bismuth selenides whose composition is close to $Bi_2(Se,Te,S)_2$ and $(Bi,Pb)_2(Se,Te,S)_3$. By means of an X-ray single-crystal study (Weissenberg photographs), Johan *et al.* (1987) pointed out that skippenite is rhombohedral with a 4.183(4), c 29.12(8) Å, V 441.3 Å^3, $Z = 3$. In addition, by analogy with the compounds having a tetradymite-like structure (Harker 1934), these authors attributed the space group $R\bar{3}m$ to skippenite. However, taking into account the fact that the skippenite crystals were strongly deformed and of a soft platy nature, Johan *et al.* (1987) did not carry out a crystal-structure refinement.

More recently, in a review of the minerals occurring at the Bambolla, Bambollita and San Miguel mines, Braith *et al.* (2001) reported a further occurrence of skippenite from Sonora, Moctezuma, Mexico. However, their study was limited to a description of the occurrence; they did not report new information on skippenite. In addition, skippenite was found from the Kurile–Kamchatka belt by Kovalenker *et al.* (1989), and from the Kochkar deposit (Spiridonov 1995).

In the course of a research project dealing with the characterization of tellurium-bearing minerals of the mineralogical collection of the Natural History Museum of Florence (Bindi & Cipriani 2003a, b, 2004a, b, Cipriani & Bindi 2004), we discovered a further occurrence of skippenite from the Kochkar deposit, southern Urals, Russian Federation (catalogue number 48034/G, labeled saddlebackite). Associated minerals are aleksite, $PbBi_2Te_2S_2$, and galena. The mineral occurs as anhedral grains up to 0.3 mm.

In this paper, we provide a physical and chemical characterization of skippenite from the Kochkar deposit, together with a refinement of the crystal structure.

OCCURRENCE

The Kochkar gold district in the East Uralian Zone of the southern Urals is located in late Paleozoic granitic gneisses of the Plast massif. Gold mineralization is associated with tabular quartz lodes that are preferentially developed along the margins of easterly trending mafic dykes (Kisters *et al.* 2000). Deformation and formation of associated gold–quartz lodes occurred close to the peak of metamorphism at conditions of the upper-greenschist to lower-amphibolite facies. Fracturing in the gneissic rocks promoted the access of mineralizing fluids (Kisters *et al.* 2000). Both ore-controlling dykes and gold–quartz lodes were subsequently overprinted by lower greenschist-facies assemblages, and an associated hydrothermal alteration postdated the gold mineralization. In the Kochkar gold deposit, mineralization is linked to mylonitized dykes, intrafolial folds and shear bands, which confirms the late nature of mineralization. All the quartz veins show evidence of recrystallization; this is evidence of a dynamic regime of formation on a regional scale.

PHYSICAL AND OPTICAL PROPERTIES

Skippenite is steel grey and shows a black streak. The mineral is opaque with a high metallic luster. Crystals are flattened along [0001], with a perfect {0001} cleavage. The dominant form is {0001}, and twinning is not observed. The micro-indentation measurements carried out with a VHN load of 25 g gave a mean value of 66 kg/mm^2 (range: 60–71), corresponding to a Mohs hardness of about 2½–3. This value is in good agreement with that found by Johan *et al.* (1987) for skippenite from the type locality (63 kg/mm^2).

In reflected light, skippenite is greyish white, weakly bireflectant, nonpleochroic, moderately anisotropic in yellowish tints. Internal reflections are absent.

Reflectance measurements were performed in air by means of a MPM–200 Zeiss microphotometer equipped with a MSP–20 system processor on a Zeiss Axioplan ore microscope. The temperature of the filament was approximately 3350 K. An interference filter was adjusted, in turn, to select four wavelengths for measurement (471.1, 548.3, 586.6, and 652.3 nm). Readings were taken for specimen and standard (SiC) maintained under the same focus conditions. The diameter of the circular area for measurement was 0.1 mm. Measurements of reflectivity (in %) for $R_{\min}$ and $R_{\max}$ are 47.8, 48.1 (471.1 nm), 49.0, 50.2 (548.3 nm), 49.1, 50.3 (586.6 nm), and 49.3, 50.7 (652.3 nm), respectively. Figure 1 illustrates the excellent agreement between the reflectance percentages obtained for the skippenite studied here with those measured by Johan *et al.* (1987) for type-locality skippenite.

CHEMICAL COMPOSITION

A preliminary chemical analysis using EDS, performed on the same crystal fragment used for the structural study, did not indicate elements ($Z > 9$) other than Bi, Te, Se and very minor Cu, Pb, Sb and S. The chemical composition was then determined with a JEOL JXA–8600 electron microprobe. Concentrations of the major and minor elements were determined at an accelerating voltage of 20 kV and a beam current of 40 nA, with variable counting times: 30 s was used for Bi, Te and Se, and 60 s for the minor elements Cu, Pb, Sb and S. For the wavelength-dispersion analyses, the following lines were used: Bi$M\alpha$, Te$L\alpha$, Se$K\alpha$, Cu$K\alpha$, Pb$M\alpha$, Sb$L\beta$, S$K\alpha$. We employed the following standards: pure bismuth (Bi), synthetic Sb_2Te_3 (Sb, Te), clausthalite (Se), pure copper (Cu), galena (Pb, S). The crystal fragment was found to be homogeneous within analytical uncertainty. The average chemical composition (15

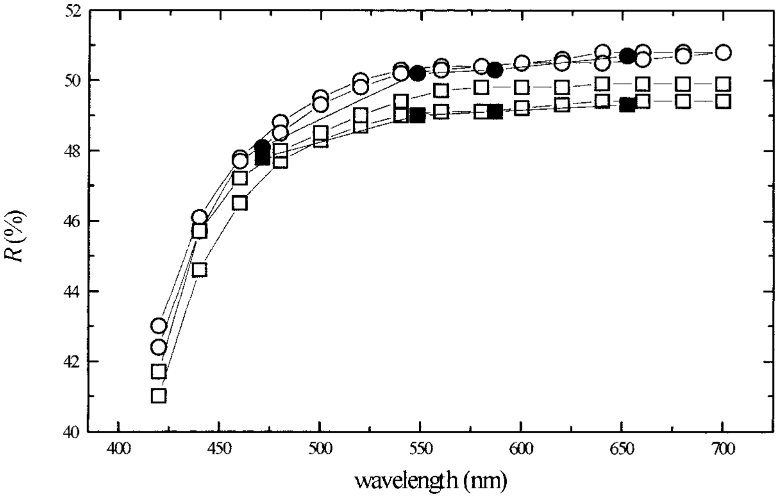

FIG. 1. Reflectivity curves for skippenite samples in air. Open symbols refer to holotype skippenite (two grains; Johan *et al.* 1987), filled symbols refer to skippenite from Kochkar deposit (this study). Circles and squares refer to $R_{\max}$ and $R_{\min}$ values, respectively.

TABLE 1. COMPOSITION OF SKIPPENITE FROM THE KOCHKAR DEPOSIT, SOUTHERN URALS, RUSSIA

element	wt.%	range	σ(%)
Cu	0.42	0.22 – 0.55	0.02
Pb	0.11	0.06 – 0.18	0.01
Bi	60.18	59.65 – 61.08	0.30
Sb	0.44	0.22 – 0.54	0.02
Sc	22.10	21.88 – 22.43	0.30
S	0.50	0.24 – 0.71	0.01
Te	15.82	15.66 – 15.99	0.20
total	99.57		

TABLE 2. DATA AND EXPERIMENTAL DETAILS FOR THE CRYSTAL OF SKIPPENITE SELECTED FOR STUDY

Space group	$R\bar{3}m$
Unit-cell parameters	
a (Å)	4.183(1)
c (Å)	29.137(3)
V (Å^3)	441.5(2)
Crystal size (μm)	10 × 25 × 25
Wavelength	Mo$K\alpha$ (50 kV, 30 Ma)
Theta range (°)	1–25
Scan mode	ω
Scan width (°)	2.75
Scan speed (°/min)	2.06
Independent reflections	131
Reflections with $F_o > 4\sigma(F_o)$	125
R_{obs} (%)	2.93
R_{all} (%)	3.00

analyses on different spots), together with ranges of element concentrations, are reported in Table 1. On the basis of 3(Se + Te + S), the formula of skippenite studied here is $(Bi_{2.06}Cu_{0.05}Sb_{0.03})_{\Sigma 2.14}\,Se_{2.00}(Te_{0.89}S_{0.11})$, which compares favorably with that reported by Johan *et al.* (1987): $(Bi_{2.05}Cu_{0.04}Pb_{0.01})_{\Sigma 2.10}\,Se_{2.00}(Te_{0.87}S_{0.13})$.

CRYSTAL-STRUCTURE REFINEMENT

A small tabular fragment (10 × 25 × 25 μm) was selected for the X-ray single-crystal diffraction study. Unit-cell parameters, determined by centering 25 high-θ (22–25°) reflections on a Nonius Mach-3 automated diffractometer, are shown in Table 2. Intensity data were collected using Mo$K\alpha$ radiation monochromatized by a flat graphite crystal in ω scan mode. Intensities were corrected for Lorentz and polarization effects, and subsequently for absorption following the semi-empirical method of North *et al.* (1968); the values of the trigonal equivalent pairs were averaged. The merging *R* for the Ψ-scan data set decreased from 18.29% before the absorption correction to 3.81% after this correction. The structure refinement was performed in the space group $R\bar{3}m$, starting from the atom coordinates of the synthetic Bi_2Se_2Te (Nakajima 1963), using the program SHELXL–97 (Sheldrick 1997). The introduction of anisotropic temperature-factors for all the atoms led to R = 2.93% for 125 observed reflections [$F_o > 4\sigma(F_o)$] and R = 3.00% for all 131 independent reflections. Neutral scattering curves for Bi, Te and Se were taken from *The International Tables of X-ray Crystallography*, volume IV (Ibers & Hamilton 1974). Inspection of the difference-Fourier map revealed that maximum positive and negative peaks were 2.00 and 1.77 e^-/Å^3, respectively. Experimental details and *R* indices are given in Table 2. Fractional coordinates of the atoms and anisotropic-displacement parameters are shown in Table 3.

A list of the observed and calculated structure-factors is available from the Depository of Unpublished Data, CISTI, National Research Council, Ottawa, Ontario K1A 0S2, Canada.

RESULTS

Skippenite is a member of the tetradymite group, as defined by Bayliss (1991). This group of minerals consists of hexagonal close-packed sheets linked by van der Waals bonding, with variable stacking sequences that give the **c** axis from 3 × 6 Å to 12 × 6 Å (Strunz & Nickel 2001). In the subgroup with the **c** axis ~5 × 6 Å, there are seven members: tellurantimony, Sb_2Te_3 (Thorpe & Harris 1973, Effenberger & Pertlik 1987), paraguanajuatite, $Bi_2(Se,S)_3$ (Shimizu *et al.* 1988), tetradymite, Bi_2Te_2S (Harker 1934, Pauling 1975), tellurobismuthite, Bi_2Te_3 (Thompson 1949, Effenberger & Pertlik 1987), kawazulite, Bi_2Te_2Se (Kato 1970), skippenite, Bi_2Se_2Te (Johan *et al.* 1987), and saddlebackite, $Pb_2Bi_2Te_2S_3$ (Clarke 1997). If we leave out saddlebackite, which has a different structure (N.J. Cook, pers. commun.), all the others are characterized by space group $R\bar{3}m$, unit-cell dimensions a in the range 4.13–4.38 Å, $28.58 < c < 33.43$ Å and $Z = 3$. The size of the unit-cell parameters for Bi-dominant members of the tetradymite group appears to be a function of the tellurium content. Indeed, the higher the content of tellurium, the larger the unit-cell dimensions (see Strunz & Nickel 2001). Data for skippenite are relatively consistent with the parameters predicted for the chemical composition observed.

On the whole, the crystal structure of skippenite refined here is topologically identical to that previously determined on the synthetic compound Bi_2Se_2Te by Nakajima (1963). It simply consists of hexagonal close-packed sheets of Bi, Se and Te, respectively (Fig. 2). The unit cell of skippenite contains 15 layers, and the stacking sequence is: A'cBAc – B'aCBa – C'bACb where A',B',C' = Te,S; A,B,C = Se and a,b,c, = Bi. This stacking sequence confirms the one inferred by Johan *et al.* (1987) in a comparison of skippenite to other members of the tetradymite group [*i.e.*, Bi_2Se_3, Strunz (1963); kawazulite, Bi_2Te_2Se, Kato (1970)].

TABLE 3. FRACTIONAL COORDINATES AND ANISOTROPIC DISPLACEMENT PARAMETERS OF ATOMS FOR SKIPPENITE

	x/a	y/b	z/c	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}	U_{eq}
Bi	0	0	0.39806(3)	0.0407(6)	U_{11}	0.0356(9)	0.0203(3)	0	0	0.0390(6)
Se	0	0	0.2116(1)	0.0274(8)	U_{11}	0.0209(9)	0.0137(4)	0	0	0.0252(6)
Te	0	0	0	0.0372(9)	U_{11}	0.0252(9)	0.0186(4)	0	0	0.0332(7)

TABLE 4. X-RAY POWDER-DIFFRACTION PATTERNS FOR SKIPPENITE

	1		2			1		2	
hkl	d_{calc}	I/I_{calc}	d_{obs}	I/I_0	*hkl*	d_{calc}	I/I_{calc}	d_{obs}	I/I_0
003	9.712	26	9.71	5	02.10	1.5383	10	1.537	4
006	4.856	10	4.85	6	20.11	1.4952	1	-	
101	3.5949	9	3.584	6	11.15	1.4233	12	1.422	5
012	3.5155	3	-		02.13	1.4088	2	-	
104	3.2436	4	3.237	2	01.20	1.3516	4	1.350	3
015	3.0766	100	3.074	10	125	1.3329	12	1.332	4
018	2.5684	7	2.562	1	128	1.2816	1	1.283	2
10.10	2.2704	37	2.267	7	11.18	1.2801	3	-	
01.11	2.1382	5	2.133	5	21.10	1.2392	8	1.239	4
110	2.0915	33	2.090	8	12.11	1.2163	1	-	-
113	2.0446	3	-		300	1.2075	4	-	-
00.15	1.9425	6	1.944	3	01.23	1.1958	2	-	-
116	1.9209	4	1.916	3	21.13	1.1684	2	-	-
10.13	1.9060	5	-		11.21	1.1562	1	-	-
021	1.8078	1	1.808	2	20.20	1.1352	2	-	-
205	1.7297	17	1.730	4	10.25	1.1095	1	-	-
10.16	1.6270	2	-		20.23	1.0381	1	-	-
208	1.6218	1	1.622	3	03.15	1.0255	2	-	-
00.18	1.6187	1	-		30.15	1.0255	2	-	-

1: d values (in Å) calculated on the basis of a 4.183(1), c 29.137(3) Å and with the atom coordinates reported in Table 2. Intensities calculated using XPOW software version 2.0 (Downs *et al.* 1993).
2: observed powder-pattern and indexing originally reported by Johan *et al.* (1987).

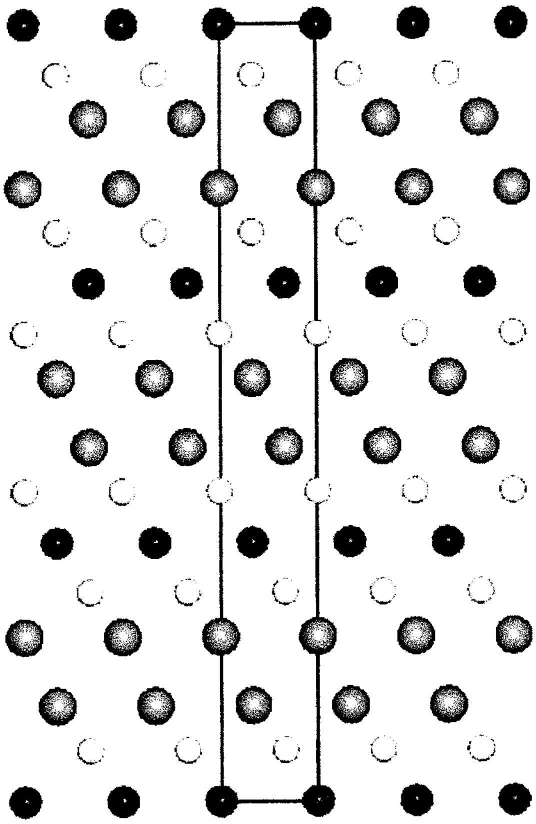

FIG. 2. The crystal structure of skippenite projected along the **a** axis. White, grey and black circles refer to Bi, Se and (Te,S) atoms, respectively. The unit cell is outlined.

The bond lengths between the sheets are: Bi–Se = 2.931(1) Å, Bi–Te = 3.064(1) Å, Bi–Bi = 4.183(1) Å, Te–Te = 4.184(1) Å, Se–Se = 3.564(3) Å, Te–Se = 4.290(2) Å, respectively. These values match closely those observed for the structure of telluronevskite, Bi_3TeSe_2, a recently approved new mineral species (Řídkošil *et al.* 2001).

In Table 4, we compare the X-ray powder pattern originally observed for the type material (Johan *et al.* 1987) with that calculated using the structural parameters obtained in this study. Calculated and observed data match closely.

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