

Nigrine – a heavy mineral aggregate indicative of pegmatites and their siliceous root zones

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Dill, H.G. & Melcher, F. (2009): Nigrine – a heavy mineral aggregate indicative of pegmatites and their siliceous root zones. [Nigrin – ein Indikator-Schwermineralaggregat für Pegmatite und ihre quarzreichen Wurzelzonen.] – Z. dt. Ges. Geowiss., 160: 191–199, Stuttgart.

Abstract: Nigrine is a black mineral aggregate composed of rutile (often enriched in Nb and/or W) and ilmenite (enriched in Mn). Nigrine occurs as heavy mineral grains in alluvial-fluvial drainage systems in areas underlain by aplitic and/or pegmatitic quartz-feldspar rocks. Three types of nigrine were established in the NE Bavarian Basement; they provide an insight into the unroofing story of bedrock deposits. Type A nigrine originates from the deeply eroded siliceous root zone of the pegmatites. This type formed in mylonitic quartz bearing shear zones in the course of pre- to synkinematic nigrinisation. Postkinematic type B nigrine originates from the highly differentiated parts of the pegmatite stocks abundant in rare metal concentrations. Postkinematic type C indicates the passage into the scheelite and wolframite mineralisation at the margin of a pegmatite district.

Kurzfassung: Nigrin ist ein schwarzes Mineralaggregat, das aus Rutil (z. T. reich an Nb und /oder W) und Ilmenit (reich an Mn) besteht. Dieses Mineralaggregat kommt in alluvialen und fluvialen Ablagerungen innerhalb oder nahe von Arealen vor, die von aplitischen und pegmatitischen Gesteinen unterlagert sind. Im nordostbayerischen Grundgebirge lassen sich drei Grundtypen von Nigrin unterscheiden. Sie geben einen Einblick in das Abtragungsgeschehen im Einzugsgebiet der Nigrin führenden Gewässersysteme. Der Nigrintyp A geht auf die quarzreichen Wurzel- bzw. Kernzonen der Pegmatite zurück. Er entstand im Zuge einer prä- bis synkinematischen Nigrinisierung in mylonitischen, Quarz führenden Scherzonen, die das Grundgebirge in unterschiedlichen Dimensionen durchsetzen. Der postkinematische Typ B unter den Nigrinen lässt sich auf hochdifferenzierte Pegmatite, die reich an seltenen Metallen (z. B. Nb, Ta, Li) sein können, zurückführen. Der postkinematisch gebildete wolframreiche Nigrintyp C markiert den Übergang in die Scheelit und Wolframit führenden Mineralisationen, die am Rande der Pegmatitgebiete liegen.

Keywords: nigrine, ilmenite, rutile, pegmatite, unroofing, Variscan, Germany

Schlüsselwörter: Nigrin, Ilmenit, Rutil, Pegmatite, Abtragungsgeschehen, Varisziden, Deutschland

1. Introduction

Rounded to subrounded dull black grains of nigrine, measuring as much as 2 cm are common in many fluvial and alluvial placer deposits, e.g., Austria, Malawi, Nepal, Sierra Leone and Russia (Bloomfield 1958, Raufuss 1973, Exel 1993, Shevelev 1997, Dill 2007, Dill & Ludwig 2008). In NE Bavaria, Germany, nigrine is rather widespread but has never been reported from the Bayerischer Wald. In the Oberpfälzer Wald, however, nigrine was described by Strunz (1961) but suspected to be rutile or cassiterite. These placer deposits have been investigated for nigrine only recently by Dill et al. (2007a) and were expanded towards the northern and southern parts of the NE Bavarian Basement to find out if the composition of nigrine is a function of the level of erosion into the various basement rocks.

Nigrine is not a mineral on its own accepted by IMA but a mineral aggregate. Many definitions have been put forward since Klaproth's first description of nigrine (1797). Ramdohr (1975) refers to nigrine as "black ferruginous rutile". In this paper nigrine is defined as a black dull to lustrous heavy mineral aggregate composed of two major constituents, namely rutile and ilmenite. It occurs only in placers proximal to the source. In distal fluvial and beach placers, nigrine is mechanically split up into rutile and ilmenite. Columbite-(Fe), pyrochlore, wolframite and zircon may be included by some nigrine types and pseudorutile is a typical replacement product of nigrine. Three working areas have been selected in the NE Bavarian basement. They share the common characteristic of nigrine being widely distributed in the drainage system but they are quite different with respect to the potential source of nigrine (Fig. 1).

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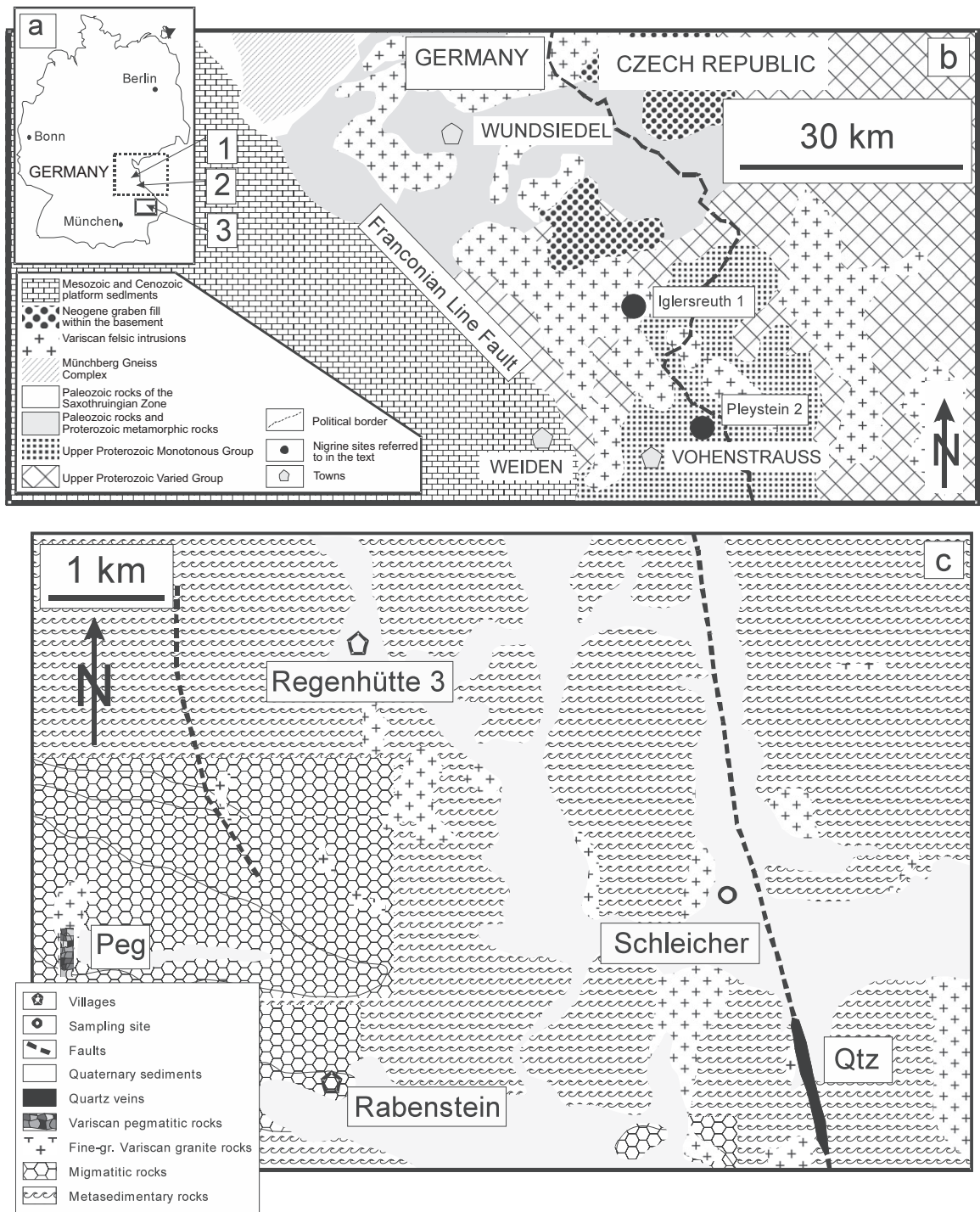


Fig. 1: Position and geology of the working areas in the NE Bavarian basement. (a) Index map to show the position of the nigrine bearing drainage systems in the various study areas. (b) Geological setting of the areas around Iglersreuth (study area 1) and Pleystein (study area 2); geology after Zitzmann 1981. (c) Close-up view of the geological setting of the area near Regenhütte (study area 3); geology after Madel et al. 1968.

Abb. 1: Lage und Geologie der Arbeitsgebiete im nordostbayerischen Grundgebirge. (a) Übersichtskarte der Nigrin führenden Gerinnensysteme in den verschiedenen Arbeitsgebieten. (b) Geologisches Umfeld in Raum Iglersreuth (Arbeitsgebiet 1) und Pleystein (Arbeitsgebiet 2); Geologie nach Zitzmann 1981. (c) Detailkarte der Geologie im Raum Regenhütte (Arbeitsgebiet 3); Geologie nach Madel et al. 1968.

This study provides a classification scheme of nigrine illustrated by images and supplements a table giving its chemical composition to facilitate provenance analyses and to assist in constraining the level of erosion in the source area.

2. Geological setting

The oldest country rocks, Upper Proterozoic in age, are exposed in the SE part of the study area (Franke 1989; Fig. 1b). Towards the NW, low to medium grade metamorphic Paleozoic and Upper Proterozoic rocks of the Saxothuringian Zone terminate the Moldanubian basement rocks. Variscan convergence in mid-Carboniferous times resulted in the folding of these rocks and in the emplacement of synorogenic granites. During the Cenozoic the basement was strongly up-

lifted and during the Neogene and Quaternary the drainage systems with their nigrine placer deposits evolved in the NE Bavarian basement.

3. Methodology

Minerals were identified by examination of thin and polished sections, SEM-EDX and X-ray diffraction analyses. Thin sections were studied under the petrographic microscope, polished sections under the ore microscope using air and immersion objectives. Electron microprobe analyses were carried with a Cameca SX100. The technique is described in Dill et al. (2007b). Back-scattered electron images (BSE) were used to depict the textural variation of nigrine grains (Figs. 2, 3).

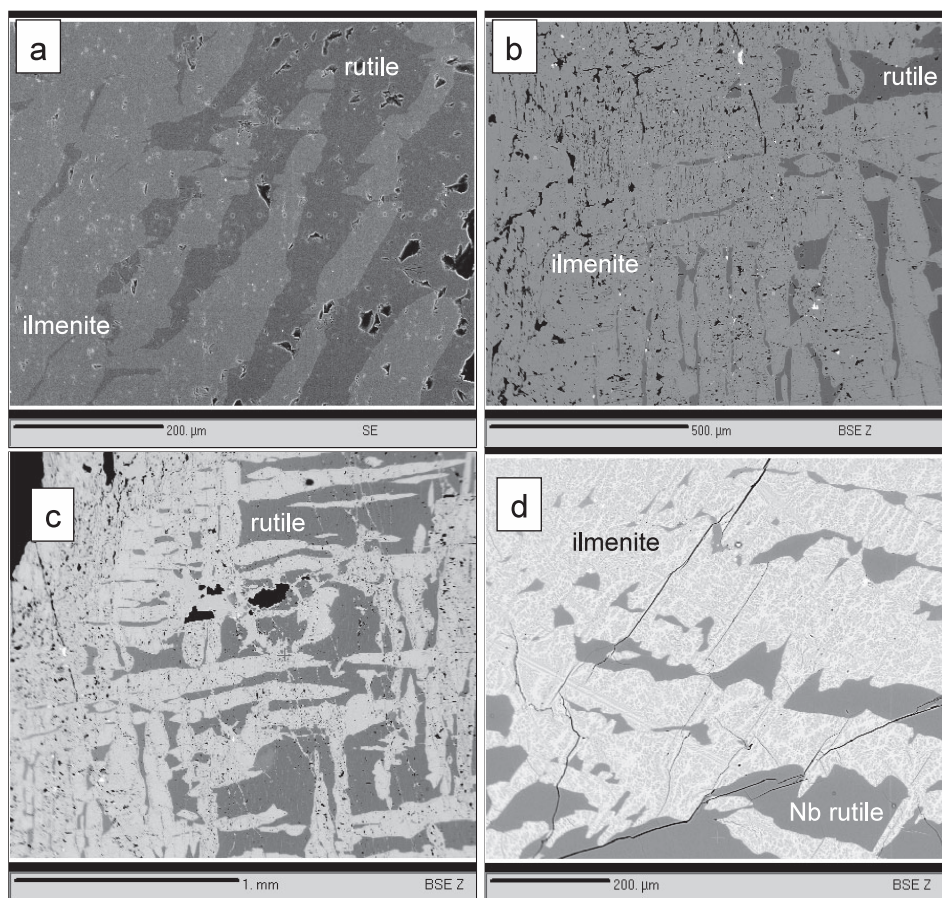


Fig. 2: Micrographs of nigrine (BSE images). (a) Nigrine type A.1, zoned grains; locality: Pleystein (study area 2). (b) Nigrine type A.1 zoned grains of rutile with ilmenite; locality: Pleystein (study area 2). (c) Nigrine type A.2 zoned grains with rim and lamellae of ilmenite; locality: Pleystein (study area 2). (d) Nigrine type A.3 insular niobian rutile rimmed by ilmenite and replaced by lamellae of pseudorutile; locality: Regenhütte (study area 3).

Abb. 2: Mikrosondenaufnahmen (Rückstrahlelektronenaufnahmen) von Nigrin. (a) Nigrintyp A.1, zonierte Mineralkörner; Fundort: Pleystein (Arbeitsgebiet 2). (b) Nigrintyp A.1, zonierte Mineralkörner aus Rutil und Ilmenit bestehend; Fundort: Pleystein (Arbeitsgebiet 2). (c) Nigrintyp A.2, zonierte Mineralkörner mit Ilmenit am Rand und als Entmischungslamellen; Fundort: Pleystein (Arbeitsgebiet 2). (d) Nigrintyp A.3, inselartiger niobreicher Rutil, umgeben von Ilmenit, der von Verdrängungslamellen aus Pseudorutil durchsetzt ist; Fundort: Pleystein (Arbeitsgebiet 2).

4. Results

4.1 Area 1 – Iglersreuth

The nigrine bearing fluvial drainage system in the northernmost study area 1 cuts into granitic rocks of the Falkenberg

and Flossenbuerg granite massifs (Wendt et al. 1986; Fig. 1b). Swarms of pegmatites and aplites may be traced across the whole Iglersreuth study area – not shown on the map for reasons of size (Forster et al. 1967). Rounded to angular black grains (4 mm in size) of nigrine in the alluvial-fluvial placer deposits have rather simple textures with slender la-

Tab. 1: Representative electron microprobe analyses (EMPA) of mineral phases included in rutile and ilmenite, and of alteration products of nigrine from the Iglersreuth (1), Pleystein (2) and Regenhütte (3) study areas in the NE Bavarian basement. (a) rutile and textures, (b) ilmenite, mineral inclusions and alteration minerals.

Tab. 1: Repräsentative Analysen der Nigrinkörner und ihrer Einschlüsse mittels Elektronenstrahlmikrosonde. Es handelt sich um Rutil, Ilmenit und deren Einschluss- und Alterationsphasen aus den Arbeitsgebieten von Iglersreuth (1), Pleystein (2) und Regenhütte (3) im nordostbayerischen Grundgebirge. (a) Rutil und Nigrintexturen, (b) Ilmenite, Mineraleinschlüsse und Alterationsminerale.

Sampling location	Type	Texture	Rutile core		Rutile lamellae/ grains		Relation to deforma- tion	Host rocks	Geomorphologi- cal and economic conclusions
			Nb ₂ O ₅	FeO	Nb ₂ O ₅	FeO			
Weissenstein	A.1a	zoned grains	0.5	0.4	3.3	2	pre- to synkine- matic nigrinisa- tion	mylonitic quartz- bearing shear zones (core)	deeply eroded siliceous root zone – area of little or no economic potential for rare metal pegmatites
Pingermühle (Zottbach)	A.1b	zoned grains with ilmenite rim	0.8–1	0.2–6	1.1–7.4	0.5–2.9			
Pflaumbach near Pleystein	A.1c	zoned grains with ilmenite rim	0.96	0.6					
Steininger Loh NW Fahrenberg	A.2	zoned grains with trellis-like ilmenite lamellae	0.3	0.5	1.1–3.5	1.1			
Schleicher near Zwiesel	A.3a	insular rutile rimmed by ilmenite and pseudorutile	0.3–6.6	0.4–6.6					
Schleicher near Zwiesel	A.3b	rutile core of nigrine grain	0.3–0.5	0.3–0.6					
Schleicher near Zwiesel	A.3c	rutile core	0.4–2.5	0.3–1.1			postkine- matic nigrinisa- tion	aplitic to peg- matitic quartz- feldspar mobili- zates (core)	moderately deep eroded – target area of rare metal pegmatites
Pingermühle (Zottbach)	B.1	exsolution lamellae of rutile in ilmenite and Nb-rutile lamellae in rutile	0.3–0.4	0.4–0.8	1.5–8.6	1.0–4.2			
Iglersreuth Tirschen- reuther Waldnaab	B.2	ilmenite with lamellae of Nb rutile			0.03– 21.0	0.2–7.0			
Schleicher near Zwiesel	B.3a	rutile lamellae interfingering with ilmenite and pseudorutile			7.0	3.7			
Schleicher near Zwiesel	B.3b	rutile lamellae in the ilmenite rim of nigrine grains			7.6–14.7	2.9–8.0			
Schleicher near Zwiesel	B.3c	rutile lamellae from the core into the ilmenite rim			2.7–4.1	1.1–1.6			
Schleicher near Zwiesel	B.3d	thin rutile lamellae within the rutile core			0.4	15	postkine- matic	quartz veins (margin)	very little to moderately deep eroded – proxi- mal to wolframite and scheelite mineralization
Pflaumbach near Pleystein	C	rutile with exsolu- tion lamellae of W-enriched rutile	0.15	0.5	0.3	2			

Sampling location	Type	Ilmenite		Inclusions					Alteration		Relation to deformation	Host rocks	Geomorphological and economic conclusions
Abbreviations: hm-lam, hematite lamellae; sph, sphalerite; po, pyrrhotite; mt, magnetite; x, present		MnO	Nb ₂ O ₅	Wolframite	Columbite	Pyrochlore	Zircon	others	Pseudorutile	Ti-Al-P-Fe ox			
Weissenstein	A.1a	1.7	0.1	X					X		pre- to synkinematic nigrinisation	mylonitic quartz-bearing shear zones (core)	deeply eroded siliceous root zone – area of little or no economic potential for rare metal pegmatites
Pingermühle (Zottbach)	A.1b	1.1		X					X				
Pflaumbach near Pleystein	A.1c	3	0.09										
Steininger Loh NW Fahenberg	A.2	1.8	0.1	X				sph, po					
Schleicher near Zwiesel	A.3a	0.8–2.9	0.0–0.4	X					X				
Schleicher near Zwiesel	A.3b												
Schleicher near Zwiesel	A.3c			X							postkinematic nigrinisation	aplitic to pegmatitic quartz-feldspar mobilizates (core)	moderately deep eroded – target area of rare metal pegmatites
Pingermühle (Zottbach)	B.1	2.4	0.22		X	X		mt, U-pyrochlore		X			
Iglersreuth Tirschenreuther Waldnaab	B.2	1.5–9.0	0.1–0.5	X	X		X		X	X			
Schleicher near Zwiesel	B.3a												
Schleicher near Zwiesel	B.3b				X								
Schleicher near Zwiesel	B.3c	1.4	0.1										
Schleicher near Zwiesel	B.3d										postkinematic	quartz veins (margin)	very little to moderately deep eroded – proximal to wolframite and scheelite mineralization
Pflaumbach near Pleystein	C												

mellae of rutile intergrown with ilmenite. Ilmenite solid solution series show a rather high pyrophanite component (MnTiO₃), expressed by MnO contents in the range 1.5–9.0 wt. % (Fig. 3b, Tab. 1). Under the ore microscope, columbite-(Fe), wolframite and zircon have been identified (Tab. 1).

4.2 Area 2 – Pleystein

Upper Proterozoic metamorphic rocks were intruded by Late Carboniferous felsic intrusive rocks including aplites and pegmatites (Forster 1965, Muecke 1981, Muecke et al. 1990, Wendt et al. 1994; Fig. 1b). Swarms of quartz veins strike in NW–SE direction across the Late Variscan granite province. Nigrine has been sampled from alluvial-fluvial placer depos-

its as well as quartz veins (Dill et al. 2007a). Nigrine grains from the Pleystein area show a varied spectrum of mineral inclusions and textures (Tab. 1). The most primitive textural type is recorded from quartz veins with zoned intergrowth of rutile (core) and ilmenite (rim; Figs. 2a, b). Zoned nigrine grains exhibit trellis-like ilmenite lamellae (Fig. 2c). Pyrrhotite and Fe bearing sphalerite are scattered as tiny grains in nigrine. Another type of nigrine shows a markedly different texture characterised by exsolution lamellae of rutile in ilmenite and Nb-rutile lamellae in rutile (Fig. 3a). This nigrine type contrasts with all other nigrine types by its variety of inclusions of columbite-(Fe) and U bearing pyrochlore (Tab. 1; Dill et al. 2007a). Another unique type of Ti compound cannot be termed a nigrine by definition because of its lack of ilmenite. Rutile is peppered with crisscrossing exsolution lamellae of W-enriched rutile (Fig. 3d).

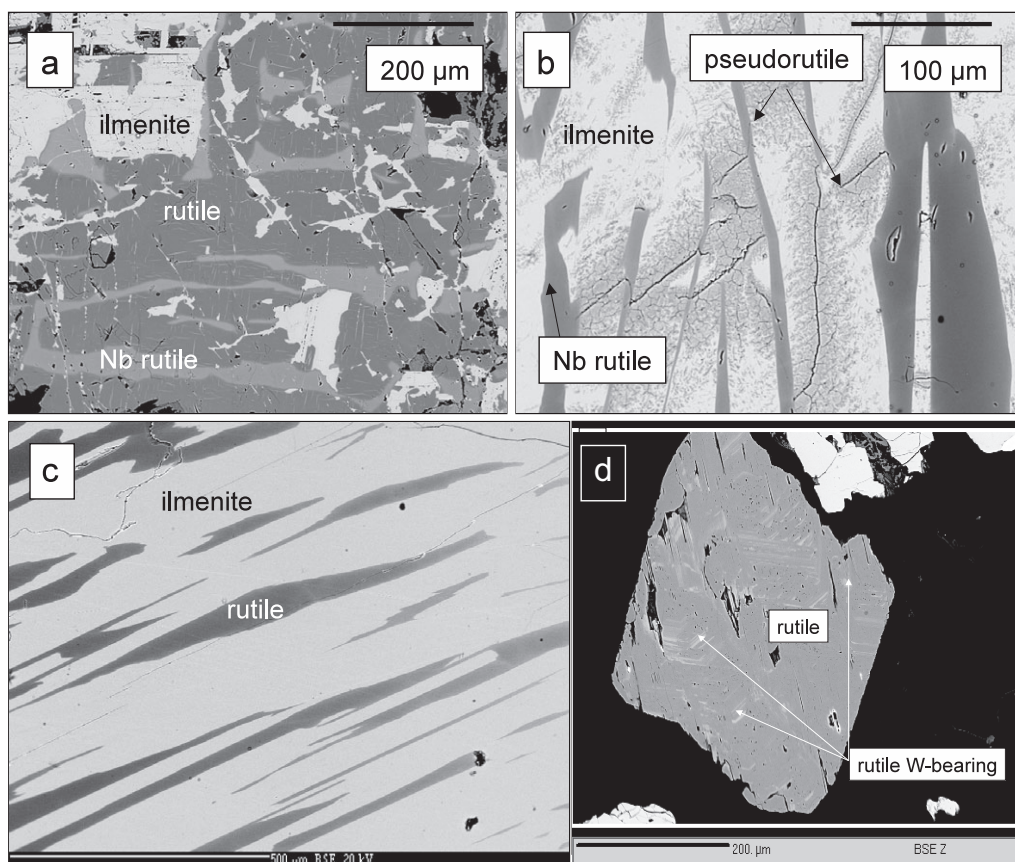


Fig. 3: Micrographs of nigrine (BSE images). (a) Nigrine type B.1 exsolution lamellae of rutile in ilmenite and Nb rutile lamellae in rutile; locality: Pleystein (study area 2). (b) Nigrin type B.2 ilmenite replaced by pseudorutile with lamellae of niobian rutile; locality: Iglersreuth (study area 1). (c) Nigrine type B.3 rutile lamellae in the ilmenite rim of nigrine grains; locality: Regenhütte (study area 3). (d) Nigrine type C. Rutile with exsolution lamellae of W-enriched rutile; locality: Pleystein (study area 2).

Abb. 3: Mikrosondenaufnahmen (Rückstrahlelektronenaufnahmen) von Nigrin. (a) Nigrintyp B.1, Entmischungslamellen von Rutil im Ilmenit und von niobreichem Rutil im Rutil; Fundort: Pleystein (Arbeitsgebiet 2). (b) Nigrintyp B.2, Ilmenit mit Lamellen von niobreichem Rutil von Pseudorutil verdrängt; Fundort: Iglersreuth (Arbeitsgebiet 1). (c) Nigrintyp B.3, Rutillamellen im Ilmenitsaum von Nigrinkörnern; Fundort: Regenhütte (Arbeitsgebiet 3). (d) Nigrintyp C, Rutil mit Entmischungslamellen von wolframreichen Rutil; Fundort: Pleystein (Arbeitsgebiet 2).

4.3 Area 3 – Regenhütte

This location in the Bayerischer Wald was selected for its position between a quartz lode striking roughly N–S and the well-known Hühnerkobel pegmatite which is aligned also N–S, similar to its fine-grained host granite of Late Variscan age (Madel et al. 1968; Fig. 1c). Two types of nigrine (1 cm in size) have been identified in the stream sediments of the tributaries of the river Regen. One type of nigrine is made up of insular niobian rutile rimmed by ilmenite which is replaced by pseudorutile (Fig. 2d). Manganese contents in ilmenite closely resemble those values reported from the neighbouring Pleystein area. Tiny spots of wolframite, less than 0.5 µm in size, were identified as inclusions in ilmenite (Tab. 1). In one of the insular nigrine, a tripartite subdivision of nigrinisation may be established (Fig. 4). It starts off with undistorted exsolution of ilmenite from Nb rutile, followed

by kinking of exsolution lamellae of ilmenite in niobian rutile and eventually ends up with normal faulting of ilmenite, whereby exsolution lamellae of ilmenite are thrown against Nb rutile.

The second type of nigrine is characterised by slender rutile lamellae interfingering with ilmenite and pseudorutile protruding out of the rutile core (Fig. 3c). Only columbite-(Fe) was found to be included in ilmenite (Tab. 1).

5. Discussion

Based upon their texture and their accessory minerals three types of nigrine were established for the NE Bavarian study areas. What is the unroofing story to be told by these nigrine types in the heavy mineral assemblage?

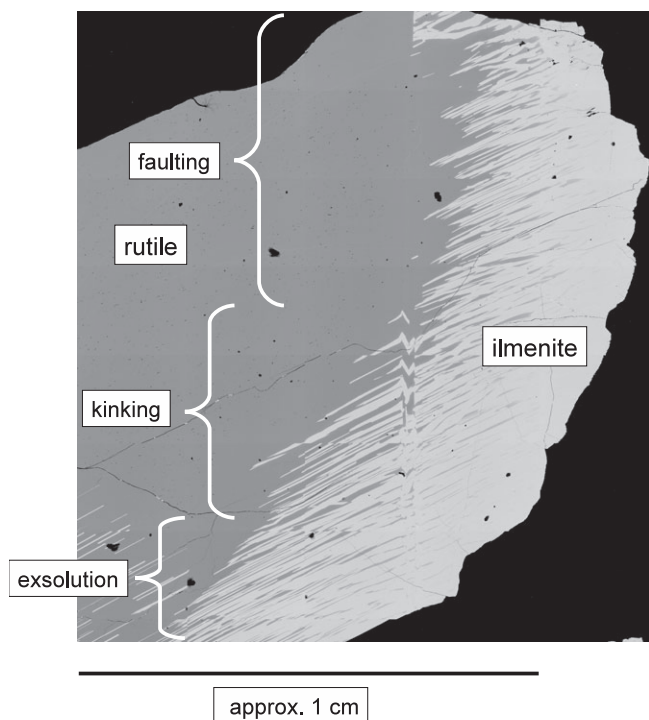


Fig. 4: Succession of chemical and mechanical processes affecting A.3 nigrine from Schleicher (Bayerischer Wald). Phase I: Undistorted exsolution of ilmenite from Nb rutile; phase II: kinking of exsolution lamellae of ilmenite in Nb rutile; phase III: normal faulting of ilmenite with ilmenite exsolution lamellae thrown against Nb rutile.

Abb. 4: Abfolge der chemischen und mechanischen Alterationsvorgänge, die den Nigrintyp A.3 bei Schleicher (Bayerischer Wald / Arbeitsgebiet 3) erfassten (Phase I: Ungestörte Entmischungslamellen von Ilmenit in niobreichem Rutil, Phase II: Knickfaltung der Ilmenitentmischungslamellen im niobreichen Rutil, Phase III: Bruchartige Verformung von Ilmenit, wobei Ilmenitentmischungslamellen „gegen Niobrutil verworfen werden“).

5.1 Pre- to synkinematic nigrine type A

Nigrine types A1a and 2 have been sampled from hard rocks and, hence, both types of nigrine may clearly be attributed to the formation of zones of silicification and quartz dykes. The sampling site in the Bayerischer Wald is located immediately west of a fault zone mineralised with quartz (Fig. 1c). The Nb specialisation in some nigrine placer samples, especially at Pleystein, cannot be ignored. Niobium is supposed to be a marker element for the pegmatitic mineralisation overlying the quartz root. It was stripped-off by erosion during unroofing of these quartz-feldspar systems west of the Hagendorf-Pleystein pegmatite province. By analogy with similar patterns described by Černý et al. (1999) from the McGuire granitic pegmatite, the intergrowth patterns between rutile and ilmenite are interpreted as exsolution structures. This process did not take place in a mechanically stable environment as observed in many postkinematic granites in the Oberpfälzer Wald (Fig. 4).

Nigrinisation, here defined as the split-up of a Fe-Ti bearing solid solution series into two discrete mineral phases, is pre- to synkinematic relative to the shearing in the quartz bearing fault system. The fault system hosting nigrine in the Bayerischer Wald forms part of the strain-stress system of the NW striking “Pfahl” zone, a mylonitic shear zone associated with brittle to ductile deformation fabrics and a conspicuous hydrothermal quartz mineralisation.

5.2 Postkinematic nigrine type B

Type-B nigrine is left unaffected by Variscan deformation and, hence, its origin is interpreted as postkinematic nigrinisation. Moreover, nigrine type B contains a variegated spectrum of inclusions mainly of the columbite solid solution series and pyrochlore solid solution series stressing the close relationship between nigrinisation and the formation of pegmatitic and aplitic mobilisates. Columbite group minerals from Iglersreuth and Pingermuehle have $\text{Nb}_2\text{O}_5/\text{Ta}_2\text{O}_5$ ratios almost identical with $\text{Nb}_2\text{O}_5/\text{Ta}_2\text{O}_5$ ratios calculated for columbite-(Fe) from the Hagendorf-South pegmatite (Forster et al. 1967). The $\text{Nb}_2\text{O}_5/\text{Ta}_2\text{O}_5$ of columbite-(Fe) from the nigrine bearing stream sediments at Schleicher is significantly lower than those of the northern part of the NE Bavarian basement and resembles the $\text{Nb}_2\text{O}_5/\text{Ta}_2\text{O}_5$ ratios of columbite-(Fe) from the Huehnerkobel pegmatite (Forster et al. 1967). There is little doubt that nigrine B is genetically related to the apical parts of the Late Variscan pegmatites, which in parts may be traced back to the deep-seated root zones or shear systems hosting type A nigrine.

5.3 Postkinematic wolframite bearing rutile type C

Type C does not qualify as nigrine *sensu stricto*, but fits into the structural pattern developed for nigrine A and B. In those sites where wolframite is scavenged from the mineralising solutions and incorporated into rutile, wolframite is no longer stable. The solubilities of rutile in the “dry” melts were investigated and compared to those of columbite, tantalite and wolframite in similar melts by Linnen (2005). Wolframite solubility increases with increasing water content in the sub-aluminous melt. According to Sasaki et al. (2003), pyrophanite and Mn enriched ilmenite crystallised in the greatly differentiated facies of the Tono pluton, NE Japan, which solidified from an intrinsically volatile-rich magma. Moreover, the oxygen partial pressure considerably affects the Fe-Mn-Ti ternary system under subsolidus conditions (Kang & Lee 2005). Elevated Mn contents and low FeO/MnO ratios in ilmenite and wolframite supposedly are due to more oxidising conditions. In a strongly oxidising environment this results in the precipitation of W bearing rutile with low Mn and Fe.

Tungsten bearing rutile formed at the margin of the Hagendorf-Pleystein pegmatite province. Such rutiles abnormally enriched in W are known from alluvial to fluvial Sn

placer deposits which have been derived from erosion of high-temperature quartz vein and granitic mineralisations (Hassan 1994, Sardi 2003). A similar scenario is supposed to have occurred in study area 2, where the primary mineralisation of W bearing rutile was eroded and only the succeeding wolframite-quartz vein mineralisation as well as the scheelite bearing calcsilicates were, in places, preserved around Pleystein (Dill 1989).

6. Summary and conclusions

The value of nigrine, found in alluvial and fluvial deposits, for geomorphologists and petrographers alike lies in its contribution to the unroofing story of the bedrock (Dill 1995).

- Type A (“low Mn wolframite nigrine”) is related to the deeply eroded siliceous parts of pegmatites. These mineral grains developed in mylonitic quartz bearing shear zones in course of pre- to synkinematic processes. The temperature of formation cannot be given, but the redox conditions are supposed to be reducing based upon the FeO/MnO ratio.
- Type B (“high Mn wolframite-columbite-pyroxhlore nigrine”) is related to highly differentiated apical parts of pegmatite stocks where rare metal concentration in form of oxidic minerals may be expected to have escaped erosion. Postkinematic nigrinisation took place in pegmatitic rocks under more oxidising conditions.
- Type C (“tungsten bearing rutile or ilmenite-depleted nigrine”) indicates the passage from the pegmatitic mineralisation into the scheelite and wolframite mineralisation at the margin of a pegmatite district where conditions are rather oxidising. Type C nigrine denotes an overall shallow level of erosion and formed under postkinematic conditions.

Nigrine is as a shelter for various types of mineral with redox sensitive elements such as Fe and Mn, preventing these minerals from decomposition by supergene alteration. Replacement minerals such as pseudorutile or P bearing “leucoxene” may record the supergene alteration affecting these pegmatites during the most recent periods of the geological history.

7. Acknowledgements

We are indebted to J. Lodziak who conducted the electron-microprobe analyses. Chemical analyses were carried out in the laboratory of BGR by F. Korte. D. Weck has carried out the XRD analyses. We kindly acknowledge the contribution of samples by G. Steyer, M. Fuessl and B. Weber. We extend our gratitude also to two anonymous reviewers for their fruitful comments to our manuscript.

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Manuscript received: 26.03.2009

Accepted for publication: 26.04.2009