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SILICIDE SPHERULES FROM PERMIAN SEDIMENTS OF THE FORE-SUDETIC MONOCLINE (SW POLAND)

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Abstract: The presence of Fe-silicides, extremely rare mineral phases were documented in the Permian Cu-bearing ore formation in the Fore-Sudetic Monocline (Polkowice-Sieroszowice and Rudna mines, SW Poland). It is a first report of their occurrence within rocks of Late Palaeozoic age. The Fe-Si alloys form spherules of various structure and chemical composition. The silicide spherules were found in the flotation concentrates prepared from the copper ore. Their composition varies from FeSi (fersilicite) to Fe₂Si₃ or Fe₄Si₉. A dominant constituent is Fe₅Si₃ (xifengite) with admixtures of P, Ti, Cr and Mn. Native Si and Ti were detected in the marginal part of spherules. The current knowledge do not permit to determine unequivocally whether the Fe-silicide spherules formed as a result of Permian cosmic dust or constitute terrestrial magmatic material of ultramafic character transported into a sedimentary basin from the adjacent terrain.

Keywords: Permian, Fe-silicide spherules, cosmic dust, ore minerals, Fe-Si alloy

Introduction

During routine research of copper flotation concentrates from the Polkowice-Sieroszowice Mine (SW-Poland, Fig. 1) the presence of very rare minerals, such as iron silicide was discovered (Muszer, 2007). On the basis of this analysis new research was designed to determine the location of their occurrence in the Permian rocks.

Silicides still remain relatively poorly investigated and exotic mineralogical compounds. First report about naturally occurring Fe-Si compounds was published by Gevorky'en (1969). Recently a newly discovered Fe-Si phase was described from the Tibetan mines (Bai et al., 2007). On the other hand, from the chemical point of view, silicides has been known for over 100 years. Alloys of Si with another metallic element, e.g. TiSi₂ and CoSi₂, found industrial use as joints of various metals in semiconductors. Natural Fe-silicides were found in meteorites (Mason, 1962; Judin and Kolomenskij, 1987), sedimentary (Gevork'yen et al., 1969; Novosielova, 1975), metamorphic (Xiongjian, 1991) as well as mafic (Bai et al., 2007) and felsic igneous

rocks (Jakabska and Rozložník, 1989). This mineral group comprise fersilicite FeSi , hapkeite Fe_2Si , gupeite Fe_3Si , suessite Fe_3Si , xifengite Fe_5Si_3 , luobusaite $\text{Fe}_{0.84}\text{Si}_2$ and ferdasilicite (leboite) FeSi_2 . Fersilicite was recorded from the Poltava sandstones of the Donyeck area in Ukraine (Gevork'yen, 1969; Gevork'yen et al., 1969). Suessite was discovered in the North Haig ureilite meteorite (achondrite) from South Australia in 1980 (Keil et al., 1982; Judin and Kolomenskij, 1987). According to Keil et al. (1982) it was formed during the fall as a result of silica reacting with carbonaceous matrix. Gupeite and xifengite were detected in cosmic dust from Yanshan province (China) in the paragenesis with kamacite, taenite, magnetite, wüstite and maghemite (Zuxiang, 1984). Similarly to fersilicite from Ukraine, they were found in heavy mineral concentrates obtained from alluvial deposits.



Fig. 1. Location of sampling points in SW Poland

Luobusaite $\text{Fe}_{0.84}\text{Si}_2$ was found at the Cr-Fe Luobusa mine in the Autonomic Xizang Region (Tibet) in 2007 (Bai et al., 2007). It occurs as irregular grains in Cretaceous ophiolitic rocks accompanied by numerous alloys of Fe-Si, Ni-Fe, Fe-Cr, Ir-Fe, W-Co, Cr-C, Ti-N, Si-Ca as well as abundant oxides, sulphides, arsenides and extremely rare Si spinels (Bai et al., 2007).

Another IMA-approved natural Fe-Si species are hapkeite and ferdasilicite. Hapkeite Fe_2Si was described from Lunar meteorite breccia Dh-280 found in the Dhofar Province, Oman (Anand et al., 2004). It occurs in weathered plagioclase together with fersilicite, Fe-Ni alloys and ferdasilicite FeSi_2 . According to Anand et al. (2004), hapkeite formed as a result of meteorite fall onto the Lunar regolith. Ferdasilicite (leboite) FeSi_2 is known from the sedimentary Poltava sequence in the Donyeck area (Ukraine) and the concentrates of metamorphic rocks from Longquan, Zhejiang Province, China (Xiongjian, 1991). In both occurrences it coexists with other Fe-Si compounds, i.e. Fe_5Si_2 , xifengite (Fe_5Si_3) and a mixture of phases with varying Fe-Si proportions. Ferdasilicite was also found in the kimberlites of Sytykanskaya (Russia) where it is accompanied by native Si and other Fe-silicides (Pankov and Spetsius, 1989). Unnamed are such phases as Fe_4Si_9 , Fe_5Si_3 and Fe_2Si_3 . They occur as

intergrowths with moissanite (SiC) found in Lower Cambrian carbonaceous rocks of the Sayan Mts, Russia (Novosielova, 1975). According to Novosielova and Bagdasarova (1979) they are of meteoritic origin.

Samples

Three samples, about 10 kg each, were chosen to investigate the presence of Fe-silicides in the Permian rocks of SW Poland (Fig. 1). The samples were: ore-bearing formation of the Polkowice-Sieroszowice copper mines (KGHM Polska Miedz S.A.), ore material processed in the Polkowice-Sieroszowice Ore Concentrator and a sample of tailing stored in the Zelazny Most tailings pond (Rudna Ore Concentrator). The first one consists of reddish-brown dolomitic shale and represents marine sediments of the Rotliegendes/Zechstein boundary (Werra cyclothem). It was obtained from the side-wall of the gallery in the G-31 mining section of the Polkowice-Sieroszowice mine (Fig. 2). The laminated aleurite-pelitic rock contains carbonates (calcite, dolomite), clay minerals and detritic quartz with minor anatase and titanite. Opaque phases constitute from 0.1 to 1% of its volume. Dominating chalcopyrite is dispersed in the rock, while subordinate chalcocite, covellite, digenite, pyrite together with uncommon bornite, native Au and electrum (up to 35.5% Ag) form laminae. Chalcopyrite aggregates reach 200 μm in diameter. Pyrite forms subhedral grains up to 50 μm across. Other sulphides rarely exceed 30 μm . Native gold and electrum form intergrowths and clusters with digenite. Au-bearing ore minerals, dominated by native Au with small admixture of Ag < 2.45 wt %, are of various size: from several to several dozens μm .

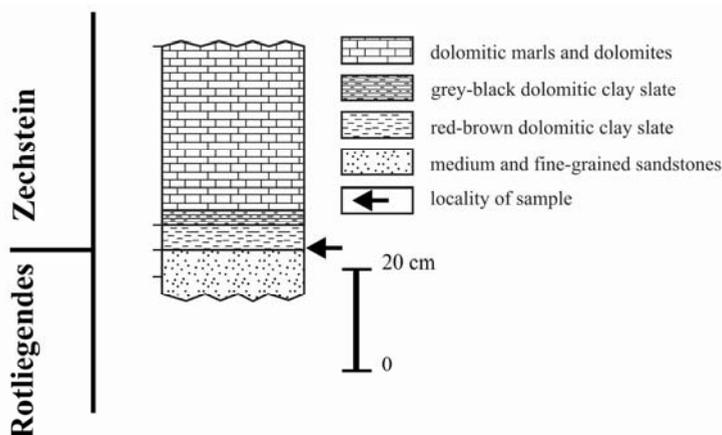


Fig. 2. Lithological profile of the side-wall of mine gallery in mining section G-31 (Polkowice-Sieroszowice mine)

The sample of material processed in the Polkowice-Sieroszowice Ore Concentrator consists of Cu-bearing, streaky dolomite and dolomite. The sample from the Rudna Ore Concentrator represents flotation tailings.

Methods

The Permian rocks were crushed in a Fritsch jaw crusher into <0.25 mm fraction and then enriched by flotation and magnetic separation (Nd-magnet, 6000 Gs and 9000 Gs). A Joy-Denver flotation cell was used to concentrate minerals and spherules. Cu- and Fe-bearing concentrates together with spherules built of various minerals constituted 0.01 and 0.02% of the initial weight of the first and second sample, respectively.

Magnetic and non-magnetic spherules were separated from other mineral phases present in the concentrates using an experimental ramp-like device developed by Muszer (2007). As a result, a satisfying number of objects was obtained to use statistical approach in describing structural and microtextural features of individual spherule types as well as to characterise their chemical composition.

Polished sections were prepared following the standard procedure used for ore minerals and metallographic material (Samuels, 1982; Muszer, 2000). DP-Mol, DP-Dur and DP-Nap Struers polishing cloths were used with precisely controlled granulation of diamond pastes.

An optiphot 2-Pol Nikon microscope was used for observations in reflected light. Ore minerals were distinguished basing on their optical and physical properties (Uytenbogaardt and Burke, 1971; Ramdohr, 1973, 1975; Criddle and Stanley, 1993; Muszer, 2000; Bernhard, 2008). Planimetric analysis, coupled with the Lucia M software, was employed to establish the proportions between minerals, spherules and microporosity in the studied samples. The spherules were subdivided into several types basing on the concentration of a dominant mineral or alloy (i.e. into aluminosilicate, silicate and silicide types).

Chemical point analyses of the spherules and accessory minerals were conveyed using SEM-515 (Philips) and JOEL JSM-55800LV electron microscopes. A MIKROSKAN MK-9 Cambridge electron microprobe was used in case of bigger mineral grains or homogeneous spherules. The analyses were carried out in the Institute of Low Temperature and Structure Research (Polish Academy of Sciences, Wroclaw), Institute of Materials Science and Applied Mechanics (Wroclaw University of Technology) and Department of Mineralogy and Petrology (Institute of Geological Sciences, University of Wroclaw). Pure (99.99%) metal of Fe, Ni, Co, Mn, Al, Cu and Zn as well as pure SiO₂ and Al₂O₃ were used as standards.

Results

In the analysed samples Fe-silicides occur within spherules, which are present in the red shale obtained at the Rotliegendes/Zechstein boundary in the Polkowice-Sierszowice mine as well as in the tailings from the Rudna Ore Concentrator. In the ore from the Polkowice-Sierszowice mine only metallic sulphides together with magnetite, magnetite-hematite and Fe-spherules were detected (Muszer, 2007).

The silicide spherules are present in parasteresis with Cu-, Zn- and Pb-sulphides as well as other minerals typical of the ore-bearing rock series of the Lubin-Glogow Mining Area: chalcocite, bornite, chalcopyrite, covellite and others. The chemical composition and microtexture of the Fe-spherules in both samples are alike. However they differ in the spherules size. In the red shale Fe-silicide spherules reach 50 μm and often exceed 150 μm in the tailings.

Spherules from red-brown shale (Polkowice-Sierszowice mine)

Magnetite, hematite, Fe-silicides, arsenopyrite, pyrrhotite, bornite, covellite, pyrite and goethite were detected in a magnetic concentrate prepared from the red shale (Polkowice-Sierszowice mine). Rock fragments incrustated by Fe-hydroxides, i.e. goethite and hydrogoethite constitute 60.45% of the sample volume. Only magnetite grains from several to 200 μm across were not intergrown with rock fragments and other minerals. Pyrrhotite spherical forms, with the diameter ranging from 2 to 25 μm , occur within rock fragments enriched in Fe-hydroxides. A 25 μm big pyrite framboids coexists with bornite. Covellite was also found in rock fragments (up to several dozens μm) impregnated with Fe-hydroxides (Fig. 3).

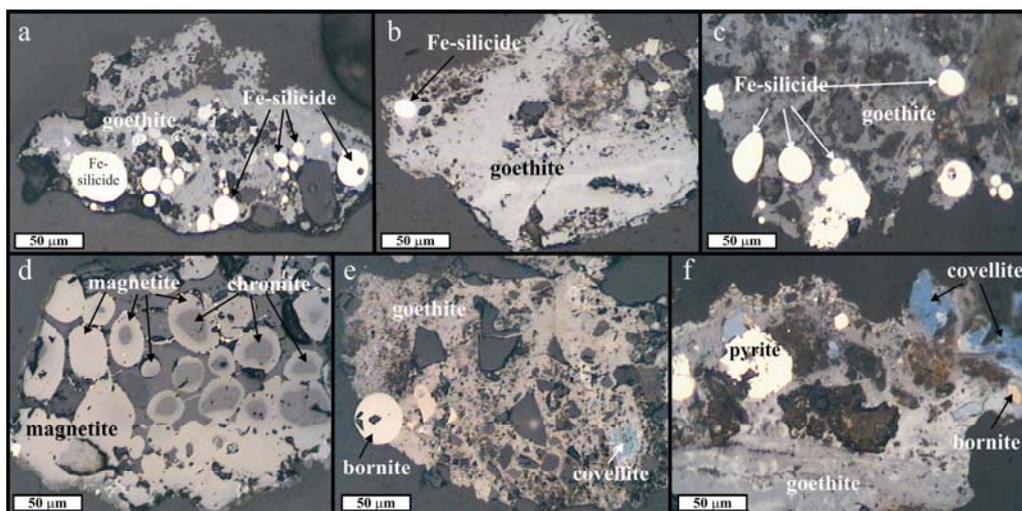


Fig. 3. Forms of spherules in a magnetic concentrate from the red shale (Polkowice-Sierszowice mine), reflected light, without analyser

The spherules from this concentrate are mainly built of Fe-silicides (Fe-spherules, Table 1, Fig. 3a–c), magnetite (Fig. 3d), silicates with goethite and recrystallized bornite framboids (Fig. 3e, Table 1). Most of the spherules (>95%) are massive (Table 2). Porous microtexture was observed only in the magnetite spherules. In this sample spherical forms are dominated by the Fe-spherules with the composition of Fe_5Si_3 with minor amounts of P, Ti, Cr and Mn (Table 3). They are small, between several and 50 μm , oval or slightly flattened, massive and relatively homogeneous as concerns their chemical composition (Table 3).

Table 1. Distribution of spherule types from the red shale (Polkowice-Sieroszowice mine)

Spherule		Spherule type	wt %	
Fe-oxide	Magnetite		5.41	13.52
	Silicate with goethite		8.11	
Iron	Iron (Fe-silicide)		86.36	83.36
Sulphide	Bornite		0.13	0.13
Total			100.00	100.00

Table 2. Distribution of microstructures in spherules from the red shale (Polkowice-Sieroszowice mine)

Spherule type	microtexture, %		Total
	porous	massive	
Magnetite	3.25	2.16	5.41
Silicate with goethite	–	8.11	8.11
Iron (Fe-silicide)	–	86.36	86.36
Bornite	–	0.13	0.13
Total	3.25	96.80	100.00

The silicide spherules are present in goethite aggregates or occur separately. Within the aggregates they occur together with magnetite, covellite and pyrite grains as well as silicate spherules (Fig. 4). The latter are very small and do not exceed 25 μm . The spherules built of bornite are of comparable size. Such forms are most probably an effect of recrystallization of framboids enclosed in the shale ore, which were originally composed of bornite.

The magnetite spherules present in the concentrate are usually porous (Table 2). The core of the porous magnetite spherules is granular, while the massive spherules contain chromite cores (Fig. 3).

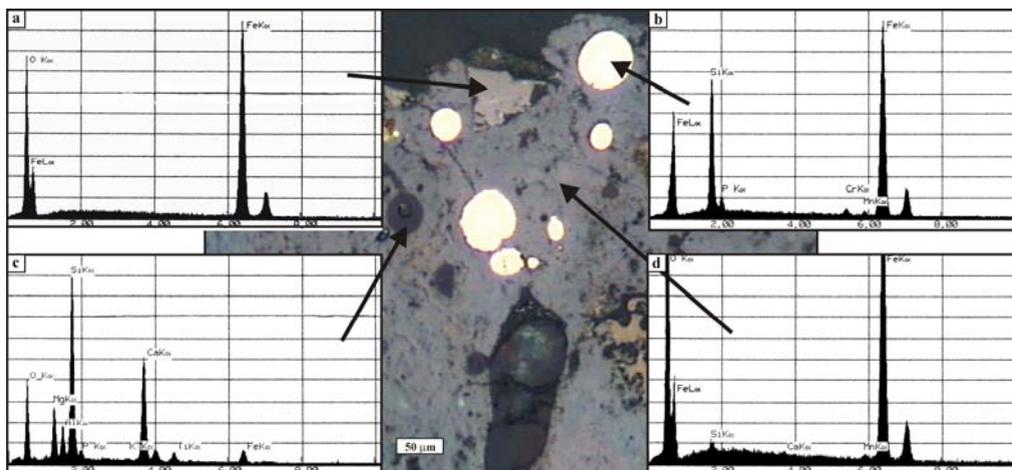


Fig. 4. Analyses of minerals in goethite aggregate from the red shale (Polkowice-Sierszowice mine). Reflected light, without analyser. Spectra of typical X-ray radiation of
 a – magnetite grain, b – Fe-Si-spherule, c – silicate spherule, d – goethite, excitation voltage 20 kV

Table 3. Contents of elements in Fe-silicide spherules from the red shale (Polkowice-Sierszowice mine)

Element	wt %									
	1	2	3	4	5	6	7	8	9	10
P	3.96	2.17	2.05	1.10	0.56	3.92	0.24	2.10	0.76	0.37
Ti	0.22	1.25	1.20	–	–	0.2	0.65	1.13	–	0.63
Cr	1.03	–	–	0.99	0.55	1.05	0.65	–	1.19	0.91
Mn	0.56	0.11	–	0.55	–	0.51	0.51	–	0.57	–
Fe	72.49	74.12	74.65	74.64	76.27	72.53	75.13	74.66	74.86	75.27
Si	21.74	22.35	22.10	22.72	22.62	21.79	22.82	22.11	22.62	22.82

Spherules from Rudna tailing

Mineralogical composition of the tailings sample from the Rudna Ore Concentrator differs significantly from those of the Polkowice-Sierszowice ore. The following phases were recognised: (with decreasing amount) bornite, pyrite (+marcasite), covellite, chalcocite (+digenite), sphalerite and galenite. Minor amounts of rammelsbergite-safflorite, zircon and various types of spherules also occur. Bornite, pyrite and covellite form over 70% of ore minerals in the sample. Magnetite, magnetite-hematite, Fe-silicides and Fe-silicates spherules predominate (Table 4). Massive microtexture is far more common than porous (Table 5).

Table 4. Distribution of spherules in a concentrate from tailings of Rudna Ore Concentrator

Spherule	Spherule type	wt %	
Fe-oxides	magnetite	81.81	90.91
	magnetite-hematite	9.09	
Fe	Fe-silicides	4.75	9.10
	Fe-silicates	4.35	
Total		100.00	100.00

The size of the spherical forms (50–250 μm) is comparable to those from the Polkowice-Sierszowice mine. Their surface is smooth with high metallic lustre. The magnetite spherules are granular and contain pores but lack discernible grain boundaries when their microtexture is massive (Fig. 5, Table 5).

The amount of magnetite spherules with skeletal magnetite crystals is low and does not exceed 15,5 % of the total spherule number. Magnetite is often replaced by hematite. Martitization develops outwards if the spherule core is empty, but in the opposite direction if the spherule do not possess granular structure. Magnetite crystals replaced a minor admixture of Mn (<1.65 wt%).

Table 5. Distribution of microstructures in spherules from tailings of Rudna Ore Concentrator

Spherule type	Microstructure, %		Total
	porous	massive	
	40.91	59.09	
Magnetite	31.82	49.99	81.81
Magnetite-hematite	9.09	–	9.09
Fe-silicides	–	4.75	4.75
Fe-silicates	–	4.35	4.35

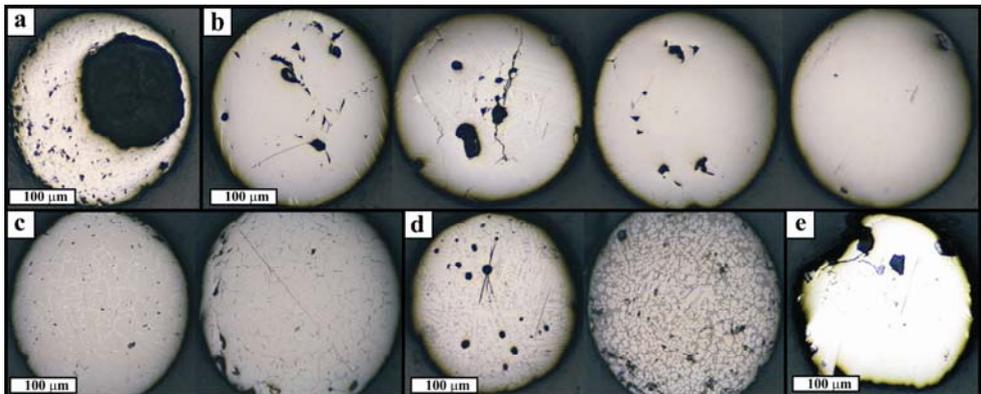


Fig. 5. Spherules in a concentrate from tailings of Rudna Ore Concentrator. Reflected light, without analyser: a – granular and porous magnetite spherule, b – magnetite spherules with invisible microtexture or of net-like texture, c – magnetite spherules with granular texture, martitization and silicates, d – magnetite spherules with skeletal texture, e – Fe-silicide spherule with inclusions of native silicon (grey)

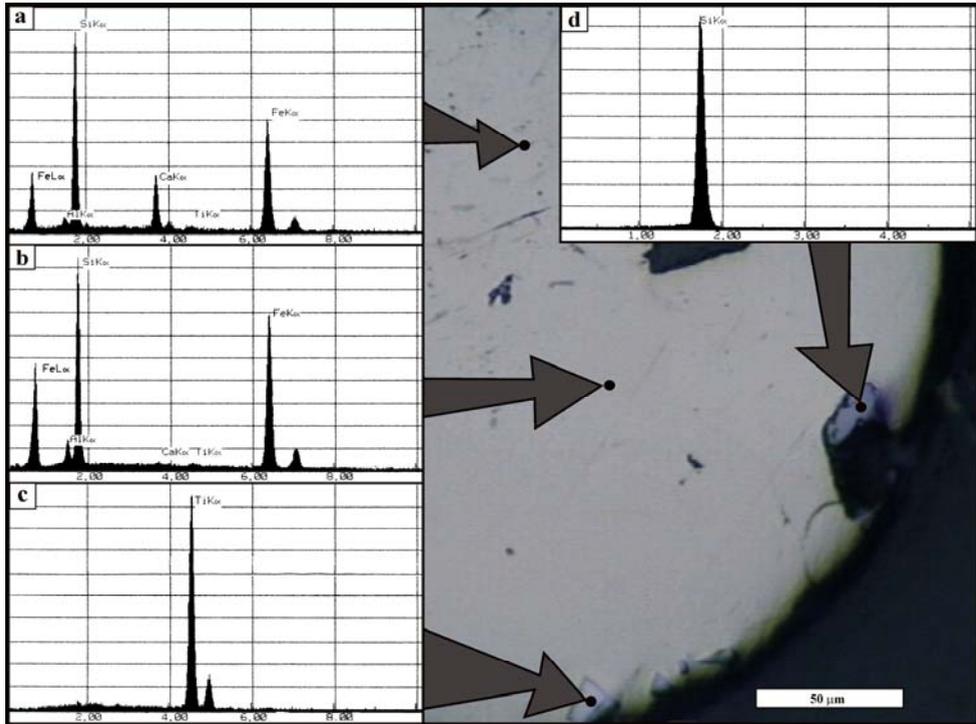


Fig. 6. Analyses of mineral phases in an Fe-silicide spherule from Rudna Ore Concentrator. Reflected light, without analyser. Spectra of typical X-ray radiation of: a – Fe_2Si_3 , b – FeSi , c – Ti crystal, d – Si crystal, excitation voltage 20 kV

Magnetite-hematite spherules are rare in this concentrate. All of them are porous with highly developed net-like structure that results from martitization of magnetite. The spherules built mainly of native Fe or Fe with admixtures of Si show a different structure that is reflected by Fe-Si compounds. Their size varies from 100 to 150 μm and the chemical composition changes over several μm . Although the differences are not readily discernible in BSE images, they become pronounced in reflected light. The composition varies from FeSi to Fe_2Si_3 or from FeSi to Fe_4Si_9 . The margins contain crystals of native Si or Ti (Fig. 6). Tabular-lenticular unmixing structures, present in central parts of the spherules, display an increase of Al and Ca (from 0 to 3.95 wt%). Ti concentration varies between 0 and 0.65 wt%. The spherules surface is rough comparing to the magnetite spherules. During polishing the silicide spherules crumble away along the margins.

Discussion and conclusion

A characteristic feature of Fe-silicides in described spherules is their occurrence in paragenesis with native Si. The latter is an extremely rare mineral, reported from the

Neogene volcanic rocks found on the Mediterranean beaches, 150 km NW of Izmir, Turkey. It forms intergrowths with moissanite (Di Pierro et al., 2003; Robinson et al., 2004). Native Si was also reported from volcanic rocks of Tolbachik in Kamchatka (Russia) and volcanic exhalations in the ophiolitic complex of Nuevo Potosí (Cuba). In the latter case it forms inclusions in native gold (Novgorodova et al., 1989). A 19-80 μm big regular octahedrons of native Si are known from the Kola Peninsula and from the Jakutia kimberlites near Sytykanskaya, Russia (Pankov and Spetsius, 1989). The crystals from the Kola Peninsula occur in paragenesis with graphite, moissanite as well as native Fe, Au and Cu-bearing Zn (Novgorodova et al., 1989). Native Si was also observed as inclusions in natural SiC and native Fe (Bauer et al., 1963) as well as in Fe-silicides, e.g. ferdasilicide Fe_3Si_7 (Marshintsev, 1990).

The samples of Permian rocks from Polkowice and Rudna belong to one ore-bearing formation on the Fore-Sudetic Monocline. Ore minerals, mainly chalcocite, bornite, chalcopyrite, covellite, galenite, sphalerite, pyrite and uncommon Ni-Co arsenides belong to medium- and low-temperature phases. The accompanying spherulitic forms are characteristic for very high crystallization temperatures (magnetite, silicide and silicate spherules). Their shape implies surface tension operating during their formation and indicates liquid or gaseous conditions (Muszer, 2007). The comparison of the textural and the elementary composition with other spherules described in the bibliography showed numerous analogies of the internal structure. Necessary high temperature may be achieved when meteoritic dust travels through upper parts of the troposphere. Alternatively, it may be related to magmatic (volcanic) terrestrial conditions. The described Fe-silicides and other magnetite and silicate spherules are exoclasts within the rocks sampled.

The current research do not permit to determine unequivocally whether the Fe-silicide spherules formed as a result of Permian cosmic dust or constitute terrestrial magmatic material of ultramafic provenance transported into a sedimentary basin from the adjacent terrain. Their co-occurrence with Fe-hydroxides resembles the position of suessite Fe_3Si from the North Haig achondrite from South Australia (Keil et al., 1982; Judin and Kolomenskij, 1987). In both cases silicides are surrounded by goethite (originally probably native Fe) but they do not yield to oxidation themselves. The spherules from the Permian rocks, composed of Fe-silicides did not oxidize either although the process transformed the primary Fe-bearing minerals into goethite. Besides, crystals of native Si and Ti were detected in the silicide spherules from the concentrate obtained from tailings of the Rudna Ore Concentrator (Fig. 5). Such paragenesis in the spherules is similar to the one described from the Luobusa Cr-Fe ore, Autonomic Xizang Region, Tibet (Bai et al., 2007).

The composition of silicide spherules varies from FeSi (fersilicite) to Fe_2Si_3 or from FeSi to Fe_4Si_9 . The spherules composed of Fe_5Si_3 (xifengite) with admixture of P, Ti, Cr and Mn dominate. This composition does not allow determining their origin. It corresponds however to silicides reported from meteorites (Judin and Kolomenskij, 1987; Novosielova and Bagdasarova, 1979) as well as the ones from sedimentary,

metamorphic and igneous rocks (Gevork'yen et al., 1969; Novosielova and Bagdasarova, 1979; Xiongjian, 1991; Bai et al., 2007).

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