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O KRYSTALECH AMALGAMU POLONIA

SUR LA FORMATION DES CRISTAUX D'AMALGAME DE POLONIUM

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J. KOUŘIMSKÝ, E. FILČÁKOVÁ:

Výzkum hadců z dobšinských hald

Исследование серпентинов из добшинских груд

(Předloženo 2. IX. 54)

Z průmyslových důvodů bylo nutné provést systematický výzkum hadců, nacházejících se v Dobšiné a zejména hadcových odpadů, které zbývají po vytěžení asbestových vláken. Vzorky odpadového materiálu byly zkoumány z různých míst hald, zejména při chemických analysách. Tato práce obsahuje rozbor mineralogický, chemický a mikroskopický s použitím elektronového mikroskopu.

Zkoumané vzorky pocházejí z hald odpadového materiálu, který zbývá po těžbě hadcového asbestu ze serpentinového tělesa na severním okraji Dobšiné. S touto exploitací chrysotilu, tvořícího zde žilky o mocnosti až 3 cm, bylo započato již v roce 1924. Chrysotil zde vznikl laterální sekrecí, t. j. vyloužením vodami z matečné hadcové horniny, jejíž geologické stáří nebylo dosud jednoznačně určeno.

Další podobná serpentinová tělesa se nacházejí na sever od tohoto hlavního, asbestem nejbohatšího tělesa a na severovýchod od Dobšiné na jihovýchodním svahu vrchu Čuntava. Mineralogicky zajímavé jsou zde dosti hojné výskyty světlezelených průsvitných krystalků granátu — andraditu, přicházejícího na puklinách těchto dobšinských hadců.

Mikroskopickým výzkumem výbrusů i práškových preparátů dobšinské látky bylo zjištěno, že její podstatnou část tvoří dvě složky, antigorit a chrysotil. Celkově bylo pozorováno větší množství antigoritu než chrysotilu, byly však zjištěny též partie, v nichž je poměr opačný. V práškovém preparátu jsou obě složky dobře rozpoznatelné při použití imersního prostředí o středním indexu lomu. Antigorit má pak indexy lomu vyšší, chrysotil nižší. Oba nerosty zhášejí rovnoběžně.

Antigorit: V preparátu byly zjištěny dva typy krystalků:
1. tabulkovité (hojnější),
2. krátce sloupcovité.

Refraktometricky s použitím imersních olejů byly stanoveny indexy lomu pro světlo sodíkové na krystalech obou typů (viz angl. text). Získané konstanty jsou značně nižší než průměrné hodnoty antigoritu, zatímco dvojlom je poněkud vyšší.

C h r y s o t i l: Tvoří v práškových preparátech dlouhá podélně rýhovaná vlákna i kratší sloupcovité krystalky, ne nepodobné antigoritu. Byl rovněž určen stanovením indexů lomu.

Porovnáme-li získané optické konstanty obou studovaných nerostů s průměrnými hodnotami jak antigoritu tak i chrysotilu, pozorujeme dosti značné rozdíly. Tyto rozdíly jsou pravděpodobně způsobeny zvětráním hadců na haldách, příp. změnou množství vody.

Že jde skutečně o nerosty serpentinové skupiny dokázalo i roentgenografické určení (methodou Debye—Scherrerovou) a výsledky diferenčních thermických analys. Zatímco pomocí roentgenu není možno bezpečně rozlišit antigorit od chrysotilu, dokazují thermické analysy, že jde o serpentin s převahou antigoritu, což je tedy zcela ve shodě s uvedenými určeními optickými.

Chemickým rozborem zkoumané hmoty jsme zjistili, že vzorky obsahující kromě značného procenta SiO_2 a MgO menší příměsi CaO, Fe_2O_3, Al_2O_3, alkalií, FeO a TiO_2. Množství těchto příměsí je z různých partií naleziště různé.

Vláknitý charakter zkoumané hmoty byl zjištěn elektronovým mikroskopem při zvětšení 4000—25.000×. Tomuto submikroskopicky vláknitému charakteru můžeme přisuzovat velký specifický povrch a na základě toho i velkou mechanickou aktivitu tohoto materiálu. Elektronovým mikroskopem byly zkoumány i některé fysikální vlastnosti této látky, jako přilnavost, pórovitost a proces tvrdnutí a tuhnutí ve směsi s cementem.

*

Исследованные образчики происходят из груд отбросов, остающихся после добывания серпентинового асбеста из серпентинового массива на северной окраине Добшины. Уже в 1924 году было начато с эксплуатацией хризотила, творящего здесь жилки до 3 см толщины. Хризотил здесь возник латеральной секрецией, т. е. разливкой воды из маточной серпентиновой горной породы, геологический век которой еще небыл до сих пор твердо установлен.

Иные подобные серпентиновые массивы находятся севернее от этого главного массива, наиболее богатого асбестом, а так-же, северо-восточнее от Добшины на юго-восточном склоне горы Чунтава Минералогически представляют интерес довольно частые здесь находки светло-зеленых прозрачных кристаллов граната — андрадита, встречающагося на трещинах этих добшинских серпентинов.

Микроскопическим исследованием шлифов и порошкообразных препаратов добшинского материала было установлено, что их основную часть составляют антигорит и хризотил. В общем было найдено больше антигорита, нежели хризотила, но местами было установлено и их обратное взаимоотношение. В порошкообразном препарате эти обе составные части ясно отличаются друг од друга при помощи имерзной среди со средним коэфициентом лучепреломления. У антигорита коэфициент лучепреломления более высокий нежели у хризотила. Оба минерала угасают параллельно.

Антигорит: В препарате было определено 2 сорта кристаллов:

1. плиткообразные (чаще),
2. короткими столбиками.

Рефрактометрически с употреблением имерзных жидкостей были определены коэфициенты лучепреломления для натронного света у обоих типов кристаллов (см. английский текст). Найденные константы гозардо меньше средних величин антигорита, в то время как двойное лучепреломление несколько больше.

Хризотил: В порошкообразных препаратах он творит длинные волокна с продольным желобкованием, а так-же и короткие столбообразные кристаллы, довольно схожие с антигоритом. Хризотил был установлен тоже определением коэфициента лучепреломления.

Сравнением полученых оптических констант обоих исследованых минералов со средними величинами как антигорита, так и хризотила, найдем довольно значительные отличия. Эти отличия являются вероятно следствием изветривания змеевиков в отбросовых грудах, а также изменением количества воды.

Что мы имеем дело с минералами серпентиновой группы, доказало и рентгенографическое определение (методой порошка), а также результаты дифференциальных анализов. В то время, как при помощи рентгена нельзя точно розличить антигорит от хризотила, термические анализы доказывают, что мы имеем дело с серпентином, где преобладает антигорит; это установление вполне совпадает с приведенными определениями.

Химический анализ образчиков показал кроме большого процентного содержания SiO_2 и MgO, также и небольшие примеси CaO, Fe_2O_3, Al_2O_3, алкалий, FeO и TiO_2. В различных частях мест нахождения количество этих примесей было различным.

Электроновым микроскопом, при увеличении в 4.000—25.000 раз, был установлен волокнистый характер исследованного материала. Этому суб-микроскопическому волокнистому характеру можно приписать большую специфическую поверхность а поэтому и значительную механическую активность этого материала. Под электроновым микроскопом подвергались испытанию и некоторые его физические свойства, как напр. смачиваемость, поровитость и процесс твердения и сгущения в смеси с цементом.

Research of the serpentine from Dobšiná

The industrial reasons induced us to search the serpentine from Dobšina and in the first place the serpentine as folling of matter which remain after the extraction of the asbestos fibres. The samples of the folling of matter were searched on the various places of the heap of dead rocks chiefly during the chemical analysis. This paper contents mineralogical, chemical and microscopic analys with the use of the electron microscope.

The tested samples cames from heaps of dead rocks of waste product which remains after the exploitation of the serpentine body on the northern border of Dobšiná. The extraction of chrysotile which here forms veins of 1¼″ of thicknes already begun in 1924. Chrysotile has been formed here through lateral secretion from the serpentine rock and the sedimentation on its fissures. These veins of asbestos are here often found mixed with quartz.

The question of the geologic age of serpentines of Dobšiná which have been formed through the decomposition of basic eruptive-rocks has not yet been uniformly solved. According to the older conceptions they have appeared in the primary mountains, according to the more modern judgment of B. KORDIUK(6) from 1941 they are of trissic formation.

The further similar serpentine bodies are to be found on the north of this principal body which is the richest in asbestos, and to north-east of Dobšiná on the south-eastern slope of the mountain Čuntava. The serpentines of Dobšiná have in their overlayers the layers of limestone, the insertions of the same kinds of limestone are also found inside the mountain. The northern body of Dobšiná contains besides the proper serpentines also thinner rests of the original roughly granulated basic rocks. In the underlayer of the powerful Čuntava zone of serpentines, which are substantially poorer in asbestos, are found layers of dolomit in its overlayers light coloured kinds of limestone of the Spiš-layers. Mineralogically interesting are the frequently light-green transparent crystals of garnet-andradite appearing on the cracks of the Dobšiná-serpentine.

The mineralogic determination of the Dobšiná material.

The microscopis research of the grinds and mineral grains of the Dobšiná material has prowed that its essential part consists of two substances — antigorite and chrysotile. On the whole, a greater quantity of antigorite than that of chrysotile has been found, but in some parts the proportion is inverted. Lesser ingredients are represented by small grains of carbonat, garnet — andradite, chromite and magnetite, which are gradually changing into haematite and finally into limonite. Further more the residua of olivine (fig. 1.) and bronzite are being found here which change into antigorite and chrysotile. The microscopic structure of the Dobšiná serpentine is minutely described by O. KALLAUNER (5).

Antigorite and chrysotile are sometimes greatly similar as chrysotile often forms here besides the typical longish fibres also shorter ones and even column-like crystals. Both of thes substances are easily distinguishable with the use of the liguid of the average refractive indice ($n=1,535$). Antigorite has in that case higher indices of refraction, chrysotile has lower ones. Both these minerals show parallel extinction.

A n t i g o r i t e: Two types of crystals have been found in the preparate:
1) The plate-form ones (formed more frequently),
2) The short columnar ones.
An imperfectly developed axial interference figure of an biaxial optically negativ mineral in one of the plate-formed crystals succeeded to investigate. Some crystals of the other type show a slight pleochroism (from colourless to the slightly greenish one). The elongation is positiv.

The refractive indices have been determinated in the crystals of both the types. They have been established refractometrically, with the use of the immersing liquids in the natrium light:

$n\alpha=1,536 \pm 0,001$ (determitated in the crystals of the 2^{nd} type)
$n\beta=1,546 \pm 0,001$ (determinated in the crystals of the 1^{st} type)
$n\gamma=1,549 \pm 0,001$ (determinated in the crystals of the 1^{st} type, 1,550 on the crystals of the 2^{nd} type).
Birefringence = 0,013

The investigated face on the crystals of the 1 st type has been considered to be the basal pinacoid (001) which is the fade of the perfect cleavage of antigorite. The investigated face of the crystals of the 2nd type was considered as the fase (010), the direction of the 2nd cleavage of antigorite. The habitual appearance of the crystals of both the types also corresponds to this crystalographic orientation. The obtained constances are considerably lower than the average values of antigorite. Also the birefringence is somewhat higher (the average of 0,008—0,011).

C h r y s o t i l e: forms long vertically striated fibers and shorted columnal crystals. Here also a very slight pleochroism (from colourless to slightly greenish resp. brownish). The elongation is positive.

These indices of refraction have been determinated:

$$n\alpha, \beta=1,522 \pm 0,001,$$
$$n\gamma=1,535 \pm 0,001.$$

These constants have been determinated in fibrous and columnal crystals with the assumption that the direction of the cleavage is a vertical prism (110) and the direction of the fibre elongation$=c\sim\gamma$. For this reason it is impossible to determine here precisely refractive indices for the optical directions X and Y.

If we compare the obtained optical constants with the values mentioned by G. C. SELFRIDGE in his work about the minerals of the serpentine group (7), we notice rather considerable differences which are obviously influenced by decomposition of the serpentines in the

Table I.

Comparison of X-ray-gram of D-material with chrysotile from Barabba Distr. and with antigorite from Antigorio Valley.

Debye—Scherrer, Ø cameras 63,66 mm, Co $K\alpha_{1,2}$ radiation = = 1,787 kX, filtered through Fe, 25 kV, 60 mA, exposition of 120 min.

No.	Dobšiná		Chrysotile		Antigorite		Note
	I.	d.	I.	d.	I.	d.	
1.	7	7,27	9	7,31	10	7,23	
2.					1	6,26	
3.			1	5,16	1	5,18	
4.	3	4,55	5	4,51	3	4,61	
5.	1	4,28			2	4,19	
6.	5	3,63	} 7	3,46	} 10	3,60	
7.	3	3,35					
8.	7	3,01					
9.	5	2,51	3	2,58	7	2,53	
10.	1	2,43	4	2,45	2	2,40	
11.	1	2,27					Admixture?
12.	3	2,09	1	2,09	4	2,16	
13.	1	1,91					
14.	1	1,87					
15.	1	1,81	1	1,82	2	1,81	
16.	1	1,73	1	1,73	1	1,73	
17.	2	1,59			5	1,57	
18.	4	1,53	5	1,53	4	1,54	
19.	1	1,51			2	1,51	
20.	1	1,45			2	1,45	
21.	1	1,42			1	1,42	
22.	2	1,31	2	1,31	2	1,31	

Intensities of reflections have been determinated visuely.

unseen in the light microscope, their length moves from 10 microns to 0,1 microns, their thickness or diameter moves from 0,1 microns to 0,05 microns (Fig 3.) On the same figure irregularly limited aggrégates on chrysotile fibres are due to the other components of the maternical stones.
This fibrous structure of the waste product-D-material-remains after glowing over 1100 °C.

For comparison of the size of fibers serves figure 4. showing normal chrysotile, these fibres are considerably longer and thicker than the fibres of the waste product.

With these ultramicro fibres of asbestos the usual adhesion to mineral powder is supplemented by a mechanical bond, the amount of this bond in amy given case depending on the specific surface.

The normal asbestos fibres have an extreme large specific surface 2.10^5 cm^2/g, it increases in the presence of ultramicro fibres of asbestos.

The large specific surface as well as the known property of asbestos fibres to catch the fine granulus of mineral powder also of cement, induced us to test some physical properties of this D-material associated with cement for use in the building industry.

On the electron microscope picture No 5. of product asbestos-cement-D-material sheeting, we can observe how particles collect more abundantly on smaller fiber of asbestos than on rough one, due to their larger specific surface. This picture shows the grinded product after secundaric hydratation process and sudden hardening in alcool (75 %).

The pictures 6a and 6b show directly the process of stiffening and hardening of a mixture of D-material and cement by adding water. The interpretation follows each small and needle crystal of hydratation product grown into a single fibre of chrysotile, confirming the view that development of crystal starst at certain preferred centers in this case these crystals grown by hardening together with the present two substances of D-material fibres of chrysotile and fine plates of antigorite forming a compact feltlike armature.

The solidity of arised material depends on the solidity of crystals— product of hydratation — that are in this case reinforced, fixed and joined by the chrysotile microfibres and plates of antigorite, their growing together is also more perfect than by cement alone whereby the porosity of the material arised is much smaller. The smaller porosity causes then a smaller water absorption.

The superiority of the quality of new building material larger solidity, smaller porosity, smaller water absorption were tested on ready made products in practical use and also in laboratory.

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heap of dead rocks resp. by the variation of the quantity of water. With consideration of these differences in the optical constants has been produced the X-ray-gram of the material investigated according to the method Debye-Scherrer. The typical samples of antigorite from the classical locality Val Antigorio in Piemont (Coll. of the Mineral. Dep. of the National Museum, Prague, inv. no. 21.376) and chrysotile from the Barabba Distr. — N. S. Wales (coll, of the same Institution, inv. no. 27.335) have been used for comparison (Tab. I., fig 2.). The obtained values have been compared with the various values of both these minerals mentioned in Hanawalts Tables (Tab. II. and III.) and in the above mentioned work of SELFRIDGE.

This comparison of X-ray-grams has also proved that it was the case of a mineral of the group of the serpentine. At the same time the results of the investigation have shown that it is impossible to recognize accurately chrysotile from antigorite with the use of X-rays as E. ARUJA (2) has already found in the rotating-crystal photograph of both these minerals. In the Debye-grams of antigorite and chrysotile we don't find the typical differences in the position of the reflections. The reflections of chrysotile is perhaps only more diffusible than those of antigorite (see ARUJA), which naturally can not serve as a sufficient criterium for distinguishing them.

That it is the question of a serpentine with the antigorite component prevailing has been also proved by the results of differential thermical analysis made by Dr. V. ŠATAVA from the Instutute of the Technology of the silicates at the High School of chemistry in Prague (8).

The chemical analysis of the Dobšina material and its research by use of the electron microscope.

Following is an average analysis of the same samples of waste material mineralogicaly tested i. e. of the samples of old heaps of dead rocks useless after the extraction of asbestos fibres:

SiO_2 %	MgO %	CaO %	Fe_2O_3 + FeO %	Al_2O_3 %	CO_2 + H_2O %
37,1 to 39,6	36,2 to 37,8	3,6 to 5,2	2,1—5,8	2,3—4	12,5—13,9

The analysis also shows varying small amounts of alkalies. (1,1 %). The single samples differ considerably, according to their origin on various places on the heaps of dead rocks, chiefly by the content of admixtures: $CaCO_3$, FeO, and Al_2O_3.

By ligth microscopic research were observed the presence of short asbestos fibres so to say microscopic fibers, the length of these is less than 0,002″, these pass sieves during the production of asbestos fibres from the original stones finely pulverised.

Research by electron microscope reveals a great quantity of much smaller fibres, ultramicroscopic fibers, entirely

Table II.

Comparison of X-ray-gram of chrysotile from Barabba Distr. with the values mentioned in Hanawalt's Tables for chrysotile from Thetford mines (Quebec — Canada) and from Chelmsford (Massachusetts — USA).

No.	Barabba		Thetford		Chelmsford	
	I.	d.	I.	d.	I.	d.
1.					4	8,0
2.	9	7,31	10	7,31	9	7,3
3.	1	5,16			1	4,93
4.	5	4,51	5	4,52	6	4,46
5.					4	4,00
6.	7	3,43	7	3,65	10	3,64
7.					2	3,01
8.					2	2,85
9.					1	2,71
10.	3	2,58	4	2,56	6	2,57
11.	4	2,45	6	2,45	7	2,44
12.					1	2,27
13.	1	2,09	2	2,09	1	2,09
14.	1	1,82			3	1,82
15.	1	1,73	2	1,73	1	1,73
16.			2	1,71	2	1,69
17.	5	1,53	6	1,53	9	1,52
18.					3	1,46
19.					1	1,41
20.	2	1,31	3	1,31	6	1,30

Table III.

Comparison of X-ray-gram of antigorite from Antigorio Valley (Italy) with the values mentioned in Hanawalt's Tabelas for antigorite from the same locality, from Griffin Range (New Zeeland) and from Mc. Konni (New Zeeland).

No.	Antigorio		Antigorio HT		Griffin R.		Mc. Konni	
	I.	d.	I.	d.	I.	d.	I.	d.
1.	10	7,23	9	7,25	10	7,28	10	7,24
2.	1	6,26			2	6,25		
3.	1	5,18			1	5,23		
4.	3	4,61	3	4,60	5	4,62	4	4,61
5.	2	4,19	4	4,18	5	4,17	2	4,18
6.							1	3,87
7.	10	3,60	9	3,62	10	3,63	10	3,61
8.			0,5	3,48				
9.			1	2,79				
10.							2	2,59
11.	7	2,53	10	2,53	9	2,55	9	2,52
12.	2	2,40	2	2,43	4	2,43	2	2,44
13.			2	2,38			2	2,41
14.							1	2,23
15.	4	2,16	7	2,16	5	2,17	7	2,16
16.			0,5	1,99				
17.	2	1,81	4	1,85	3	1,83	4	1,82
18.			4	1,79			3	1,78
19.	1	1,73	5	1,73			3	1,74
20.			0,5	1,70				
21.	5	1,57	7	1,57	6	1,57	5	1,56
22.	4	1,54	7	1,54	5	1,64	5	1,53
23.			1	1,53				
24.	2	1,51	5	1,51				
25.			1	1,47	4	1,46	1	1,46
26.			1	1,45				
27.	1	1,44	1	1,44				
28.	1	1,42	2	1,42				
29.			1	1,39				
30.			2	1,34	2	1,35		
31.	2	1,31	6	1,32	4	1,32	4	1,31
32.	1	1,30	2	1,30				
33.	1	1,28	3	1,28	1	1,27	2	1,28
34.			3	1,26			1	1,26
35.	1	1,21	1	1,21	3	1,21	2	1,21

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EXPLANATION OF PLATES.

Fig. 1. Strong corrodes residua of olivine in the material of Dobšiná, X nicols, X nat. size.
Fig. 2. X-ray-grams of D-material, of chrysotile from Barabba Distr. and of antigorite from Antigorio Valley.
Fig. 3. Showing he relative positions and intensities of the lines for serpentine from Dobšiná, chrysotile from Barabba Distr. and antigorite from Piemont (Anigorio Valley).
Fig. 4. Electron microscope picture of material from Dobšiná (X 21.000).
Fig. 5. Electron microscope picture of normale chrysotile—asbestos fibers from Dobšiná (X 4.000).
Fig. 6. Electron microscope picture of product asbestos-cement-D-material sheeting (X 21.000).
Fig. 7a, b. Electron microscope pictures, showing directly the process of stiffening and hardening of mixture of D-materiál and cement by adding water (X 21.000).