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ANALYSIS OF PROPERTIES AND TRIBOLOGICAL WEAR OF THE Co-Cr ALLOYS USED FOR PROSTHETIC CONSTRUCTIONS

ANALIZA WŁASNOŚCI I ZUŻYCIA TRIBOLOGICZNEGO STOPÓW Z UKŁADU Co-Cr STOSOWANYCH NA KONSTRUKCJE PROTETYCZNE

Key words: Co-Cr-Mo-W alloys, microstructure, abrasive wear, prosthetic constructions.

Abstract The paper presents the results of investigations performed in the scope of the tribological wear of prosthetic cast alloys Co-Cr with micro-additions Mo and W. Abrasive wear resistance tests were carried out by means of a T-05 tester in the roller-block friction system in dry sliding metal-metal contact. A qualitative and quantitative evaluation of the alloy microstructure was made in correlation with hardness and abrasive wear. The analysis of the abrasive wear resistance of the examined alloys included an analysis of such parameters as the relative mass loss of the sample, the mean friction coefficient value, and the wear depth. It was established that the examined alloys Co-Cr-Mo-W are characterize by a low value of the friction coefficient, independent of the tribological test duration, which points to the lack of changes in the wear mechanism with the increase of the test duration time. The abrasive wear resistance of the tested materials are related to the morphology of the phases at the alloys microstructure after the solidification. A similar width of the interdendritic areas results in a similar degree of mass loss for the examined materials.

Słowa kluczowe: stopy Co-Cr-Mo-W, mikrostruktura, zużycie ścierne, konstrukcje protetyczne.

Streszczenie W opracowaniu zaprezentowano wyniki badań zużycia tribologicznego protetycznych odlewniczych stopów Co-Cr z mikrododatkami Mo i W. Badania odporności na zużycie ścierne wykonano przy użyciu testera T-05 w układzie trącym rolka–klocek, które przeprowadzono w styku ślizgowym suchym w warunkach kontaktu metal–metal. Dokonano jakościowej i ilościowej oceny mikrostruktury stopów w korelacji z twardością i ich zużyciem ściernym. Analiza odporności na zużycie ścierne badanych stopów obejmowała analizę takich parametrów jak, relatywny ubytek masy próbki, średnia wartość współczynnika tarcia oraz głębokość wytarcia. Stwierdzono, że badane stopy Co-Cr-Mo-W charakteryzują się niską wartością średniego współczynnika tarcia, niezależną od czasu trwania próby tribologicznej, co wskazuje na brak zmian mechanizmu zużywania wraz ze wzrostem czasu trwania próby. Odporność na zużycie ścierne badanych materiałów związana była z morfologią faz obserwowanych w mikrostrukturze stopów po procesie krzepnięcia. Podobna szerokość obszarów międzydendrytycznych skutkuje podobnym stopniem zużycia masowego badanych materiałów.

INTRODUCTION

One of the most frequently used non-precious metal alloys in medicine and prosthetics are Co-Cr alloys with Mo and W additions. Cobalt-based alloys as-cast are characterize by low ductility and fatigue strength **[L. 1, 2]**, high abrasive wear resistance **[L. 3–6]**, and very good corrosion resistance **[L. 7–10]**. Co-Cr-Mo-W alloys as-cast are applied in the production of skeletal prostheses, bridges, and crowns. The elements of clamp sets made

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of Co-Cr alloys prevent loosening of the movable parts of the prosthesis during its everyday use, and the alloys' resistance to permanent deformation prevents the loss of retention and stability of the prosthesis. It is also important that the alloys exhibit high strength properties, especially in the aspect of fatigue strength in the scope of the occlusal forces **[L. 11]**.

A typical microstructure of cobalt alloys obtained after casting is characterizes by the significant segregation of the chemical composition within the observed dendritic structure, which is caused by a large concentration difference between the liquid phase and the solid phase for a small difference in temperatures **[L. 13]**.

Moreover, in the microstructure of alloys from the Co-Cr system, we observe the presence of hard $M_{23}C_6$-type carbide precipitates in the interdendritic areas **[L. 12, 13]**. The precipitation of carbides with a high content of alloy elements can cause alloy brittleness as well as a drop in the pitting and crevice corrosion resistance **[L. 14]**.

The study **[L. 15]** discusses the results of investigations performed in the area of the abrasive wear resistance of cobalt-based alloys from the Remanium 2001 (CoCrMoW) and Wironit LA (CoCrMo) groups, which were made by means of the Miller apparatus **[L. 16]**. The abrasion process included cycles of 2, 4, 6, 8, 12, and 16 hrs.

The abrasion tests were carried out in a suspension of artificial saliva and SiC powders of different granularities, 1000–500 µm and 45 µm. The tests showed that, regardless of the chemical composition or the applied granularity of the abrading particles, the dependences of the relative mass loss on the time are linear in character.

The effect of alloy hardening with time was observed in no examined cases. It was established that a higher tendency for abrasive wear is exhibited by the Remanium 2001 alloy **[L. 15]**. In the case of an abradant mixture with SiC particles of sizes above 500 µm, the values of the relative mass loss obtained for the Wironit LA alloy were twice as low as the values obtained for the Remanium 2001 alloy **[L. 15]**. In the case of abrasive wear, in the suspension of artificial saliva and SiC powder with granularity 45 µm, similar mass losses were observed for both examined alloys. In the case of large particles of the abrading powder, most probably, the phenomenon of their "sliding" on the dendritic arms took place, which causes a slight abrasion of the soft cobalt shield. In a situation when the size of the particles has been reduced over ten times, to the average dimensions at the level of 45 µm, free abrasion of the matrix between the arms of the hard dendrites took place. In the case of powders with large granulation (>500 µm), for the Wironit LA alloy, one could observe the process of sliding on the numerous dendritic arms with a small degree of matrix abrasion. For the Remanium 2001 alloy, a similar process took place; however, due to a much lower concentration of dendrites, the matrix underwent a much more significant abrasion. In the case of abrading particles with the mean size 45 µm, the abrasion of the matrix in both alloys was much more facilitated, which caused an increase of the wear of both examined alloys **[L. 15]**.

The above investigation results of the abrasion wear of prosthetic alloys Co-Cr with micro-additions Mo and W point to the possibility of obtaining interesting results of further tests performed in the scope of the effect of the microstructure on the tribological properties of prosthetic alloys. The tribological examinations presented in the paper, performed with the use of a T-05 tester in the roller-block friction system, as well as the analysis of the obtained results will make it possible to better understand the microstructural properties of the examined alloys in correlation with their wear resistance. Liquidation of abrasive particles and the removal of the corrosive environment allow overcoming the influence of the other parameters on the tests result. A consequence of such methodology is an opportunity to focus at the aspects which reduce Co-Cr alloys wear resistance in a most significant way.

MATERIALS AND RESEARCH METHODOLOGY

The subjects of the investigations were Co-Cr-Mo-W alloys used to make skeletal prostheses, combined works, implants, and metal frameworks for ceramics. The chemical compositions of the alloys Co-Cr alloys with micro-alloyed Mo and W, Co-Cr-Mo-W (commercial name Remanium 2001) and Co-Cr-W-Mo (commercial name Heraenium P) are presented in **Table 1**.

Table 1. Chemical composition (% mas.) of tested Co-Cr alloys
Tabela 1. Skład chemiczny (% masowy) badanych stopów Co-Cr

Name of the alloy	Element, (% mas.)						
	Co	Cr	Mo	W	Si	C	Others
Remanium 2001	63.0	23.0	7.3	4.3	1.6	max. 0.35	Mn, N < 1.0
Heraenium P	59.0	25.0	4.0	10.0	1.0		

The tests samples were centrifugally cast by means of the lost wax method. The first stage of the casting process was the preparation of the wax models of specified dimensions for each alloy of 20x4x4 mm (dimensions of the samples required for abrasive tests with the use of a T-05 tester in a friction system roller-block).

Next, the wax models were placed in a ring, which was filled with refractory mass based on phosphates. After the casting, the rings were placed in a pressure chamber, with the pressure set to 0.4 MPa, for the time of 20 minutes, for a proper binding of the refractory mass. After the mass had been bound, the formed crucible was placed into a furnace and the process of annealing began at the rate of 7°C /min.

During the annealing, two isothermal stops were made: the first one at the temperature of 250°C, for the time of 20 minutes, in order to vaporize water from the casting ring and for the wax to evaporate, and the second one at the temperature of 600°C, when a transformation of the silica occurs. The end of the remelting process took place at the temperature of 950°C, and the casting process then took place with the use of a Vulcan 3-550 furnace. After the casting, the samples were removed from the furnace and cooled in the open air.

The following stage of the experiments was the removal of the refractory mass and mechanical sandblasting on an Ecoblast Kombi machine with the use of 200 μm granularity sand and with the application of 0.6 MPa pressure. The last step of sample preparation was mechanical removal of the gating channels.

The measurement of the samples was performed by the Vickers method. The hardness was examined on a ZWICK/ZHU 187.5 hardness tester with the load of 98.7 N (HV10). The number of measurements was 10, performed in randomly selected areas of the sample.

In order to reveal the microstructure of the examined Co-Cr alloys, the microsections were subjected to chemical etching with the use of the following etching reagent: 1 portion of HNO_3 + 3 portions of HCl. The microsections prepared in this way underwent observations with the use of an electron scanning microscope.

In the microstructure images taken with the use of scanning electron microscope, the size of the interdendritic areas as well as the free distance between them were measured using the computer image analysis software Metilo. Also, the percentage volume fraction of the interdendritic precipitations in the examined Co-Cr alloys was estimated. The abrasive wear resistance tests with the use of a T-05 tester in the roller-block friction system were conducted in a dry sliding contact under the conditions of a metal-metal contact under the load of 100 N and for two friction paths of 500 and 1000 m. The counter specimens were a ϕ 49.5 mm ring made of steel 100Cr6 with the hardness 55 HRC. Hard high carbon steel countersample give an opportunity for investigation of material wear resistance under the high tribological strain. Tribological tests under the axial loads conditions also enabled the consideration of the material behaviour during material surface finishing after casting. The spindle worked at 136 rpm. The surfaces of the examined samples after abrasion underwent profilometric tests performed with the use of an optical profilometer WYKO NT9300.

THE RESULTS OF RESEARCH

The hardness of the alloys tested was 406 ±10 HV10 (Remanium 2001) and 455 ±15 HV10 (Heraenium P), respectively. **Figure 1** presents microstructures of the analysed materials.

The microscopic observations revealed a dendritic character of the crystallization of the tested material's matrix. One can clearly see a contrast between the dendritic areas and the interdendritic ones. The dendritic arms are constituted by a solid solution of alloy elements in the β-Co matrix. In turn, in the interdendritic areas, one can observe numerous precipitates. The observed precipitates are distributed uniformly within the material's volume **(Figs. 1a, c)**. In the case of the Remanium 2001 alloy, much larger precipitates of the carbide phase are observed than in the case of the Heraenium P alloy **(Figs. 1a-d)**.

For both examined alloys, the precipitates have an irregular shape. A higher heterogeneity in the aspect of the size of the observed precipitates was documented in the case of the Remanium 2001 alloy **(Figs. 1a, b) [L. 12, 13, 17]**. Based on the microscopic documentation, quantitative analyses of the examined microstructures were performed. These measurements confirmed that, for the Remanium 2001 alloy, an over twice as high volume fraction of the interdendritic precipitates is observed in the material's structure. A similar relation

a) b) c) d)

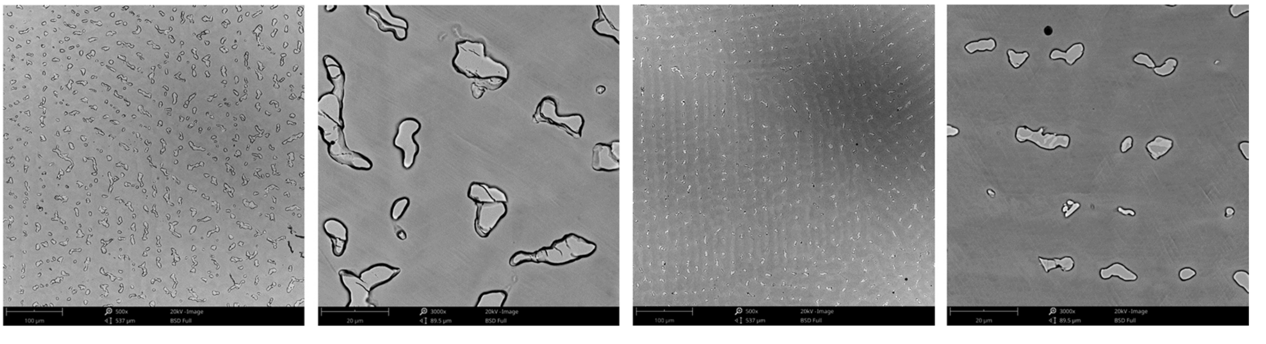


Fig. 1. Microstructures of tested alloys: Co-Cr-Mo-W (Remanium 2001) (a, b) and Co-Cr-W-Mo (Heraenium P) (c, d)
Rys. 1. Mikrostruktury badanych stopów: Co-Cr-Mo-W (Remanium 2001) (a, b) and Co-Cr-W-Mo (Heraenium P) (c, d)

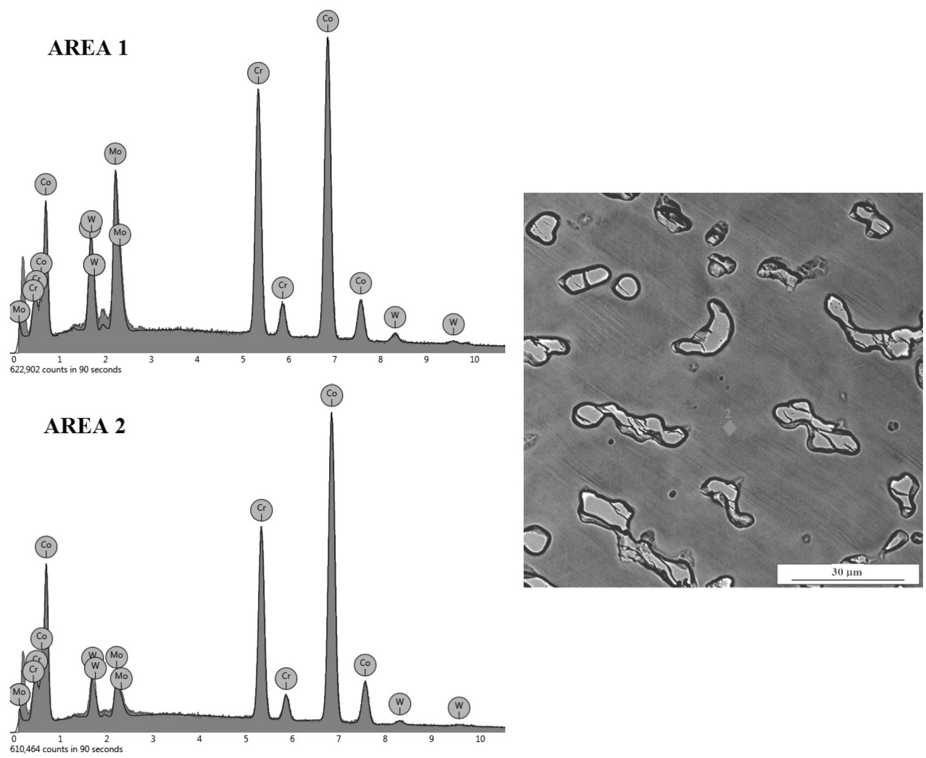
was observed for the mean particle size. In the case of the Remanium alloy, a much larger scatter was also observed in the aspect of the mean particle size; however, for both materials, the scatter of the observed particles' sizes is significant. The distance between the particles, measured as a cross section through a dendritic arm, was about 25% longer for the Remanium 2001 alloy than for the Heraenium P alloy. The amount of precipitations in reference to 1 mm^2 of the alloy's surface area was also analysed. The results of the quantitative analysis for each analysed alloy have been compiled in **Table 2**.

In order to determine the segregation degree of the chemical composition of the carbide phase and the dendritic matrix, an EDS analysis of the microsections was performed. A significant non-uniformity of the chemical composition between the dendrite area and the precipitate area was observed. The highest degree of segregation into the interdendritic areas was exhibited by molybdenum and tungsten. The degree of segregation into the interdendritic areas was lower for Cr. In the interdendritic areas, one should expect the presence of complex carbides type $M_{23}C_6$.

Additionally, these carbides can be surrounded by a small amount of phase σ, which would explain the elevated values of Mo and W in the interdendritic areas **[L. 12, 17]**. The results of the analyses of the chemical composition performed on the microsections are presented in **Figures 2** and **3**.

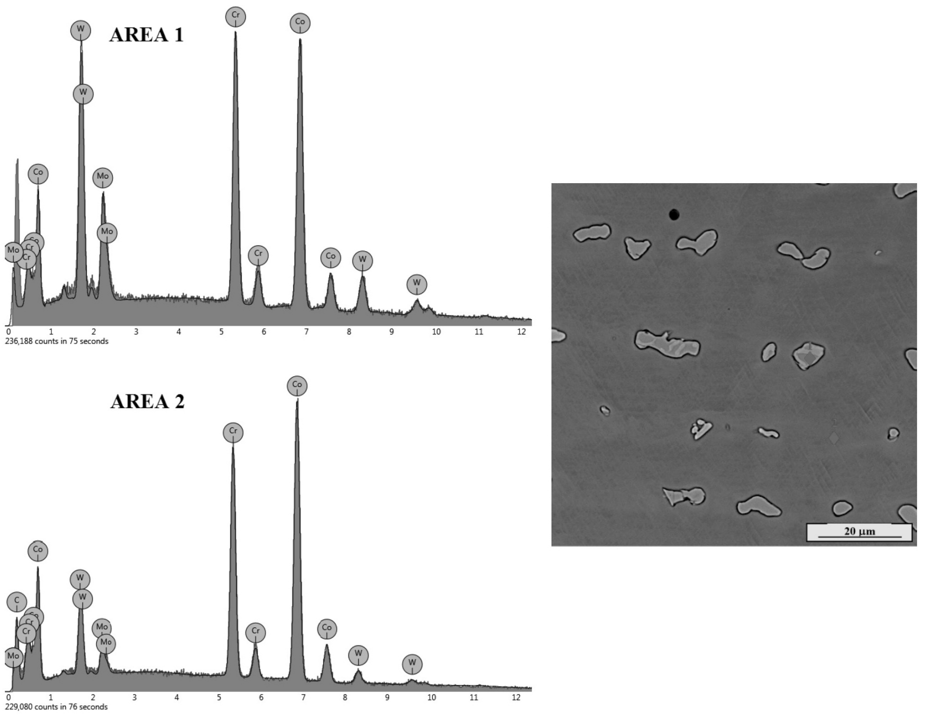
Table 2. Stereological parameters of the microstructure of tested Co-Cr alloys
Tabela 2. Parametry stereologiczne mikrostruktury badanych stopów Co-Cr

Name of the alloy	Relative volume fraction of interdendritic precipitations,%	Average area of interdendritic precipitations, µm^2	Distance between interdendritic regions, µm	Relive number of particles per mm^2
Co-Cr-Mo-W Remanium 2001	17.5 ±0.9	73 ±36	23 ±9	1718 ±86
Co-Cr-W-Mo Heraenium P	6.3 ±0.5	22 ±15	17 ±8	2229 ±110



Area	Chemical composition, mass %				
	Co	**Cr**	**Mo**	**W**	**Si**
1	47.0	20.0	21.4	7.5	4.1
2	67.4	22.2	5.7	4.7	-

Fig. 2. EDS analysis results from the areas presented at the microstructure of Remanium 2001 alloy (Co-Cr-Mo-W)
Rys. 2. Wyniki analiz EDS z mikroobszarów przedstawionych na obrazie mikrostruktury stopu Remanium 2001 (Co-Cr-Mo-W)



Area	Chemical composition, mass %			
	Co	Cr	Mo	W
1	39.0	22.5	13.0	25.5
2	64.1	25.0	2.2	8.7

Fig. 3. EDS analysis results from the areas presented at the microstructure of Heraenium P alloy (Co-Cr-W-Mo)
Rys. 3. Wyniki analiz EDS z mikroobszarów przedstawionych na obrazie mikrostruktury stopu Hearenium P (Co-Cr-W-Mo)

The observation of the wear area of the tested materials made it possible to establish that, in the case of each tribological test variant and for each examined material, the same wear mechanism is observed. On the surface of the investigated materials, abrasion wear realized by the micro-machining and micro-ridging mechanism was observed. Plastic deformation of the examined materials' surface was also present, as well as sticking of the wear products into the surface of the tested materials **(Fig. 4)**.

The tribological test results for the examined materials are presented in **Table 3**. The mass loss for a sample set on two frictions paths, 500 m and 1000 m, was analysed. The samples' mass loss and the depth of the wear areas created as a result of the tribological tests were taken into consideration. The mean wear depth was determined based on the analysis of the cross sections of the wear profiles. Moreover, the mean friction coefficient for each analysed tribological test variant was determined.

a) b) c) d)

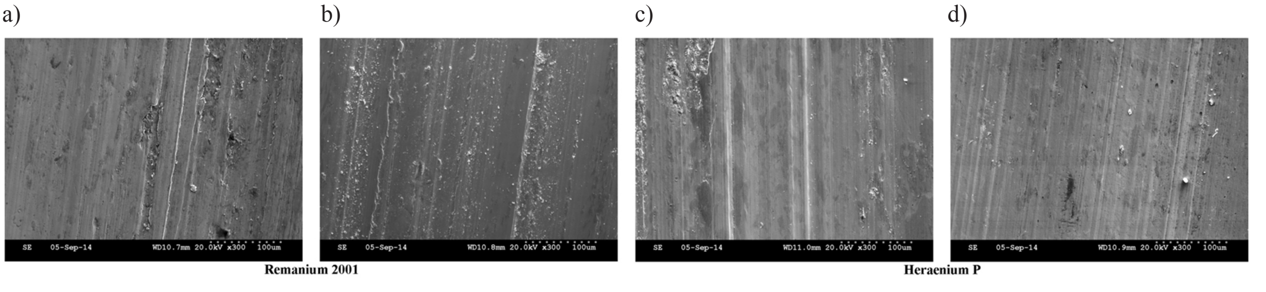


Fig. 4. Sample scanning images of worn surfaces of tested Remanium 2001 (Co-Cr-Mo-W) alloys (a, b) and Heraenium P (Co-Cr-W-Mo) (c, d) subjected to the abrasion process with the T-05 tester
Rys. 4. Przykładowe obrazy zużytych powierzchni badanych stopów Remanium 2001 (Co-Cr-Mo-W) (a, b) i Heraenium P (Co-Cr-W-Mo) (c, d) poddanych procesom ścierania za pomocą T-05 tester

Table 3.Tribology tests results for the investigated alloys Co-Cr
Tabela 3. Wyniki badań tribologicznych badanych stopów Co-Cr

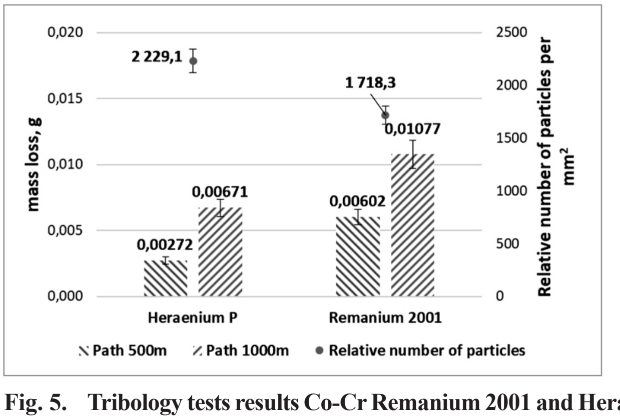
Name of the alloy	Path, m	Δm, g	Wear Depth, µm	Average Friction Coefficient
Co-Cr-Mo-W **Remanium 2001**	500	0.00602 ±0.00028	94.3 ±9.8	0.293 ±0.021
	1000	0.01077 ±0.00039	118.0 ±11.8	0.393 ±0.027
Co-Cr-W-Mo **Heraenium P**	500	0.00272 ±0.00014	55.1 ±6.9	0.360 ±0.040
	1000	0.00671 ±0.00009	68.9 ±5.4	0.379 ±0.039

The results are also compiled in a graphic form in reference to the stereological parameters of the microstructures of the examined materials presented in **Table 2 (Fig. 5)**.

Increasing the path of friction twice resulted in over a double mass loss for alloy Cr-Cr-W-Mo (Heraenium P). In the case of alloy Co-Cr-Mo-W (Remanium 2001), less than a double mass loss of the sample with an increase of the path of friction is observed. Comparing the results obtained for alloys Co-Cr with micro-additions Mo and W, it can be stated that, for both analysed paths of friction, i.e. 500 m and 1000 m, a clearly higher abrasive wear resistance of the Heraenium P alloy is observed. The alloy is characterized by a higher relative abundance of the particles in the microstructure **(Fig. 5a)**. In the case of the analysis of the wear depth, similarly to the mass loss, the Heraenium P alloy is characterized by a lower value of wear depth for both paths of friction. It is observed that the width of the dendrite arms for alloy Co-Cr-W-Mo (Heraenium P) is also lower **(Fig. 5b)**. However, the relative number of particles is significant **(Table 2, Fig. 5a)**, with an almost 30% increase of the amount of particles per 1 mm^2 of the surface of the examined alloys. This is confirmed in the hardness of the tested materials, where the Heraenium P alloy characterizes in hardness about 50 HV10 higher. The strong hardening of the material connected with the precipitation mechanism as well as the hardening of the material's matrix with alloy elements **(Fig. 3)** translated to an improvement of its wear resistance.

Not without importance in the context of the study **[L. 15]** is the mean width of the dendrites, which can constitute an area with an elevated susceptibility to abrasive wear. Additionally, the disproportionate increase of the wear depth in the Heraenium P alloy in reference to the mass loss with an elongated path of friction makes it possible to suspect that the hardening of the alloy reduced its tendency for plastic deformation during the friction. This assumption is confirmed by the comparison of the mass losses for the Heraenium P alloy after a 500 m path of friction and the Remanium 2001 alloy after 1000 m. One can observe that the mass of the material removed from the surface for both alloys was comparable. In turn, no such balance is observed in respect of the depth of the created wear areas. This makes it possible to conclude that, as a result of the operation of significant forces during the tribological tests, for alloy Co-Cr-Mo-W (Remanium 2001), a significant participation of the plastic deformation of the material's surface as a result of the friction test as well as spalling of the particles from the surface of the wear area can be assumed. This is also proved by the change in the mean value of the coefficient of friction, where, for alloy Co-Cr-W-Mo (Heraenium P), the coefficient of friction in both of the path of friction variants remains at a similar level. For the Remanium 2001 alloy, an increase of the mean coefficient of friction is observed **(Table 3)**.

a)



b)

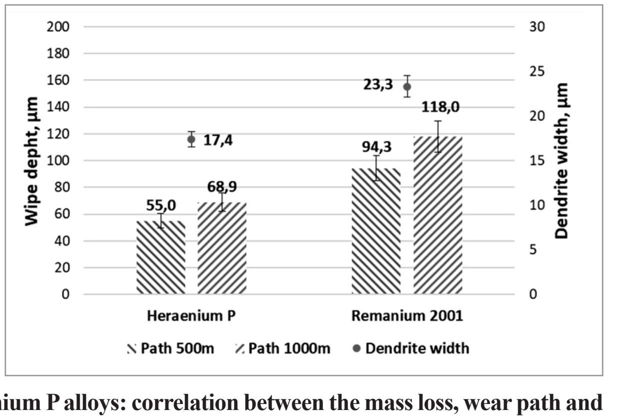


Fig. 5. Tribology tests results Co-Cr Remanium 2001 and Heraenium P alloys: correlation between the mass loss, wear path and volume fraction of precipitations (a); correlation between the wipe depth, wear path and average dendrite width (b)

Rys. 5. Wyniki testów tribologicznych stopów Co-Cr Remanium 2001 i Heraenium P: korelacja między ubytkiem masy, ścieżką zużycia a udziałem wydzieleń międzydendrytycznych (a); korelacja między głębokością zużycia, drogą tarcia a szerokością obszarów dendrytycznych (b)

CONCLUSIONS

The performed investigations make it possible to draw the following conclusions:

1. The examined alloys are characterized by a dendritic microstructure with a significant heterogeneity of the chemical composition. In the interdendritic areas, numerous precipitates are observed, which can be identified as complex carbides $M_{23}C_6$. Moreover, a significant segregation of Mo and W into the interdendritic precipitates occurs.
2. An increased volume fraction of the carbide phase in the microstructure of the examined alloys Co-Cr does not result in their effective hardening or improvement of their abrasive wear resistance. Important aspects are the number of the particles and the distance between them. The more fine particles distributed in the matrix at smaller distances, the better is the material's abrasive wear resistance.
3. The presence of larger precipitates in the microstructure as well as the higher tendency of the material for plastic deformation increase the mean value of the friction coefficient as a result of a prolonged time of tribological operation.
4. The analysis of the abrasive wear in a tribological contact steel-alloy Co-Cr with micro-additions Mo and W makes it possible to establish that these alloys are characterized by a significant degree of wear in the contact metal-metal. The use of high carbon steel enables a characterization of the material's behaviour in the context of its subtractive manufacturing. Especially the plastic deformation of the material surface layer could have resulted in the lower quality of the surface after manufacturing. Increasing the time of friction resulted in the changing of the friction force, especially in the case of the material with the lower volume fraction of the precipitations and higher average area of the dendrite.

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