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Calculating minor constituents in synthetic corundum abrasives

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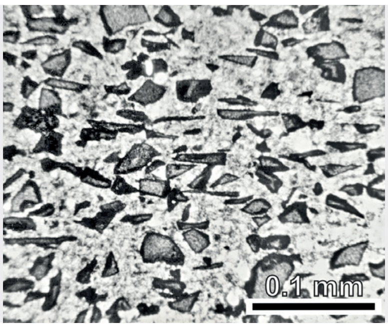
Abstract: Electromelted corundum and SiC are the most frequently used industrial abrasives. The final products of electromelting of Al_2O_3 and bauxites contain contaminants. Their content and present glass phases adversely affect the functional properties of the abrasives. It is shown that the content of the contaminants can be calculated from chemical analysis. The calculation is similar to that used to determine the theoretical analysis of clay and other raw materials for the manufacture of ceramics (e.g. calculation by Kollauner-Matejka). An example of such calculation is shown in the case of the brown i.e. the most common corundum and the results are compared with those, obtained by microscope analysis as a reference. These calculations can be used for quality control during production and also for monitoring of quality changes during research and development of new types of electro corundum. The computed compositions are in good agreement with those found by the microscope analysis. The content of the contaminants was also monitored as a function of particle size of the abrasives. A certain correlation was observed between the crystal size and the classification method of the particle size. The content of the deleterious contaminants decreased with decreasing size of the crystals from large to medium whilst it increased as the crystals further decreased from medium to fine particles; it could reach up to 20 % in the worst case.

Key words: synthetic corundum abrasives, cracking grinding wheels, brown corundum, mineral composition, calculating

Graphical abstract



Highlights

- Electromelting of Al_2O_3 and bauxites at production of corundum and SiC, as the most frequent industrial abrasives, produces contaminants.
- The content of the contaminants can be calculated from chemical analysis (e.g. Kollauner-Matejka calculation method), which results are in good agreement with those from microscope analysis.

Introduction

Each abrasive tool consists of abrasive grains, their bond and pores, i.e. components of a solid and gasiform nature. Abrasive grains represent a special mineral phase and the bond is either anorganic, for example the synthetic resin, or the rubber-based materials. The variability of individual components is great. Their properties and distribution in the abrasive tool directly affect also its physical-mechanical properties. Petrological and mineralogical procedures have made it possible to solve some technological problems of many years and to integrate the control methods for verifying the quality of abrasive grains.

Definition of abrasive materials and abrasive tools

The abrasive can only be a material with higher hardness than the material we want to work with and which is rigid enough to withstand the mechanical destruction. The abrasive tool can also be described as the abrasive grains connected with a metal, ceramic, glass or organic bond. These grains are connected in a defined geometric shape, most frequently as an abrasive wheel. The parameters that define the quality of abrasive tool at present are as follows: (a) type of abrasive; (b) type of bond; (c) abrasive grain size; (d) shape of abrasive grains; (e) hardness of the tool (which means the resistance of abrasive grain to mechanical break out of the wheel); (f) structure of the tool determined by pore volume or volume of abrasive grains used.

Type of abrasive

An abrasive is a material that indicates the same or greater hardness than the workpiece and is resistant to temperature and chemical reactions. In the past, silicious sand, garnet and emery (a natural rock made of corundum) were used as abrasives, later joined by the diamond dust.

During the industrial revolution in the 18th and 19th centuries, following the requirements of the evolving technologies, the synthesis of hard substances was achieved (Baumann, 1962; Kamencev, 1950; Polubelova et al., 1968; Novotný & Turnovec, 1967; Turnovec, 1971; Hořínek & Turnovec, 1970; Turnovec & Illášová, 2009, 2011).

An overview of the main natural and synthetic types of abrasives can be seen in Chart 1. The hardest abrasive is diamond, the softest one is quartz. The methodology of studying abrasive materials when it comes to preparation and making cuts is a rather complex. Therefore, a methodology of studying the cuts was developed. The first researchers who studied the cuts in the reflected light were Filonenko and Lavrov (1958). In the following text we focus on silicon carbide and corundum abrasives as well as on abrasive tools.

Silicon carbide (SiC, carborundum) was independently synthesized by Acheson (1892) and Schützenberger (1892). Acheson´s technology is still in use today (Turnovec & Kleander, 1970; Kleander, 1971). It is produced in the electric resistance furnaces bearing the Acheson´s name and it is made of silicious sand and carbon raw material, which is a petroleum coke (Fig. 1). It has been experimentally demonstrated that the initial temperature for SiC formation is 1200–1400 °C. The main reactions and formation of α-SiC occur in the range of 1700–2300 °C.

The electro-melted corundum, as an essential raw material for the production of abrasive grains, contains, in addition to the corundum phase, also other accompanying minerals and their total content may exceed 10 %. The basic types of corundum abrasive materials are:

a) monocorundum (named as Alucryst special);
b) brown corundum (also known as Normalcorundum) and its microcrystalline modification;
c) white corundum (named as Elektrit, Alundum, etc.);
d) red corundum (called Rubin);
e) alloyed corundum (also known as zirconium, vanadium, manganese-titanium corundum, etc.).

Monocorundum is made by crystallization of Al_2O_3 dissolved in a sulphide melt by melting a mixture of bauxite or industrial aluminum oxide with a suitable sulphide (most often pyrite) in an electric furnace. This production was patented by Hagelund in 1922 and the technology is still used today. Unlike other corundum materials, monocorundum is chemically the cleanest material with the best cutting properties. The main raw material for brown corundum is bauxite. The white corundum is produced without any additives, while adding Cr_2O_3 produces the red corundum and adding other additives participate in producing other alloyed corundums.

For corundums produced by melting an industrial alumina, the main pollutant is β-corundum and the glass phase. The influence of other accompanying components is negligible. During the crystallization process in alloyed corundums, adulterants (e.g. baddeleyte in Zr-corundum) often appear as the catalyzers that favorably influence the resulting structure and increase the toughness of the resulting abrasive. For brown corundum made from bauxites, the detrimental accompanying components are carbides and titanium nitrides, formed during over-reduction of the bauxite melt. These components, although only in the trace content, significantly increase their volume C in the critical temperature range of 500–600 ºC and cause their destruction. The formation of accompanying components can be quantified from the chemical composition. An indicator is that, in addition to nitrides and carbides, the greater quantities of anorthite or mullite glass phase are formed. On the contrary, the presence of titanium in the structural lattice of corundum crystals is a benefit because during the burning, the toughness of the abrasive grains is increasing.

Mineral composition and properties of abrasive grains

The functional properties of corundum abrasive grains are influenced both by the composition and structure of the melting products and by the technology of their production. In general, the quality of all abrasive grains is determined by their size and granularity, mechanical strength, shape, mineral composition and structure, and by the content of inclusions (metal and magnetic particles). Standard ČSN 224012 defines an abrasive grain as a crystal or abrasive material particles whose width do not exceed 5 mm and the ratio of the largest dimension to the smallest does not exceed 90 % of the grains.

The influence of the mineral composition of abrasive grains was studied for addressing the causes of cracking the abrasive tools (with ceramic bonding) already during the burning. During the burning, not only a ceramic bonding mass is formed, but also mutual reactions between the abrasive grains and the liquid phase of the bond can be noticed. The gradual heating results in the diffusion of Al_2O_3, which is caused by the aggressiveness of the alkali oxides of bond, especially by Na_2O, represented in congruently forming liquid phase (Turnovec, 1984).

Cracking of ceramically bonded brown corundum grinding wheels

On cracked wheels, the boundaries between grains and bond were observed – first under binoculars, later on polished sections in the reflected light. It has been shown that the cracks are present in the grain-bond interface as well as within the grains themselves. In the wheels around the cracks already formed during burning, kidney-like formations, later identified as anosovite and rutile, were found on the grain surface and at the points of cracking. These are impurities (oxygen-free titanium compounds), which, during oxidation burning between 500–600 ºC, extremely increase their volume and cause anomalous expansion of the abrasive grains. This is the evidence that the bauxite used was over-reduced during melting of brown corundum (which was a fatal mistake; water infiltrating the bauxite was up to 11 %, which caused the over-reduction with the same total amount of bauxite and reducing agent).

The functional properties of the abrasive depend on the content of the accompanying components (mainly β-corundum in white and hexaaluminate in brown corundum or glass phases). Similar to the rational analyses of ceramic raw materials (e.g. Kollauner-Matějka calculations), the mineral composition of corundums can be calculated. As an example, calculations for brown corundum are given. Its melting conditions are close to equilibrium. They take place in the liquid phase and the subsequent crystallization is sufficiently slow. Crystallization occurs in the CaO – Al_2O_3 – SiO_2 system, near the Al_2O_3 peak (Fig. 1). According to the Rankin's diagram (Filonenko & Lavrov, 1958), three final crystallization products are possible:

I. corundum; mullite; anorthite;

II. corundum; anorthite;

III. corundum, calcium hexaaluminate; anorthite.

This is a simplification that does not take into account the other accompanying components, which are the MgO and TiO_2 compounds, mostly the spinellides and taosite. The proportion of spinellides is only trace to negligible, but, most importantly, it does not affect functional properties. The situation is different for titanium compounds (Turnovec, 1969, 1971, 1982a). A crystallization diagram is shown in Fig. 2.

From the simple ratio of oxides bound to individual components, it is clear that the most preferred melting product is the one in which, in addition to corundum, only anorthite is present. The least preferred is a melt allowing the formation of hexaluminate. Based on the steichiometric ratios of the individual oxides, the proportions of the main components can be calculated from the analytical data. Chemical analysis is a part of quality control and one of the basic quality parameters and is listed in the Czechoslovak Standard ČSN 22 4044. Material conforming to ČSN standard can have a very variable mineral composition thus relating to its functional properties.

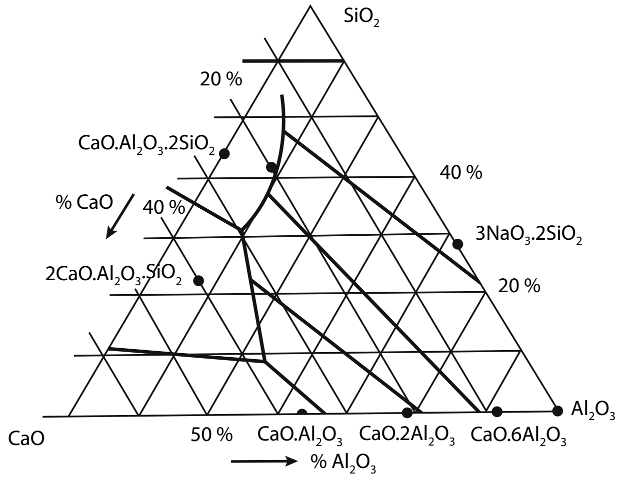


Fig. 1. Three phase diagram CaO – Al_2O_3 – SiO_2, near the top of the Al_2O (Filonenko & Lavrov, 1958).

The originally proposed calculation method (Turnovec, 1970) was quite demanding. During practical use it was simplified and made more effective (Turnovec, 1982b). *Denoting variables and auxiliary variables:*

A. Al_2O_3 content determined by analysis;

B. SiO_2 content determined by analysis;

C. TiO_2 content determined by analysis;

D. CaO content determined by analysis.

H1 – content of Al_2O_3 in anorthite; H2 – content of Al_2O_3 in calcium hexaaluminate; H3 – content of Al_2O_3 in mullite; H4 – content of Al_2O_3 in taosite.
Resulting variables:
AN = anorthite content in %; MUL = mullite content in %; HXL = hexaaluminate content in %; TA = taosite

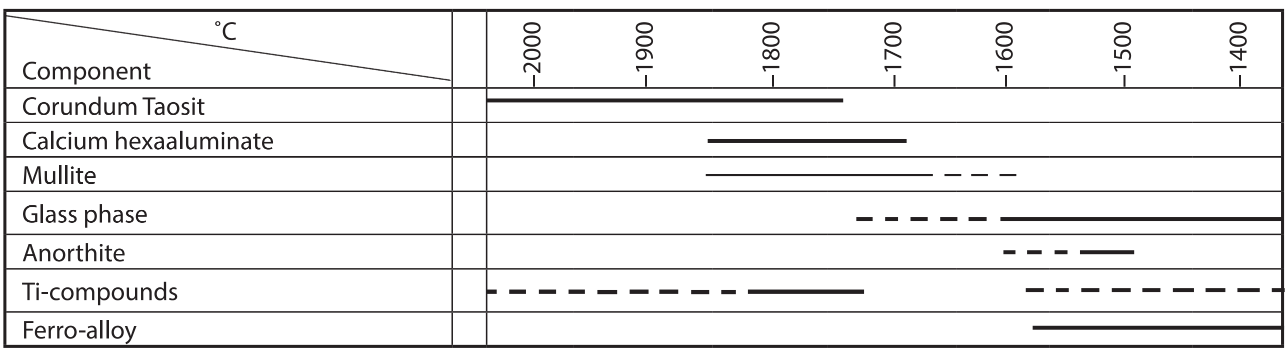


Fig. 2. The situation is different in the presence of compounds containing titanium (Turnovec, 1969, 1971, 1982a).

content in %; KC = corundum content in %; KF = physical corundum content, i.e. with taosite in %; KM = corundum module, i.e. ratio of Al_2O_3 content in corundum phase to Al_2O_3 content in accompanying components.

Method for calculating mineral composition

The first step – deciding the ratio D : B (CaO : SiO_2).
There are three possibilities:
a) the ratio is greater,
b) the ratio is 0.467,
c) the ratio is smaller.

The second step
possibility a)
AN = 1.58 × B × 1.467;
H 1 = AN – B × 1.467;
HXL = 11.91 × (D – 0.467 × B);
H 2 = HXL – (D + 0.467 × B).
Possibility b)
AN = 1.58 × (B + D);
H 1 = AN – B × 1.467.

Possibility c)
AN = 1.58 × D × 3.142;
H 1 = AN – 3.142 × D;
MUL = 3.546 × (B – 2.141 × D);
H 3 = MUL – (B + 2.141 × D).

The third step – a futher calculation common to all three
TA = 2,276 × C;
H 4 = TA – C;
KC = A – (H 1 + H 2 + H 4);
KF = KC + TA.

The fourth step – a calculation of the corundum module
KM = (KF – C) : (H 1 + H 2 + H 3)

Key to the variables and auxiliary variables used:
A. Al_2O_3 content from the analysis;
B. SiO_2 content from the analysis;
C. TiO_2 content from the analysis;
D. CaO content from the analysis.

H 1 – Al_2O_3 content in anorthite;
H 2 – Al_2O_3 conte nt in calcium hexaaluminate;
H 3 – Al_2O_3 content in mullite;
H 4 – Al_2O_3 content in taosite.

The resulting variables:
AN = content of anorthite in %;
MUL = content of mullite in %;
HXL = content of hexaaluminate in %;
TA = content of taosite in %; KO = content of corundum in %;
KF = content of physical corundum (total Al_2O_3 incl. taosite) in %;
KM = corundum modulus i.e. a ratio between Al_2O_3 in the corundum phase and Al_2O_3 in the remaining constituents.

Tab. 1
Mineral composition of brown corundum calculated from analytical data and determined microscopically.

Mineral composition of brown corundum calculated from analytical data and determined microscopically														
	Chemical composition				Calculation of mineral composition							Microscopic analysis		
sample	Al_2O_3	SiO_2	TiO_2	CaO	AN %	MUL %	HXL %	TA %	KC %	KF %	KM	Corundum %	Calcium hexaaluminate	Glass phase*
1	96.88	0.94	1.09	0.64	2.18	-	2.39	2.48	93.37	95.86	27.03	93.5	2.6	3.9
2	95.63	0.64	2.00	1.35	1.48	-	12.52	4.55	81.64	86.19	7.38	86.2	12.3	1.5
3	93.79	1.82	2.90	0.84	4.17	-	-	6.60	88.56	95.16	59.29	95.0	-	5.0
4	96.79	1.06	1.35	0.55	2.46	-	0.65	3.07		96.64		95.0	st	5.0
5	95.06	2.50	1.80	0.17	0.84	7.57	-	4.10	87.74	91.84	17.95	90.4	-	9.6
6	95.17	2.54	1.20	0.53	2.63	4.98	-	2.73	91.37	94.10	24.45	90.5	-	9.5
7	94.56	0.71	2.10	1.35	1.65	-	12.13	4.78	80.82	85.60	7.55	86.0	12.1	1.9

Notes: The designation of the components of the calculation is kept as for the calculation formulas AN = anortite, MUL = mullite, TA = content of taosite, HXL = calcium hexaaluminium, KC = physical corundum, taosite, KF = content of physical corundum (total Al_2O_3 incl. taoiste), KM = corundum modulus i.e. a ratio between Al_2O_3 in the corundum phase and Al_2O_3 in the remaining constituents.

* The glass phase represents all translucent components filling the spaces between microscopic analyses of corundum crystals, no distinction is made when the main constituents are anortite or mullite about the glass phase.

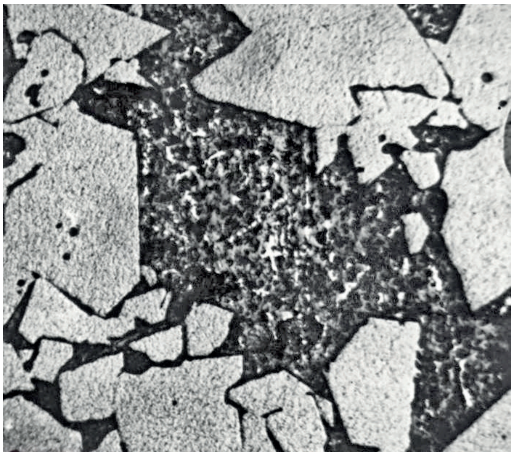


Fig. 3. Anorthite glass phase surrounded by corundum (Turnovec, 1982b). Width of view 80 μm.

Conclusion

The methods and approach of petrologists and mineralogists can significantly help in assessing the properties of abrasives and abrasive tools and many other technical materials, especially in their development. At the end, we summarize some examples where petrology contributed to solving technological problems in the field of abrasives: determination of mineral phases resulting from the production of abrasive materials; determining the structures and size of the crystals formed; solving the technological problem in the production of brown corundum (over-reduction); quantification of mineral composition from analytical data; evaluation of thermal expansion and its elimination by annealing; evaluation of abrasive grain shapes.

Comparison of calculations with microscopic analysis and basic chemical composition is shown in the attached table. As the table shows, the difference between the quantified and microscopically determined composition is only small. Conversions can be used to monitor quality during research work, but also for statistical evaluation of technical production control (Tab. 1). The agreement between the calculated and microscopically determined composition is relatively high.

The contents of the accompanying components were also monitored depending on the size of the abrasive grains. It has also been shown that there is a certain regularity depending on the size of the crystals and the size sorting method. From the largest to the medium grains, the pollutants content decreases, from the medium to the fine grains, pollutants again increase and can reach up to 20 % in the unfavorable case. The study of the polished sections has proved to be very effective in identifying individual mineral phases, but also in the study of the internal construction of abrasive tools and structures as described for natural rocks.

Calculation methods of the rational, i.e. mineral compositions from the chemical composition are quite accurate. The ability to determine the crystallization type by the calculation method (i.e. the presence of anorthite or mullite glass phase as indicators of the presence of other compounds) becomes a useful advantage in evaluating the properties of corundum abrasives and abrasive tools. The quantification of the mineral phases in electro-melted corundum enables to solve not only the abrasive or refractory phases but also technological problems (especially the over-reduction of the melting), which cannot be detected solely by analytical data. Through the computer algorithm, the results are available parallel with the analytical data, which allows technological interventions, increasing or decreasing the reducing agent content in the melt. It is advisable to ensure the formation of the anorthite glass, as the mullite phase increases, a danger of over-reduction of blocks becomes real. Timely quantification and adjustment of the batch brings a significant saving of funds, because the thermal adjustment (modification) of the over-reduced materials is very expensive and unnecessary, if the reduction is managed properly.

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Výpočet zastúpenia sprievodných zložiek v umelom korundovom brúsive

Elektrónovaný korund a SiC sú najčastejšie používané priemyselné brúsivá. Konečné produkty elektrolytického spracovania Al_2O_3 a bauxitov obsahujú kontaminanty. Ich obsah a obsah sklených fáz nepriaznivo ovplyvňuje funkčné vlastnosti brúsiv. Ukázalo sa, že obsah kontaminantov možno vypočítať chemickou analýzou. Výpočet je podobný výpočtu, ktorý sa použil na stanovenie teoretickej analýzy hliny a iných surovín na výrobu keramiky (napr. výpočty podľa Kollaunera-Matejku). Príklad takéhoto výpočtu je uvedený na zistenie obsahu hnedého, najbežnejšieho korundu a výsledky sa porovnávajú s výsledkami získanými mikroskopickou analýzou ako referencie. Tieto výpočty sa môžu použiť na kontrolu kvality počas výroby a tiež na sledovanie zmien kvality počas výskumu a vývoja nových typov elektrokorundu.

Každý brúsny nástroj sa skladá z brúsnych zŕn, spojiva a pórov, teda zo zložiek pevnej a plynnej povahy. Brúsne zrná sú špeciálnou minerálnou fázou a spojivo je buď anorganické, môže to byť syntetická živica, alebo materiály na báze kaučuku. Pestrosť jednotlivých zložiek je veľká. Od ich vlastností a rozloženia v brúsnom nástroji priamo závisia aj fyzikálno-mechanické vlastnosti. Petrologické a mineralogické postupy umožnili vyriešiť niektoré mnohoročné technologické problémy a doplniť kontrolné metódy na overovanie kvality brúsnych zŕn. Od obsahu sprievodných zložiek (hlavne β-korundu v bielom a hexahlinitanu v hnedom korunde, prípadne sklených fáz) závisia funkčné vlastnosti brúsiva. Podobne, ako sa vyčísľujú racionálne analýzy keramických surovín, možno vyčísliť minerálne zloženie korundov. Ako príklad uvádzame výpočty v prípade hnedého korundu. Podmienky jeho tavenia sa približujú rovnovážnym. Prebiehajú v tekutej fáze a následná kryštalizácia je dostatočne pomalá. Kryštalizácia nastáva v systéme CaO – Al_2O_3 – SiO_2, v oblasti blízkej vrcholu Al_2O_3. Poznáme tri konečné produkty kryštalizácie: 1. korund, mullit, anortit; 2. korund, anortit; 3. korund, hexahlinitan vápenatý, anortit.

Ide o zjednodušenie, ktoré neberie do úvahy ďalšie sprievodné zložky. Tými sú zlúčeniny MgO a TiO_2, teda prevažne spinelidy a taosit. Zastúpenie spinelidov je iba stopové až bezvýznamné a neovplyvňuje funkčné vlastnosti. Iná situácia je v prípade zlúčenín titánu. Z jednoduchého pomeru oxidov viazaných na jednotlivé zložky je zrejmé, že najvýhodnejší je taký produkt tavby, v ktorom sa okrem korundu bude vyskytovať iba anortit. Najmenej výhodná je tavba umožňujúca vznik hexahlinitanu. Na základe stechiometrických pomerov jednotlivých oxidov možno z analytických údajov vypočítať zastúpenie hlavných zložiek. Chemická analýza je súčasťou kontroly kvality a patrí medzi základné kvalitatívne parametre. Je uvedená v ČSN 22 4044. Materiál zodpovedajúci ČSN môže mať veľmi variabilné minerálne zloženie a s tým súvisia aj jeho funkčné vlastnosti.

Metódy aj prístup petrológov a mineralógov môžu významne napomôcť pri posudzovaní vlastností brúsiva a brúsnych nástrojov, ale aj mnohých ďalších technických hmôt, najmä pri ich vývoji. V závere zrekapitulujeme niektoré príklady, keď k riešeniu technologických problémov v odbore brúsiv prispela práve petrológia: stanovenie minerálnych fáz vznikajúcich pri výrobe brúsnych materiálov, stanovenie štruktúr a veľkosti vznikajúcich kryštálov, vyriešenie technologického problému pri výrobe hnedého korundu (preredukovanie), vyčíslenie minerálneho zloženia z analytických údajov, hodnotenie tepelnej rozťažnosti a jej odstránenia žíhaním, hodnotenie tvarov brúsnych zŕn. Porovnanie výpočtov s mikroskopickou analýzou a základné chemické zloženie je v pripojených tabuľkách. Ako vyplýva z tabuliek, rozdiel medzi vyčísleným a mikroskopicky stanoveným zložením je iba malý, nepatrný. Prepočty možno využiť pri sledovaní kvality počas výskumných prác, ale aj na štatistické hodnotenie technickej kontroly výroby. Zhoda medzi vypočítaným a mikroskopicky zisteným zložením je pomerne vysoká. Obsah sprievodných zložiek sa sledoval aj v závislosti od veľkosti brúsnych zŕn. Ukázalo sa, že aj tu existuje istá zákonitosť, podmienená veľkosťou kryštálov a spôsobom veľkostného triedenia. Od najväčších zŕn k stredným obsah prímesí klesá, od stredných k jemným opäť škodlivín pribúda a ich obsah môže v nepriaznivom prípade dosiahnuť až 20 %. Štúdium nábrusov sa ukázalo ako veľmi efektívne pri identifikácii jednotlivých minerálnych fáz, ale aj pri štúdiu vnútornej stavby brúsnych nástrojov.

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