



Tomasz Durejko, Michał Ziętała, Magdalena Łazińska*

Military University of Technology, Department of Chemistry and New Technologies, Department of Advanced Materials and Technologies
ul. Kaliskiego 2, 00-908 Warsaw, Poland

* Corresponding author. E-mail: mlazinska@wat.edu.pl

Otrzymano (Received) 02.03.2011

ANALYSIS OF SURFACE LAYER OF Fe40Al SINTERED WITH ADDITION OF NANO- Al_2O_3 OXIDE CERAMICS AFTER ELECTRICAL DISCHARGE MACHINING

The influence of the parameters of wire electrical discharge machining on the surface layer of Fe40Al based sinters with Al_2O_3 nanoceramics has been studied. The properties of the sinters surface layer were controlled by electric discharge machining roughing and finishing with 0.25 mm wire, where the operating conditions were: time of interval (t_p) and amplitude of current (I_A). On the basis of the load capacity curve, parameters describing the investigated sinters against abrasive wear were estimated. Changes in the materials microstructure in the sinters surface layer were also defined. Analysis of the sinters surface texture (ST) exhibited a slight influence of the kind of treatment (roughing, finishing). The smallest values of roughness were obtained for a current value of $I_A = 40$ A for 100 μs long intervals, after finishing treatment ($Ra = 1.46$ μm) and roughing treatment ($Ra = 1.68$ μm). The treatment parameters influence the condition of the treated surface. The parameters of the load capacity curve are also influenced by the discharge machining technological parameters. Final treatment improves theoretical abrasive wear (Rpk). The Rpk parameter values decrease with an I_A current decrease. The best resistance to abrasive wear ($Rpk = 2.4$ μm) was found for sinters after finishing treatment with a 100 μs long interval (t_p) and current of $I_A = 40$ A. The sinters microstructure after discharge machining does not exhibit significant changes. The width of the layer exhibiting changes is just a few micrometers and is independent of the operating parameters.

Keywords: FeAl with nano- Al_2O_3 composite sinters, wire electrical discharge machining (WEDM), surface texture (ST)

ANALIZA STANU WARSTWY WIERZCHNIEJ SPIEKÓW Fe40Al Z DODATKIEM NANOCERAMIKI TLENKOWEJ Al_2O_3 PO OBRÓBCE ELEKTROEROZYJNEJ

W pracy przeanalizowano wpływ parametrów obróbki elektroerozyjnej na stan warstwy wierzchniej spieków na osnowie fazy Fe40Al domieszkowanych nanoceramiką Al_2O_3 . Strukturę geometryczną powierzchni (SGP) uzyskanych spieków kształtowano za pomocą zgrubnej i wykańczającej obróbki elektroerozyjnej drutem o średnicy 0,25 mm, sterując czasem przerwy t_p oraz amplitudą natężenia prądu I_A . Na podstawie krzywej nośności wyznaczono parametry charakteryzujące badane spieki pod względem odporności na zużycie ściernie. W celu określenia zmian strukturalnych warstwy wierzchniej po cięciu elektroerozyjnym przeprowadzono badania mikroskopowe przygotowanych zglądów metalograficznych. Analiza SGP spieków wykazała nieznaczny wpływ rodzaju zastosowanej obróbki (zgrubna, wykańczająca). Najmniejszą wartość chropowatości powierzchni po obróbce wykańczającej ($Ra = 1,46$ μm) w porównaniu do powierzchni po obróbce zgrubnej ($Ra = 1,68$ μm) uzyskano dla natężenia prądu $I_A = 40$ A z czasem trwania przerwy między impulsami t_p równym 100 μs . Wraz ze wzrostem natężenia prądu I_A odnotowano wzrost chropowatości powierzchni. Zredukowane parametry nośności zmieniają się w zależności od przyjętych parametrów obróbki elektroerozyjnej. Przeprowadzenie obróbki wykańczającej poprawia odporność powierzchni na zużycie ściernie (Rpk). Wartość parametru Rpk maleje wraz ze zmniejszeniem natężenia prądu I_A . Największą odporność powierzchni na zużycie ściernie ($Rpk = 2,4$ μm) odnotowano dla spieku po obróbce wykańczającej z czasem przerwy $t_p = 100$ μs i natężeniem prądu $I_A = 40$ A. Mikrostruktura spieków po obróbce elektroerozyjnej nie wykazuje znaczących zmian. Niezależnie od parametrów obróbki szerokość zmienionej warstwy wynosi kilka mikrometrów.

Słowa kluczowe: spieki kompozytowe FeAl z nano- Al_2O_3 , obróbka elektroerozyjna, struktura geometryczna powierzchni (SGP)

INTRODUCTION

Dynamic development in the energetic, automotive and aviator industries fosters material design/development with various assumed properties, designated for exploitation in a given environment. New alloys are being researched by engineers to face high

requirements of exploitation in aggressive, high-temperature and corrosive environments. Recently, intensive research in the field of heat-resistance materials based on intermetallic alloys have been done. These alloys may be a great opportunity to substitute classical

heat-resistant materials, being exploited at a high temperature, close to the melting point and simultaneously facing increasing requirements stated by construction engineers. Intermetallic alloys from the Fe-Al phase diagram can be included in this group of alloys. They can be obtained inter alia by powder metallurgy of technically pure iron and aluminum powders [1]. Doping with alumina particles is significant while the material is being exploited at a high temperature [2, 3]. Controlled fragmentation of the structure and strengthening with alumina ceramics particles allow one to control the properties of FeAl alloys, especially in the field of high temperature exploitation (high level of ductility, high resistance towards metals creeping etc.). The presence of oxide ceramics (high abrasive wear resistance), abnormal increase of yield strength with temperature increase and lower cohesive strength in the grain boundaries than for AS cast materials, these alloys are classified as hard to machine materials [1, 4, 5].

Research being conducted in the Department of Advanced Materials and Technologies of MUT reveals that a suitable solution of the FeAl intermetallic alloys shaping problem is the application of electrical discharge machining with a numerically controlled wire cutter (WEDM) [4]. This technology allows one to obtain elements with complex geometry and with a precisely shaped surface texture (ST) thanks to the optimization of treatment energetic parameters (amplitude, impulse duration, interval duration) [4, 6]. Treatment parameter optimization allows one to control surface layer geometry, defining its exploitation features. In recent work, the results of ST analysis of the sinters after electrical discharge machining roughing and finishing depending on the operating parameters have been presented. Optimization of the finishing treatment of Fe40Al intermetallic alloys with nano-Al₂O₃ ceramics have been done. Furthermore theoretical assessment of the abrasive wear based on Abbott-Fireston load capacity curve analysis was done [7-9].

MATERIALS AND INVESTIGATION METHODS

The investigated material was obtained by means of a three-step technological process. The starting batch material was a mixture of technically pure aluminum and composite Fe-nAl₂O₃ powder (2% vol. addition of nano Al₂O₃ ceramic, obtained by low-energetic milling in a Fritsch ball-mill with, a velocity of 200 rpm and milling time 20 minutes [10]) in 60 to 40% at. iron to aluminum ratio. The mixture was homogenized in a turbulent mixer. The obtained powder mixtures were pressed at ambient temperature at a pressure of 300 MPa. The cylindrical sample was 20 mm in diameter and 4 mm in height. They were initially sintered at low pressure (10⁻²mBar) at 1050°C under 40 MPa. Consolidation and initial sintering were done using an induction heating apparatus (ELBIT). To homogenize the structure, the obtained samples were heated at 1200°C for 1 hour in an argon atmosphere.

After a full cycle of heat treatment, the sinters were machined with an electric discharge device with a numerically controlled wire cutter BP-97d. During cutting (perpendicular to pressing direction) a brass wire of 0.25 mm in diameter and durability of 500 MPa was used. The operating conditions were controlled by the duration of the interval between impulses t_p and current amplitude I_A (Table 1). For fixed values of the interval and current amplitude I_A , roughing treatment was done, mainly the Fe40Al sinter was cut and next finishing treatment, consisting of cutting and four sparking steps was done (without treated surface penetration).

TABLE 1. Parameters of wire electric discharge machining (WEDM) of Fe40Al + n-Al₂O₃ sinters
TABELA 1. Parametry cięcia elektroerozyjnego spieków Fe40Al + n-Al₂O₃

Treatment option	Kind of treatment	I_A [A]	t_p [μ s]
1-z	roughing	80	100
1-w	finishing, - 4 steps	80	100
2-z	roughing	56	100
2-w	finishing, - 4 steps	56	100
3-z	roughing	40	100
3-w	finishing - 4 steps	40	100

The metallographic experiments (quantitative) and point chemical composition analysis (EDS) were done by using a PHILIPS XL30/LaB₆ electron microscope. The sinters surface was observed by means of a BSE detector immediately after electrical discharge cutting. The samples were embedded in thermosetting conductive resin and then polished with a STRUERS PLANPOL 3 on a disc, by using diamond suspension.

To assess the influence of the electrical discharge cutting parameters on the materials strengthening near the surface layer area, linear micro-hardness measurements were done by means of the Vicker's method (SHIMADZU M micro-hardness measurements device).

The macro surface texture (ST) of the sinters after electrical discharge cutting was done by utilizing a PGM-1C profilometer. The amplitude parameters, including Ra and Rz were estimated on the basis of the obtained roughness profile. Three-dimensional roughness profiles were recorded for the investigated samples. The Abbott-Firestone load capacity curves were plotted, on the basis on the investigated surface roughness profile, to assess quantitatively the structures texture parameters (ST), crucial to abrasive wear [8].

RESULTS AND DISCUSSION

Structure analysis of the Fe40Al sinters with an addition of alumina nanoceramics after a full cycle of heat treatment was based on the microscopic observation and quantitative analysis of the chemical composition. Structural investigations of the sinters revealed chemical homogeneity of their matrix (Fe40Al phase) and

micro- and nano-metric oxide particles (Al_2O_3) distributed into the grain bulk and among the FeAl grain boundaries of the matrix (Fig. 1).

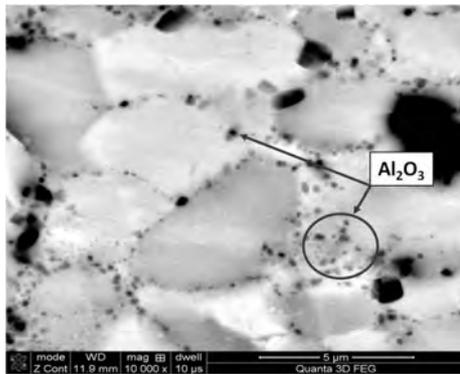


Fig. 1. SEM microstructure of Fe40Al sinter with addition of 2% vol. Al_2O_3 [10]

Rys. 1. Mikrostruktura SEM spieku Fe40Al z dodatkiem 2% obj. Al_2O_3 [10]

Surface analysis of the material admixed with ceramics after electrical discharge finishing treatment in the micro approach (BSE detector) exhibited its isotropy, independent of the treatment operating conditions (Fig. 2). Erosion energy is crucial to the size of appearing craters.

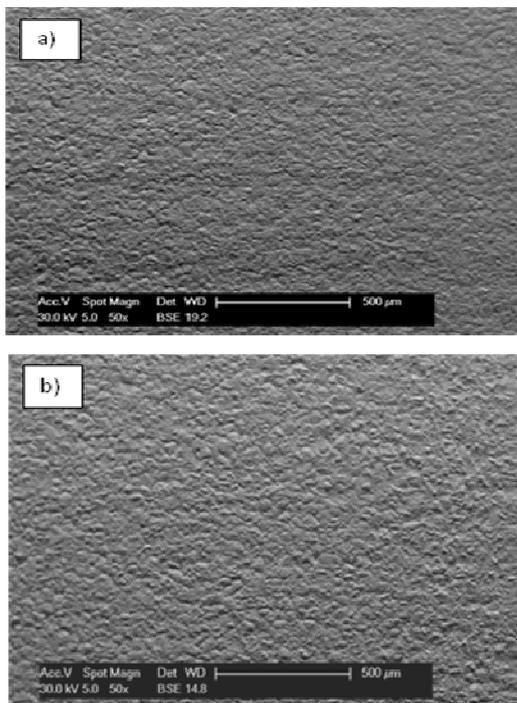


Fig. 2. Surface of sinters after WEDM finishing treatment: a) Fe40Al+2% vol. nAl_2O_3 ($I_A = 40$ A, $t_p = 100$ μs), b) Fe40Al+2% vol. nAl_2O_3 ($I_A = 80$ A, $t_p = 100$ μs)

Rys. 2. Powierzchnia spieków po wykańczającej obróbce elektroerozyjnej: a) Fe40Al+2% obj. nAl_2O_3 ($I_A = 40$ A, $t_p = 100$ μs), b) Fe40Al+2% obj. nAl_2O_3 ($I_A = 80$ A, $t_p = 100$ μs)

Melting and removal of matrix material (Fe40Al intermetallic alloy) occurs as a result of electric discharge

roughing treatment, but the ceramics is only “micro-cut” by the wire, filling bottom of the crater. During the sparking treatment, without material penetration by the wire, the remaining “sharp” peaks of craters are cut and the ceramics is a dielectric material, limiting the matrix melting process. The formation of new craters and deepening of the existing ones does not take place.

The obtained images of the surface layer exhibit negligible changes in the sinters with a nano-ceramics microstructure (Fig. 3).

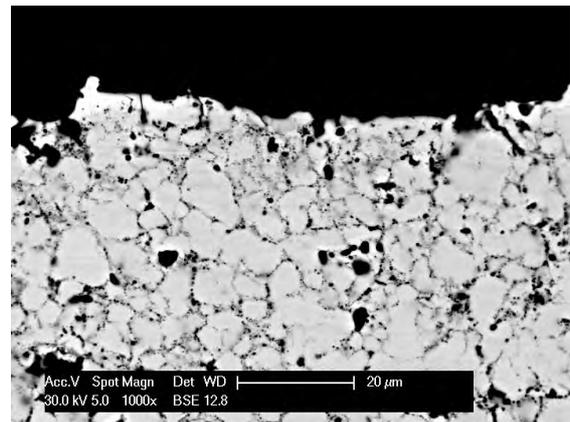


Fig. 3. SEM microstructure of sinter surface after wire electric discharge machining finishing (WEDM) ($I_A = 40$ A, $t_p = 100$ μs)

Rys. 3. Mikrostruktura SEM powierzchni spieków po obróbce elektroerozyjnej wykańczającej ($I_A = 40$ A, $t_p = 100$ μs)

An area with an almost white color was spotted. Independent of the treatment operating conditions, the thickness of the changed layer was just a few micrometers (Fig. 3) and comparable with all the treatment options.

Linear analysis of the chemical composition of the surface layer after electrical discharge cutting has shown that it differed from the intermetallic FeAl matrix. Increased participation of oxygen and aluminum, and a reduced iron content in the subsurface zone was noticed (Fig. 4).

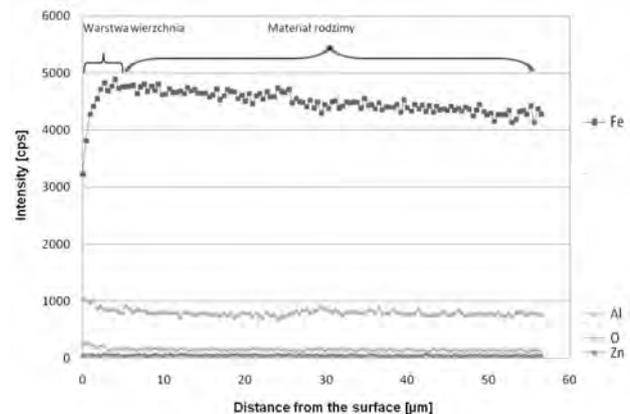


Fig. 4. Linear analysis of chemical composition of surface layer after WEDM ($I_A = 80$ A, $t_p = 100$ μs)

Rys. 4. Liniowa analiza składu chemicznego warstwy wierzchniej po obróbce elektroerozyjnej ($I_A = 80$ A, $t_p = 100$ μs)

The obtained roughness profiles for all the treatment options (Fig. 5) are in agreement with the surface images in the micro scale (Fig. 2). In the case when the sinter with an addition of ceramics was cut, a decrease of the current to the value of 40 A and application of a longer interval (100 μs) between pulses results in surface "smoothing" up to the $P_c = 4,02 \mu\text{m}$ level (Fig. 5a).

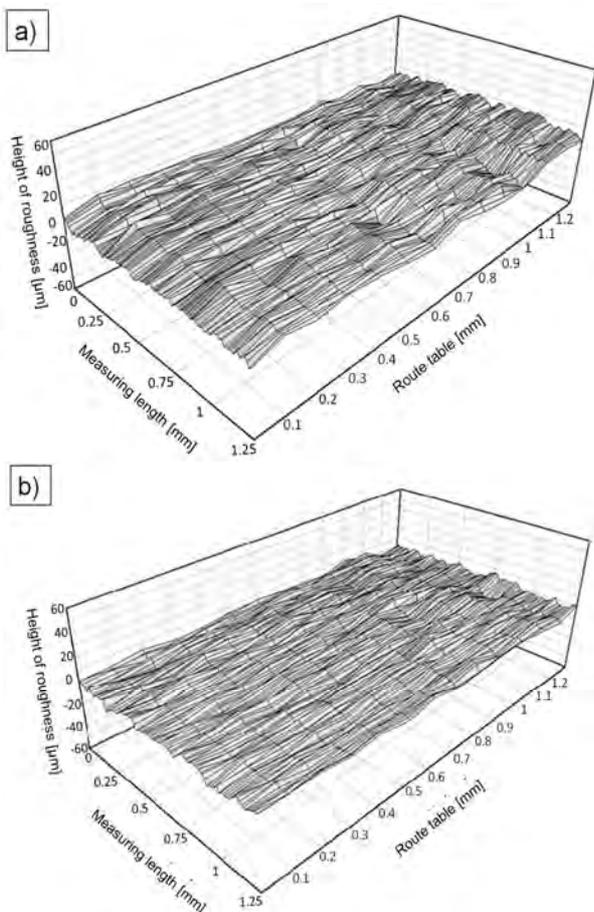


Fig. 5. 3D roughness profile of sinter surface after EDM finishing: a) Fe40Al+2% vol. nAl_2O_3 ($I_A = 40 \text{ A}$, $t_p = 100 \mu\text{s}$), b) Fe40Al+2% vol. nAl_2O_3 ($I_A = 80 \text{ A}$, $t_p = 100 \mu\text{s}$)

Rys. 5. Profil chropowatości 3D powierzchni spieków po wykańczającej obróbce elektroerozyjnej: a) Fe40Al+2% obj. nAl_2O_3 ($I_A = 40 \text{ A}$, $t_p = 100 \mu\text{s}$), b) Fe40Al+2% obj. nAl_2O_3 ($I_A = 80 \text{ A}$, $t_p = 100 \mu\text{s}$)

R_a , being the average arithmetic deviation of the profile roughness from the average value, was set as a representative parameter in this work. The basic values of the roughness parameters of the sinter surface with an addition of 2% of alumina nanoceramics after electric discharge cutting were set in Table 2. Surface texture analysis of this type of sinter after electrical discharge treatment shows a minor influence of the kind of treatment (roughing, finishing) on the R_a parameter. The smallest values of surface roughness after finishing treatment in comparison to roughing treatment were found for option 3 (Tab. 2) (R_a decrease was 13.1%). The surface roughness after WEDM finishing treatment with a current of $I_A = 40 \text{ A}$ and interval of $t_p = 100 \mu\text{s}$ (for option 3) was $R_a = 1.46 \mu\text{m}$.

TABLE 2. Main roughness parameters of investigated surface of sinters after wire electric discharge machining (WEDM)

TABELA 2. Podstawowe parametry chropowatości powierzchni spieku po obróbce elektroerozyjnej

Variant WEDM	Type of WEDM	I_A [A]	t_p [μs]	R_a [μm]	R_z [μm]
1-z	roughing	80	100	2.33	14.572
1-w	finishing	80	100	2.15	13.256
2-z	roughing	56	100	1.84	12.731
2-w	finishing	56	100	1.74	13.529
3-z	roughing	40	100	1.68	11.560
3-w	finishing	40	100	1.46	8.473

The load capacity curve of the sinters surface after electric discharge cutting was also investigated. The Abbott-Firestone curve represents the materials participation in the surface roughness as a function of the materials length functions of profiles set at a given level [8, 9]. Figure 6 shows typical load capacity curves for chosen contour lines of the 3D profile. The curves are S-shaped, which is typical for random distributed irregularities.

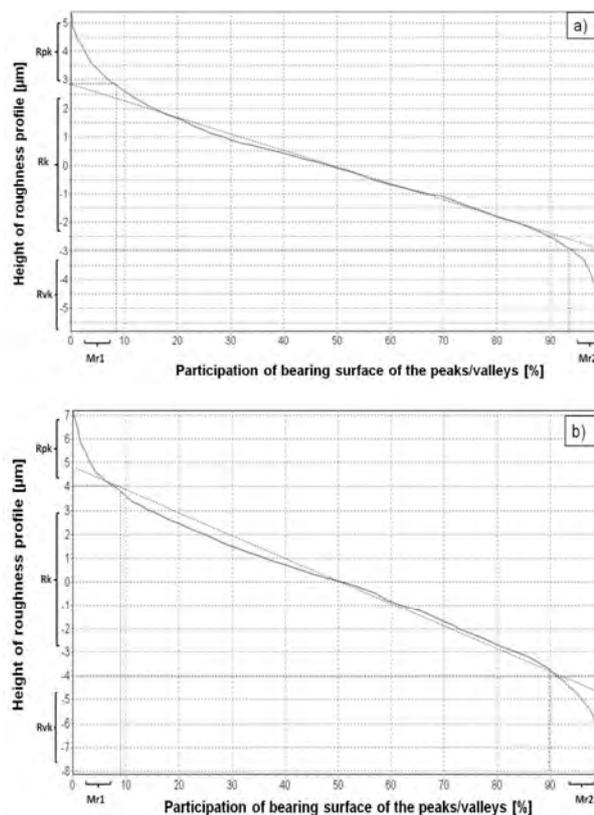


Fig. 6. Load capacity curves of sinters after EDM finishing: a) Fe40Al+2% vol. nAl_2O_3 ($I_A = 40 \text{ A}$, $t_p = 100 \mu\text{s}$), b) Fe40Al+2% vol. nAl_2O_3 ($I_A = 80 \text{ A}$, $t_p = 100 \mu\text{s}$)

Rys. 6. Krzywe nośności spieków po wykańczającej obróbce elektroerozyjnej: a) Fe40Al+2% obj. nAl_2O_3 ($I_A = 40 \text{ A}$, $t_p = 100 \mu\text{s}$), b) Fe40Al+2% obj. nAl_2O_3 ($I_A = 80 \text{ A}$, $t_p = 100 \mu\text{s}$)

On the basis of the Abbott-Firestone curves, the reduced height of the R_{pk} peaks and R_{vk} valleys, roughness height of the R_k core and load capacity participa-

tion peaks Mr1 and valleys Mr2 were estimated. The quantitative analysis of the curve was done in accordance to the ISO 13565 standard [11]. Reduced parameters describing the sinter surface with 2% vol. additives of Al₂O₃ nanoceramics changes, depending on the technological option of the electric discharge treatment (Tab. 3).

TABLE 3. Parameters of load capacity curve for surface of Fe40Al+nAl₂O₃ sinters after wire electric discharge machining roughing and finishing (EDM)

TABELA 3. Parametry krzywej nośności dla powierzchni spieków Fe40Al+nAl₂O₃, po zgrubnej i wykańczającej obróbce elektroerozyjnej

Variant WEDM	Parameters of load capacity curve					
	<i>Rpk</i> [μm]	<i>Rvk</i> [μm]	<i>Rk</i> [μm]	Mr1 [%]	Mr2 [%]	Mr2-Mr1
1-z	6.8	4.5	7.6	9	90	81
1-w	4.7	4.8	6.7	10.5	92	81.5
2-z	5.2	7.7	5.7	9.5	88.5	79
2-w	3.6	9.1	5.2	10	90	80
3-z	2.6	3.2	6	9	90	81
3-w	2.4	3.8	5.6	9	93	84

Reduced height of the *Rpk* peaks characterized this part of the surface which will be removed during the “lapping in” and initial period of the work. This parameter should be treated as the resistance towards abrasive wear of the investigated surface.

In the case of nAl₂O₃ doped material, the final treatment improves resistance towards abrasive wear of the surface, excluding the case with a current of $I_A = 40$ A and 100 μs long interval, where the *Rpk* parameter is constant, independent of the kind of treatment (Tab. 3). One should notice that *Rpk* is influenced by the energetic parameters of the treatment - a long with a reduction in current, the *Rpk* parameters decrease (Tab. 3). The smallest value of *Rpk*, equal to 2.4 μm was found for the surface after finishing treatment with a current density of $I_A = 40$ A and 100 μs long intervals. A lower value of *Rpk* means high resistance towards abrasive wear of the investigated surfaces.

The height of the core roughness of the profile, *Rk*, informs about the height of the surface irregularities during “lapping in”. The value of this parameter is independent of the height of the peaks and deepness of the valleys of the profile.

The influence of the operating parameters on the *Rk* for the investigated surfaces analysis, implies the statement that the highest load capacity after “lapping in” will be for the sinter with the addition of nano-ceramics after final electrical discharge treatment (option 2-w: $t_p = 100$ μs, $I_A = 56$ A) – $Rk = 5.2$ μm.

A decrease of the core roughness was observed where the current was decreased to 56 A and the *Rk* value was constant till 40 A. The kind of the treatment (roughing or finishing) does not influence the surface irregularities of the sinters after the “lapping in” period.

Differences between the shares of load capacities of the peaks Mr1 and valleys Mr2 for the surface of the sinter with the addition of nanoceramics are comparable, however, the treatment parameters influence on these parameters is negligible. Significant differences between the Mr1 and Mr2 parameters evidences a high load capacity of the sinters surface after the “lapping in” period.

The reduced depth of the *Rvk* valleys is a parameter describing the ability of the shape surface to maintain a lubricant on the surface. An increase of the *Rvk* parameter was noticed after final treatment with a current of 56 A and time of interval of $t_p = 100$ μs in comparison to the surface after roughing treatment with the same current parameters. The influence of the kind of treatment (roughing, finishing) on the *Rvk* parameter was not found in the case of treatment with a current of $I_A = 80$ A and $I_A = 40$ A. However, it was found that treatment with the smallest value of current ($I_A = 40$ A) results in the smallest value of *Rvk* parameter, $Rvk = 3.2$ μm. The highest ability to maintain a lubricant on the surface had the sinter surface doped with Al₂O₃ nanoceramics after finishing treatment with a current of $I_A = 56$ A and interval of $t_p = 100$ μs.

CONCLUSIONS

On the basis on the current and previous results [4] it can be stated that there is a possibility to shape Fe40Al sinters doped with alumina nanoceramics geometry by electric discharge machining. The kind of treatment and operating conditions are crucial for surface texture (ST), therefore are also crucial for abrasive wear resistance and lubricant maintenance. Doping with nanoceramics (dielectric, hard material) does not diminish the effectiveness of the electric discharge machining of Fe40Al based materials. What is more, it influences positively the quality of the obtained surface. Doping Fe40Al sinters with homogenously distributed alumina nanoceramics in bulk and grain boundaries successfully blocks and inhibits micro-crack propagation towards the materials bulk during machining.

Load capacity dependency on the machining energetic operating conditions is similar to the dependency observed for the *Ra* parameter. A different tendency was noticed for the reduced depth of cavities (*Rvk*). A correlation between surface roughness and load capacity parameters was also found. A roughness increase causes an increase of values like reduced height peaks, core roughness of the profile, although the highest value of the reduced depth of the valleys was found for an average value of roughness.

The best “smoothing” of the surface ($Ra = 1.46$ μm) of the sinters with 2% vol. addition of alumina nanoceramics was obtained after finishing treatment (four sparking steps) with application of the following parameters: $I_A = 40$ A and $t_p = 100$ μs. The highest value of abrasive wear resistance ($Rpk = 2.4$ μm) was noted

for the sinter surface after finishing treatment with an interval of $t_p=100 \mu\text{s}$ and current of $I_A = 40 \text{ A}$. It was noticed that material sintered with an addition of alumina nanoceramics after final electric discharge machining (operating conditions: $t_p = 100 \mu\text{s}$, $I_A = 56 \text{ A}$) has the highest load capacity of the surface during the “running in” period ($Rk = 5.2 \mu\text{m}$) and the highest ability of lubricant maintenance ($Rvk = 9.1 \mu\text{m}$).

Acknowledgements

The authors are grateful for financial support from Polish Ministry of Science and Higher Educations, Grant No: O N508 004434.

REFERENCES

- [1] Bojar Z., Przetakiewicz W., Metallic materials with intermetallic phases, Warszawa 2006.
- [2] Morris D.G., Possibilities for high-temperature strengthening in iron aluminides, Intermetallics 1998, 6, 753-758.
- [3] Garcia Oca C., Munoz-Morris M.A., Morris D.G., High temperature structural coarsening of an ODS FeAl, Intermetallic 2003, 11, 425-434.
- [4] Durejko T., Zrański Z., Sulej S., Constitution of surface layer of FeAl+Al₂O₃ sinters by electric discharge micro-machining, Kompozyty (Composites) 2008, 8, 2, 190-194.
- [5] Patejuk A., Durejko T., Influence of reinforcing phase on abrasive wear of FeAl-Al₂O₃ composites, Przegląd Mechaniczny 2005, 3.
- [6] Khanra A.K., Patra S., Godkhindi M.M., Electrical discharge machining studies on e reactive sintered FeAl, Bull. Mater. Sci. 2006, 29, 3.
- [7] Tomasik J., Biało D., Application of curve of carrier participation parameters to porous surfaces description, Mechanik 2003, 1, 26-28.
- [8] Oczóś K.E., Liubimov V., Surface geometry, Oficyna Wydawnicza Politechniki Rzeszowskiej, Rzeszów 2003.
- [9] Adamczak S., Surface geometry metrology. Outlines of shape, waviness and roughness, WNT, Warszawa 2008.
- [10] Strąg A., Structural stability of Fe40Al based sinters with addition of alumina nanoceramics, Masters' thesis WAT, Warszawa 2010.
- [11] ISO 13565-1: 1996 Geometrical product specification (GPS) - Surface texture: Profile method - Surface having stratified functional properties.