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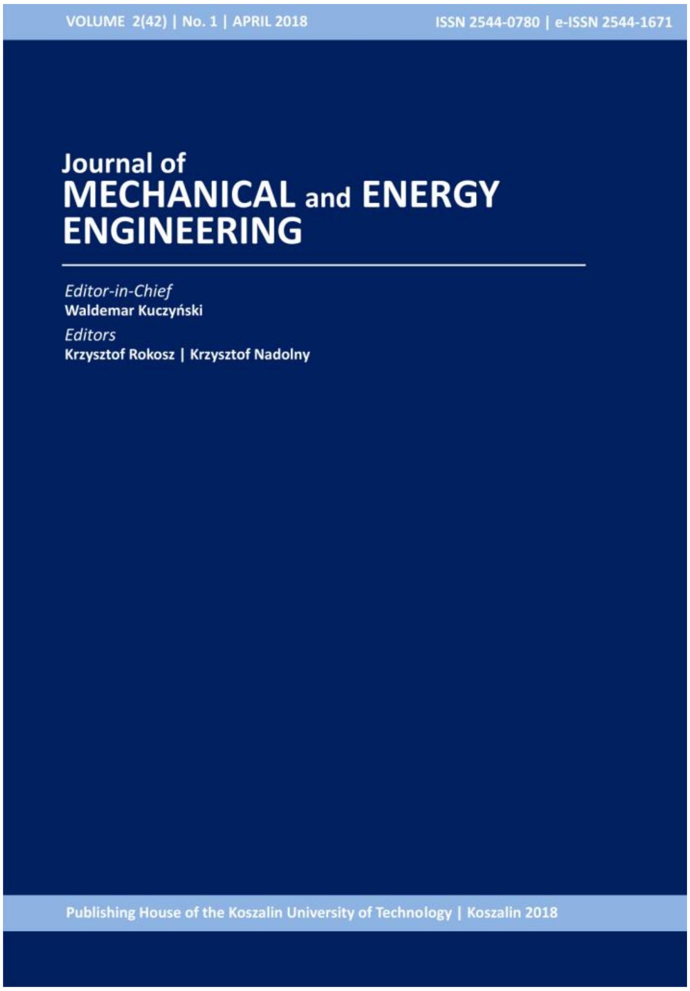

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SEM-BASED IMAGING AND ANALYSIS OF SURFACE MORPHOLOGY OF THE TRIZACT™ ADVANCED STRUCTURED ABRASIVES

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Abstract: This paper shows that imaging and analysis of morphological features of the surface of modern structured abrasives in the pre-machining state can be carried out by means of electron microscopy supported by image processing and analysis techniques. The acquisition of SEM micrographs for active surfaces of the four (A6, A30, A65, A160 grades) monolayer abrasive discs 237AA (3M) with Trizact™ abrasive grains was carried out by the use of Quanta 200 Mark II (FEI Company) high-resolution scanning electron microscope. Visual analysis allowed for the observation of abrasive grains mainly in terms of the occurrence on their surface of various defects resulting from the technology of their production. For a parametric analysis, the authors used the Fiji 1.51s software (J. Schindelin et al.) which, in turn, made it possible to determine the values of the basic geometrical parameters characterizing the abrasive grains in the selected area of the active surface of assessed abrasive tool. The observation and measurement instruments used in the experimental studies described in this work and the proposed methodology may present an interesting alternative approach to the assessment of the surface morphology of advanced structured abrasives.

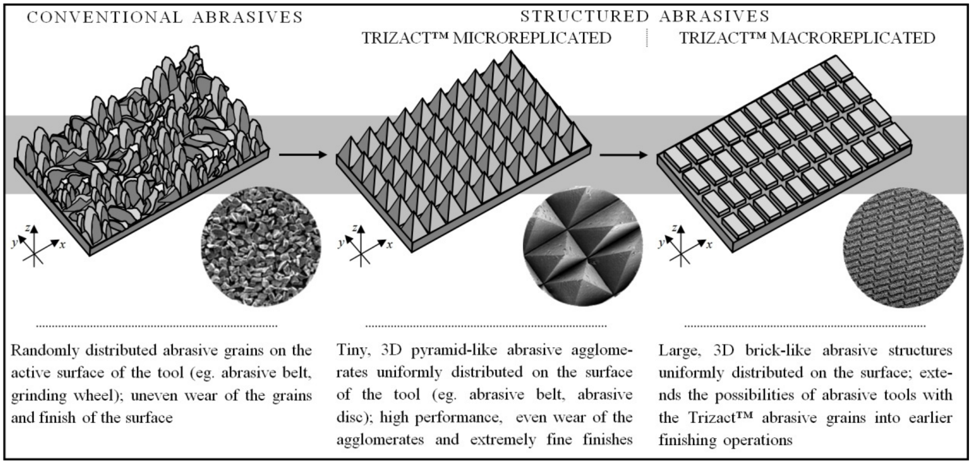
Keywords: Scanning electron microscopy, image processing and analysis, structured abrasives, monolayer abrasive disc, Trizact™

1. INTRODUCTION

In many modern construction solutions, it is extremely beneficial to use elements with surfaces characterised by a much higher requirements regarding their exploitational or utility features. Obtaining such features often involves the use in surface finishing process of other types of abrasive tools than commonly used grinding wheels. One of the types of such tools are, among others, flexible tools in the form of monolayer abrasive discs with a modern Trizact™ abrasive coating [1, 2]. Using of innovative Trizact™ abrasive grains is a completely new approach to the grinding process, both in terms of the specific geometry of the tool used and the method of its production. In the 1960s, the 3M (Maplewood, MN, USA) developed the so-called replication process, which became the basis of modern technology of producing characteristic 3D (structured) surfaces of abrasive grains.

These structured surfaces were defined by Evans and Bryan in the work [3] as *those where the surface structure is a design feature intended to give a specific functional performance.* For the Trizact™ (pyramid-like) abrasive grains, this technology is called *microreplication* as shown by Fletcher et al. in the work [4]. For the structures of a different shape (brick-like) and slightly larger geometrical dimensions, this process is called *macroreplication*. The evolution of abrasive materials, from conventional to structured ones, using the above mentioned micro- and macroreplication processes are presented in Fig. 1.

The Trizact™ abrasive grains are produced in the form of tetragonal pyramids with a square or rectangular base (with a variable apex angle from a range between 60° and 90°) from a set of elementary aluminium oxide

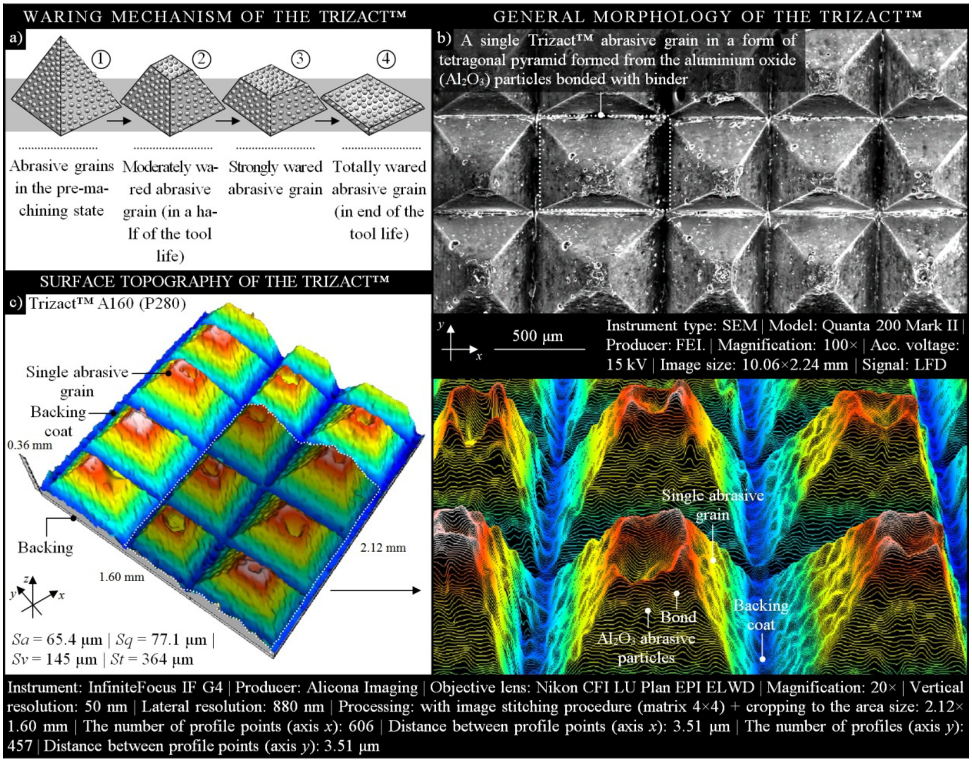


Randomly distributed abrasive grains on the active surface of the tool (eg. abrasive belt, grinding wheel); uneven wear of the grains and finish of the surface

Tiny, 3D pyramid-like abrasive agglomerates uniformly distributed on the surface of the tool (eg. abrasive belt, abrasive disc); high performance, even wear of the agglomerates and extremely fine finishes

Large, 3D brick-like abrasive structures uniformly distributed on the surface; extends the possibilities of abrasive tools with the Trizact™ abrasive grains into earlier finishing operations

Fig. 1. Evolution of abrasives – from conventional to structured



Instrument type: SEM | Model: Quanta 200 Mark II | Producer: FEI. | Magnification: 100× | Acc. voltage: 15 kV | Image size: 10.06×2.24 mm | Signal: LFD

Sa = 65.4 μm | Sq = 77.1 μm | Sv = 145 μm | St = 364 μm

Instrument: InfiniteFocus IF G4 | Producer: Alicona Imaging | Objective lens: Nikon CFI LU Plan EPI ELWD | Magnification: 20× | Vertical resolution: 50 nm | Lateral resolution: 880 nm | Processing: with image stitching procedure (matrix 4×4) + cropping to the area size: 2.12×1.60 mm | The number of profile points (axis x): 606 | Distance between profile points (axis x): 3.51 μm | The number of profiles (axis y): 457 | Distance between profile points (axis y): 3.51 μm

Fig. 2. Characteristic of basis features of the Trizact™ abrasive grains: a) schematic representation of the waring mechanism; b) general morphology of the grains; c) surface topography and close-up of the grains (see details in the text below)

(Al_2O_3) particles bonded with binder as described Eleková and Lipa, Goossens et al. as well as Zaborski and Pszczołowski in the works [5-7]. As a result, abrasive agglomerates are formed, from which homogeneous active surfaces of the abrasive tools characterized by precisely defined stereometry are produced. The possibility to choose the abrasive material and to control the size of individual abrasive agglomerates allows the production of tools with strictly defined cutting properties, allowing to increase the predictability of material removal efficiency and to obtain a high quality surface finish.

The Trizact™ abrasive grains production technology makes them characterized by a specific mechanism of its wear during work. This mechanism, presented by Zaborski and Pszczołowski in the work [7], consists in the even uncovering of successive layers of elementary particles of the agglomerate (that are not involved in the process), after they have been chipped off from the previous layer until to its base, which is schematically presented in Fig. 2a. The morphology of Trizact™ abrasive grains (A6 (3M) / P2000 (FEPA)) on the mono- layer abrasive disc 237AA (3M) acquired by the scanning electron microscope Quanta 200 Mark II (FEI Company, Hillsboro, OR, USA) and surface topography (A160 (3M) / P280 (FEPA) obtained by advanced focus variation microscope InfiniteFocus® IF G4 (Alicona Imaging GmbH, Graz, Austria) is presented in Fig. 2b-c.

Monolayer abrasive discs containing structured abrasives are often used in a wide range of modern processes such as grinding, lapping and polishing realized in aerospace, automotive and precision industry as well as in numerous applications in medical and optical sectors. Surface finishing by Trizact™ abrasive grains is carried out for conventional and hard-to-cut materials (stainless steels, nickel and cobalt alloys), nonferrous metals (copper, bronze and aluminium) composite materials, hard ceramic materials (silicon carbide) as well as brittle materials (optical glass (Borofloat™), glass ceramics (Zerodur™). An overview of some selected applications of the abrasive tools with Trizact™ abrasive grains is given in the Table 1.

2. EXPERIMENTAL STUDIES

2.1. The main goals of the studies

The works were divided into two phases. In Phase I, the authors focused on observation and visual analysis of the abrasive tool active surface, where the mainly a various types of defects of abrasive grains (i.e. those being the remainder of the production process of Triazact™) were sought after. A visual analysis in this phase of the experimental works has been extended to include parametric analysis carried out in next phase (Phase II). The main goal of this analysis was to determine the space configuration and the values of the basic geometrical parameters characterizing the abrasive grains in the selected area of the active surface of the assessed abrasive tool.

2.2. Characteristic of the sample

A set of four abrasive discs in the pre-machining state with an external diameter of $d = 75$ mm, with the Trizact™ abrasive grains were selected for the experimental studies. Their general characteristics are given in Table 2.

Tab. 1.Examples of selected applications the abrasive tools with Trizact™ abrasive grains in modern science and technology areas

Process	Element(s)	Material(s)	References
Grinding	MIM workpiece	316L stainless steel	Yang and Tsai [10]
	Optical elements	Phosphate glass	Marino et al. [13]
		Borosilicate glass	Johnson at al. [20]
	Display glass for mobile electronics	Gorilla™ glass, soda-lime glass	Na and Zheng [17], Zheng et al. [18], Zheng and Na [19]
Lapping	Optical elements (brittle optical substrates)	Borofloat™, BK7, Pyrex™, window glass	Fletcher et al. [4, 8]
		Borofloat™, BK7, quartz	Cho et al. [11], Kim et al. [12]
Polishing	Large hydro-power spherical valve	309L stainless steel	Hazel et al. [9]
	Flat plate	316L stainless steel	Goossens et al. [6]
	TSCA-E components	Ti-6-4 heat-resistant alloy	Axinte et al. [15, 16]
	Optical elements (large aperture mirrors)	Borofloat™	Johnson et al. [14]

Tab. 2.General characteristics of abrasive tools with Trizact™ abrasive grains used in experimental studies

Sample No.	Grade		
	3M $^{1)}$	FEPA $^{2)}$	
1	A6	P2000	Producer: 3M Designation: 237AA Tool type: abrasive disc Backing type: semi-flexible Use: dry Pressure: light and medium
2	A30	P600	
3	A65	P280	
4	A160	P120	

$^{1)}$ due to the unique construction of Trizact™ abrasive tools, the producer has developed an original grading system. Grade is defined by the average particle size given in µm, and begins with an A, $^{2)}$ grain size determined by the Federation of European Producers of Abrasives (FEPA)

2.3. Observation-measurement instrument

An assessment of the active surface of abrasive tools is generally difficult and it does not always bring the expected results. The obtained results are not always reliable, which makes it difficult or even impossible to correctly interpret them. The use of conventional contact (stylus) measurement techniques [21] in this case is limited, in principle, to non-contact methods, whereby optical and electron methods (such as, e. g. focus-variation microscopy [22], confocal laser scanning microscopy [23] and scanning electron microscopy [24]) play a significant role.

During the experimental studies, an advanced scanning electron microscope Quanta 200 Mark II produced by FEI Company (Hillsboro, OR, USA) was used. The general characteristic of this instrument was given in Table 3, a general view in Fig. 3, whereas some of its applications were presented by Borkowski et al. and Kapłonek et al. in the works [25, 26].

Tab. 3.General characteristics of scanning electron microscope used in experimental studies

Instrument type	Model	Producer
SEM	Quanta 200 Mark II	FEI Company, (Hillsboro, OR, USA)
Configuration and features		
Components: detectors: SEI (Everhadt-Thornley SED, low-vacuum SED (LFD), gaseous SED (GSED)), BEI (Solid-state (BSED), Gaseous SED (GSED)) , specimen stage: eucentric goniometer stage (4-axis motorized) Features: magnification range: 30– ~1,000,000×, vacuum pressure in the specimen chamber: < 0.0006 Pa (HVM), 10–130 Pa (LVM), accelerating voltage = 0.2–30 kV, resolution (using HVM): 3.0 nm at 30 kV SEI, 4.0 nm at 30 kV BSE, 10 nm at 3 kV SEI, (using LVM): 3.0 nm at 30 kV SEI, 4.0 nm at 30 kV BSE, < 12 nm at 3 kV SEI Software: Dedicated FEI software		

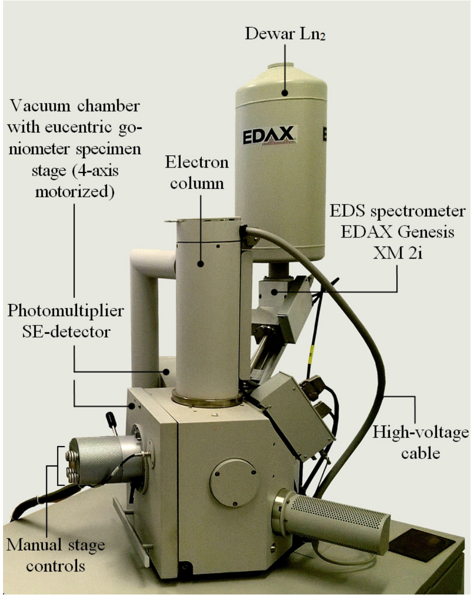


Fig. 3. General view and the main parts of high-resolution scanning electron microscope Quanta 200 Mark II produced by FEI Company used in the experimental studies

The microscopic observations were additionally supported by image processing and an analysis of the acquired SEM micrographs. In this case, open source Fiji 1.51s (Fiji = Fiji is Just ImageJ) (J. Schindelin et al.) software was used. This program is an advanced version of the well-known non-commercial Java-based image processing and analysis ImageJ software extended by a number of bundled plugins. Fiji was widely described by Schindelin et al. in the work [27].

3. RESULTS AND DISCUSSION

3.1. Visual analysis of the SEM micrographs

In Phase I of the carried out experiments, which focused on an observation and a visual analysis of the abrasive tool active surface, generally magnifications from 50× to 1000× were used. The observations of the selected areas of the active surface of the abrasive tool, containing usually several or a dozen of abrasive grains, were carried out at magnifications 50×–100×, whereas the details of the morphology of single abrasive grains were observed at magnifications from 300×–500×. In special cases (details of the technological defects), magnification up to a max.1000× was used. Representative examples of the selected SEM micrographs presenting Trizact™ abrasive grains with and without visible technological defects are given in Fig. 4. The properly produced single abrasive grains are shown in Fig. 4a. Side walls of the pyramids are smooth with locally occurring fine impurities. The pyramid's cone is bevelled, which does not affect the efficiency of machining. A different defect is presented in Fig. 4b. During the forming of the abrasive grain, air bubbles accumulate on its side walls. The released air leaves cavities visible on the shown SEM micrographs. Cavities can occur singly or in groups, even in relatively large areas of side walls surfaces of the Trizact™ abrasive grains. The average values of the selected geometrical parameters determined for one of such groups (Fig. 4b – B) are as follows: $An = 0.12$ mm^2, $P = 0.03$ mm, $l = 0.01$ mm, $w = 0.007$ mm, $F_{min.} = 0.007$ mm, $F_{max.} = 0.01$ mm). A single small abrasive agglomerate sometimes can also occur on the side walls. Such a situation is presented in Fig. 4c. The average values of the geometrical parameters of an example group of such agglomerates (Fig. 4c – B) are as follows: $An = 0.24$ mm^2, $P = 0.02$ mm, $l = 0.01$ mm, $w = 0.007$ mm, $F_{min.} = 0.007$ mm, $F_{max.} = 0.01$ mm). Other defects in a form of dented side walls and worn edges of the side walls are shown in Fig. 4d. The above mentioned defects may occur on the surface of wall grains in various combinations (i.e. on the grains there may be defects of one type or their combination). Nevertheless, the described defects do not have any significant impact on the efficiency of the machining process, and their occurrence is caused by the specific process of producing the abrasive tool.

Fig. 4. Collection of selected SEM micrographs presenting a typical technological defects of the Trizact™ abrasive grains: a) properly produced abrasive grains without visible technological defects; b) abrasive grains with visible technological defects (group of air bubbles remnants); c) abrasive grains with visible technological defects (single abrasive agglomerates on the side walls); d) abrasive grains with visible technological defects (dented side walls and worn edges of the side walls)

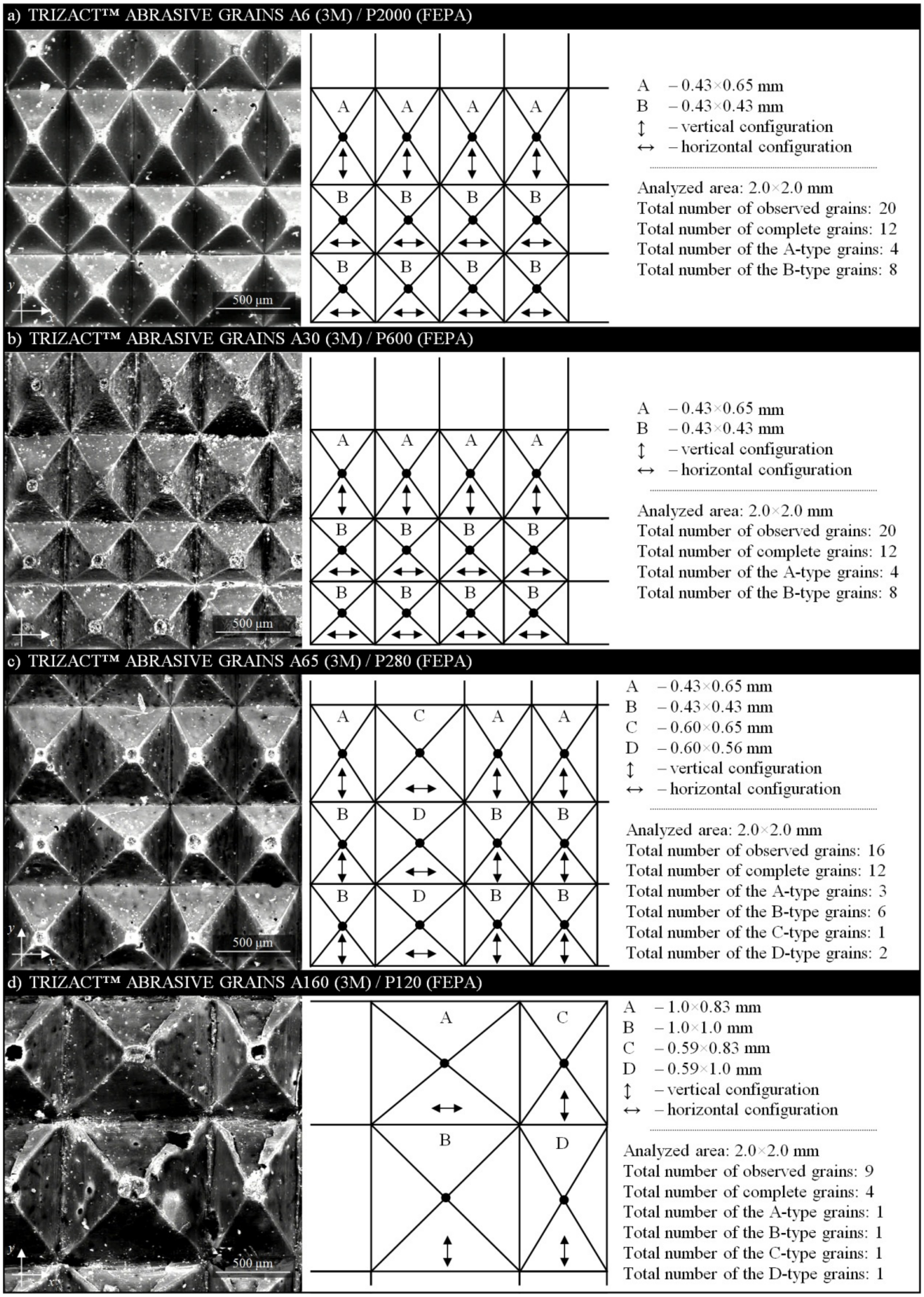
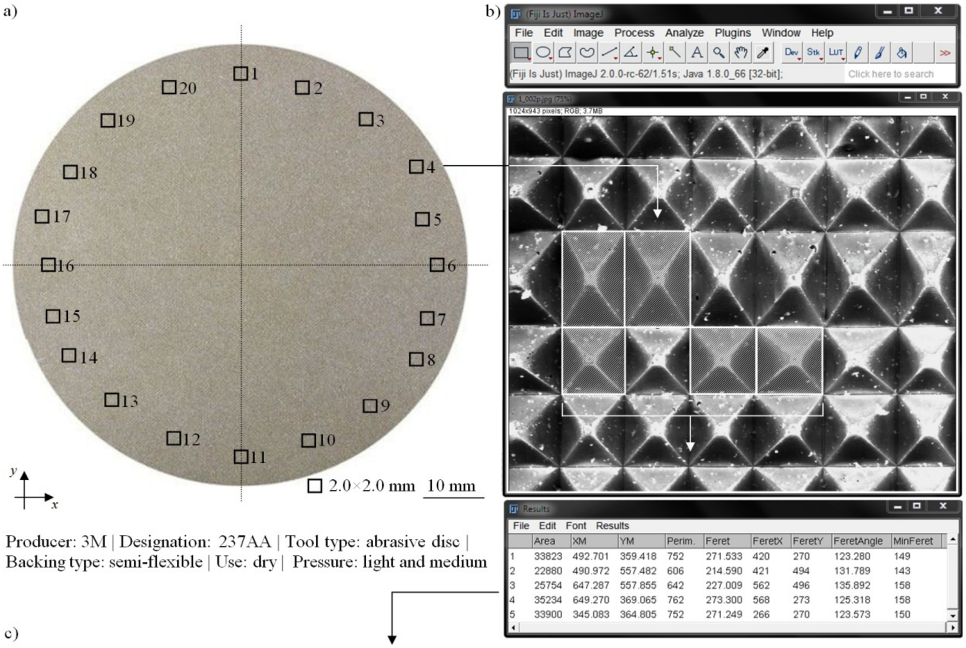


Fig. 5. Collection of selected SEM micrographs presenting the Trizact™ abrasive grains located on the active surfaces of abrasive discs with corresponding its space configurations (vertical or horizontal) as well as the values of selected geometrical parameters calculated by Fiji 1.51s software for: a) Sample 1 (Area 11); b) Sample 2 (Area 5); c) Sample 3 (Area 18); d) Sample 4 (Area 15)



Producer: 3M | Designation: 237AA | Tool type: abrasive disc | Backing type: semi-flexible | Use: dry | Pressure: light and medium

(Fiji Is Just) ImageJ 2.0.0-rc-62/1.51s; Java 1.8.0_66 [32-bit];

```
   Area   XM       YM       Perim.  Feret    FeretX  FeretY  FeretAngle  MinFeret
1  33823  492.701  359.418  752     271.533  420     270     123.280     149
2  22880  490.972  557.482  606     214.590  421     494     131.789     143
3  25754  647.287  557.855  642     227.009  562     496     135.892     158
4  35234  649.270  369.065  762     273.300  568     273     125.318     158
5  33900  345.083  364.805  752     271.249  266     270     123.573     150
```

c)

Designation	Abrasive grain type	Parameter	l, mm	w, mm	Ar	F_{min}, mm	$F_{max.}$, mm	An, mm^2	P, mm
A6 (3M) / P2000 (FEPA)	A	Average	0.430	0.653	1:1.5	0.429	0.776	96.356	2.150
		Std. dev.	0.013	0.041		0.202	0.200	1.667	0.764
	B	Average	0.436	0.437	1:1	0.426	0.627	68.274	1.775
		Std. dev.	0.039	0.046		0.216	0.253	1.870	0.160
A30 (3M) / P600 (FEPA)	A	Average	0.439	0.656	1:1.5	0.429	0.775	95.672	2.143
		Std. dev.	0.031	0.046		0.142	0.137	3.347	0.279
	B	Average	0.430	0.439	1:1	0.429	0.627	67.909	1.775
		Std. dev.	0.041	0.043		0.103	0.064	4.431	0.394
A65 (3M) / P280 (FEPA)	A	Average	0.671	0.513	1:1.3	0.513	0.845	119.378	2.370
		Std. dev.	0.074	0.105		0.131	0.243	2.487	0.290
	B	Average	0.554	0.496	1:1.1	0.496	0.743	95.337	2.102
		Std. dev.	0.329	0.307		0.298	0.268	7.221	0.571
	C	Average	0.671	0.542	1:1.2	0.598	0.898	138.937	2.549
		Std. dev.	0.191	0.178		0.209	0.288	3.855	0.145
	D	Average	0.589	0.548	1:1.2	0.548	0.805	112.028	2.277
		Std. dev.	0.296	0.362		0.361	0.226	6.870	0.489
A160 (3M) / P120 (FEPA)	A	Average	1.010	0.844	1:1.2	0.843	1.373	293.783	3.709
		Std. dev.	0.430	0.532		0.532	0.362	34.006	0.653
	B	Average	1.041	0.989	1:1.1	0.986	1.431	353.290	4.058
		Std. dev.	0.095	0.048		0.314	0.107	37.926	0.485
	C	Average	0.832	0.619	1:1.3	0.619	1.035	117.842	2.901
		Std. dev.	0.073	0.018		0.018	0.017	0.457	0.412
	D	Average	0.989	0.613	1:1.6	0.613	1.163	209.43	3.204
		Std. dev.	0.142	0.053		0.053	0.085	1.280	0.232

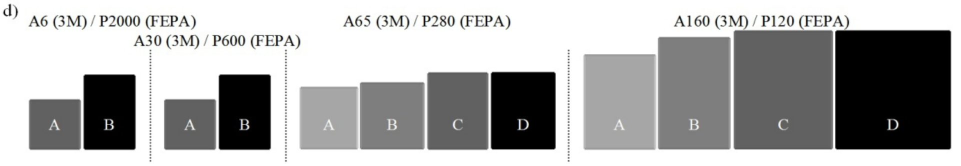


Fig. 6. Parametric analysis of the SEM micrographs of the active surfaces of abrasive discs with Trizact™ abrasive grains: a) disc divided into 20 areas for which analysis were carried out; b) windows of the Fiji 1.51s software during the geometrical analysis of the abrasive grains; c) results obtained of the analyses; d) grain types (criterion: geometrical dimensions) occurring on active surfaces of given analysed abrasive disc

3.2. Parametric analysis of the SEM micrographs

After making the general characteristics of the morphology of Trizact ™ abrasive grains (observation and comparison of various sizes grains and an analysis of defects occurring on their surfaces (Fig. 4)), the Phase II of experimental studies was started. This phase included a parametric analysis related to the determination of the basic geometrical parameters of abrasive grains located on the active surface of a given sample (abrasive disc). Visual presentations of this phase of experimental studies are presented in Fig. 5 and 6.

On each of the surface of the samples (Fig. 6a) twenty areas were marked (size: 2.0×2.0 mm, located evenly on the circumference of the abrasive tool) for which SEM micrographs were acquired. The acquisition process was realized for all the surfaces with the same parameters (magnification: 100×, accelerating voltage: 15 kV, sig-nal: LFD) by the use of a scanning electron microscope Quanta 200 Mark II produced by FEI Company. The processing of the obtained SEM micrographs and a geo-metrical analysis of the contained therein abrasive grains was carried out with Fiji 1.51s software (Fig. 6b).

The obtained results of the above-mentioned process for selected areas are presented in Fig. 5. A set of results for abrasive grains A6 (3M) / P2000 (FEPA) is given in Fig. 5a. At the analyzed field of view (FOV) (Area 11) twenty grains are located, wherein twelve of them are complete. These grains are represent by two types:

- A (form: rectangle, size: 0.43×0.65 mm configuration: vertical),
- B (form: square, size: 0.43×0.43 mm, configuration: horizontal).

Although A-type abrasive grains are larger, there are fewer of them (four), than B-type grains (eight) in the analyzed area. The same types of grains in the same configurations and geometrical dimensions are on the active surface of the A6 (3M) / P2000 (FEPA) sample in analysed Area 6 (Fig. 5b). Four types of abrasive grains can be observed on the active surfaces of the abrasive discs – A65 (3M) / P280 (FEPA) and A160 (3M) / P120 (FEPA). The following grain types can be represented on the first of these discs, in the analyzed Area 18 (Fig. 5c):

- A (form: rectangle, size: 0.43×0.65 mm configuration: vertical),
- B (form: square, size: 0.43×0.43 mm, configuration: vertical),
- C (form: rectangle, size: 0.60×0.65 mm configuration: horizontal),
- D (form: rectangle, size: 0.60×0.56 mm, configuration: horizontal).

The total number of abrasive grains observed in the FOV is sixteen, wherein twelve of them are complete. Individual grain types occur in the following number: A-type grains: three, B-type grains: six, C-type grains: one and D-type grains: two. The most interesting of the observed active surface is the one containing A160 (3M) / P120 (FEPA) abrasive grains. There are only nine of them in the FOV, of which only four are complete. These grains (Fig. 5d) represent four types:

- A (form: rectangle, size: 1.0×0.83 mm configuration: horizontal),
- B (form: square, size: 1.0×1.0 mm, configuration: vertical),
- C (form: rectangle, size: 0.59×0.83 mm configuration: vertical),
- D (form: rectangle, size: 0.59×1.0 mm, configuration: vertical).

An example of SEM micrographs acquired for selected areas of active surface of the abrasive discs with given abrasive grains and values of its basic geometrical parameters is presented in Fig. 5. It has been extended by the values of other geometrical parameters calculated for all of the analysed areas and given in an averaged form. This set of parameters included: An, P, $F_{min.}$, $F_{max.}$, w, l and Ar. The obtained results are given in Fig. 6c and they are substantially at a similar level to the values shown in Fig. 5. The aspect ratio given in Fig. 6c, which is defined as the quotient of the length and width of the measured abrasive grain, indicates a large dimensional diversity of the analyzed abrasive grains, and hence their different shape – square (1:1) and rectangle (1:1.1-1:1.6). These differences, for all the analyzed grain types, are shown graphically in Fig. 6d. The geometrical dimensions also determine other parameters, such as the surface area An and the perimeter P. The values of the first parameter are in the range from 67.90 (A6 (3M) / P2000 (FEPA)) to 353.29 mm^2 (A160 (3M) / P120 (FEPA)), while the second from 1.77 to 4.05 mm, which give an increase of more than five and more than two times for the listed above abrasive grains, respectively.

4. CONCLUSIONS

On the basis of the imaging and analysis of the morphology of the Trizact™ advanced structured abrasives by the use of SEM technique presented in this paper, the following detailed conclusions can be drawn:

1. Produced by the use of microreplication technology a Trizact™ abrasive grains are characterized by a different morphology than conventional abrasive grains. Their structure is regular (in the form of tetragonal pyramids with a square or rectangular base) same as their arrangement on the surface of the abrasive tool (Fig. 2).

2. The specific production technology results in the occurring various, typical for this abrasive grains class, defects – dented side walls and worn edges of the side walls, etc. (Fig. 4). Despite their occurrence, they do not have a significant influence on the course and efficiency of the machining process realized by the abrasive tool.
3. Depending on the grade (Tab. 2), Trizact™ abrasive grains have different geometric dimensions, form (square, rectangle) and they are located in different configurations (horizontal and vertical) on active surface of the given abrasive tool (Fig. 5). The number of a full grains observed in FOV (2.0×2.0 mm for all the analyzed SEM micrographs) decreases with increasing grade from 12 (A6 (3M) / P2000 (FEPA)) to 4 (A160 (3M) / P120 (FEPA)).
4. An extremely useful observation technique in the issues related with assessment of the morphology of Trizact™ abrasive grains is SEM microscopy. This technique allows an observation of grain surfaces in a wide range of magnifications (in the work the magnification in a range from 50× to 1000× was used) depending on the expected effects – general analysis of active surface of the given abrasive tool, an analysis of the morphology details of individual abrasive grains.
5. An important aspect of the carried out experimental studies was the extension of the SEM observation by acquisition (realized by advanced scanning electron microscope Quanta 200 Mark II produced by FEI Company) and then the processing and parametric analysis of the SEM micrographs presenting the active surfaces of the abrasive tools. The tasks related to the image analysis are usually carried out by specialized computer software. In this work, a non-commercial Java-based ImageJ 1.51s software was successfully used (Fig. 6b).
6. The authors intend to continue work from this area in the future, including an assessment of Trizact abrasive grains morphology after the machining process, in particular conventional and non-conventional materials made of metals and their alloys.

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Nomenclature

Symbols

An – surface area, mm^2
Ar – aspect ratio, –
P – perimeter, mm
l – length, mm
w – width, mm
$F_{min.}$ – minimal value of the Feret diameter, mm
$F_{max.}$ – maximal value of the Feret diameter, mm

Acronyms

FEPA – Federation of European Producers of Abrasives
FOV – Field of View
HVM – High-Vacuum Mode
LVM – Low-Vacuum Mode
MIM – Metal Powder Injection Molding
SEM – Scanning Electron Microscope(y)
TSCA-E – Targeted Safety Critical Aero-Engine

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