

VAVŘÍNITE, Ni₂SbTe₂, A NEW MINERAL SPECIES FROM THE KUNRATICE Cu–Ni SULFIDE DEPOSIT, CZECH REPUBLIC

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ABSTRACT

Vavřínite, Ni₂SbTe₂, is a new mineral species discovered at the Kunratice Cu–Ni sulfide deposit, Czech Republic. It was observed in one polished section, where it occurs as a lath-shaped grain 20 μm long and as one additional small inclusion. Vavřínite occurs at grain boundaries between grains of pentlandite. The other associated minerals are pyrrhotite, chalcopyrite, violarite, Ni-bearing pyrite, melonite, sperrylite and altaite. In plane-polarized reflected light, the synthetic analogue of vavřínite is white-cream, weakly bireflectant, and weakly pleochroic from pale light brown to pale brown. Between crossed polars, it is anisotropic, without internal reflections. Values of reflectance in the range 400 – 700 nm are tabulated. Synthetic Ni₂SbTe₂ has a silver grey color and metallic luster. It is brittle with a perfect {0001} cleavage. The Vickers microhardness (VHN₁₀) for synthetic Ni₂SbTe₂ is 92 kg/mm² (range 73.4–111.7), which corresponds to a Mohs hardness of ~2. The calculated and measured densities are 7.88 and 7.79 g/cm³, respectively. The average result of three wavelength-dispersion electron-microprobe analyses of vavřínite is Ni 22.92, Fe 1.29, Pd 1.29, Sb 23.65, Bi 0.33, Te 49.95, total 99.43 wt.%, which corresponds to (Ni_{1.93}Fe_{0.11}Pd_{0.06})Σ_{2.10}(Sb_{0.96}Bi_{0.01})Σ_{0.97}Te_{1.93}; synthetic Ni₂SbTe₂ gave Ni 23.85, Sb 24.33, Te 51.77, total 99.95 wt.%, corresponding Ni_{2.01}Sb_{0.99}Te_{2.00} (basis: five atoms per formula unit). The mineral is hexagonal, space group *P6₃/mmc*, with *a* 3.9090(2), *c* 15.6820(9) Å, *V* 207.52(2) Å³ and *Z* = 2. The strongest lines in the powder-diffraction pattern of the synthetic analogue [*d* in Å(*hkl*)] are: 3.3848(13)(100), 2.8421(81)(103), 2.0704(16)(106), 1.9556(100)(110), 1.6114(23)(203), 1.2437(20)(213) and 1.1291(14)(300). The mineral is named in honor of Ivan Vavřín in recognition of his investigations of tellurium minerals and significant contributions to research on Cu–Ni sulfide deposits.

Keywords: vavřínite, new mineral species, Ni–Sb telluride, electron-microprobe data, reflectance data, synthetic Ni₂SbTe₂, X-ray-diffraction data, crystal structure, Kunratice, Czech Republic.

SOMMAIRE

La vavřínite, Ni₂SbTe₂, est une nouvelle espèce minérale découverte dans le gisement de sulfures à Cu–Ni de Kunratice, en République Tchèque. Nous avons trouvé un seul cristal lamellaire 20 μm de long dans une lame polie et aussi une petite inclusion. La vavřínite est située entre des grains de pentlandite. Lui sont associés pyrrhotite, chalcopyrite, violarite, pyrite nickelifère, mélonite, sperrylite et altaite. En lumière réfléchie polarisée parallèle, l'analogue synthétique de la vavřínite est blanc-crème,

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faiblement biréfléctant, et faiblement pléochroïque, allant de brun pâle à brun. Entre nicols croisés, elle est anisotrope, sans réflexions internes. Nous présentons les valeurs de réflectance entre 400 et 700 nm. Le Ni_2SbTe_2 synthétique a une couleur gris argent et un éclat métallique. Il est cassant, avec un clivage {0001} parfait. La microdureté de Vickers (VHN_{10}) pour l'analogue synthétique est 92 kg/mm^2 (valeurs dans l'intervalle 73.4–111.7), ce qui correspond à une dureté de Mohs d'environ 2. Les valeurs calculées et mesurées de la densité sont 7.88 et 7.79 g/cm^3 , respectivement. Le résultat moyen de trois analyses de la vavřinite, effectuées avec une microsonde électronique, est: Ni 22.92, Fe 1.29, Pd 1.29, Sb 23.65, Bi 0.33, Te 49.95, pour un total de 99.43% (poids), ce qui correspond à $(\text{Ni}_{1.93}\text{Fe}_{0.11}\text{Pd}_{0.06})_{\Sigma 2.10}(\text{Sb}_{0.96}\text{Bi}_{0.01})_{\Sigma 0.97}\text{Te}_{1.93}$; le Ni_2SbTe_2 synthétique a donné Ni 23.85, Sb 24.33, Te 51.77, pour un total de 99.95%, ce qui correspond à $\text{Ni}_{2.01}\text{Sb}_{0.99}\text{Te}_{2.00}$ (normalisation sur une base de cinq atomes par formule unitaire). Le minéral est hexagonal, groupe spatial $P6_3/mmc$, avec a 3.9090(2), c 15.6820(9) Å, V 207.52(2) Å³ et $Z = 2$. Les raies les plus intenses du spectre de diffraction X (méthode des poudres) de l'analogue synthétique [d en Å(hkl)] sont: 3.3848(13)(100), 2.8421(81)(103), 2.0704(16)(106), 1.9556(100)(110), 1.6114(23)(203), 1.2437(20)(213) et 1.1291(14)(300). Le nom choisi honore Ivan Vavřín en reconnaissance de ses études des minéraux de tellurium et ses contributions importantes en recherche ciblant les gîtes minéraux sulfurés de Cu–Ni.

(Traduit par la Rédaction)

Mots-clés: vavřinite, nouvelle espèce minérale, tellurure de Ni–Sb, données obtenues avec microsonde électronique, données de réflectance, Ni_2SbTe_2 synthétique, données de diffraction X, structure cristalline, Kunratice, République Tchèque.

INTRODUCTION

In the context of mineralogical investigations of the abandoned Kunratice Cu–Ni sulfide deposit in North Bohemia, Czech Republic, Vavřín & Frýda (1998a) encountered a tellurium-bearing assemblage composed of pyrrhotite, pentlandite, chalcopyrite and an unknown phase of composition Ni_2SbTe_2 . The mineral is probably identical to the unnamed phase Ni_2SbTe_2 reported from a Cu–Ni sulfide deposit in southwestern China (Fleischer *et al.* 1976). We chose to name the mineral *vavřinite* [vavřzhinite] in honor of mineralogist Dr. Ivan Vavřín (*b.* 1937) of the Czech Geological Survey, for his role in long-term investigations of tellurium minerals and his significant contributions to research on Cu–Ni sulfide deposits. The mineral and mineral name have been approved by the Commission on New Minerals and Mineral Names of the IMA (CNMMN 2005–045). The holotype material (one polished section) is deposited at the mineral collection of the National Museum, Prague, Czech Republic, under catalogue number P1p 10/2005. Our purpose here is to document the physical and chemical attributes of the new species, and to shed light on its crystal structure.

OCCURRENCE AND ASSOCIATED MINERALS

The sample containing vavřinite was found on a dump of the abandoned Kunratice Cu–Ni sulfide deposit, located near Šluknov, in northern Czech Republic ($50^\circ 58' 35'' \text{ N}$, $14^\circ 26' 34'' \text{ E}$, Fig. 1). The deposit is hosted by a Cadomian basaltic dyke of calc-alkaline affinity that cuts a granodiorite of the Lusatian Massif (Fediuk *et al.* 2000). The dyke is rich in Te (182 ppm) (E. Jelínek, pers. commun. 2005). It is composed of Ca-rich plagioclase (41%), clinopyroxene (32%), olivine (14%) and biotite (4%). The accessory minerals are mainly Ti-bearing magnetite, ilmenite, and chromite (Fediuk *et al.* 2000).

According to Vavřín & Frýda (1998a), the dominant minerals in all polished sections are pyrrhotite, chalcopyrite, pentlandite and violarite. Common accessories are said to be galena, Ni-bearing pyrite (up to 2.5 wt.% Ni), scheelite and sperrylite. Sperrylite, mainly embedded in pyrrhotite and less commonly in chalcopyrite, represents a major Pt-bearing phase. Baddeleyite also occurs as a rare accessory mineral.

In the Kunratice Cu–Ni sulfide deposit, the tellurides occur as anhedral inclusions enclosed by pyrrhotite. Vavřín & Frýda (1998a) reported that the tellurium-bearing mineralization consists predominantly of melonite and altaite. Melonite was found as oval grains from 4 to 12 μm in size, commonly enclosed by pyrrhotite. In addition, Vavřín & Frýda (1998a) observed two unnamed Pd-bearing phases: $\text{PdNi}(\text{Sb,Bi})\text{Te}_2$ and $\text{Pd}_2(\text{Ni,Fe})_2\text{BiTe}_6$. A detailed investigation of these two unnamed phases is currently in progress.

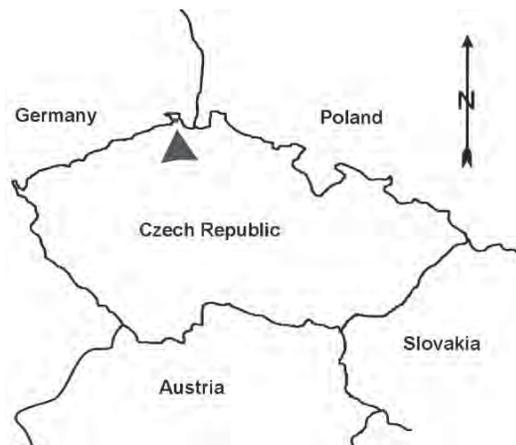


FIG. 1. Location of the Kunratice Cu–Ni sulfide deposit.

The new mineral was discovered in a polished section as a lath-shaped grain 20 μm long embedded between pentlandite grains (Fig. 2). A narrow rim of violarite occurs along the grain boundaries of the pentlandite. Close to the described lamella of the new mineral, there is an additional small grain (<5 μm in length). The wavelength-dispersion spectra acquired on this small grain showed, in addition to nickel, antimony, and tellurium, $\text{FeK}\alpha$ and $\text{SK}\alpha$ peaks. The intensities of the individual peaks in all spectra were found to vary strongly, indicating that the surrounding violarite contributed to the spectrum. Consequently, the small size of the grain precluded any reliable chemical analysis with an electron microprobe. Nevertheless, electron back-scattered diffraction (EBSD) identified this small additional grain as vavřínite (see below). The back-scattered electron (BSE) image of the mineral association is shown as Figure 2.

According to Vavřín & Frýda (1998a), the Cu–Ni sulfide deposit in Kunratice can be compared to the Cu–Ni mineralization in the Ransko Massif (Czech Republic), which is known for its indications of primary magmatic Cu–Ni sulfide ores (Vavřín & Frýda 1998b). In contrast, the narrow rim of violarite along the grain boundaries of pentlandite represents an alteration product of pentlandite. Consequently, taking into account the textural evidence in Figure 2, we infer that vavřínite was formed as a product of low-temperature hydrothermal activity during or soon after the evolution

of the rim of violarite. However, a detailed explanation of vavřínite formation requires further study.

EXPERIMENTAL PROCEDURES

Vavřínite could not be isolated for a thorough characterization owing to its very small grain-size. Therefore, the analogue was synthesized. This synthetic Ni_2SbTe_2 phase was used to determine physical properties, to measure the reflectance, to collect powder data, and to refine cell parameters. A similar approach had also been used in the case of milotaitite (Paar *et al.* 2005). The synthetic analogue of vavřínite was prepared using the silica glass tube method. High-purity elements, tellurium (99.999%), antimony (99.99%) and nickel (99.995%), were used as starting materials. Before being used for synthesis, the nickel was heated for one hour in a stream of H_2 at 400°C and then again for one hour at 800°C. Stoichiometric amounts of Ni, Sb and Te were loaded into a silica glass tube and sealed under vacuum. The sample was heated in a programmable furnace at 400°C for three weeks, and then slowly cooled to room temperature at the rate of 15°C/h. The resulting product is composed exclusively of crystals of Ni_2SbTe_2 ; no other compounds were detected.

Chemical analyses of the synthetic material were performed using a CamScan CS 3200 scanning electron microscope equipped with the WDS Microspec 3PC system (acceleration voltage 25 kV, beam current

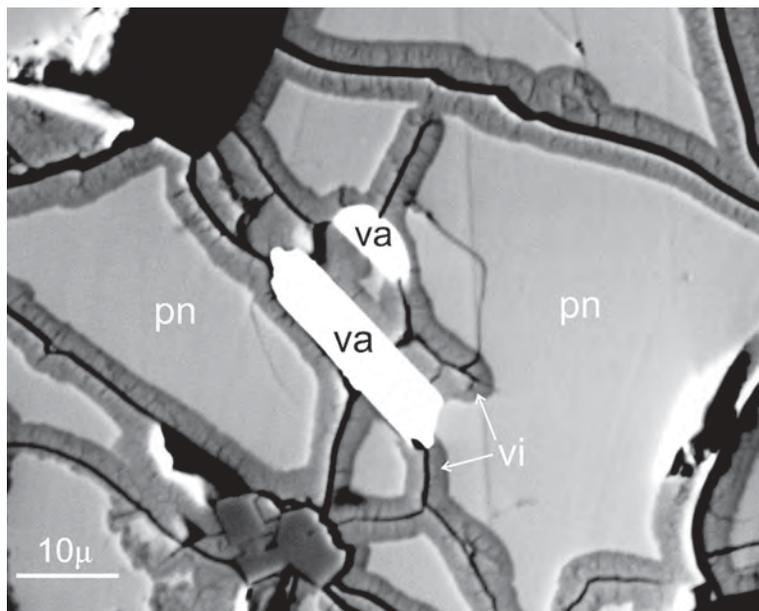


FIG. 2. BSE image of vavřínite and associated minerals. Symbols: va: vavřínite, pn: pentlandite, vi: violarite.

16.6 nA, beam diameter 1 μm , $\phi(\rho Z)$ correction). The following X-ray lines and standards were used: NiK α (pure Ni), SbL α (pure Sb) and TeK α (pure Te).

Reflectance measurements were carried out in air with an Ortholux Pol I (Leitz) microscope equipped with a photomultiplier MPV-1 (Leitz) and a Veril S-200 transmission-interference filter (Leitz). A WTiC reflectance standard was used as a reference material. Microhardness was determined with a PMT-3 tester.

Data for the crystal-structure study were acquired using a four-circle single-crystal diffractometer (Nonius Kappa CCD) at the Centre of Molecular Structures at the Faculty of Science, Charles University, Prague. The program COLLECT (Nonius 1997-2000) was used for data collection. Unit-cell refinement and data reduction were carried out with the program HKL-SCALEPACK (Otwinowski & Minor 1997). After correcting the data for absorption (Spek 2001), the structure refinement was performed with SHELX97 (Sheldrick 1997) using the initial structure-model for synthetic Ni₂SbTe₂ provided by Reynolds *et al.* (2004).

The powder-diffraction data for the synthetic material were collected on a STADIP STOE diffractometer in transmission geometry (CoK α radiation). The sample was placed between mylar foils, and the data were acquired with a position-sensitive detector.

To ensure the identity of natural material and the synthetic analogue, an electron-diffraction scan of back-scattered electrons (EBSD) was performed. We used a CamScan CS 3200 scanning electron microscope (SEM) combined with an EBSD system manufactured by HKL Technology, Habro, Denmark (accelerating voltage 20 kV, working distance 33 mm, tilt angle of the sample 70°). The sample was prepared for investigation by etching the mechanically polished surface with a colloidal silica (OP-U) for 30 minutes to reduce surface damage. The EBSD patterns were collected and processed using a proprietary computer program CHANNEL5 provided by HKL Technology (2004). The

center of six Kikuchi bands was automatically detected using the Hough transform routine (Schmidt *et al.* 1991) with a resolution of 50 (internal Hough resolution parameter in the HKL software). The solid angles calculated from the patterns were compared with a synthetic Ni₂SbTe₂ match unit containing 80 reflectors to index the patterns.

CHEMICAL COMPOSITION AND PHYSICAL PROPERTIES

According to Vavřín & Frýda (1998a), the chemical composition of the natural grain (based on five atoms per formula unit) is: (Ni_{1.93}Fe_{0.11}Pd_{0.06}) Σ 2.10(Sb_{0.96}Bi_{0.01}) Σ 0.97Te_{1.93} [the mean result of three wavelength-dispersion electron microprobe (WDS) analyses]. This composition is close to the ideal formula Ni₂SbTe₂. The empirical formula of the synthetic analogue, based on five atoms, is Ni_{2.01}Sb_{0.99}Te_{2.00} (mean result of five WDS analyses). This empirical formula also matches very well the ideal formula, Ni₂SbTe₂, obtained from the crystal-structure refinement (below). Results of the chemical analyses and resulting empirical formulae are summarized in Table 1.

In plane-polarized reflected light, the synthetic analogue of vavřínite appears white-cream, and weakly pleochroic from slightly light brownish to slightly brown. A pink shade may also be observed. The birefractance is weak. Between crossed polars, the mineral is strongly anisotropic, without internal reflections. This strong anisotropy is also visible on the natural grain of vavřínite. Reflectance data (R_{\min} , R_{\max}) were measured in air and are summarized in Table 2. Figure 3 shows the reflectance curves obtained for synthetic Ni₂SbTe₂ as well as those reported by Picot & Johan (1977) for melonite, NiTe₂, and breithauptite, NiSb.

The synthetic material has a silvery grey color and a metallic luster, and is opaque. The cleavage, most probably parallel to {0001}, is perfect. The phase is brittle. The microhardness (10 g load), determined on six individual grains (20 indentations), ranges between

TABLE 1. ELECTRON-MICROPROBE DATA FOR VAVŘÍNITE AND SYNTHETIC Ni₂SbTe₂

	a) Natural material * (3 analyses)			b) Synthetic material (5 analyses)		
	wt.%	Range	<i>apfu</i>	wt.%	Range	<i>apfu</i>
Ni	22.92	22.54 - 23.35	1.93	23.85	23.69 - 24.02	2.01
Fe	1.29	1.24 - 1.33	0.11	-	-	-
Pd	1.29	1.29 - 1.34	0.06	-	-	-
Sb	23.65	23.56 - 23.78	0.96	24.33	24.13 - 24.52	0.99
Bi	0.33	0.23 - 0.52	0.01	-	-	-
Te	49.95	50.06 - 49.78	1.93	51.77	51.34 - 52.14	2
Total	99.43			99.95		

* Data from Vavřín & Frýda (1998).

TABLE 2. REFLECTANCE DATA FOR THE SYNTHETIC ANALOGUE OF VAVŘÍNITE, MEASURED IN AIR

λ (nm)	R_{min} (%)	R_{max} (%)	λ (nm)	R_{min} (%)	R_{max} (%)
400	46.6	48.5	560.0	50.5	56.9
420	48.0	50.4	580.0	51.4	58.0
440	47.5	51.2	589.0	51.7	59.3
460	47.4	52.0	600.0	52.1	58.9
470	47.5	52.5	620.0	52.9	59.7
480	47.7	52.9	640.0	53.9	60.4
500	48.0	53.9	650.0	53.3	60.6
520	48.7	54.9	660.0	54.1	60.8
540	49.4	55.8	680.0	54.6	61.2
546	49.7	56.1	700.0	55.0	61.4

The standard wavelengths (COM) are in bold.

TABLE 3. POSITIONS AND DISPLACEMENT PARAMETERS (\AA^2) OF ATOMS IN SYNTHETIC Ni_2SbTe_2

	x	y	z	U_{eq}	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Ni	0	0	0.1657(1)	0.012(1)	0.011(1)	0.011(1)	0.015(1)	0	0	0.0039(5)
Sb	$\frac{1}{3}$	$\frac{2}{3}$	$\frac{1}{4}$	0.0082(9)	0.006(1)	0.006(1)	0.012(1)	0	0	0.0031(5)
Te	$\frac{1}{3}$	$\frac{2}{3}$	0.58704(6)	0.0086(9)	0.007(1)	0.007(1)	0.010(1)	0	0	0.0055(6)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

The space group of vavřinite is group $P6_3/mmc$ (#194); the unit-cell parameters are a 3.9090(2), c 15.6820(9) \AA , V 207.52(2) \AA^3 .

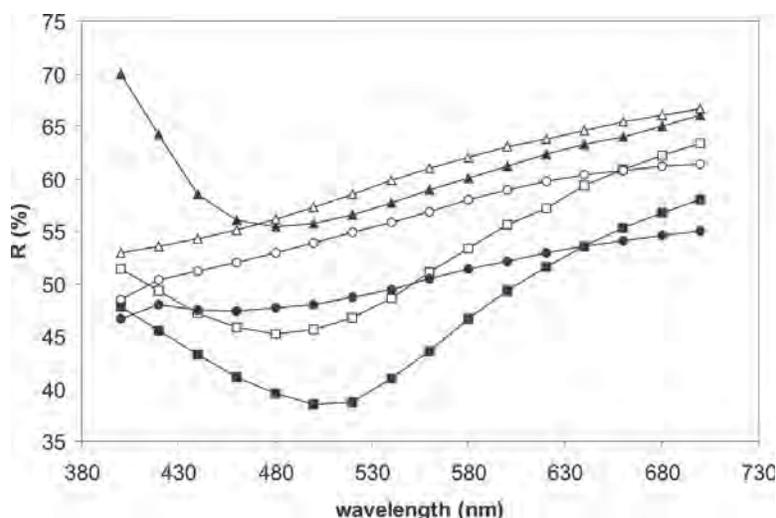


FIG. 3. Reflectance curves for vavřinite, breithauptite and melonite in air. Circles refer to vavřinite, squares and triangles refer to breithauptite and melonite, respectively (Picot & Johan 1977). The reflectance values (R %) are plotted *versus* wavelength.

73.4 and 111.7 kg/mm^2 (mean of 92 kg/mm^2). It corresponds to a Mohs hardness of 2. The density calculated on the basis of the empirical formula is 7.88 g/cm^3 . The density measured by weighing in toluene yields 7.79 g/cm^3 .

CRYSTALLOGRAPHIC DATA AND RELATIONSHIP WITH OTHER MINERALS

The crystal structure of the synthetic phase was refined using the initial structural model provided by Reynolds *et al.* (2004) to an R_1 index of 5.81% for 101 observed unique reflections [$I > 2\sigma(I)$] and corrected for absorption with an empirical correction (Spek 2001). The structure is hexagonal, with a space group $P6_3/mmc$, cell parameters a 3.9090(2), c 15.6820(9) \AA , V 207.52(2) \AA^3 , and $Z = 2$. Atom positions and aniso-

tropic displacement parameters are given in Table 3. A table of observed and calculated structure-factors is available from the Depository of Unpublished Data on the MAC web site [document Vavřinite CM45_xxx].

The structural model proposed by Reynolds *et al.* (2004) was in principle confirmed, as the refined fractional coordinates do not deviate by more than 0.01 from the starting values, although we reported slightly different lattice parameters. This difference may be attributed to different methods of the synthesis.

The unit-cell parameters refined from powder-diffraction data by means of the program BURNHAM (Burnham 1962) based on 19 reflections (Table 4) between 7.817 and 1.129 \AA are: a 3.9115(2), c 15.698(2) \AA , V 207.99(3) \AA^3 .

The structural identity of vavřinite and the synthetic material was confirmed by results of the electron back-

scattered diffraction (EBSD) study. The EBSD patterns (also known as Kikuchi patterns) obtained from the natural material (three measurements from a lath-shaped grain and two measurements from the adjacent small grain) were found to match the patterns generated from the structure data of synthetic Ni_2SbTe_2 yielded by our single-crystal refinement of the structure (Fig. 4).

The values of the mean angular deviation (MAD, *i.e.*, goodness of fit of the solution) between the calculated and measured Kikuchi bands range between 0.316° and 0.785° . These values reveal a very good match; as long as values of mean angular deviation are less than 1° , they are considered as indicators of an acceptable fit (HKL Technology 2004).

The EBSD and chemical study support the identity of the natural and synthetic materials and consequently render legitimate the use of the synthetic phase for a complete characterization of vavřinite. However, it should be noted that in general, the positive match obtained with EBSD is not a conclusive proof of structural identity, since EBSD cannot necessarily distinguish between closely similar structures that differ in a few reflectors observable in specific orientations only. Nevertheless, the positive match between calculated and measured patterns collected on two natural grains in mutually different orientations seems to provide a solid base for identification in this case.

The crystal structure of Ni_2SbTe_2 was described in detail by Reynolds *et al.* (2004) and by Laufek *et al.* (2005). Therefore, we will not review it thoroughly here. Its schematic graphical representation is shown in Figure 5. Reynolds *et al.* (2004) described it as a “hybrid” of breithauptite (NiSb) and melonite (NiTe_2) with an elongate *c* axis. The resulting five-layered structure of vavřinite consists of the stacking sequence CABAC (A: Ni, B: Sb, C: Te). The interlayer Te – Te distance is 3.543 \AA . We note that this interatomic distance is longer than in Te_2 -pair-containing phases

(2.763 \AA in HfTe_2 and 2.793 \AA in ZrTe_2 ; ICSD 2003) or in pure Te (2.84 \AA), but shorter than the van der Waals distance of 4.4 \AA observed in pure tellurium (Emsley 1989). The weak interlayer bonding of Te atoms perpendicular to the *c* axis results in a perfect cleavage along $\{0001\}$ and the plate-like morphology of the crystals. The crystal structure of vavřinite represents a unique structure-type; no structural analogues in the mineral kingdom are known. The most closely related species is hexatestibiopanickelite $[(\text{Ni},\text{Pd})_2\text{SbTe}]$. Both minerals crystallize in the same space-group, $P6_3/mmc$, but the crystal structure of hexatestibiopanickelite displays the mixing of Sb and Te atoms on a single crystallographic site, whereas the vavřinite structure has Sb and Te atoms on distinct crystallographic positions.

TABLE 4. X-RAY POWDER-DIFFRACTION DATA FOR SYNTHETIC Ni_2SbTe_2

<i>l calc.</i>	<i>l meas.</i>	<i>d meas.</i>	<i>d calc.</i>	<i>h k l</i>
3	4	7.8171	7.8460	0 0 2
12	13	3.3848	3.3875	1 0 0
4	5	3.1073	3.1100	1 0 2
80	81	2.8421	2.8433	1 0 3
3	3	2.6158	2.6153	0 0 6
15	16	2.0704	2.0701	1 0 6
100	100	1.9556	1.9558	1 1 0
2	2	1.8980	1.8977	1 1 2
1	2	1.7507	1.7503	1 1 4
20	23	1.6114	1.6113	2 0 3
6	5	1.5664	1.5662	1 1 6
5	5	1.5508	1.5502	1 0 9
6	7	1.4218	1.4216	2 0 6
1	1	1.3082	1.3076	0 0 12
1	2	1.2804	1.2803	2 1 0
19	20	1.2437	1.2436	2 1 3
3	4	1.2151	1.2148	2 0 9
7	7	1.1500	1.1499	2 1 6
15	14	1.1291	1.1291	3 0 0

Values of *d* (measured) and *d* (calculated) are expressed in \AA . Intensities of the strongest seven lines are given in bold.

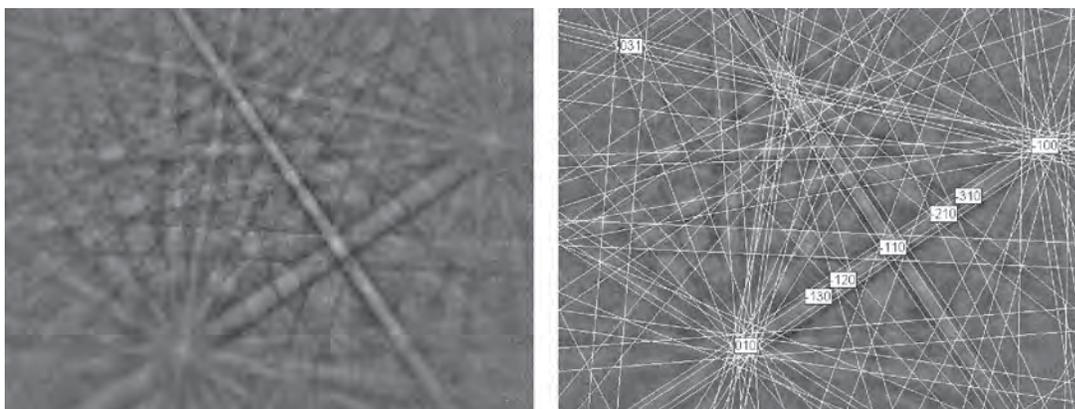


FIG. 4. EBSD image of natural vavřinite; in the right pane, the Kikuchi bands are indexed.

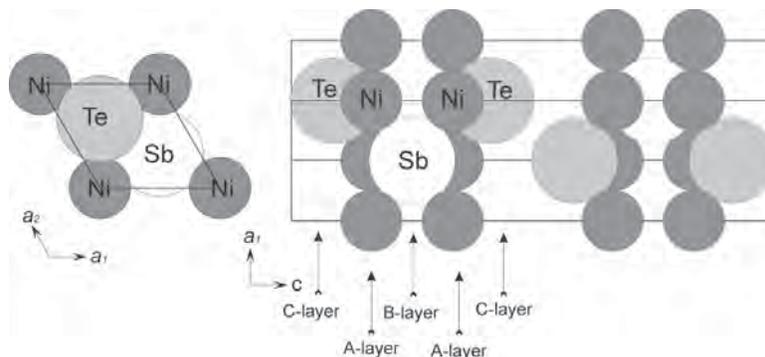


FIG. 5. Crystal structure of synthetic Ni_2SbTe_2 . This view emphasizes the five-layer structure of synthetic Ni_2SbTe_2 with the stacking sequence CABAC (A: Ni, B: Sb, C: Te). Projections onto a_1 – a_2 and a_1 – c planes.

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