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Auger/AES surface film measurements on AISI 316L biomaterial after magnetolectropolishing

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Abstract

Auger Electron Spectroscopy (AES) was used to study the elemental composition of surface film formed on AISI 316L biomaterial after its magnetolectropolishing (MEP) under varied conditions of the magnetic field intensity and the process current density applied. The results of the analyses are compared with those obtained on the samples after a conventional electropolishing (EP) under two different current densities. Changes in general elements' contents of the steel surface film, concerning Fe, O, Cr, and Ni, after EP and MEP processes, have been analyzed. The studies allowed to find out MEP parameters at which the important components/elements of the surface film reach maximum (Cr) and/or minimum (Ni) values. It appears the AES results, mainly with higher amounts of Cr and lower Ni, obtained on 316L biomaterial samples after MEP under selected parameters, are more advantageous than the ones after EP. That way the AES measurements results allowed to indicate the MEP process to be more advantageous for AISI 316L biomaterial treatment.

Keywords: Auger Electron Spectroscopy (AES), magnetolectropolishing (MEP), surface film analysis.

Analiza Augera/AES warstwy wierzchniej stali AISI 316L jako biomateriału po magneto-eklepatorowaniu

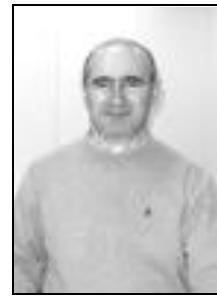
Streszczenie

Spektroskopia elektronów Augera (AES) posłużyła do zbadania składu warstwy wierzchniej (WW) stali AISI 316L jako biomateriału, po magnetoeklepatorowaniu (MEP) przy różnych warunkach natężenia pola magnetycznego i gęstości prądu. Wyniki analiz porównano z wynikami AES uzyskanymi na próbkach stali po konwencjonalnym eklepatorowaniu (EP) przy dwu gęstościach prądu. Analizie poddano zmiany składu chemicznego WW stali, głównie Fe, O, Cr, i Ni, po EP i MEP. Badania pozwoliły na określenie maksymalnych wartości Cr i minimalnych wartości Ni po EP i MEP przy wybranych parametach. Wyniki pomiarów AES pozwoliły na określenie warunków MEP, przy których skład WW jest najbardziej korzystny, ze względu na wzrost zawartości Cr i zmniejszenie Ni. Wykazano, że proces MEP znacznie korzystniej niż EP wpływa na uzyskanie oczekiwanej stanu WW stali 316L jako biomateriału.

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Slowa kluczowe: spektroskopia elektronów Augera (AES), magneto-eklepatorowanie MEP, analiza warstwy wierzchniej.

1. Introduction

The stainless steels are important group of biomaterials commonly used in surgery for implants, prepared for substituting human organs [1, 2]. Using these biomaterials as bare-metals requires their inert behaviour, good corrosion resistance, and biocompatibility. In many cases the processes of electrolytic polishing or electropolishing (EP) fulfill the requirements [3-5]. Recently the possibility to improve many surface properties arrived by using a magnetic field [6-9], and the process has been named as magnetolectropolishing (MEP) [6, 7].

Several surface analytical techniques have been used to determine the composition of the surface film formed after EP/MEP processes [10-13], e.g. X-ray Photoelectron Spectroscopy (XPS) [14-16], and Auger Electron Spectroscopy (AES) [13, 17-20]. AES is one of the most widely used analytical techniques for chemical analysis of the outmost material comprising solid surface or interface. AES uses a primary electron beam to excite the sample surface. Secondary electrons that are emitted as a result of a de-excitation process of a doubly ionized atom are analyzed and their kinetic energy is determined. Auger electron energy is characteristic of the element from which it was emitted. A peak or signal at a certain energy in the resulting Auger spectrum identify the presence of a particular element at the surface. The intensity of the signal indicates how much of a given element is present.

The technique is sensitive to the top few atomic layers of the surface, and must be carried out in ultra-high vacuum. AES analysis provides detailed information on the elemental composition of materials and, in certain cases (when valence electrons are involved) on the chemical states of surface atoms.

By combining Auger analysis with ion etching of the surface, a composition profile of elements as a function of depth may be achieved. The AES depth profile raw data consist of peak-to-peak elemental signals as a function of the sputtering time but a more commonly used in-depth composition image is given by the elemental concentration as a function of the erosion depth. Therefore, a conversion of the measured sputtering time into sputtering depth and that of the acquired signal intensities to elemental concentration are required. In order to determine the erosion rate, a reference material of certificate thickness is used [17].

Quantitative analysis is obtained using reference materials and the elemental sensitivity factors (SF). The relative sensitivity, S_x , between any element, X , and silver can be obtained from the standard Auger spectra acquired with the same instrumental setting using:

$$S_x(E_p) = \left(\frac{A+B}{A} \right) \frac{I_x}{S_{Ag} I_{Ag}} \quad (1)$$

where I_x and I_{Ag} are the peak-to-peak amplitudes of the element X and silver, respectively. S_{Ag} is the silver sensitivity factor, and A and B are the chemical formulae indices of the compounds X_A, Y_B .

The relative atomic concentration C of an element x in a sample is given by

$$C_x = \frac{I_x / S_x}{\sum_i (I_i / S_i)} \quad (2)$$

where I_x is the intensity of the Auger signal from the unknown specimen, and S_i is the relative sensitivity factor of pure element i . The summation is for the corresponding ratios for all other elements present in the sample.

2. Experimental method

The bulk chemical composition of austenitic AISI 316L stainless steel was studied and given in Table 1.

Tab. 1. Bulk chemical composition of AISI 316L SS, wt %
Tab. 1. Skład chemiczny stali austenitycznej AISI 316L, % wag

Element Content	C	Si	Mn	P	S	Cu
0.02	0.39	1.30	0.032	0.011	0.28	
Element Content	Cr	Ni	Mo	W	V	Co
16.92	10.38	2.01	0.04	0.09	0.19	
Element Content	B	Al	Sn	N	Fe	Balance
0.0019	0.007	0.014	0.0405			

Two sets of austenitic stainless steel samples of dimensions $25 \times 5 \times 1$ mm have been prepared for the investigation. The first one was electropolished (EP) in the transpassive region at the anodic current density of 500 A/dm^2 (EP500) and at about 1000 A/dm^2 (EP1000). The second set of samples was treated by electropolishing using a magnetic field (magnetoelectropolishing MEP) of intensity 66 mT up to 450 mT , and current density from 50 A/dm^2 up to 800 A/dm^2 , e.g. MEP66/180 up to MEP450/800, leaving all other conditions unchanged.

The set up for electropolishing consisted of a potentiostat and a controller. The studies were carried out for a broad range of current densities (up to 1200 A/dm^2), potential to over 10 V , up to 20 V vs. MSE (mercury-sulphate electrode), and electrolyte temperature of 60°C at the temperature control equalled $\pm 1^\circ \text{C}$. For the studies a proprietary sulphuric/orthophosphoric acids mixture electrolyte was used [7-9, 12-16, 18-22].

Auger measurements were performed in a PHI model 4200 Thin Film Analyzed, equipped with a variable resolution cylindrical Mirror Analyzer (CMA) and a coaxial electron gun (Fig. 1).



Fig. 1. The PHI model 4200 set-up used for AES measurements
Rys. 1. Stanowisko PHI model 4200 do badań powierzchni próbek metodą AES

Combining the AES measurements with argon bombardment, the surface layer can be gradually removed and the analysis can be carried out on new layers exposed after each sputtering cycle. This has been known as depth profiling analysis and provides the relative concentrations of elements as a function of depth.

The velocity of surface erosion is described by an instantaneous sputtering rate SR which determines the mean eroded depth as a function of the sputtering time.

A method to obtain the erosion rate SR is to measure the time required to sputter through a layer of known thickness, for instance a metallic evaporation layer or an oxide layer. A 100 nm SO_2 thin film on silicon was used as thickness certificate reference material and the ion gun parameters were fixed so to obtain a suitable erosion rate [17].

The system base pressure was $2.1 \times 10^{-7} \text{ Pa}$. As a rule, the interface time t was determined between 84% and 16% of the intensity change at the interface (Fig. 2). A 2.44 nm/min erosion rate was obtained for the silicon dioxide.

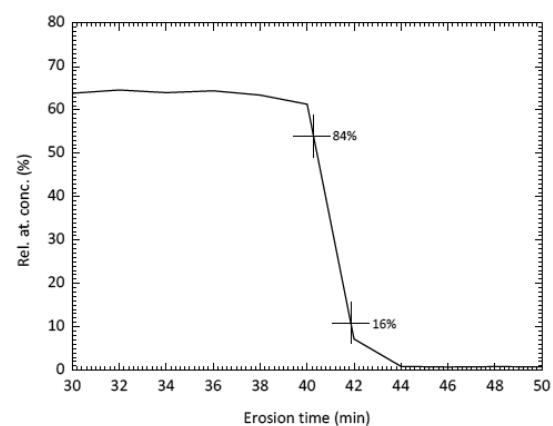


Fig. 2. Oxygen region. Interface time measured between the 16 and 84 % of the intensity change at the interface
Rys. 2. Zasada pomiaru AES dla zakresu intensywności zmian (16÷84) % (zakres tlenu)

Depth profiles were obtained by alternating sputtering and acquisition cycles. A 2 keV Ar^+ beam, rastered over an area of $2 \times 2 \text{ mm}$, sputtered the samples while a 3 keV , 450 nA electron beam excited the Auger electron.

3. Results of the study

3.1. Surface analysis

Scans of the whole range of Auger electron energies ($20 \div 1020$ eV) were carried out by detecting and counting the number of Auger electrons. The analysis gives information about the presence of elements and contaminants on the sample surface region ($2 \div 20$ atomic layers). By taking into account the SF's of the elements detected, quantification is possible. This method is useful in identification of the unknown elements and estimating their concentration on the sample surface.

Figures 3-9 show the AES selected spectra acquired on the surface of MEP450/50, MEP450/500, MEP225/50, MEP225/1000, EP500, and EP1000 samples, respectively. Sulphur, oxygen, chromium, nickel, and iron are of special attention in the studies, with different concentration detected on surface of all samples. There are some other elements, such as chlorine, carbon, potassium, calcium, nitrogen and sodium which can be attributable to common contamination caused by samples manipulation and/or contact with air.

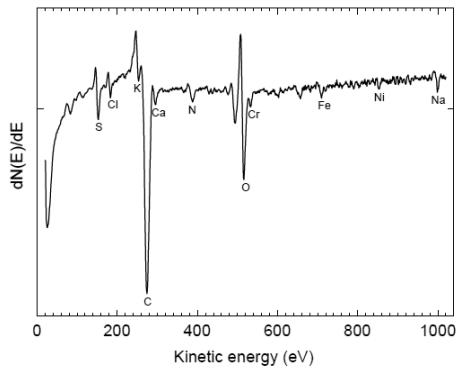


Fig. 3. AES survey on the sample MEP450/50
Rys. 3. AES dla próbki MEP450/50

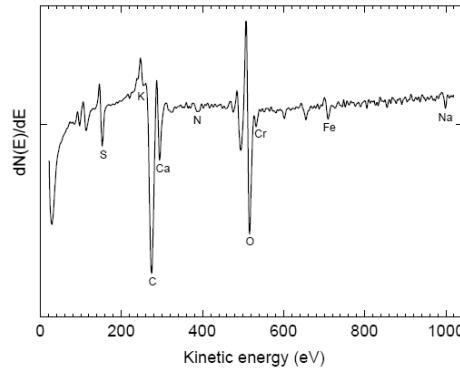


Fig. 7. AES survey on the sample MEP225/1000
Rys. 7. AES dla próbki MEP225/1000

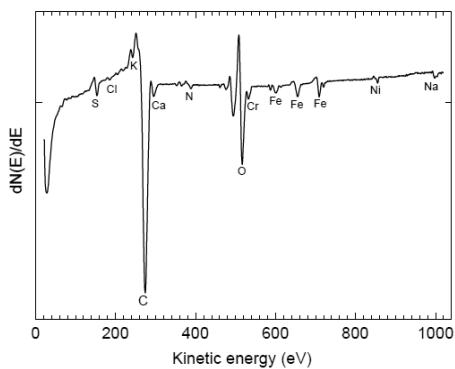


Fig. 4. AES survey on the sample MEP450/500
Rys. 4. AES dla próbki MEP450/500

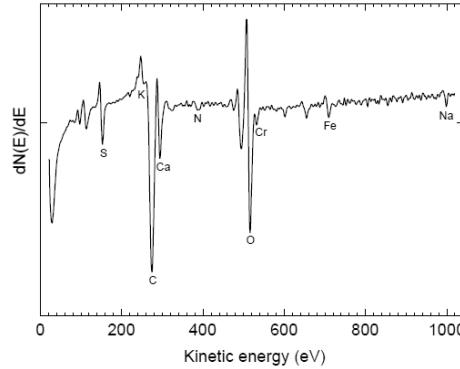


Fig. 8. AES survey on the sample EP1000
Rys. 8. AES dla próbki EP1000

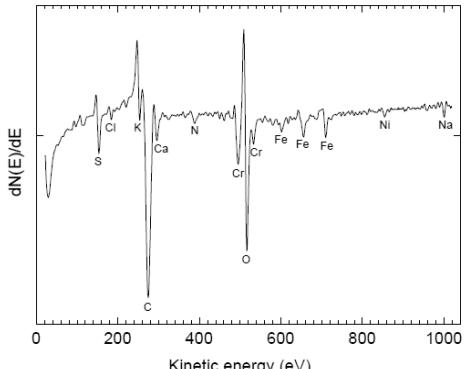


Fig. 5. AES survey on the sample MEP225/50
Rys. 5. AES dla próbki MEP225/50

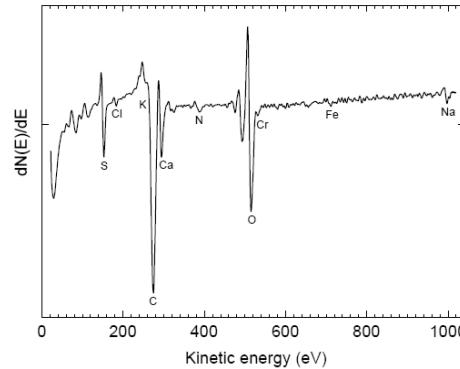


Fig. 9. AES survey on the sample EP1000
Rys. 9. AES dla próbki EP1000

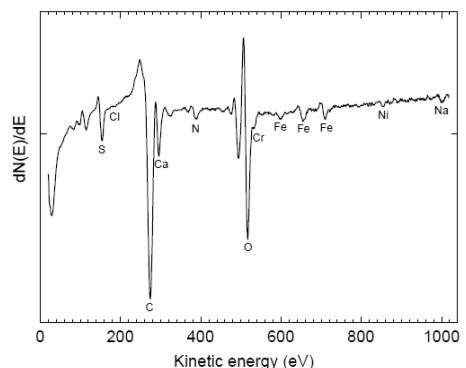


Fig. 6. AES survey on the sample MEP225/500
Rys. 6. AES dla próbki MEP225/500

The relative atomic concentration of elements measured on surface of all samples has been reported in Fig. 10. All samples show a significant contamination of carbon and oxygen. The former ranges from 49% to 76% and the latter from 10% to 21%. Other elements greatly suffer of a "shadow effect" due to the high concentration of C and O₂. Iron concentration does not exceed the 10%, and the MEP225/50 and MEP450/50 samples only, show a value of approximately 8%. Chromium and nickel contents are lower than 3%; samples MEP450/200, MEP225/1000, and EP1000 do not present nickel on surface. Other elements named "contamination elements" show a surface concentration smaller than 10%.

Considering summation of four basic elements ($\Sigma E4$) in the steel sample, iron, chromium, nickel and oxygen only, the highest Cr/ $\Sigma E4$ ratio of 0.0807 was obtained for MEP225/50, the lowest 0.0397 for MEP225/1000 with the biggest ratio for the sample electropolished without the magnetic field, EP500 being 0.0673. That result of chromium ratio is consistent with our other studies

performed on this material using the X-ray Photoelectron Spectroscopy (XPS) [12, 14, 21, 22]. Zero nickel was detected for MEP450/200, and at both MEP225/1000 and EP1000.

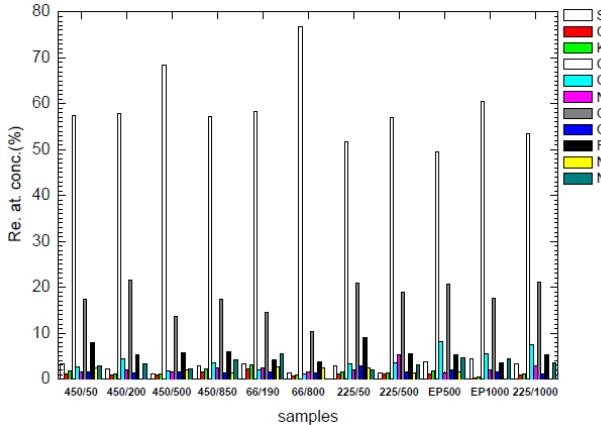


Fig. 10. Relative atomic concentration measured on surface of all samples
Rys. 10. Względna zawartość atomowa zmierzona na powierzchni próbek

3.2. Depth profile analysis

The AES depth profile measurements were performed for all studied samples. Excepting for the EP1000 sample (Fig. 11), where a thicker oxidation layer was detected (above 10 nm), no remarkable differences can be observed (mostly from 4 nm to 7 nm) when comparing the elemental composition as a function of depth.

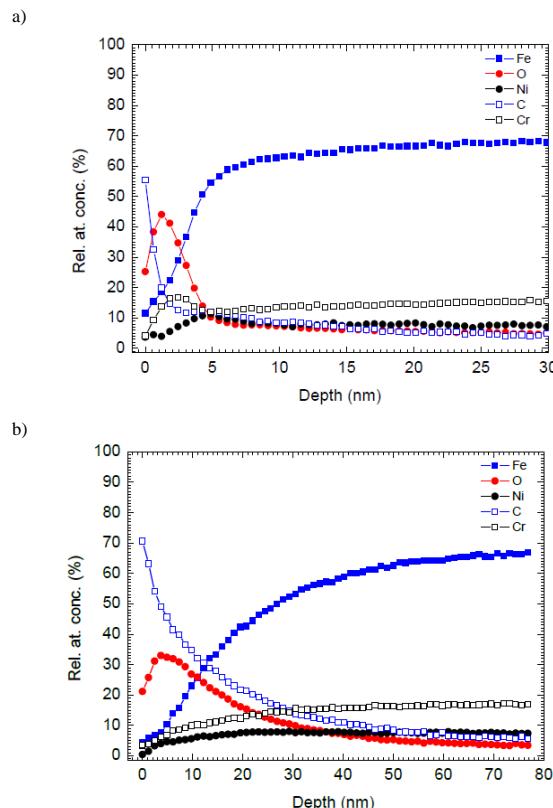


Fig. 11. AES depth profile on the sample: a) MEP225/50, b) EP1000
Rys. 11. Analiza profilowa AES dla próbek: a) MEP225/50, b) EP1000

Oxygen, iron, nickel and chromium regions have been individually reported in Figures 12-15. These pictures allow to appreciate the small differences in the depth compositional profiles, i.e. thickness of the outermost oxide layer of iron

(Figs. 13a, b), nickel (Figs. 14a, b), and chromium (Figs. 15a, b) surface enrichment phenomena.

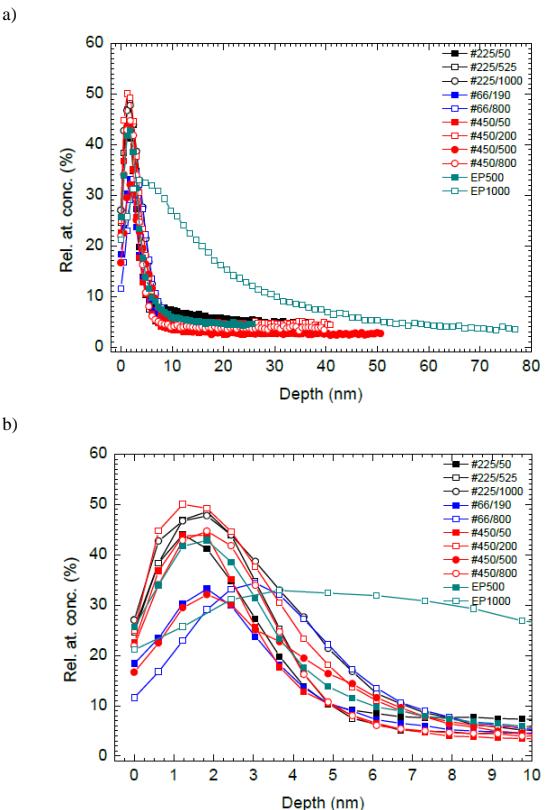


Fig. 12. Oxygen depth profile of all samples: a) depth range up to 80 nm,
b) depth range up to 10 nm
Rys. 12. Analiza zmiany zawartości tlenu dla wszystkich próbek:
a) zakres głębokości do 80 nm, b) zakres do 10 nm

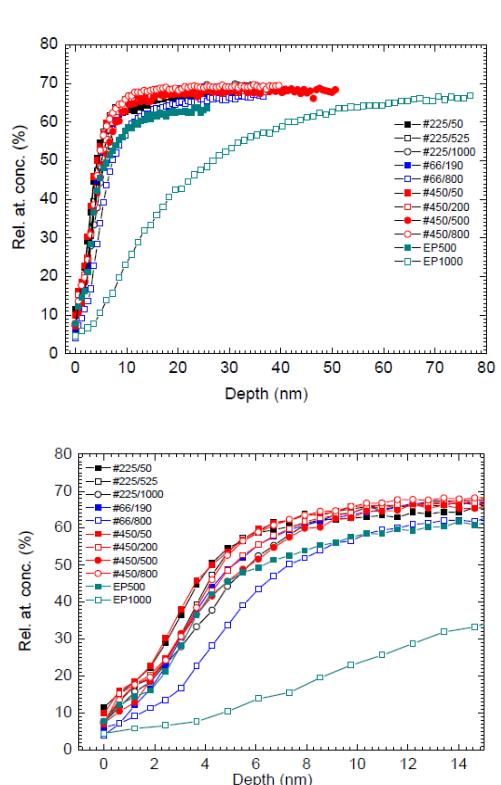


Fig. 13. Iron depth profile of all samples: a) depth range up to 80 nm,
b) depth range up to 15 nm
Rys. 13. Analiza zmiany zawartości żelaza dla wszystkich próbek:
a) zakres głębokości do 80 nm, b) zakres do 15 nm

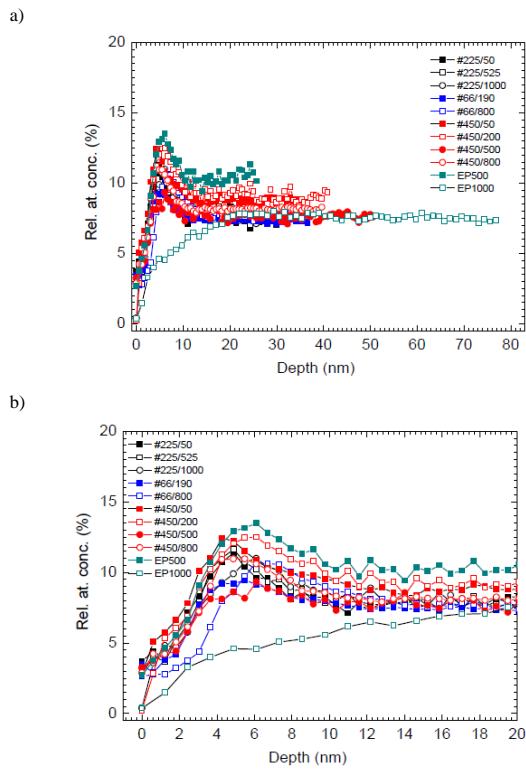


Fig. 14. Nickel depth profile of all samples: a) depth range up to 80 nm, b) depth range up to 20 nm

Rys. 14. Analiza zmiany zawartości Ni dla wszystkich próbek: a) zakres głębokości do 80 nm, b) zakres do 20 nm

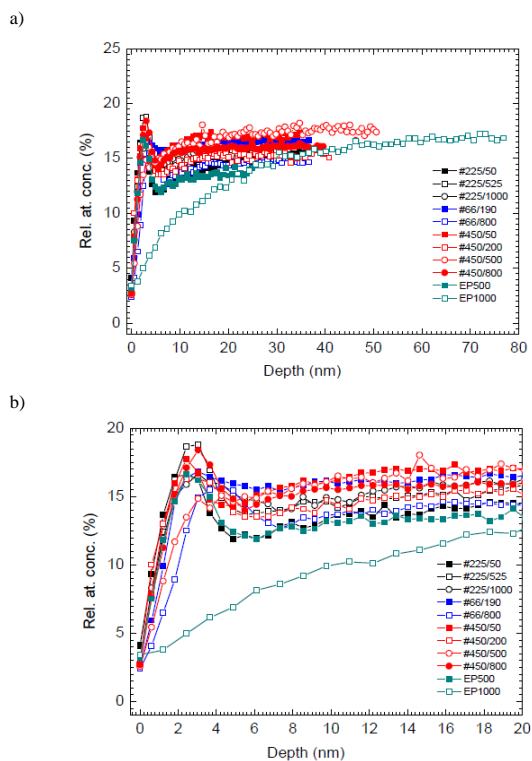


Fig. 15. Chromium depth profile of all samples: a) depth range up to 80 nm, b) depth range up to 20 nm

Rys. 15. Analiza zmiany zawartości Cr dla wszystkich próbek: a) zakres głębokości do 80 nm, b) zakres do 20 nm

One may notice a big deviation from the courses in Figs. 12-15 for EP1000 sample. However, it should be admitted that such extreme treatment conditions are improbable to be applied in practice.

4. Conclusion

The Auger spectra acquired on the as-received surfaces reveal a remarkable contamination of carbon. Its high concentration (>50%) has a “shadow” effect on the elements composing the sample matrix (Fe, Ni, Cr and O). Surface is also affected by trace amounts of a variety of elements (S, Cl, Ca, K, N and Na) due to contamination or process residue.

Except for the EP1000 sample, depth profile analyses show on outermost oxide layer whose thickness does not exceed the 5 nm for all samples. Carbon and other contaminants (not reported here) rapidly decrease with the erosion time. A sub-surface enrichment layer can be observed for both chromium and nickel. To conclude, the EP1000 sample clearly shows remarkable discrepancies in the depth compositional analysis compared to the others. As for the small differences (oxide thickness, C contamination, Ni and Cr behaviour) in the other samples, they can be easier observed in the expanded pictures (Figs. 12b, 13b, 14b, 15b).

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RECENZJE

Wczoraj, dziś i jutro polskiej informatyki

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Ryszard Tadeusiewicz, Jan Węglarz, Kazimierz Wiatr

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ta bardzo interesująca książka, będąca podsumowaniem stanu aktualnego i perspektyw rozwoju technologii i infrastruktury informacyjnej w Polsce, jest zbiorem wystąpień „najlepszych z najlepszych” polskich informatyków w poszczególnych obszarach problemowych, wygłoszonych podczas Światowego Zjazdu Inżynierów Polskich, który odbywał się w Warszawie w dniach 8-10 września 2010 r. Książka została wydana w ramach obchodów Światowego Dnia Społeczeństwa Informatycznego, pod honorowym patronatem Prezydenta Rzeczypospolitej Polskiej.

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Historię polskiej informatyki, obejmującej okres sześćdziesięciu lat, prezentuje mgr inż. Jerzy S. Nowak, członek założyciel Polskiego Towarzystwa Informatycznego w 1981 r. Autor prezentuje pierwsze informacje o komputerach, sięgające lat 40-tych minionego stulecia, wskazuje osoby które w sposób szczególny przyczyniły się do opracowania konstrukcji, a następnie produkcji polskich komputerów. Przywołane zostają konstrukcje komputerów pamiętane jeszcze przez starsze pokolenie informatyków, wyprodukowane w Zakładach Elwro we Wrocławiu, a mianowicie: Odra-1003, 1013, 1204, 1304, 1305, 1325. Ponadto autor syntetycznie przedstawił osławione komputery Jednolitego Systemu RIAD, a także minikomputery produkowane w Polsce.

Kolejny rozdział, opracowany przez Prof. Andrzeja Skowrona, został poświęcony twórcy polskiej szkoły sztucznej inteligencji,

nieżyącemu już Prof. Zdzisławowi Pawlakowi. Niewątpliwie największym osiągnięciem Prof. Z. Pawlaka są fundamentalne prace związane z zaproponowaną przez Niego teorią zbiorów przybliżonych. O olbrzymim znaczeniu tej teorii niech świadczy fakt, że od czasu ukazania się pierwszej publikacji Profesora Pawlaka na temat teorii zbiorów przybliżonych w 1982 roku, na świecie w języku angielskim opublikowano ponad 4000 pozycji. Do książki Prof. Andrzej Skowron dołączył kompletną listę prac Prof. Pawlaka, zawierającą 294 pozycje.

Prof. Jan Węglarz wraz ze swoim współpracownikiem dr inż. Maciejem Stroińskim prezentują wieloletni rozwój i aktualny stan infrastruktury informatycznej Polski. Prof. Węglarz odegrał w stworzeniu i rozwoju polskiej e-infrastruktury absolutnie kluczową rolę. Autorzy przedstawiają bazę superkomputerowo-sieciową infrastruktury polskiej nauki, określającą uwarunkowania polskiej informatyki na koniec pierwszej dekady XXI wieku.

Dr inż. Wacław Iszkowski przedstawia przedsiębiorstwa informatyczne, a także przedsiębiorców mających istotny wpływ na przemiany w polskiej gospodarce po upadku komunizmu. Polityka przeplata się tutaj z gospodarką oraz techniką informacyjną. Interesujące jest prześledzenie przetargów i zawierania kontraktów na systemy i usługi informatyczne.

Ostatni rozdział książki, poświęcony polityce wspierania prac naukowych i wdrożeniowych w obszarze informatyki, opracował senator RP, przewodniczący Komisji Nauki, Edukacji i Sportu Senatu RP, Dyrektor Akademickiego Centrum Komputerowego CYFRONET AGH w Krakowie, Prof. Kazimierz Wiatr. Opracowanie prezentuje działania rządu i parlamentu w obszarze informatyki oraz wskazuje na trudności i ograniczenia. Obraz kreślony przez Prof. Wiatra wydaje się być optymistyczny, wieloletnie plany strategiczne Polski przewidują dalszy rozwój Społeczeństwa Informacyjnego w Polsce.

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