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AN OCCURRENCE OF PGM AND Ag TELLURIDES AND Te-BEARING ELECTRUM IN THE KRZEMIANKA Fe-Ti-V DEPOSIT (SUWAŁKI ANORTHOSITE MASSIF, NE POLAND)

A b s t r a c t. Intensive studies of oxide Fe-Ti ores and accompanying them sulphides in Krzemianka revealed an aggregate of PGM ores. Very small sizes of the grains have excluded routine optical measurements, essential in mineralogical identification. Chemical analyses in microareas have confirmed preliminary identification of the following ore minerals: merenskyite (PdTe₂), Pt-merenskyite (5.26% Pt), Ag-merenskyite (1.87% Ag), Au-merenskyite (8.13% Au), Te-bearing electrum (81.80% Au, 11.90% Ag, 3.15% Te), hessite Ag₂Te containing a Pd admixture (Pd-bearing hessite?), possibly stuetzite Ag_{5-x}Te₃. Tentatively identified phases include Se-bearing altite Pb(Te,Se) orclausthalite PbSe, as well an Ag-Pd-Te mineral: Ag-bearing empressite (Pd,Ag)Te, sopcheite Ag₄Pd₃Te₄ or tellargpalite (Pd,Ag)_{4+x}Te. Tellurides of platinum, palladium, gold and silver are epigenetic in relation to typical Fe-Ti ores and may represent an unusual, hydrothermal stage of ore precipitation.

Key-words: merenskyite, hessite, Te-electrum, Krzemianka Fe-Ti deposit, NE Poland

INTRODUCTION

Mineralogical and geochemical investigations of vanadium-bearing titanomagnetite ores (Fe-Ti-V association) containing subordinate Cu-Ni-(Co) and Cu-Fe sulphides have been carried out by several research groups on core samples from an anorthosite massif occurring in the area of Krzemianka, Udryń, Jeleniewo and Jezioro Okrągłe (NE Poland). For the first time ever one of the present authors (W.S.) found there ore minerals of platinum group metals (PGM), i.e. of Pd and Pt, which form a paragenesis with Au, Ag, Te, Se and Pb. The results of previous studies of Kucha et al. (1977), Kubicki and Siemiątkowski (1979), Speczik et al. (1980), Kucha (1982), Kozłowska and Wiszniewska (1990), and Wiszniewska (1998) did not record ore minerals of these

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elements. Only Kubicki and Siemiątkowski (1979) did report traces of Pb, Ag, Se, Te and Mo, established in spectral analyses. Witold Salamon found in cores from Krzemianka a sample containing minerals from the PGM group when preparing materials for his Assoc. Prof. dissertation. L. Kubica supported his observations with several chemical analyses in microareas and Salamon and Banaś (2000) published the first results on this finding. It was established that the aggregate with the PGM minerals has the size of about 0.1 mm and is associated with a copper sulphide. The following minerals were preliminarily identified: electrum, merenskyite and its Pt-, Au- and Ag-bearing varieties, hessite, sopcheite (?), a Pb-Se mineral (?). Prolonged efforts to find further occurrences of PGM-bearing minerals have been unsuccessful.

As a result, the small inclusion visible only in immersion under high magnifications of the ore microscope represents a mineralogical curiosity unknown up to now within the sulphides of the Krzemianka deposit. Detailed microscope observations combined with EDS chemical analyses were continued and have indicated that the fine aggregate in question is more complex than preliminarily reported. Probable empressite (Ag-Pd telluride), stuetzite $Ag_{5-x}Te_3$ and a Pd-Te-Pb-Se phase have been added into the list of components, while the chemical composition of all the minerals has been re-examined or analysed anew.

METHODS OF INVESTIGATIONS

An OPTON-AXIOPLAN microscope was used in reflected light investigations. Chemical analyses in microareas were carried out with a Jeol-Superprobe JXA-733 in the RDS mode at accelerating voltage of 20 kV. Standards and spectral lines were as follows: metallic Au, Ag, Pd, Pt, Fe, Cu, Se; PbTe for both elements, pyrite for S (K α line). ZAF corrections were calculated using an original JEOL program.

RESULTS OF MICROSCOPE AND CHEMICAL ANALYSES

When exploring the Krzemianka ores with the aim to carry out necessary ore microscope and geochemical investigations for geological reports and to establish technology of ore processing, a substantial amount of samples was collected. Apart of Ti-Fe-V oxides, a due attention has been paid to sulphide minerals. They represent 0.5–1.5 vol.% of ore minerals in the deposit (Kucha et al. 1977; Kucha 1982), whose reserves have been established at 214 million tones of the Fe-Ti-V ore (Siemiątkowski 1998).

After finding in the late 1990s traces of platinoids and determining their probable localization within the ores (Salamon, Banaś 2000), intensive mineralogical and geochemical investigations (microscope examinations in reflected light with immersion and under the highest possible magnifications) were undertaken to discover further concentrations of PGM-bearing minerals. The results of these observations carried out up to date indicate that further search for PGM mineralization within the titano-36 magnetite ores of the anorthosite massif in the Krzemianka area (NE Poland) should focus on the sulphides of the chalcopyrite type. The sulphide associations of mooihoekite and haycockite are the most suspected carriers of PGM-bearing minerals as one of them, probably mooihoekite, co-occurs with the PGM inclusion found. Kucha et al. (1977) and Kucha (1982) described these two chalcopyrite-related sulphides in paragenesis with two varieties of pyrrhotite, pentlandite, Co-pentlandite, smithite (Fe,Ni)₉S₁₁, thiospinel (Fe,Ni)(Co,Ni)₂S₄, Co-pyrite, cubanite (CuFe₂S₃) and Ni-Co mackinawite. This list should be completed with millerite (NiS) and previously detected (Kubicki, Siemiątkowski 1979) sulphides: pyrite, marcasite, bravoite, violarite, bornite, chalcocite, linneite and sphalerite. Unfortunately, the search has been unsuccessful and none of many samples checked have contained PGM-bearing minerals.

The single PGM aggregate found so far occurs partly within a silicate, partly along its border with a sulphide and, eventually, in the sulphide itself, being of the hockey stick shape and of the length about 0.1 mm. The sulphide has its microscope properties, i.e. colour, reflectance R, almost imperceptible bireflectance ΔR , distinct anisotropy, lack of internal reflections, similar to those of chalcopyrite, and the EDS composition 33.09 wt.% S, 31.01 wt.% Fe and 35.90 wt.% Cu (Table 1, anal. 13; Phot. 1–3). These features are the closest to mooihoekite, whose chemical composition is 32.32–33.30 wt.% S, 31.66–32.01 wt.% Fe, 35.19–36.80 wt.% Cu, while the average VHN (50 G) hardness 243.6 kG/mm² (Kucha 1982). It may be accepted with a high probability that the PGM-bearing minerals occur mainly just in mooihoekite (Mo — Phot. 1, see also Table 1).

The PGM aggregate described is composed of two parts. The first is represented by an elongated grain, designated A, with a length of about 60 μ m and a width locally reaching 5–10 μ m, localized within a silicate (Phot. 1, 2). Part B of the aggregate, making an open angle extension with part A, is localized mainly between the silicate and the mooihoekite, but its fragment also penetrates the latter. Its length is about 35 μ m and the thicknesses of both parts are comparable (Phot. 2). Both grains are composed of minerals with metallic lustre and high reflectance. Very small diameters of individual grains of the aggregate have made detailed optical measurements impossible, in particular those of R and VHN. As a result, the authors had to base on general optical observations, supported by EDS chemical determinations in microareas (microprobe analyses). In the case of very small grains, EDS measurements can be burdened with some error, mainly due to matrix effects, affecting also calculations of atomic proportions. All these problems make part of mineral identification tentative.

The first section of the grain A (Phot. 1; Fig. 1, points 1, 2, 3) is built of a white mineral with R about 65%, identified as **merenskyite** (PdTe₂). Other optical properties are indeterminable because of a small size of the inclusion. Microprobe chemical analyses in points 1, 2, 3 of the grain indicate the presence of pure palladium telluride (Table 1, anal. 1, 2, 3), and an X-ray emission spectrum of this phase is shown in Figure 4-1. The same grain A analysed in next points (Fig. 1, points 4, 5) has also the composition approximating that of merenskyite (Table 1, anal. 4, 5; Fig. 4-4, 4-5) but with some amounts of Ag and Pt. The contents of the latter (up to 5.26%) may indicate Pt-bearing merenskyite.

TABLE 1

EDS composition of mineral phases from the PGM-bearing aggregate, Krzemianka (upper figures in wt.%, lower figures in atomic proportions)

Mineral	12	merenskyite PdTe		merenskyite Pd _{0.950} Te _{2.000}	Pt-merenskyite Pd _{0.925} Pt _{0.029} Ag _{0.064} Te _{2.000}	Pt-merenskyite Pd _{0.887} Pt _{0.100} Te _{2.000}	stuetzite? Ag4.901Te3.000 + chalcopyrite/mooihoekite	Pt-merenskyite Pd _{0.935} Pt _{0.068} Te2.000 + chalcopyrite/mooihoekite	Au-merenskyite Pd _{0.942} Au _{0.170} Pt _{0.058} Te _{2.000}	hessite Ag _{2.047} Te + chalcopyrite/mooihoekite	
S	11	n.d.	n.d.	p.u	0.27	n.d.	1.49	11.41	n.d.	12.17	
Se	10	n.a.	n.a.	n.a.	n.a. n.a. n.a.		n.a.	n.a.	n.a.	n.a.	
Си	6	n.d.	n.d.	n.d.	0.20	0.27	1.43	11.14	1.11	12.52	
Fe	8	n.d.	.p.u	n.d. 0.20 0.17		0.17	1.43	10.62	0.91	11.59	
Pb	7	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a	
Te	6	71.32 0.5589	71.50 0.5603	71.62 0.5613	69.20 0.5423	68.72 0.5385	41.01 1.0000	46.09 2.0304	61.81 1.0000	23.03 0.1805	
Pt	5	n.d.	n.d.	n.d.	1.55 0.0079	5.26 0.0269	n.d.	2.40 0.0690	2.75 0.0291	n.d.	
Pd	4	28.68 0 2694	28.50 0.2678	28.38 0.2667	26.71 0.2509	25.43 0.2389	n.d.	17.98 0.9492	24.29 0.4711	0.84 0.078	
Ag	З	n.d.	n.d.	n.d.	1.87 0.0173	0.14 0.0013	54.64 1.6337	0.36 0.0184	0.99 0.0189	39.85 0.3694	
Чu	2	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	8.13 0.0850	n.d.	
Analytical point	1	1	5	3 5		4 7		4 0		6	

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12	empressite ?	$Ag_{0.539}Pd_{0.332}Pt_{0.023}Te_{1.000}$	merenskyite Pd _{0.957} Pt _{0.048} Te _{2.000} +	chalcopyrite/mooihoekite	Pt-merenskyite Pd _{0.887} Pt _{0.099} Te _{2.000}	+ chalcopyrite/mooihoekite	mooihoekite	Te-electrum	hessite	$Ag_{1.979}Pd_{0.047}Te_{1.000}$	merenskyite	$Pd_{0.938}Pt_{0.027}Te_{2.000}$	otinalization - Catiladianala	ciausulalite: + illereliskylle
11	n.d.		9.37		1.08		33.09	n.d.	n.d.		-	п.а.	n.d.	
10	n.a.	n.a. n.a.		n.a.		n.a.	n.a.	n.a.		n.a.		6.64	0.0841	
6	1.09	0.0395	9.05		2.11		35.90	1.90	0.94	0.0148	1.35		n.d.	
8	0.77	0.0315	8.72		1.90		31.01	1.25	69.0	0.0123	1.11		0.29	
7	n.a.		n.a.		n.a.		n.a.	n.a.	n.a.			п.а.	70.94	0.3424
6	55.50	1.0000	50.80	0.3981	65.74	1.0000	n.d.	3.15	36.26	0.2842	60.69	1.0000	16.42	0.1287
5	1.98	0.0232	1.85	0.0095	4.96	0.0493	n.d.	n.d.	n.d.		1.43	0.0073	n.d.	
4	15.37	0.3320	20.27	0.1905	24.33	0.4437	n.d.	n.d.	1.43	0.0134	27.01	0.4687	5.71	0.0536
3	r.d. 25.29 n.d. 25.29 0.5389 n.d. n.d.			n.d.		.p.u	11.90	60.67	0.5624	-	л.a.	n.d.		
2			.p.u		n.d.		n.d.	81.80	n.d.		-	п.а.	n.d.	
1	10		11		12		13	14	15		16		17	

cont. table 1

n.d. — not detected, n.a. — not analysed.

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Fig. 1. Back-scattered electron image of the PGM-bearing aggregate, Krzemianka. Analytical points 1–17



Fig. 2. Back-scattered electron image of the PGM-bearing aggregate, Krzemianka. Analytical points 14, 15, 16



Fig. 3. Back-scattered electron image of the PGM-bearing aggregate, Krzemianka. Analytical point 17

Merenskyite with traces of Ag and Pt has also been identified in grain B (Table 1, anal. 7, 11, 12, 16; Fig. 1, points 7, 11, 12, 16; Fig. 4-7, -11, -12, -16). Another EDS analysis reveals **Au-bearing merenskyite** in point 8 (Fig. 1; Table 1: anal. 8; Fig. 4-8). Directly contacting with the Au-bearing merenskyite there occurs an inclusion with the length of about 5 µm (Fig. 2, Phot. 2, 3), resembling native Au. The optical features and the chemical analyses indicate it to represent **Te-bearing electrum** (Table 1, anal.14; Fig. 4-14).

Among other phases, a palladium telluride with an admixture of Pt and considerable amount of Ag is worthy noticing (Fig.1, point 10; Fig. 4-10, Table 1, anal. 10); its composition is similar to that of **empressite** (PdTe), but confirmation of this mineral requires further investigations.

Worthy consideration is the phase analysed by the microprobe in point 17 (Fig. 3) within grain A. This inclusion gives a conspicuously high reflection of electrons in a scanning image. Its microprobe chemical composition confirms the presence of major platinum group elements (Table 1, anal. 17) but indicates also a substantial amount of Pb. Not being recognized for sure as a separate mineral, this phase may represent either a selenide-telluride of lead and palladium (Pb,Pd)TeSe or a mixture of clausthalite and merynskyite. The atomic proportion of Pd and Te equal to 1:2 suggests rather the second solution to be more probable. This phase (phases?) also requires further studies to be fully identified.

Under the reflected light microscope, on both sides of part A of the PGM aggregate are localized two larger, irregular grains marked C and one smaller marked 1 (Phot. 2), particularly well visible in oil immersion. Using dry objectives, the grains C reveal the



Fig. 4-1. X-ray spectrum of merenskyite (point 1, Figure 1)



Fig. 4-5. X-ray spectrum of Pt-merenskyite (point 5, Figure 1)



Fig. 4-7. X-ray spectrum of Pt-merenskyite (point 7, Figure 1)



Fig. 4-4. X-ray spectrum of Pt-merenskyite (point 4, Figure 1)



Fig. 4-6. X-ray spectrum of stuetzite(?) (point 6, Figure 1)



Fig. 4-8. X-ray spectrum of Au-merenskyite (point 8, Figure 1)





Fig. 4-14. X-ray spectrumof Te-electrum (point 14, Figure 2)



Fig. 4-10. X-ray spectrum of empressite(?) (point 10, Figure 1)



Fig. 4-12. X-ray spectrum of Pt-merenskyite (point 12, Figure 1)



Fig. 4-15. X-ray spectrum of hessite (point 15, Figure 2)



features of an anisotropic substance, with reflectance about 18% but significantly lowered in immersion. The components of the aggregates C are grey, in places with brownish tint, while internal reflections point to their highly heterogeneous structure. Heterogeneity found and diversification of chemical composition of this substance is reflected in differentiated surface colours of the fields 1 and 2 (Phot. 2). A trained observer can see these colour shades under the microscope, overprinted by suggestive colours of internal reflections. Other optical features, such as weak anisotropy and relief of hardness, are not sufficient to identify this aggregate.

Microprobe chemical analyses carried out, e.g. at the contact of parts A and B of the PGM aggregate (grey substance, also bordering mooihoekite, see Phot. 2) and in points 6 (Fig. 1) and 15 (Fig. 3) indicate silver tellurides (Fig. 4-9, -15). They represent **hessite** Ag₂Te (Table 1, anal. 9, 15) in points 9 (on the border between parts A and B: Fig. 1) and 15 (close to the Te-bearing electrum inclusion mentioned above: Fig. 2, 3). Recalculation of analysis 6, where Ag and Te are major elements, gives an Ag:Te ratio different than that of hessite; the mineral has been tentatively identified as **stuetzite** Ag_{5-x}Te₃ (Fig. 4-6, comp. EDS spectra 4-9 and 4-15).

SUMMARY AND CONCLUSIONS

Among the phases whose compositions have been established using EDS analyses, a full positive identification can be made in three points (analyses 1, 2, 3, Table 1), in which pure palladium telluride indicates the presence of **merenskyite** PdTe₂. Rather correct identifications are those made in the analytical points 4, 5, 7, 8, 11, 12 and 16: the mineral phases represent members of Pt-bearing merenskyite. Of these analyses, those numbered 5 and 12 give the highest contents of Pt, around 5%. Worthy noticing is Au-Pt-Ag merenskyite, containing about 8% Au and 2.7% Pt (Table 1, point 8). Also relatively easy in identification are Ag-Te phases: almost pure **stuetzite** (Ag_{5-x}Te₃,

Table 1, point 6) and **Pd-bearing hessite** (Ag,Pd)₂Te (Table 1, points 5 and 15). Equally unquestionable is the result of the analysis 14, typical of **Te-bearing electrum**: 81.80% Au, 11.90% Ag, 3.15% Te. Some problems have been encountered when identifying a mineral whose composition is given by the analysis 10. As its full optical recognition is not possible (too small size!), it must suffice to consider microprobe chemical determinations that indicate **empressite** AgTe, but due to the presence of palladium may correspond to the compositions of sopcheite Ag₄Pd₃Te₄ (Orsoev et al. 1982) or $(Pd,Ag)_{4+x}$ Te (Naldrett 1981). The values revealed by the analysis 10 (Table 1) are similar also to those of the sopcheite Ag₄Pd₃Te₄ described by Piestrzyński et al. (1994). The result of the analyses 13 indicates a pure sulphide of the chalcopyrite group, probably **mooihoekite**.

In many other analyses listed in Table 1 there are some amounts of Cu, Fe and S that must be associated with the cryptocrystalline of Cu-Fe sulphide(-s), either directly in the analytical points (intergrowths and/or inclusions) or immediately below them (matrix effect). For this reason, the contents of Cu, Fe and S in the analyses other than No.13 have been interpreted as associated with a mineral of the chalcopyrite group (mooihoekite) that is intergrown with or underlies palladium or silver tellurides. Atomic proportions suggest just such an interpretation.

A complicated geochemical and mineralogical problem represents the microprobe determination shown in Figure 4-17. Probably only Pb and Se do represent the mineral analysed, while Te and Pd are related to its nearest neighbourhood. If the Te were incorporated into this mineral, the phase would represent **Se-bearing altaite** Pb(Te,Se) or, simply, a **telluro-selenide of palladium and lead**, moreover that altaite does not form solid solution either with galena or clausthalite. However, optical data suggest the presence of **clausthalite** PbSe, and the EDS chemical composition can result from cryptocrystalline intergrowths of this mineral with merenskyite or represents another matrix effect.

The PGM-bearing mineral formation has been described for the first time in the Fe-Ti-V ores of the Krzemianka region, so genetic hypotheses are very tentative. It is the more so that the results presented are based on observations of a single mineral accumulation and most of mineral identifications have been inferred from EDS chemical determinations. Considering small or very small sizes of the mineral phases, these microprobe analyses are not always sufficient to draw sound conclusions.

The analysis of microscope textures suggests that the sulphide assemblage studied is composed of pyrrhotite, pyrite, pentlandite and Co-pentlandite (older association), intergrown with or overgrown by younger copper sulphides, i.e. chalcopyrite itself or chalcopyrite-related minerals (mooihoekite). Pyrrhotite is quantitatively dominating among ore minerals and makes often intergrowths with other sulphides. Both pyrite I and pyrrhotite reveal the signs of simultaneous fracturing, the feature non existing in the remaining sulphides. Contacts of almost all ore minerals, except for pyrite, show metasomatic corrosion, e.g. chalcopyrite in the form of veinlets intersects pyrrhotite and pentlandite or partly replaces both of them.

The nest of copper sulphide with the PGM assemblage must have been formed by metasomatosis in the silicate in which it mainly occurs. The described ore aggregate (parts A and B) has sharp boundaries with mooihoekite and a silicate, and could have

precipitated as a result of a fracture-filling process. Such a process may represent a phenomenon related to concentration of PGM minerals during the hydrothermal stage, although majority of PGM occurrences known so far is associated with crystallization of ultrabasic or basic rock complexes.

The vanadium-bearing titanomagnetite ores with the sulphide assemblage in the Krzemianka area (not exploited for environmental and economic reasons) can be compared, for instance, with such well-known deposits as Sudbury, Bushveld, Norylsk, and Kola Peninsula. To make any comparison, one should bear in mind that the Polish deposit is practically devoid of elements or minerals of the platinum group, and their traces described in the present paper are without any economic value. The parallels, however, may reveal some common features that may help define further directions of prospecting for the PGM accumulations of practical value within the Krzemianka ores. Although our intensive search has not been successful, it is obvious that a program should focus on extensive sampling and analysing (assays for trace elements combined with ore microscopy at the highest possible magnifications). Continuation of comparative studies seems also to be necessary.

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WYSTĄPIENIE TELURKÓW PLATYNOWCÓW I SREBRA ORAZ ELEKTRUM W ZŁOŻU RUD Fe-Ti-V KRZEMIANKA (MASYW ANORTOZYTOWY SUWAŁKI, POLSKA NE)

Streszczenie

Badania mikroskopowe tytanomagnetytowych rud tlenkowych i towarzyszących im siarczków w złożu Krzemianka ujawniły obecność submikroskopowego agregatu minerałów platyno-, złoto- i srebronośnych. Szczegółowe badania chemiczne w mikroobszarze, przy zastosowaniu EDS, pozwoliły zidentyfikować następujące fazy mineralne: merenskit PdTe₂ i jego odmiany — platynonośną (5,26% Pt), srebronośną (1,87% Ag) i złotonośną (8,13% Au), hessyt Ag₂Te z domieszką Pd (do 1.4% Pd), Te-electrum (81,80% Au, 11,90% Ag, 3,15% Te). Możliwa jest też obecność stuetzytu Ag_{5-x}Te₃, empressytu (Ag,Pd)Te, sopcheitu Ag₄Pd₃Te₄, lub pokrewnych telurków Ag i Pd, a także clausthalitu PbSe lub ałtaitu PbTe. Wydaje się prawdopodobne, że powstanie opisanego zespołu minerałów zawierających Au, Ag, Pd, Pt, Te, Se i Pb jest związane z nieznanym szerzej stadium działalności hydrotermalnej w obrębie anortozytowego masywu Suwałki, obserwacje mikroskopowe wskazują bowiem na ich wyraźnie późniejsze pochodzenie w stosunku do rud tytanomagnetytowych. MINER. POLON. Vol. 35, No 1, 2004



Phot. 1. A nest of mooihoekite (Mo) in a silicate (K). At the border of both phases a PGM-bearing aggregate is visible; its combined length is *ca* 0.1 mm



Phot. 2. A blow-up of the PGM-bearing aggregate from Phot. 1, composed of parts A, B and C. Part A within the silicate: white, reflectance >60%. Part B between the silicate and mooihoekite (Mo), partly within the latter: pale blue with a pinkish tint, reflectance lower than that of A (*ca* 45%). At the border of B an inclusion with optical properties close to native gold is visible. Parts C a heterogeneous mineral substance, grey, with reflectance close to that of sphalerite (17%). The substance reveals strong internal reflections in immersion, with diversified colour effects in blue-brownish shades; they mark fields 1 and 2. The colours of patchy internal reflections are the same. Grey-blue overgrowths 1 and 2 on the grains A and B contain inclusions of hessite, positively identified in microprobe analyses.

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Phot. 3. A blow-up of the grain of native gold from Phot. 2, present in grain B. The grain contains an admixture of silver, actually representing Te-bearing electrum (vide X-ray spectrum in Fig. 4-14 and chemical analysis 14 in Table 1). Optical features determinable: saturated yellow colour, very high reflectivity *ca* 80%.

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