

Enhancing of ZA-27 alloy wear characteristics by addition of small amount of SiC nanoparticles and its optimisation applying Taguchi method

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Abstract

The objective of this work was to investigate the influence of the addition of a small amount of SiC nanoparticles on the mechanical characteristics and wear resistance of ZA-27 alloy. The ZA-27 alloy-based nanocomposites were produced by a relatively cheap compocasting process preceded by mechanical alloying. Reinforcing elements were the silicon carbide (SiC) nanoparticles with an average size lower than 50 nm and in very small amounts of 0.2, 0.3 and 0.5 wt. %. Wear tests were realized on a block-on-disc tribometer under lubricated sliding conditions, at two sliding speeds (0.25 and 1 m/s), two normal loads (40 and 100 N) and a sliding distance of 1000 m. Optimisation of the SiC amount was performed by applying the Taguchi method, showing that the SiC amount of 0.5 wt. % is optimal for the given testing conditions. Prediction of the results and wear maps were also conducted. The analysis of variance showed that the SiC amount has the greatest influence on wear rate (70.8 %), followed by the normal load (19.8 %), and the sliding speed (3.9 %), while the influences of all interactions between these factors did not have any significant influence.

1. Introduction

The ZA-27 alloy is one of the commercial zinc-aluminium alloys for foundry and die castings, with the highest amount of aluminium of all standardised alloys [1]. In comparison to ZA-8 and ZA-12 alloy, it has lower density and higher strength (high strength-to-weight ratio) [2]. The ZA-27 alloy is also successfully used as a matrix for metal matrix

composites (MMCs) in combination with different elements, such as SiC [3-6], Al₂O₃ [7-9], zircon, garnet, fly ash and graphite [10]. The results of these studies have shown that the mechanical characteristics and wear resistance of ZA-27 alloy can be significantly improved in this way. Recently, based on the positive results with MMCs, there is an increasing interest for researches on metal matrix nanocomposites (MMnCs) with ZA-27 alloy matrix and incorporated nanoparticles. Influences of various nanoparticles have been investigated, namely SiC [11-14], Al₂O₃ [11,12,15,16], ZnO [17],

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TiB₂ [18], B₄C [19,20], graphite [21], graphene [22], and its combinations, Al₂O₃ and graphite [23,24], B₄C and graphite [25] and B₄C and graphene [26].

Although the SiC particles are the mostly used reinforcement in MMCs, their application in MMnCs is surprisingly significantly lower. There are only a few research papers which investigate ZA-27 alloy-based nanocomposites with the addition of SiC nanoparticles. Hayajneh et al. [13] have produced the ZA-27 alloy-based nanocomposites through the stir casting technique and investigated their corrosion behaviour. The size of used SiC nanoparticles was between 45 and 60 nm and the amounts were 2, 3 and 4 wt. %. The first thing they noticed is that higher weight fractions (6 and 9 wt. %) are difficult to mix within the matrix due to the problems of the nanoparticles wettability. The nanocomposites retain the dendritic structure of the base alloy; however, the addition of SiC particles has resulted in a finer structure since the dispersed nanoparticles restricted the movement of grain boundaries and hinder grain growth. The uniform corrosion resistance of the nanocomposites has been improved over the base alloy, but the pitting corrosion has been more pronounced at nanocomposites.

Babic et al. [14] have investigated compocasted ZA-27 alloy-based nanocomposites reinforced with 1, 3 and 5 vol. % SiC (average particle size was 50 nm). They have noticed that the SiC nanoparticles tend to agglomerate and form clusters, causing local porosity of the nanocomposites. The addition of SiC nanoparticles has decreased the nanoindentation hardness, which has been lower for all nanocomposites in comparison to the base alloy, although the higher content of SiC nanoparticles has been followed by the increase in nanoindentation hardness. Tribological tests have been performed under dry sliding conditions in linear (reciprocating) sliding mode. The static counter-body was a 1.5 mm diameter alumina ball. The authors used very small normal loads (50, 100, 150 and 200 mN) and average sliding speeds (2, 4, 6 and 8 mm/s), which gave a short sliding distance of only 500 mm. Results have shown that at higher normal loads wear resistance has been improved significantly with the addition of 1 vol. % SiC nanoparticles, while a further increase of the SiC amount has resulted in a slight improvement of the wear resistance. On the other hand, the coefficient of friction of all materials has been similar and improvement over the base alloy has been noticed only when the lowest normal load and the lowest sliding have been applied.

The idea to use a very small amount of nanoparticles is relatively new, although it has been shown in the review by Rohatgi and Schultz [27] that for some MMnCs addition of a small percentage of nanoparticles can affect specific material properties. In our previous studies [11,12] we have analysed the microstructure, mechanical and erosive wear properties of the ZA-27 alloy-based nanocomposites reinforced with 0.2, 0.3 and 0.5 wt. % SiC nanoparticles (average size was less than 50 nm), which have been produced through the compocasting process. The addition of nanoparticles has produced a finer structure of the matrix and has improved the macrohardness and compressive yield strength through the enhanced dislocation density strengthening mechanism [11]. According to the results of the solid particle impingement (particles impact angle was 90°) erosion tests of the same materials [12], the presence of nanoparticles has led to a slight increase in erosive wear resistance due to the increase of ductility of nanocomposites. The aim of this paper was to investigate the influence of small addition of SiC nanoparticles on the wear resistance of ZA-27 alloy-based nanocomposites in lubricated sliding conditions.

2. Experimental details

2.1 Materials

The chemical composition of the matrix material, zinc-aluminium alloy ZA-27, was according to the ASTM standard [1]. The SiC nanoparticles (particle size < 50 nm) were added to the matrix in the following amounts: 0.2, 0.3, and 0.5 wt. %. The matrix material is obtained by semi-solid processing (thixocasting), while the compocasting process with mechanical alloying pre-processing (ball milling) was used for producing the nanocomposites. In ball milling, metal chips of the matrix alloy were mechanically alloyed with nanoparticle reinforcements. The process has been carried out in the air, at room temperature, with a rotational speed of 500 rpm, using alumina balls of 10 and 14 mm in diameter (with a 60:40 percentage ratio), for 60 minutes. The metal chips-to-nanoparticles weight ratio was 3:1, while the weight ratio alumina ball-to-milling mixture was 5:1 [12]. The apparatus used for the semi-solid processing of matrix alloy and nanocomposites is described elsewhere [28], as well as the production process parameters and a detailed description of the experimental procedure [11,12].

All nanocomposites and matrix material have microstructures that are similar and non-dendritic, while the matrix material contains larger α phase particles, indicating a more homogeneous structure of nanocomposite matrices. Furthermore, the size of η phase regions (rich in zinc) was reduced in all nanocomposites when compared to the matrix material. The maximum porosity and clustering of nanoparticles were noticed in nanocomposite with 0.5 wt. % of SiC nanoparticles.

2.2 Mechanical testing

Microhardness measurements were performed on plate-like samples ($16 \times 12 \times 6$ mm) using the Vickers hardness tester with 500 g load (HV 0.5) and dwell time of 15 s. For each sample, at least five measurements were performed to get the representative average values of microhardness.

Nanoindentation tests included hardness and modulus of elasticity measurements. These tests were carried out on Nanoindentation tester NHT² under the assumption that all tested materials have Poisson's ratio of 0.33. Each material was measured in nine positions, according to the prescribed scheme (Fig. 1a), and the average value is calculated. The shape of the individual indent (Fig. 1b) corresponded to the used Berkovich diamond indenter (modulus of elasticity 1141 GPa, Poisson's ratio 0.07). The tests were conducted under linear loading, with a maximum load of 50 mN and pause at a maximum load of 15 s. The loading and unloading rate was 100 mN/min, and the approach and retract speed was 1500 nm/min.

2.3 Wear testing

Experimental research of the wear characteristics was performed by using the same plate-like samples that were used for microhardness measurements. Wear behaviour was determined under lubricated sliding conditions, on a tribometer with a block-on-disc contact geometry (line contact of 6 mm). Lubrication was provided by mineral gear oil (ISO VG 220, ISO L-CKC/CKD) through the rotation of the disc which was sunk into the oil container.

The blocks were produced from tested nanocomposites and matrix material, while the discs were made of hardened and tempered steel 42CrMo4 (51–54 HRC). Approximate values of surface roughness (R_a) were 0.14–0.17 μm (blocks) and 0.6–0.7 μm (discs). The tests were conducted over a 1000 m sliding distance at sliding

speeds of 0.25 and 1 m/s and normal loads of 40 and 100 N. Lubricant temperature was continuously monitored during each test and the values at the end of the test were averaged. Since it did not differ too much, the approximate steady-state value of lubricant temperature of 30 °C could be adopted for all tests. The wear scars on the blocks were measured after each test to calculate the wear volume and to compute the wear rate. They were also used to calculate the average pressure on contact surfaces at the end of the test, which in some cases went up to 21 MPa.

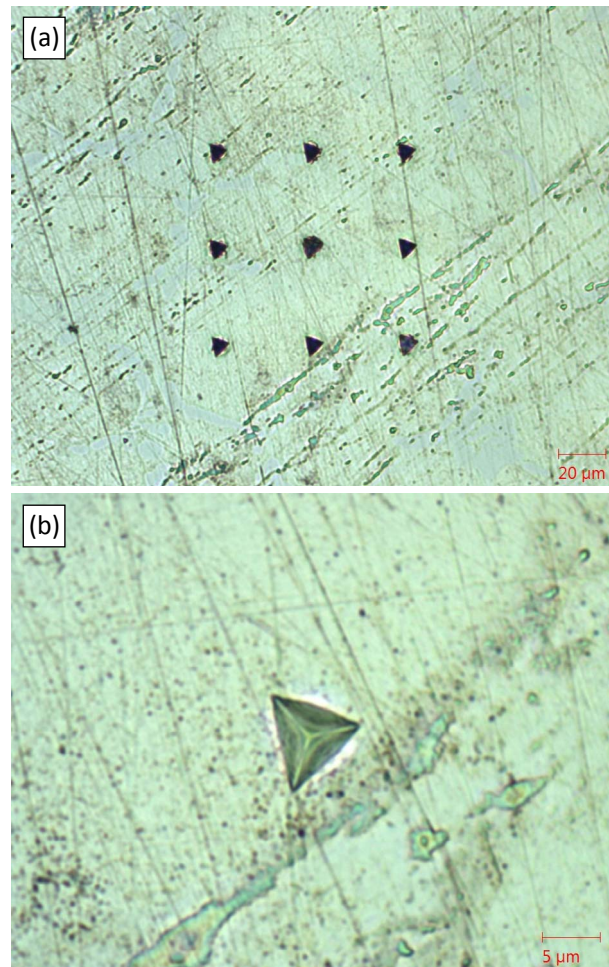


Figure 1. Nanoindentation testing of the nanocomposite with 0.5 wt. % SiC: (a) position of the indents and (b) shape and size of the individual indent

2.4 Experimental design

Experimental design can be defined as the process of doing research in a methodical and controlled way, with the goal of maximizing accuracy and enabling specific conclusions. In general, the goal is to determine the impact of a factor or independent variable on a dependent variable [29]. In the present study, the dependent variable was the wear rate of the tested materials,

while the factors that affect this variable were SiC amount, sliding speed and normal load, which were selected for experimental design (Table 1). The first factor (SiC amount) had four levels, and the other two factors (sliding speed and normal load) had two levels.

Table 1. Influencing factors and their levels

Factor	Unit	Level			
		1	2	3	4
SiC amount	wt. %	0	0.2	0.3	0.5
Sliding speed	m/s	0.25	1.00		
Normal load	N	40	100		

The Taguchi method applies a set of statistical methods and is often used in experimental design. One of the convenient things in the Taguchi method is that it uses special orthogonal arrays to study all the design factors with a minimum of experiments [30]. The experiments in this study were conducted according to the L16 orthogonal array, which is obtained by applying the mixed level design of the Taguchi method. The selection of the orthogonal array is based on the condition that the degrees of freedom of the orthogonal array should be greater than or equal to the sum of degrees of freedom of factors and their interactions [31]. The orthogonal array is formed with Minitab 16 statistical package.

The obtained experimental results were transformed into a signal-to-noise (S/N) ratio. The signal was the mean value, while the noise was the standard deviation [32]. The S/N ratio represents the scattering around a target value, and the larger S/N ratio value signifies lower scattering [33]. Usually, three types of problems (characteristics) are recognised, i.e. smaller-is-better, nominal-is-

best, and larger-is-better [34]. In this study, the smaller-is-better characteristic is applied and the S/N ratio is calculated according to the following equation:

$$S/N = -10 \log \frac{1}{n} (y_1^2 + y_2^2 + \dots + y_n^2), \quad (1)$$

where y_1, y_2, \dots, y_n are the experimental results and n is the number of experiments (which in our case was 16).

3. Results and discussion

3.1 Micro- and nanohardness and modulus of elasticity

The results of the microhardness and nanoindentation testing are shown in Figure 2. The microhardness results showed good repeatability of the results since the calculated standard deviations were below 8 %. The microhardness of the matrix alloy was comparable with the value that is prescribed by the standard for sand/die cast ZA-27 alloy of 113/119 HB [1]. The tested matrix alloy was thixocasted, i.e. its dendritic structure was changed to the non-dendritic which probably influenced the increase of hardness. In addition, microhardness is measured with a lower load than macrohardness thus showing slightly higher hardness values [35]. Following this trend, values of nanohardness are higher than the values of microhardness due to the load which was approximately 100 times lower. Results of the nanoindentation testing (both, nanohardness and elastic modulus) show a higher variation of the values, expressed through a higher value of the standard deviation (SD), which was in the same cases higher than the acceptable 10 %.

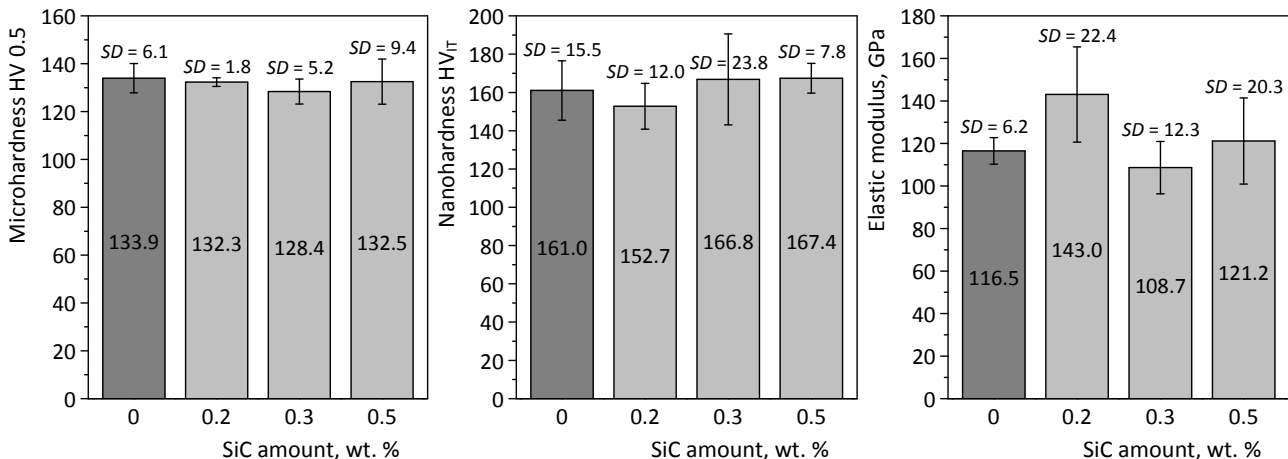


Figure 2. Microhardness, nanohardness and elastic modulus with corresponding standard deviations (SD) of tested materials

All tested materials achieved more-or-less similar hardness values, which was expected due to their similar microstructures. A higher amount of nanoparticles usually increase hardness more, but the hardness of composites also strongly depends on the interface between reinforcements and matrix as well as on the distribution of the reinforcements [36]. The low influence of SiC nanoparticles can be explained by the fact that their amount was very small. In other words, the average SiC internanoparticle distance in the ZA-27 matrix was probably larger than the average Vickers microhardness indentation diagonal (85 μm), whereas the Berkovich indenter produced an even smaller indentation with approximately 7 to 8 μm of the equilateral triangle (Fig. 1b). Therefore, the measured hardness values of nanocomposites are most probably values of the matrix. Elastic modulus values were also similar, except for nanocomposite with 0.2 wt. % SiC. The standard deviation for this nanocomposite was relatively high so this difference in elastic modulus value should be considered with caution. This is expected since the modulus of elasticity is the property that depends on the bond strength between the atoms in the material and structure,

while the heat treatment regime or cold plastic processing has little influence on it [37].

3.2 Wear rate

The results of the wear tests for different combinations of factors are presented in Table 2, together with the calculated S/N ratio values. Wear rates were calculated for the whole testing period, i.e. they represented the total wear rates. Table 2 also shows the values predicted by the application of the Taguchi method. The wear rates of the matrix (thixocasted ZA-27 alloy) were comparable with the values obtained in our previous research conducted under similar testing conditions [38,39]. Wear rate of the commercial ZA-27 alloy [38] and thixocasted ZA-27 alloy [39], tested under lubricated sliding conditions (mineral engine oil SAE 15W-40, ACEA E3) in linear contact, at a sliding speed of 0.5 m/s and a normal load of 100 N were $4.54 \times 10^{-4} \text{ mm}^3/\text{m}$ and $4.30 \times 10^{-4} \text{ mm}^3/\text{m}$, respectively. Nanocomposites wear rates varied with the SiC amount, normal load and sliding speed, but all were lower than the wear rate of the matrix alloy and in some cases, it was up to ten times lower.

Table 2. Experimental design using L16 orthogonal array, wear test results and predicted values

Test no.	SiC amount, wt. %	Sliding speed, m/s	Normal load, N	Experimental results		Predicted values	
				Wear rate $\times 10^{-4}$, mm^3/m	S/N ratio, dB	S/N ratio, dB	Wear rate $\times 10^{-4}$, mm^3/m
1	0.0	0.25	40	1.8153	-5.1791	-3.7129	1.5334
2	0.0	0.25	100	4.3038	-12.6770	-14.1579	5.1038
3	0.0	1.00	40	1.4761	-3.3822	-4.8938	1.7567
4	0.0	1.00	100	4.0986	-12.2527	-10.7671	3.4543
5	0.2	0.25	40	0.4360	7.2096	6.3102	0.4836
6	0.2	0.25	100	1.2205	-1.7308	-0.9064	1.1100
7	0.2	1.00	40	0.4864	6.2606	7.0168	0.4458
8	0.2	1.00	100	0.5480	5.2248	4.3720	0.6045
9	0.3	0.25	40	0.4019	7.9179	7.2743	0.4328
10	0.3	0.25	100	1.1617	-1.3018	-0.6047	1.0721
11	0.3	1.00	40	0.3565	8.9590	9.5585	0.3327
12	0.3	1.00	100	0.4522	6.8932	6.2512	0.4869
13	0.5	0.25	40	0.2091	13.5912	13.5723	0.2096
14	0.5	0.25	100	0.8282	1.6377	1.6017	0.8316
15	0.5	1.00	40	0.1742	15.1777	15.3743	0.1703
16	0.5	1.00	100	0.4040	7.8729	7.9755	0.3992

Experimental results were analysed by using the analysis of variance (ANOVA) technique to determine the influence of each factor (SiC amount, sliding speed and normal load), as well as the influence of their interactions on the wear rate of tested materials. The ANOVA was carried out for a significance level of 5 %, i.e. for a confidence level of 95 %. The results of the analysis are shown in Tables 3 and 4. Table 3 shows the response table for S/N ratios, and it contains a row for the average S/N ratio for each factor level, delta and rank. Delta is the difference between the maximum and minimum S/N ratio for each factor, and the rank is determined based on the delta value (a higher delta means a higher influence of the factor on the wear rate of tested materials). According to this analysis, SiC amount is the most significant factor followed by normal load and sliding speed.

Table 3. Response table for S/N ratios

Level	SiC amount, wt. %	Sliding speed, m/s	Normal load, N
1	-8.3728	1.1835	6.3193
2	4.2410	4.3442	-0.7917
3	5.6171		
4	9.5699		
Delta	17.9426	3.1607	7.1111
Rank	1	3	2

Table 3 ranks factors without taking into account the influences of factors interactions on the wear rate of tested materials. By performing the ANOVA, the percentage contribution of both, influencing factors and their interaction was determined (Table 4). Sources with a p-value less than the significance level of 0.05 (5 %) were considered to have a statistically significant

contribution. The influence of each factor and factors interaction is presented through the percentage contribution in the last column of Table 4. The contribution of the most significant factor (SiC amount) was 70.8 %, while the normal load and sliding speed had a much lower contribution, i.e. 19.8 and 3.9 %, respectively. All interactions had contributions that could be neglected since they were statistically insignificant.

Graphical interpretation of the results shown in Tables 3 and 4 are presented in Figures 3 and 4, which show the main effects plot for S/N ratios and interaction plot for S/N ratios, respectively. Plot lines practically show the significance of the particular factor or interaction of factors, i.e. the more inclined the line from the horizontal, the more significant the factor or interaction. Knowing this, it is interesting to notice that the SiC amount is the most significant factor only because of the high differences between matrix alloy (without SiC) and nanocomposite with the lowest amount of SiC (0.2 wt. %). If we extract this influence, we could say that normal load has the highest influence on the wear rate of nanocomposites.

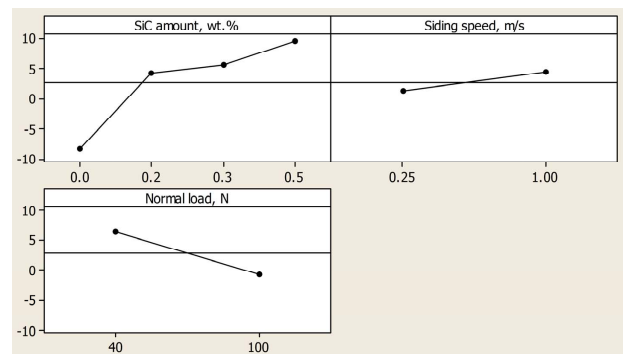


Figure 3. Main effects plot for S/N ratios (smaller-is-better characteristic)

Table 4. Analysis of variance table for S/N ratios

Source	Degree of freedom	Sequential sums of square	Adjusted sums of square	Adjusted mean square	F-value	p-value	Percentage contribution
SiC amount	3	722.68	722.676	240.892	54.42	0.004	70.8
Sliding speed	1	39.96	39.960	39.960	9.03	0.057	3.9
Normal load	1	202.27	202.270	202.270	45.70	0.007	19.8
SiC amount × sliding speed	3	6.91	6.914	2.305	0.52	0.697	0.7
SiC amount × normal load	3	14.16	14.155	4.718	1.07	0.480	1.4
Sliding speed × normal load	1	21.01	21.010	21.010	4.75	0.118	2.1
Residual error	3	13.28	13.280	4.427			1.3
Total	15	1020.27					100.0

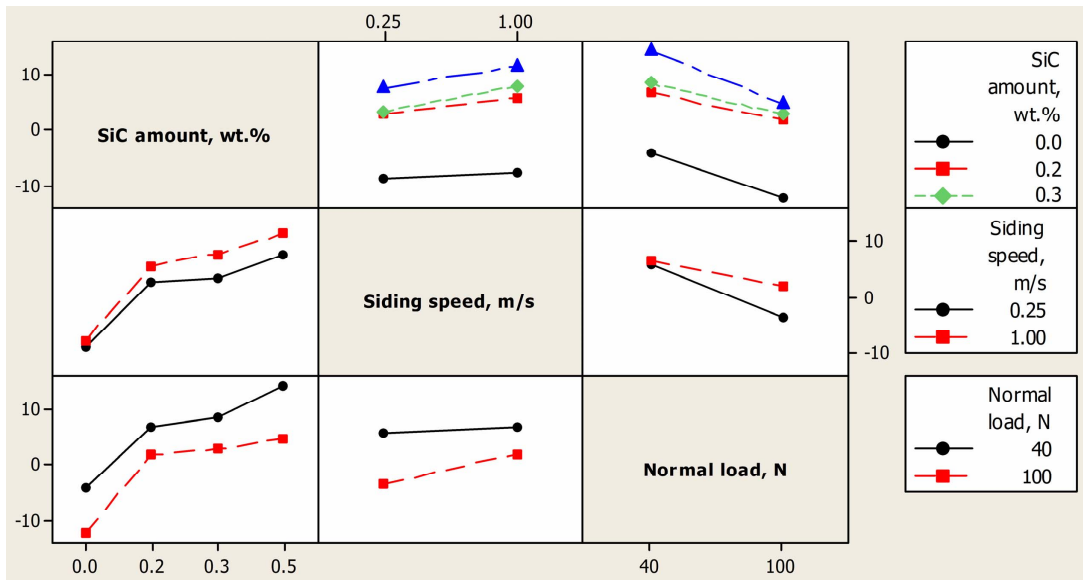


Figure 4. Interaction plot for S/N ratios (smaller-is-better characteristic)

The results of the wear rate prediction, obtained by using the linear regression model, are shown in Figure 5. The wear rates are presented in the form of wear maps, showing wear rate dependence on sliding speed and normal load (Fig. 5a), SiC amount and sliding speed (Fig. 5b) and SiC amount and normal load (Figs. 5c and d). As expected, testing under a higher normal load caused higher wear rates and the difference was notable. On the other hand, testing under higher sliding speed caused slightly lower wear rates. As previously noticed, the

influence of added SiC nanoparticles on wear rate was the most significant. However, it should be noticed that the highest decrease in wear rate was noticed between the matrix material and nanocomposites with 0.2 wt.% SiC, while the further increase of SiC amount reduced the wear rate of nanocomposites significantly less. It is possible that contact temperature, together with contact pressure, induced some of the strengthening mechanisms that usually follow the addition of nanoparticles [40].

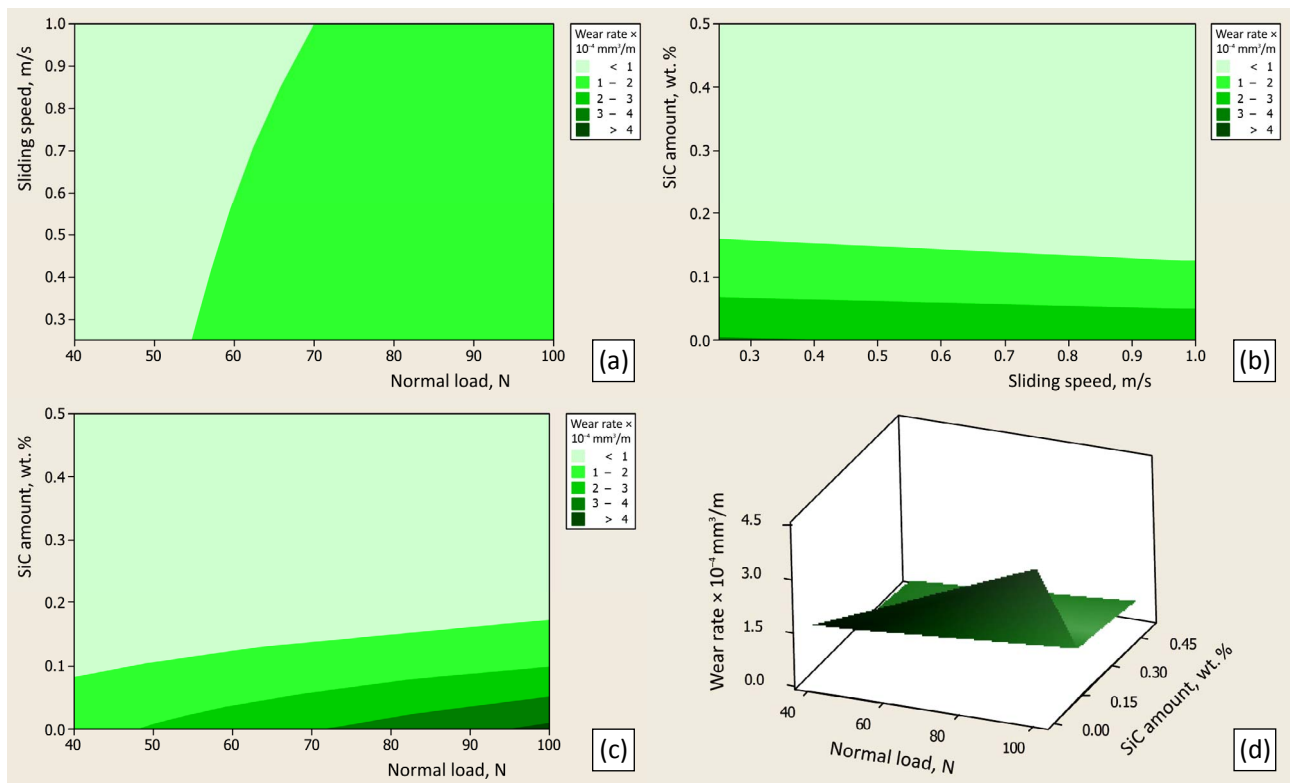


Figure 5. Wear maps for the tested materials, i.e. dependence of wear rate on: (a) sliding speed and normal load, (b) SiC amount and sliding speed and (c) and (d) SiC amount and normal load

The problem with the use of the Taguchi method is that each combination of test parameters is applied only to one sample, so the repeatability of the results is questionable. Further discussion on the impact of the amount of SiC nanoparticles is beyond the scope of this paper and would necessitate detailed microstructural analysis, as well as, worn surface analysis to explain the present wear mechanisms and to prove the possible existence of nanoparticles strengthening effect.

A normal probability plot of residuals (Fig. 6) was used to verify the assumption that the residuals are normally distributed. Figure 6 practically gives a comparison between the experimental results (red dots in Fig. 6) and the predicted values, which were obtained by the linear regression model. As could be noticed the prediction of wear rate with the formulated model is acceptable. The obtained R^2 (R-squared) value of 0.9869 and adjusted R^2 value of 0.9344 indicate a good fit of the model to the input data ($R^2 = 1$ is a perfect fit).

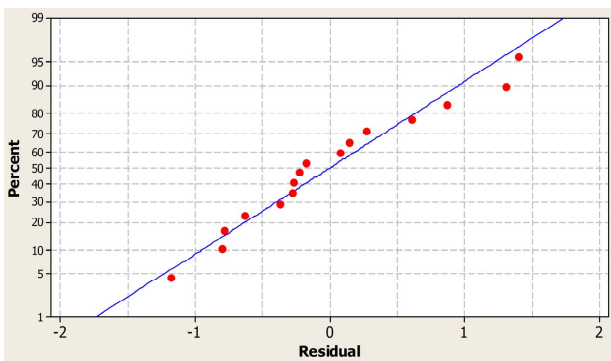


Figure 6. Comparison of the predicted values obtained by the linear regression model with experimental results

4. Conclusions

The main aim of the research was to investigate the influence of the addition of a small amount of SiC nanoparticles on the mechanical characteristics and wear resistance of ZA-27 alloy. Wear characteristics were examined in lubricated sliding conditions. Tested nanocomposites were produced by a relatively cheap process which implied the infiltration of the previously treated nanoparticles (mechanical alloying of the matrix alloy scrap with nanoparticles) into the semi-solid matrix.

Micro- and nanohardness and modulus of elasticity of all tested materials were similar, i.e. there was no obvious influence of the addition of SiC nanoparticles. Most probably that the amount of added nanoparticles was too small to

mechanically reinforce the matrix or to cause any significant strengthening effect. On the other hand, the addition of SiC nanoparticles resulted in a noticeable increase in the wear resistance of tested nanocomposites. However, the difference in wear resistance between the nanocomposites was very small, suggesting that the SiC amount is not of such importance. Maybe the increased contact temperature in combination with contact pressure (Hertz line contact) caused different strengthening mechanisms in nanocomposites to take effect and provided their higher wear resistance. Nonetheless, this should be studied further by applying detailed microstructural and worn surface analysis and other advanced surface characterisation techniques.

Analysis of variance (ANOVA) confirmed that the main influence on the wear rate of tested materials had the addition of SiC nanoparticles (70.8 %), followed by the influences of normal load (19.8 %) and sliding speed (3.9 %). The interaction of these three factors had a statistically insignificant influence on the wear rate. The increase of normal load increased the wear rate, while the increase of SiC amount and sliding speed decreased the wear rate of tested materials. Taguchi technique was used for planning the experiment as well as for the prediction of the wear rate values. These values were used for the formation of several wear maps for tested materials (wear rate vs. sliding speed vs. normal load; wear rate vs. SiC amount vs. sliding speed and wear rate vs. SiC amount vs. normal load).

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