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EMISSION SPECTRA FOR ANALYSING ELEMENTS IN CLUES USING OPTICAL METHOD

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The following research was dedicated to the investigation of emission spectra of different elements and their use in the analysis of clues. The used equipment was a spectrometer, the “Red Tide USB-650”, and an application called “Overture”. The spectrometer has a spectral range from 350 nm to 1000 nm. It’s a very lightweight and portable device, not taking up much space and weighting only 190 g. The sensitivity of the device is 75 photons at a wavelength of 400 nm. The optical resolution of the spectrometer is 2 nm, while the program can display accurate graphs with a resolution of up to 0.1 nm. The spectrometer’s integration time ranges from 3 ms to up to 65 seconds, but typically it doesn’t exceed 15 seconds. For very precise data recording the spectrometer can also use the optic fibres. The used program has a library of emission spectra of different elements. The experiment is performed by making the substance to emit photons by exciting it. The light from this substance is received by the spectroscopy and the data are transmitted to the computer. The program generates an “Intensity-Wavelength” graph right away. But to analyse and to detect elements in clues a graph is not the only required thing. Luckily, we have access to the emission spectra library built into the program. Those can be enabled and disabled to detect if the given element is present in the earlier analysed substance. Such a method can be used to detect elements in clues using spectral analysis at remarkable speeds and accuracies.

Keywords: emission spectra, clue, resolution, sensitivity, photon.

Cercetări de față sunt dedicate studiului spectrelor de emisie a diferitor elemente și utilizarea lor în analiza probelor în criminalistică. Echipamentul folosit era spectrometrul „Red Tide USB-650” și aplicația „Overture”. Spectrometrul are diapazonul spectral de la 350 nm până la 1000 nm. Este un dispozitiv foarte ușor și portativ, nu ocupă mult spațiu și cântărește numai 190 g. Sensibilitatea dispozitivului este 75 fotoni la lungime de undă de 400 nm. Rezoluția optică a spectrometrului este 2 nm, pe când programul poate afișa grafice precise cu rezoluția de până la 0,1 nm. Timpul de integrare a spectrometrului variază de la 3 ms până la 65 s, dar de obicei nu depășește 15 s. Pentru înregistrarea datelor cu precizie foarte înaltă spectrometrul de asemenea poate folosi fibre optice. Programul utilizat are o bibliotecă de spectre de emisie a diferitor elemente. Experimentul se realizează prin emiterea de substanță la excitarea ei a fotonilor. Lumina de la aceasta substanță este primită de spectroscopul și datele se transmit la calculator. Programul imediat generează dependența „Intensitate-Lungime de undă”. Dar pentru a analiza și a detecta elementele din probe graficul nu este suficient. Din fericire, avem acces la biblioteca de spectre de emisie încorporat în program. Acestea pot fi activate și dezactivate pentru a detecta dacă elementul dat este prezent în substanță analizată anterior. Așa metodă poate fi folosită pentru detectarea elementelor din probe în criminalistică utilizând analiza spectrală cu viteză și precizie remarcabilă.

Cuvinte-cheie: spectrul de emisie, dovadă, rezoluție, sensibilitate, foton.

INTRODUCTION

The following article explains how the elements found in clues can be analysed using spectral methods by a spectrometer and software. By measuring the emission spectra of excited substances it was possible to detect elements in the sample.

Every known single element has a different emission spectrum. It can be compared with a type passport for every known substance. But to understand this concept of emission spectra, it’s necessary to know the concept of emission of photons from substances.

To explain this concept Bohr’s atom model will be used. Niels Bohr first proposed that electrons can only gain and lose energy by jumping from one allowed orbit to another, absorbing or emitting electromagnetic radiation with a frequency ν determined by the energy difference of the levels according to the Planck relation:

$$\Delta E = E_2 - E_1 = h\nu \quad (1)$$

Bohr stated that photons are produced when electrons move from a lower energy state to a lower one, giving off that energy in the form of an electromagnetic wave – a photon (fig. 1). The wavelength of the photon

can either be derived from the Planck relation, where $\nu = \frac{1}{T}$, or using the Rydberg formula:

$$\frac{1}{\lambda_{if}} = R \left(\frac{1}{n_f^2} - \frac{1}{n_i^2} \right) \quad (2)$$

where R is the Rydberg constant, which is different for various elements.

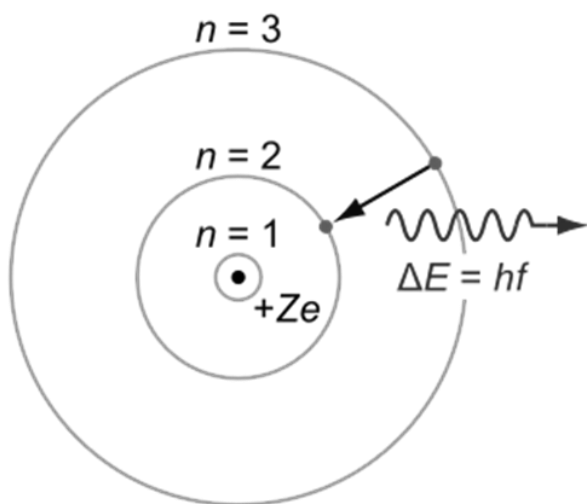


Fig. 1. A hydrogen atom according to Bohr's model, emitting a photon at a wavelength of 656 nm, which would be a red visible light

For every atom there are multiple different emission spectra. They have their own intensities and wavelengths. For example, hydrogen has 4 spectra in the visible range. This series of spectra is called the Balmer series. Photons of this series are emitted when electrons transition from a higher orbit to the second orbit (fig. 2).

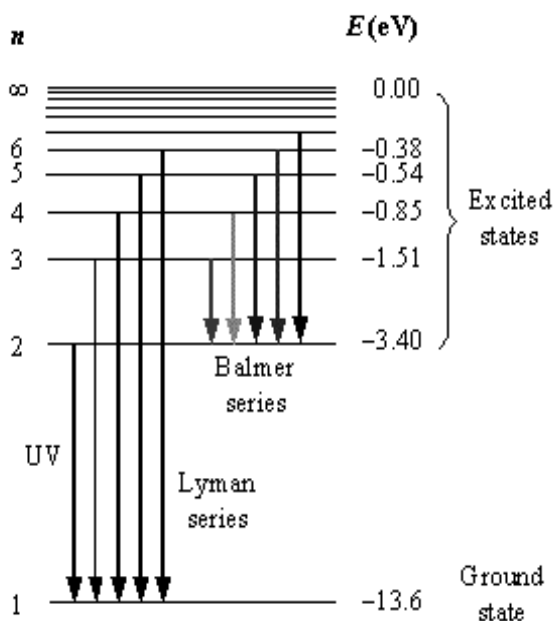


Fig. 2. Electron transitions from different energy states

There are other series for the hydrogen atom, like the Lyman (Ultraviolet) and Paschen, Brackett, Pfund, Humphreys (Infrared) (fig. 3).

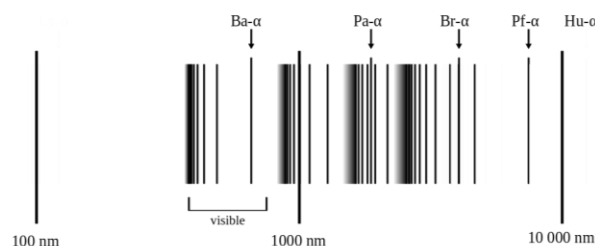


Fig. 3. The emission spectra of the hydrogen

There is also another way of demonstrating the different electron transitions occurring in a hydrogen atom and the photon energies according to the Rydberg equation.

Such a representation helps to visualise the energy of the photons emitted from different electron transitions.

Again, we see that emitted photons have different energies depending on the exact electron transitions that occur inside the atoms. Along with that the energies vary depending on the atom emitting the photon, so this again proves that the emission spectra of all atoms are different.

But they vary not only from atom to atom. The emission spectra vary from isotope to isotope as well. Although the nucleus charge of isotopes is the same, the mass is different. This affects the emission spectra by a slight amount. To explore this phenomenon hydrogen and its isotope deuterium will be used as examples.

Deuterium has an additional neutron in its core, which changes the mass of the nucleus. By expanding the Rydberg equation we get the following formula:

$$E_i - E_f = \frac{\mu_x Z^2 e^4}{(4\pi\epsilon_0)^2 2h^2} \left(\frac{1}{n_f^2} - \frac{1}{n_i^2} \right) \quad (3)$$

where μ_x is the reduced mass of the atom X. Since the equation depends on this mass, isotopes have slightly different emission spectra.

Looking at the image (fig. 4) we can see that both emission spectra of deuterium have a slightly shorter wavelength than the hydrogen spectra, but only by a very slight amount.

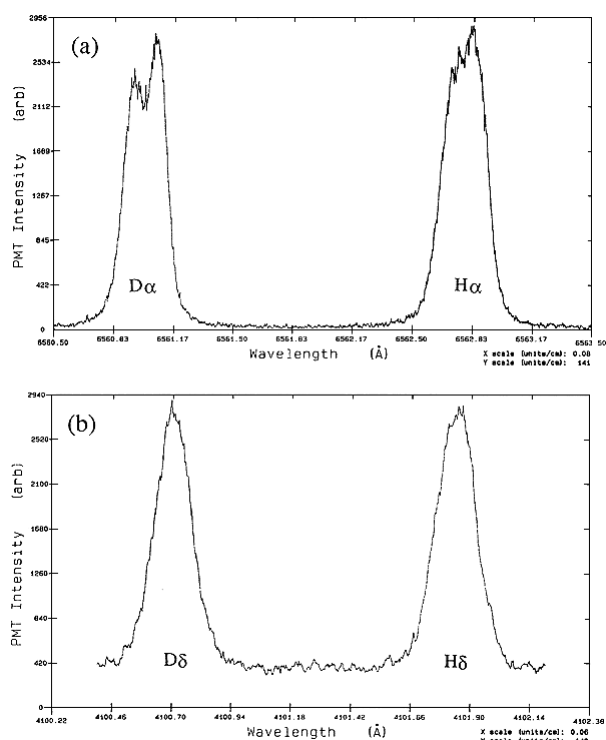


Fig. 4. Comparison of the slightly different (by only about 0.2 nm) emission spectra of hydrogen and deuterium:

- Emission spectra of D_{α} and H_{α}
- Emission spectra of D_{δ} and H_{δ}

But since they vary, it's possible to distinguish deuterium and hydrogen in the experimental studies. This relates to all the elements and isotopes, since the same thing applies to them.

ATOMIC EMISSION SPECTROSCOPY

All of the previously introduced concepts are the basis of atomic emission spectroscopy (AES). It's a method of chemical analysis used to determine the amount of an element in a certain substance or sample.

It is a very precise method of doing this kind of analysis, due to the fact that some spectrometers have a very high sensitivity, and allow measurements with great precision.

Optical methods also allow distinguishing atoms from its isomers if they are present in the sample being analysed, since the emission lines of isomers slightly differ.

It is performed by exciting the atoms of a substance sample using various methods. The data is collected with a spectroscope and

is sent to a computer. Then, according to the emission spectra of different elements, the resultant spectrum is matched with the emission spectra. If there is a match it means that the substance, the emission spectrum of which was used, is present in the sample.

There are various methods of performing this analysis. The atoms can be excited by a flame, which gives the atoms thermal energy or, for analysing metallic elements in substances, spark and arc emission spectroscopy can be used. A spark with high voltage excites the atoms and they emit photons.

EQUIPMENT

To perform AES, a spectrometer and computer are required. The used spectrometer was the "Red Tide USB-650", and the program was "Overture".

Red Tide USB-650 spectrometer (fig. 5) [1-3]:

- Dimensions: 89.1×63.3×34.4 mm
- Weight: 190 g;
- Range: 350-1000 nm;
- Sensitivity: 75 photons at 400 nm;
- Integration time: 3 ms to 60 s (usually up to 15 s);
- Optical resolution: ~2 nm;
- Able to work both with and without optical wire.



Fig. 5. Red Tide USB-650 spectrometer

Overture software (fig. 6):

- Contains a library of sample emission spectra of different elements which can be activated and disabled;
- Able to take screenshots of multiple spectra for comparison;

- Allows to work with emission, absorption and transmission spectra;
- Able to save spectra and export data for building graphs and further research.

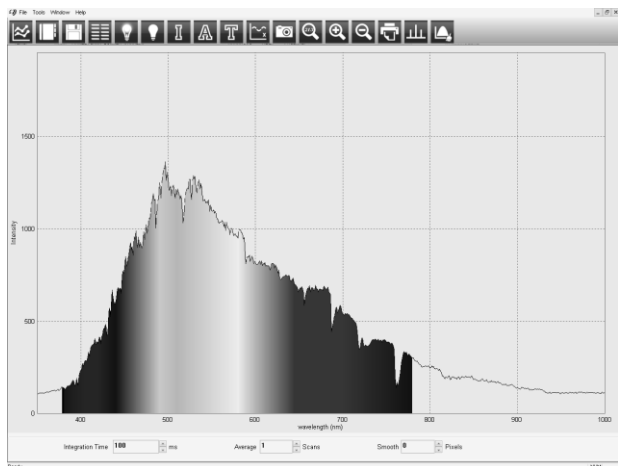


Fig. 6. Overture software interface

EXPERIMENT

After setting up the experimental equipment we decided to try and detect the elements in a simple light source – a lamp installed on the ceiling of the room. By precisely pointing the spectrometer at the light source (fig. 7) we were able to get a good image of the emitted spectrum. (Note the minor error on the bottom - it's the background noise of the spectrometer. It can be cancelled in the program).

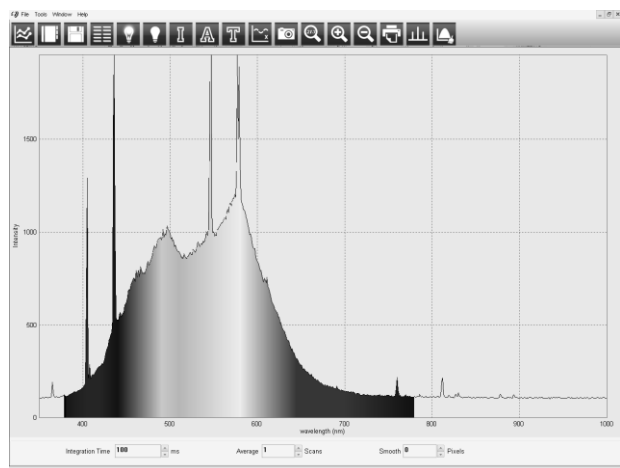


Fig. 7. Spectrum received from the device while pointing at the source

The software used in the experiment had a database of emission spectra of different elements integrated into it (fig. 8).

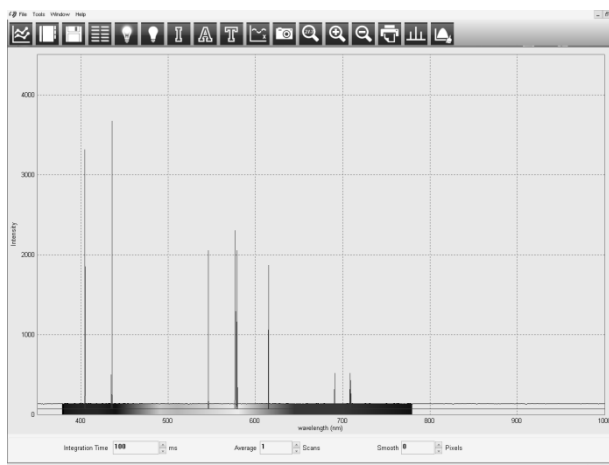


Fig. 8. Mercury emission spectrum, taken from the integrated database

The process of matching sample spectra with the spectrum of the light source was easy. It was done by simply disabling and enabling different emission spectra in the database, and matching them with the emission lines of the spectrum (fig. 9).

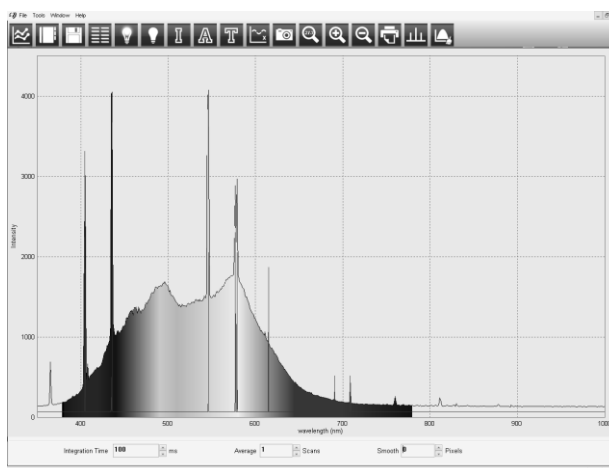


Fig. 9. The mercury emission spectrum matched with the received spectrum

After turning on the mercury emission spectrum in the software it matched the emission lines peaking from the continuous spectrum. This meant that the source contained mercury, and proved the fact that the lamp emitting the photons was a mercury lamp.

Due to the limited resolution of the spectrometer it would be very hard to precisely detect the amount of substance emitting the light, but using latest laboratory technologies it is possible to do this job with great precision.

APPLICATIONS IN CRIMINOLOGY

This method of analysis has a vast variety of applications, one of which is the analysis of elements in clues in criminology. Performing this analysis using optical methods has a lot of advantages compared to other methods.

Firstly, it's very fast. In chemical analysis for example in some cases it could take a lot of time to detect the elements making up a certain substance. Optical methods are much faster compared to some other methods. In criminology, this time could be crucial.

Secondly, optical methods are very accurate. Since the intensity depends on the quantity of a certain element, it's possible to detect the amounts very precisely.

Optical methods allow very precise and rapid detection of elements in clues.

CONCLUSIONS

The analysis of elements in clues using optical methods is a very large part of criminology. Due to the fact that atoms have their fixed emission spectra, it is possible to use them as passports in the detection of elements. Using the latest technologies it is possible to work with substances with astonishing precision. But even using standard equipment it is possible to detect certain elements in substance samples using optical methods.

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INVESTIGAȚII EXPERIMENTALE ASUPRA UNOR CARACTERISTICI FIZICO-CHIMICI DE DETERMINARE A CALITĂȚII APEI

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Caracteristicile fizico-chimice joacă un rol esențial la determinarea calității apei. Unele din aceste caracteristici sunt: conductivitatea electrică, concentrația ionilor de hidrogen, oxigenul dizolvat. Determinarea conductivității apei oferă informație asupra gradului de impuritate în eșantioanele analizate. Cercetările asupra determinării concentrației ionilor de hidrogen reprezintă gradul de aciditate sau alcalinitate a apei, caracteristici care direct influențează sănătatea populației. Lipsa de oxigen din apă duce la pierderea caracterului de prospețime, apa devenind nepotabilă. Caracteristicile fizico-chimice experimentale au fost investigate cu ajutorul senzorului multifuncțional de determinare a calității apei PS-2169.

Cuvinte-cheie: apă, calitate, conductivitate, pH, oxigen dizolvat.

Physical and chemical characteristics play an essential role in determining water quality. Some of these features are: the electrical conductivity, hydrogen ion concentration and dissolved oxygen. Determination of the water conductivity offers information of the degree of impurity in the analyzed samples. Investigation of hydrogen ion concentration provides information about the acidity or alkalinity of water, characteristics that directly influence the human health. Shortage of oxygen in the water leads to loss of character of freshness, the water becoming the undrinkable. Experimental physico-chemical characteristics were investigated with Water Quality Multi Measure Sensor PS-2169.

Key words: water, quality, conductivity, pH, dissolved oxygen.

INTRODUCERE

Apa este un nutrient de bază a corpului uman și este esențială pentru viața populației. Aceasta susține digestia alimentelor, adsorbția, transportul și utilizarea de substanțe nutritive și eliminarea toxinelor și a deșeurilor din organism. Apa este, de asemenea, esențială pentru prepararea alimentelor și cerințele pentru pregătirea alimentelor sunt incluse în discutarea cerințelor de consum [1].

Consumul total de apă include apa potabilă, apa din alte băuturi, precum și apa (umiditatea) în produsele alimentare. Un consum scăzut de apă a fost asociat cu unele boli cronice, iar un consum corespunzător de apă previne efectele nocive (în primul rând acute) de deshidratare, care includ anomalii metabolice și funcționale. Starea de hidratare este indicatorul principal utilizat pentru apă. Activitatea fizică și condițiile de mediu au o influență semnificativă asupra nevoilor de apă. Totuși, un consum excesiv al apei conduce la intoxicație, care poate pune viața în pericol datorită manifestării hiponatremiei (micșorarea cantității de sodiu în sânge) ceea ce poate duce la edema sistemului nervos central, congestie

pulmonară, și slăbiciune musculară. În circumstanțe neobișnuite hiponatremia poate apărea datorită consumului excesiv de lichide, înlocuirea sodiului, sau ambele în timpul sau după efort fizic prelungit. Hiponatremia simptomatică de exercițiu este de obicei asociată cu mai mult de 6 ore de exerciții stresante prelungite. Toxicitatea acută de apă a fost raportată ca urmare a consumului rapid al unor cantități mari de lichide care au depășit considerabil rata de excreție maximă a rinichilor de 0,7-1,0 l/oră [2].

Consumul zilnic de apă variază în funcție de diferiți factori, cum ar fi: temperatura, umiditatea, activitatea fizică și alți factori care variază larg. Consumul mediu de apă (lichid) de persoană pe zi calculat conform cercetărilor din diferite surse reprezintă 1,63 litri. Totuși, un volum mai mare, de ~2 litri/zi, a fost adoptat ca reprezentând consumul de persoană pe zi [3].

Consumul de apă potabilă variază într-un oraș modern între 100 și 530 litri pe zi de persoană [4], ajungându-se chiar și la valori de 600 litri pe zi [5]. Totuși, în anul 2007, la nivel global consumul domestic de apă constituia doar 5 %, iar în industrie fiind consumate 9 %

din cantitatea totală (în cadrul întreprinderilor, la prelucrarea materiei prime ș.a.), cea mai mare cantitate (86 %) fiind utilizată în agricultură (irigații, creșterea animalelor ș.a.) [6]. În prezent aceste valori reprezintă 8 % pentru consumul domestic, 12 % industrie și 80 % agricultură [5].

Întrucât apa reprezintă unul dintre cele mai importante elemente în viața și activitatea umană, evaluarea și asigurarea calității acesteia îi revine un rol foarte important.

Un rol deosebit în evaluarea calității apei îi revine atât indicilor de calitate (organoleptici, fizico-chimici, microbiologici, toxicologici, radioactivi, etc.), cât și metodelor de determinare a lor, prezentate în Documentele Normative în vigoare [7].

Apa plată este produsă sub control sanitar, dar proveniența ei poate fi atât de la un izvor, cât și de la un puț de adâncime, verificat și avizat sanitar anume pentru comercializare și consum. Principala calitate a apei plate, în comparație cu cea livrată de rețeaua publică de alimentare cu apă, este aceea că ea nu a străbătut țevile rețelei urbane de aducțiune și, deci, nu conține clor rezidual, nu preia și nu poartă produsele rezultate din depunerile pe țevă, din oxidarea metalului, din alte procese, foarte fine, care pot avea loc în timpul transportului [8].

Conductivitatea, pH, temperatura și oxigenul dizolvat sunt indici fizico-chimici care joacă un rol important la aprecierea calității apei. Lucrarea are ca obiectiv exemplificarea măsurătorilor acestor parametri în vederea verificării calității apei.

Scopul lucrării este efectuarea studiului de cercetare asupra indicilor de calitate a eșantioanelor analizate și corespunderea valorilor obținute cu normele stabilite în Documentele Normative.

METODICA CERCETĂRII ȘI EȘANTIOANELE

Cercetările asupra apei plate s-au efectuat pe un lot de eșantioane prelevate din ape îmbuteliate, a căror mărci comerciale sunt pe piața Republicii Moldova.

Drept indici ai proprietăților fizico-chimice a fost selectată conductivitatea electrică, determinată cu ajutorul senzorului

multifuncțional de determinare a calității apei (Water Quality MultiMeasure Sensor PS-2169) și în special cu senzorul de testare a conductivității. De asemenea, prin aceeași metodă, cu senzorul multifuncțional, s-au mai investigat și concentrația ionilor de hidrogen din apă, adică pH-ul și s-a determinat oxigenul dizolvat (DO_2), fiecărui din indicii investigați corespunzându-i senzorul cu electrod specific.

Conductivitatea (sau în mod specific, conductivitatea electrolică) este definită ca abilitatea unei substanțe de a conduce curentul electric. Este inversul rezistivității. Toate substanțele posedă conductivitate într-o oarecare măsură, dar suma variază foarte mult, de la extrem de mici (izolatoare, cum ar fi benzenul și sticla) la foarte mari (argint, cupru și metale în general). Un interes sporit în industrie este în măsurarea conductivității lichidelor care, în general, constau din compuși ionici dizolvați în apă. Valoarea conductivității acestor soluții sunt plasate între valorile izolatoarelor și a conductorilor metalici. Conductivitatea poate fi măsurată destul de ușor prin mijloace electronice, care permit un test simplu, valoarea căreia poate spune multe despre calitatea apei [9].

Pentru a începe prelevarea măsurătorilor și achiziționarea de date s-a conectat senzorul multifuncțional de determinare a calității apei la interfața PASPORT, așa cum este ilustrat în fig. 1 și ulterior la calculator și se dă click pe butonul de start pentru a începe procedura de colectare a datelor.

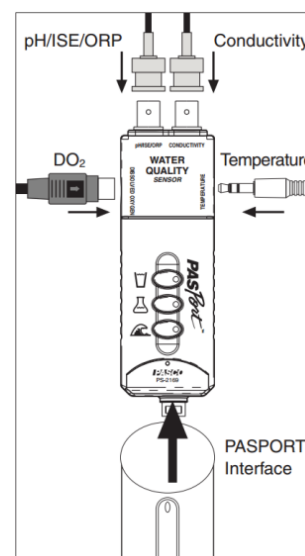


Fig. 1. Senzorul multifuncțional de determinare a calității apei cu modalitatea de conexiune la interfața PASPORT

Componenta senzorului de conductivitate (fig. 2) măsoară conductivitatea electrică a soluțiilor apoase. Ea determină conductivitatea prin aplicarea unei tensiuni alternative la celula electrodului de la capătul sondei și măsoară curentul rezultat [10].

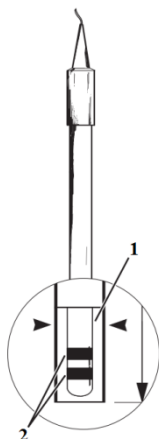


Fig. 2. Electrocul de conductivitate:
1-tijă de sticlă; 2-conductori de platină platinăți

Înainte de a utiliza electrocul de conductivitate este necesară înmuierea capătului său în apă distilată pentru 5÷10 minute, apoi, electrocul se conectează la senzorul multifuncțional și se începe prelevarea datelor nu înainte de a selecta unul dintre cele trei intervale de măsurare: de la 0 $\mu\text{S/cm}$ până la 1000 $\mu\text{S/cm}$, de la 0 $\mu\text{S/cm}$ până la 10000 $\mu\text{S/cm}$ sau de la 0 $\mu\text{S/cm}$ până la 100000 $\mu\text{S/cm}$, în cazul nostru de la 0 $\mu\text{S/cm}$ până la 10000 $\mu\text{S/cm}$. Capătul electrodului se scufundă în eșantionul analizat și se așteaptă puțin până se stabilizează citirea de date. Operațiunea se repetă pentru fiecare eșantion nu înainte ca electrocul să fie clătit în apă distilată. Instalația pentru determinarea conductivității eșantioanelor analizate este prezentată în fig. 3.

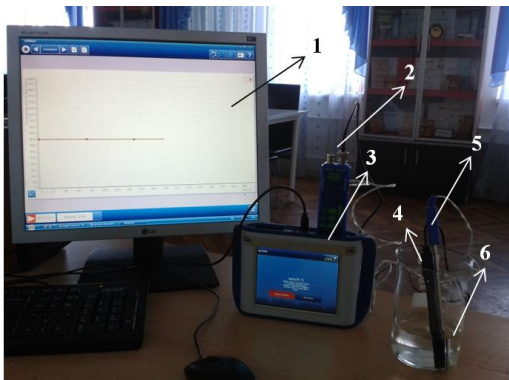


Fig. 3. Instalația de determinare a conductivității apei:
1 – calculator, 2 – senzor multifuncțional, 3 – sistem de achiziții de date (SPARK SLS), 4 – electrocul de conductivitate, 5 – electrocul de temperatură, 6 – pahar gradat cu eșantionul analizat

În metale odată cu creșterea temperaturii conductivitatea scade. Pentru a observa dependența de temperatură a conductivității în apă am conectat la multi-senzor și o probă din oțel inoxidabil pentru măsurarea temperaturii.

De asemenea, cu ajutorul conductivității se pot estima solidele dizolvate total (TDS) cu următoarea formulă de calcul:

$$TDS \text{ (mg/l)} = \frac{\sigma \text{ (}\mu\text{S/cm)}}{2} \quad (1)$$

unde σ – conductivitatea eșantionului analizat.

Determinarea valorii pH a eșantioanelor analizate a fost făcută cu electrocul pH al multisenzorului de determinare a calității apei. pH este prescurtarea de la „pondus hydrogenii” și reprezintă logaritmul cu semn schimbat al concentrației ionilor din soluție [11]. Concentrația ionilor de hidrogen al unei ape pure la temperatura camerei este 7, adică neutră. Sub valoarea de 7 apa devine acidă, iar peste valoarea de 7 devine bazică sau alcalină [12].

Electrocul pH se conectează la portul corespunzător și se începe procedura de colectare a datelor de la interfața SPARK SLS (fig. 4).



Fig.4. Instalația de determinare a concentrației ionilor de hidrogen: 1 – senzor multifuncțional, 2 – sistem de achiziții de date (SPARK SLS), 3 – electrocul pH, 4 – proba de analizat

Electrocul pH produce o tensiune proporțională cu pH-ul soluției în care este cufundat. Această tensiune este măsurată de multi-senzor, care și reprezintă pH calculat. Înaintea oricărei măsurători electrocul se clătește cu apă distilată.

Se scoate cu grijă sticluța de depozitare de pe capătul electrodului, astfel încât să nu se verse soluția de stocare. Inelul și capacul

flaconului se împing în sus, spre mânerul electrodului. Capătul electrodului se clătește cu apă distilată. Dacă se observă bule de aer în bulbul electrodului, acesta se agită ușor în jos (similar cu agitarea în jos a unui termometru). Se începe prelevarea datelor. Electrocul se plasează în soluția de măsurat și se așteaptă câteva minute până se stabilizează citirea datelor de la computer.

Determinarea cantității de oxigen dizolvat în apă este predominantă în analiza apelor, deoarece, oxigenul este necesar supraviețuirii și creșterii multor organisme. Pentru această determinare s-a folosit componenta DO_2 a multi-senzorului.

Cantitatea de oxigen dizolvat în apă depinde de temperatura apei, presiunea aerului și de conținutul în substanțe oxidabile și microorganisme. Scăderea cantității de oxigen din apă duce la pierderea caracterului de prospețime, un gust fad, devenind nepotabilă. De asemenea, scăderea oxigenului reduce capacitatea de autopurificare a apelor naturale, favorizând persistența poluării cu toate consecințele nedorite [13].

Electrodul de oxigen dizolvat este dotat cu sondă polarografică constituită dintr-un catod din Pt și dintr-un anod din Ag înconjurați de soluție de clorură de potasiu ($KCl_{(aq)}$), iar în vârf are o membrană ce separară mediul respectiv de soluția de analizat (fig. 5).



Fig. 5. Părțile componente ale electrodului de oxigen dizolvat: 1 – anod din Ag , 2 – soluție de electrolit ($KCl_{(aq)}$), 3 – carcasa sondei, 4 – izolatorul catodului, 5 – catod din Pt , 6 – membrana

Senzorul se bazează pe măsurarea curentului electric produs în urma reacției chimice care are loc pe sondă. Această reacție implică recuperarea moleculelor de oxigen (O_2) și oxidarea atomilor de argint (Ag), la anod. Printre electrozi trece o diferență de potențial

de 0,7 V, care și susține decurgerea reacției redox dorită.

Când sonda oxigenului dizolvat este plasată într-un mediu apos, cum ar fi apa, care conține oxigen, molecule de O_2 difuziază printr-o membrană subțire în soluția de electrolit, care înconjoară electrozii sondei.

Această membrană este semipermeabilă și permite trecerea O_2 dizolvat, dar nu permite trecerea majoritatea altor molecule care pot interfera cu cursul normal al reacțiilor chimice de pe electrozi. Aceste reacții produc electroni care cauzează trecerea curentului prin circuitul senzorului. Deoarece rata de difuzie depinde de concentrația oxigenului dizolvat, cantitatea de molecule O_2 va fi direct proporțională cu concentrația oxigenului dizolvat în soluția de testare. Când sonda este plasată într-un mediu apos, care conține oxigen dizolvat, moleculele O_2 difuză prin membrană și reacționează cu moleculele de apă (H_2O) în prezența electronilor de la catod, pentru a forma ioni de hidroxid (OH). Ionii de hidroxid difuzează la anod și reacționează cu atomii de argint, formând oxidul de argint (Ag_2O), H_2O , și electroni liberi. Prin urmare, numărul de electroni produși în timpul reacției redox va fi aproape direct proporțional cu concentrația de O_2 dizolvat în soluția de testare.

După procedura de calibrare se începe colectarea datelor. S-a scos flaconul de stocare de la sondă și s-a scufundat capătul sondei în soluția eșantionului. S-a agitat ușor sonda pentru prevenirea epuizării oxigenului localizat la capătul sondei și se așteaptă până se stabilizează datele. Înainte de a efectua o altă măsurare la o probă diferită electrodul s-a clătit cu apă distilată.

REZULTATE EXPERIMENTALE ȘI INTERPRETĂRI

Indicii de calitate caracteristici pentru apă oferă informația corespunzătoare calității acesteia. În acest scop, rolul principal le revin metodelor experimentale de analiză, cu ajutorul cărora se determină corespunderea acestor caracteristici cu normele stabilite în Documentele Normative. Caracteristicile fizico-chimice investigate au permis determinarea conductivității și dependența

acestea de temperatură, concentrația de oxigen dizolvat și a concentrației ionilor de hidrogen.

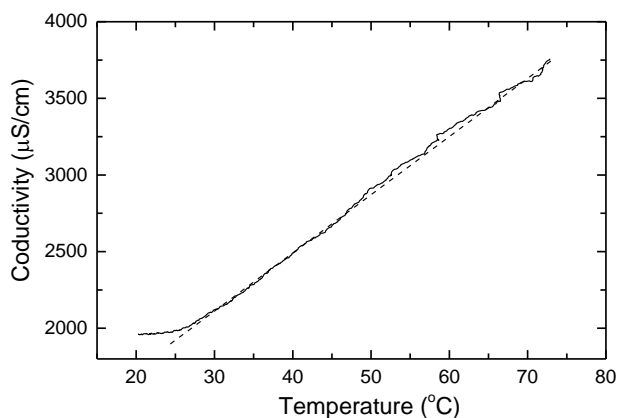
În metale, odată cu creșterea temperaturii conductivitatea scade. Pentru a observa dependența de temperatură a conductivității în apă s-a conectat la multi-senzor și o probă din oțel inoxidabil pentru măsurarea temperaturii.

În experiment, viteza de încălzire a apei cu temperatura a fost de 5,4 °C/min. În fig. 6 este prezentată dinamica conductivității apei plate în intervalul de temperaturi de la 20 până la 73 °C. Din grafic observăm o creștere liniară a conductivității apei cu temperatura, descrisă prin funcția liniară:

$$\sigma = \sigma_0 + mt$$

unde viteza de creștere a conductivității

$$m = 54,7 \mu\text{S} \cdot \text{cm}^{-1} \cdot ^\circ\text{C}^{-1},$$



iar $\sigma_0 = 1020 \mu\text{S}/\text{cm}$.

Fig. 6. Dependența conductivității de temperatură

Creșterea conductivității apei este cauzată de mărirea mobilității ionilor cu temperatura, atunci când pH este în continuă descreștere.

Toate datele experimentale obținute în urma testelor efectuate pentru caracteristicile analizate sunt prezentate în tabelul de mai jos:

Nr. Eșantion	Denumire	pH	DO ₂ (mg/l)	Conductivitatea la 25°C (μS/cm)	TDS (ppm)
1	Aqua Uniqa	8,75	13,6	536	268
2	Dorna	8,96	10,0	267	133,5
3	Gura Căinarului	8,49	13,8	2634	1317
4	Morșinskaia	8,48	18,5	179	89,5
5	OM	8,32	14,1	369	184,5
6	Varnița	8,95	18,2	1984	992

CONCLUZII

În baza experimentului efectuat asupra conductivității s-a constatat că nivelul de impuritate este mai ridicat doar la un singur eșantion, valoarea căruia a depășit norma de 2500 μS/cm, stipulată în Documentele Normative, valorile celorlalte eșantioane fiind sub limita specificată. În urma investigațiilor efectuate s-a observat că la mărirea liniară a temperaturii apei cu 5,4°C/min, conductivitatea electrică crește cu 54,7 μS·cm⁻¹·°C⁻¹. În urma determinării solidelor dizolvate total nici o probă nu a depășit limita de 1500 mg/l.

La cercetarea concentrației ionilor de hidrogen am observat că nici un eșantion analizat nu întrece limitele stipulate (6,5÷9,5), acest indice variind de la 8,32÷8,96, deci putem spune că apa este alcalină.

În cazul oxigenului dizolvat, cea mai mare concentrație o are eșantionul nr. 4 (18,5 mg/l) iar cea mai mică de 10,0 mg/l o are eșantionul nr. 2. Astfel, toate probele întrec concentrația maximă admisibilă, mai mult de 4 și mai puțin de 10 mg/l.

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REGULATION OF RADIATION TRANSMITTANCE THROUGH ELECTRO-OPTIC TECHNOLOGIES

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There are three fundamental mode of heat transfer: diffusion, convection and radiation. Although our modern windows are really good at blocking heat transferred from diffusion and convection, a lot of energy is able to pass through them in the form of electro-magnetic waves, mostly in the visible and infrared spectrum. It has been proven that even transparent curtains will afford adequate protection from optical radiation in the ultraviolet region, but that all of them will transmit high percentages of infrared radiation, while the solar irradiance is high in the infrared spectrum. Using a system of polarizers active specifically for the range of the IR spectrum with the highest irradiance and a liquid crystal cell (LCC) we can alter the energy transmittance of windows without blocking visible light. This way, using less energy than usual conditioning systems we can control the temperature of the environment, without rendering our windows opaque in the visible spectrum. This is achieved by using a system of combined Kerr and Pockels cells, with high electro-optic constant, low energy expenses and longitudinal applied voltage.

Keywords: radiation transmittance, diffusion, convection, radiation, polarizer.

Există trei moduri fundamentale ale transferului de căldură: difuzie, convecție și radiație. Deși ferestrele noastre moderne sunt foarte bune pentru blocarea căldurii transferate de la difuzie și convecție, o mare energie este capabilă să treacă prin ele în formă de unde electromagnetice, mai ales în spectrul vizibil și infraroșu. S-a dovedit că, chiar dacă perdele transparente vor permite o protecție adecvată împotriva radiațiilor optice în regiunea ultravioletă, cu toate acestea ele vor transmite un procent ridicat de radiații infraroșii, întrucât iradierea solară este mare în spectrul infraroșu. Folosind un sistem de polarizatoare, active, în mod special, pentru gama de spectrul infraroșu cu cea mai mare iradiere, și o celulă de cristal lichid (CCL) putem modifica transmisia de energie prin ferestre fără a bloca lumina vizibilă. Astfel, folosind mai puțină energie decât pentru sistemele obișnuite de condiționare putem controla temperatura mediului, fără a face ferestrele noastre opace în spectrul vizibil. Acest lucru este realizat prin utilizarea unui sistem de celule combinate Kerr și Pockels, cu constante electro-optice înalte, cheltuieli mici de energie și tensiuni joase aplicate longitudinal.

Cuvinte-cheie: transparența radiației, difuzie, convecție, radiație, polarizator.

THE PROBLEM

As noticed before, air conditionings are a good countermeasure for over-heated rooms but a bad one for ecology.

For instance, let's analyze the thermal equilibrium of a sky-craper. Almost all the outer surface of a sky-craper is covered in windows. If there were no air conditioningsystems the temperature would easily reach 40 °C. This means that a major amount of energy is lost to cool down the building.

The problem is that these air conditioning systems are fighting the heat that is already inside a closed room. Even though they are cooling down the air using the air convection effect, they are not able to combat the most important source of heat, Infrared radiation.

But, what would happen if we stopped the heat before it had entered the room?

HYPOTHESIS

We propose to decrease by half the amount of energy spent on air conditioning of the buildings with big surface of windows such as sky-crappers. We would do that by placing on the windows an additional heat-filtering layer. Also, we purpose a method that will automatically adjust the amount of filtered heat, such that the temperature inside would be constant.

GOALS AND EXPECTED OUTCOMES

The primary goals of this project are:

1) Creating an optical device (additional window layer) that can regulate the amount of

radiation which passes through the optical device.

2) Proving the fact that it is possible to control the amount of radiation which passes through the optical device using a variable AC source.

3) Finding the theoretical dependence between the voltage applied on the optical device and the energetic transmittance of the device.

4) Applying this additional layer on a regular window, in order to control the radiation passed through the window as a function of the voltage applying on the device.

5) Creating a program that can control the voltage applied on the additional layer as a function of the temperature inside the room. In this way, if the temperature inside increases, the voltage on the device is also increased and as a result, the amount of heat that passes through the window will be decreased and the temperature will be automatically

RESEARCH METHODS

The first step was analyzing the fundamental methods of heat transfer: diffusion, convection and radiation. Although our modern windows are really good at blocking heat transferred from diffusion and convection, a lot of energy is able to pass through them in the form of electro-magnetic waves, mostly in the visible and infrared spectrum.

The stages of our research:

1) Assembling a liquid crystal cell by placing a liquid crystal cell with variable birefringence between two polarizers, thus creating an optical device with a variable transmittance.

2) Then, we assembled an installation suitable for visible light that proved our theory. As the light source we used a red laser with the wavelength of 700 nm.

3) After gathering and analyzing the data, we plotted the dependence of the optical device transmittance as a function of the voltage applied on it. We noticed that the phase shift of the light passed through the liquid crystal cell is a power dependence of the voltage applied on the cell.

$$\Delta\varphi = c \cdot U^\beta \quad (1)$$

4) After analyzing the graphs we determined the parameters of our installation, like the value of β , which is $\beta = 1.85$. Finally we deduced a

theoretical dependence of the transmittance of our device as a function of voltage applied on it.

5) After obtaining a working optical device (for visible spectrum), we set our goal to create a similar device which would work in the same manner but this time with infrared light (800-1500 nm).

6) The final step was creating software that regulates the voltage applied on the liquid crystal cell as a function of the temperature inside the room. Which means that if the temperature inside raises, the voltage on the liquid crystal cell rises, and as a result the temperature decreases.

7) Explaining the way we can use the technology and assemble a smart window.

DETAILED EXPLANATION OF EACH STEP

We assembled a liquid crystal cell, which is a component of our optical device.

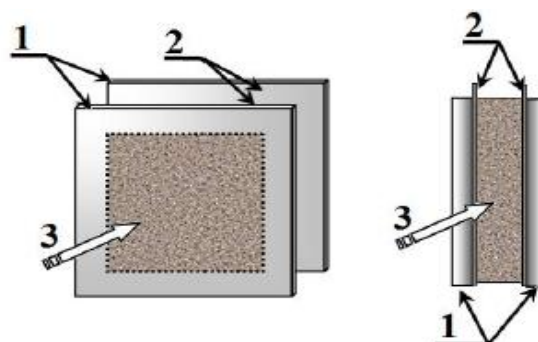


Fig. 1. Two thin polycarbonate protective layers (1), inner surface of the cell which is covered with zinc oxide doped with aluminum (which is used as a transparent conductor) (2), saturated KH_2PO_4 solution (3)

The cell is made up of two polycarbonate protective layers that are covered on the inner side with a transparent conductive material (zinc oxide doped with aluminum). And inside a saturated KH_2PO_4 solution is found.

We chose specifically the KH_2PO_4 solution because after analyzing many solutions that have a variable birefringence like nitrobenzene, Lithium Niobate and others, we came to the conclusion that the solution not only has an optimal electro-optical constant, but is also cheap and available.

EXPERIMENT

The experimental installation sketch is shown in fig. 2.

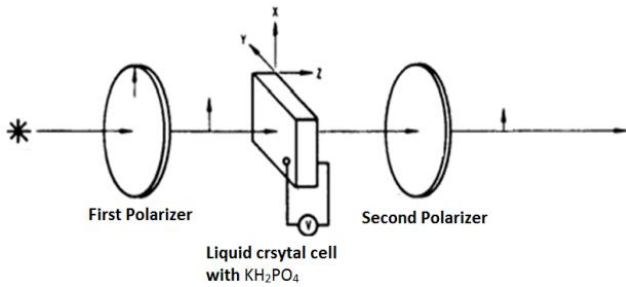


Fig. 2. Experimental installation sketch

The main goal of this experiment is to prove that radiation transmittance is a function of the voltage applied on the device. Our optical device represented as 3 independent layers (2, 3, 4) are shown in the fig. 3.

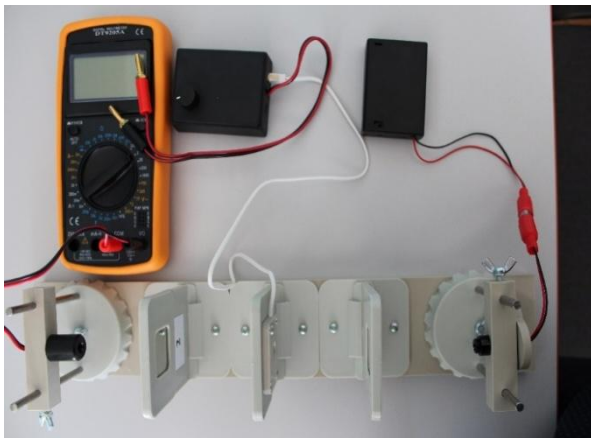


Fig. 3. Optical device

In the photo (from left to right):

- 1) Photoreceptor that gives voltage output;
- 2) Polarizer 2;
- 3) Liquid crystal cell;
- 4) Polarizer 1;
- 5) A red light source (700 nm).

Also we have a multi-meter for calculating the voltage on the photoreceptor, and also a variable AC source for the liquid crystal cell.

First and second polarizers have parallel polarization planes.

Then we start our experiment:

- Initially the voltage on the cell was zero, so the light passes without meeting any obstacles.
- Then we start to apply voltage on the cell and we notice that the voltage on the photoreceptor decreases.

- We measure with the voltmeter the dependence of the voltage on photodetector as a function of the voltage applied on the liquid crystal cell, and then we plot the data.

To determine the intensity of the light that emerges out of our optical device we used the following formulas, which we deduced from Malus law:

$$I_{emerged} = I_0 / 2 \cdot \sin^2(\Delta\phi/2) \tag{2}$$

We also know that $I \sim U$, thus we obtain:

$$(\Delta\phi) = \arcsin \sqrt{\frac{U}{U_{max}}} \tag{3}$$

where $\Delta\phi$ is the phase shift created in the infrared wave after it passed through the birefringent medium.

In order to find the theoretical formula for $I_{emerged}$ as a function of voltage applied on the optical device, we analyze the graph of the voltage applied at the liquid crystal cell as a function of the voltage on the photoreceptor. Then we plot the graph (fig. 4).

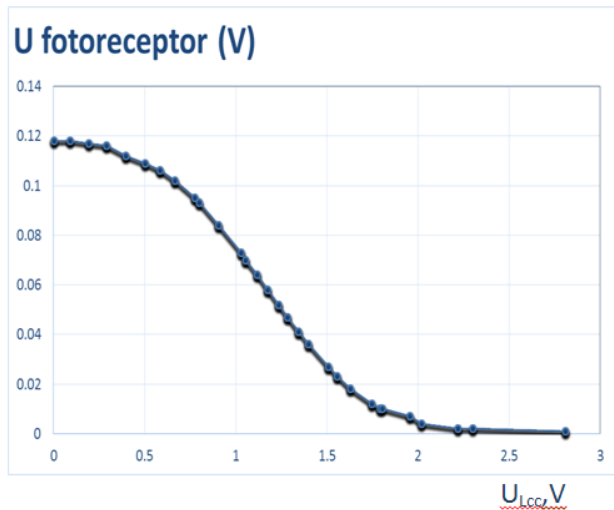
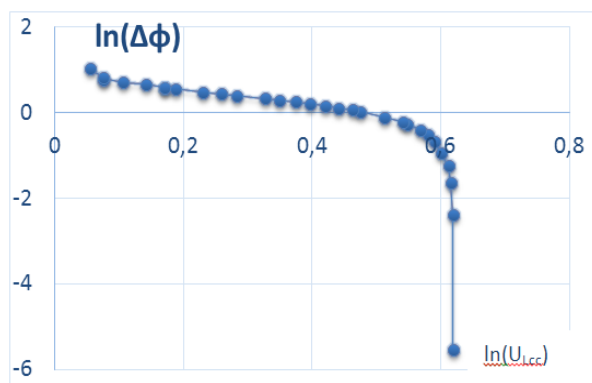


Fig. 4. Graph of the voltage applied at the liquid crystal cell as a function of the voltage on the photoreceptor

Then we supposed that the phase shift is a power function of the voltage applied on the liquid crystal cell (formula (1)).

Then in order to determine the β constant, we plot the graph of $\ln(\Delta\phi)$ function of $\ln(U_{LCC})$ (fig. 5).


 Fig. 5. Dependence $\ln(\Delta\phi)$ function of $\ln(U_{Lcc})$

So, from the graph above we find that the tangent of the line is β , and its value is $\beta=1.85$. It's a quite interesting result, because using a solution of KH_2PO_4 and applying transversal voltage, we created a cell, similar to a Pockels one, but in which the difference of refractive indexes depends on the voltage applied as roughly U^2 which is similar to a Kerr cell.

The final formula we obtained, which defines the Intensity of the light that emerges from the optical device as a function of the voltage applied on the liquid crystal cell.

$$I_{\text{emerged}} = I_0/2 \cdot \sin\left(\frac{c \cdot U^{1.85}}{2}\right) \quad (4)$$

After obtaining the final formula, we now know the dependence and we created a similar device, but whose aim is to block specific wavelengths, infrared wavelengths (750-1500 nm). This is important because many buildings are heated not only because of convection or conduction, but also due to the radiation. In fig. 6 the red zone is occupied by radiation which passes the atmosphere and thus can reach the windows. A significant surface of the "red zone" is in the infrared spectrum which means that by regulating this radiation, we could adjust the temperature of buildings.

We propose software which would relate the voltage applied on the electro-optic cell and the desired temperature inside the room. Also, the software has a temperature regulating function; that would mean that if you set a desired temperature, for example 25 °C, the window will be auto-regulating the voltage on the optic device in such a way, that the amount of infrared radiation passed through the window will be able to heat the room maximum to 25 °C.

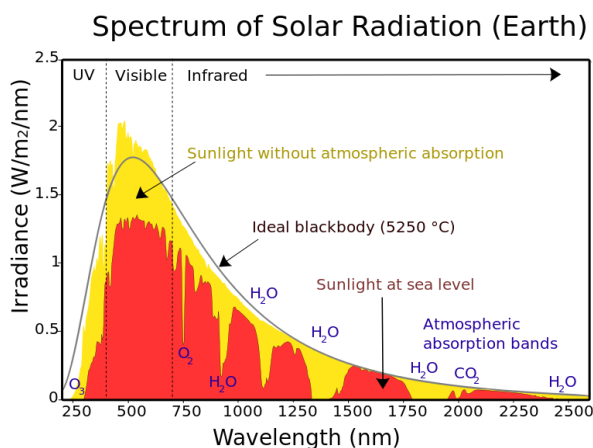


Fig. 6. Spectrum of solar radiation at the Earth

This is how it works:

The temperature inside the room is read by a device named thermo-copper, which works on the Seebeck effect. Temperature is represented as a voltage. Our software has one input and one output. For the input we have the voltage from the thermo-copper. The output is the voltage on the optical device. We then set the device to execute one simple command, to keep the ratio:

$$\frac{\text{Voltage from the thermo-copper}}{\text{Voltage on the optical device}} = \text{constant} \quad (5)$$

The whole idea is that when the temperature inside a building is higher than the desired one, the system varies the applied voltage such that the transmittance of the infrared radiation decreases and therefore the temperature decreases.

Explaining the way we assemble a smart window.

To revise: a smart window will be composed of a regular window with an additional layer that we created. The layer will be made up of small and thin "optical devices" connected in parallel such that the voltage would be the same on every "optical device".

The efficiency of this product is much higher than the efficiency of the air conditionings, because it is working at small voltages (1-5 volts) and don't require big amounts of energy.

Take for example a large 12,000 BTU air conditioner, running six hours each day will consume 270 kWh, at a cost of \$36.26 per month. However, our Smart window will consume the maximum amount of 15\$.

RISKS AND SAFETY

The system will operate on a maximum value of 10 Volts (AC). The created electric field inside the windows will be of 10 to the 6th magnitude. Although the intensity is high, it will act only on a small interval of 10 microns, which is unreachable for humans. On the other hand, the electric field intensity outside the window will be 0, so will pose no threat for the user. Also in case of a leakage, the substances used in the windows are non-toxic and can be easily be cleaned.

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THE EXPERIMENTAL STUDY OF REAL AND IDEAL HARMONIC OSCILLATORS**Evtodiev Ig.¹, Koss A.², Evtodiev S.^{3*}**¹Moldova State University, 60 A. Mateevici, MD-2009, Chisinau, Republic of Moldova²Theoretical High School „Orizont”, 22/1 N. Dimo Str., MD-2004, Chisinau, Republic of Moldova³Î.M. „Didact Vega” SRL, Interdisciplinary Science Center, 200 Ștefan cel Mare și Sfint Ave., MD-2004, Chisinau, Republic of Moldova*e-mail: didact.vega@yahoo.com

The following research was dedicated to studying and experimenting with harmonic oscillators of all kinds. The used equipment was a portable computer, the “PASCO Spark”, a force sensor, 3 different springs and weights. The portable computer is responsible for storing the data received from the force sensor, and constructing a “Force-Time” graph. The force sensor has a range of ± 50 N, an accuracy of 0.1 N and a recording speed of up to 1000 Hz. It is also equipped with a reset button to automatically set it to 0 N. The springs used in the experiment differed in damping. The first spring had a very low damping coefficient, so after the weight was released they continued oscillating for a very long time, fading away only very slightly. The second spring had a medium damping coefficient, so the time it took for the oscillations to fade was much shorter, then with the first spring. The third spring however had a very high damping coefficient, so the oscillations only continued for about 6 seconds. The experiment itself was performed by hanging a weight on the spring, and starting the data recording as soon as the weight is dropped. In all 3 experiments the data was recorded at a frequency of 100 Hz. Afterwards, the computer built “Force-Time” graphs for every experiment. This allowed us to visualize the difference between the 3 springs. The computer used in the experiment has a very useful ability of generating functions for graphs. This can be done in order to study ideal harmonic oscillations. The function generated from the graph continues forever, and can be used for further research in the field of beats, which are automatically calculated and simulated by the computer. Thanks to the precise data recording equipment human errors can be minimized, which leads to very little inaccuracies in calculations.

Keywords: harmonic oscillator, sensor, accuracy, simulation.

Cercetări de față sunt dedicate studierii și experimentării cu oscilatoare armonice de toate tipuri. Echipamentul utilizat era calculator portativ „PASCO Spark”, senzor de forță, 3 arcuri diferite și greutăți. Calculator portativ este responsabil de stocarea datelor primite de la senzorul de forță și de construirea dependenței „Forță-Timp”. Senzorul de forță are diapazonul ± 50 N, precizie de 0,1 N și viteza de înregistrare de până la 1000 Hz. Acesta este, de asemenea, echipat cu un buton de resetare pentru a-l ajusta automat la 0 N. Arcurile folosite în experiment diferă după coeficientul de elasticitate. Primul arc avea coeficientul de elasticitate foarte mic, astfel încât după ce arcul era încărcat cu greutate ei continua să oscileze pentru un interval de timp îndelungat, dispărea doar foarte încet. Al doilea arc avea coeficientul de elasticitate mediu, astfel timpul de amortizare era mult mai scurt, decât la primul arc. Însă al treilea arc avea coeficient de elasticitate foarte mare, întrucât oscilațiile au continuat numai aproximativ 6 s. Însăși experimentul a fost efectuat prin afirmarea greutății de arcul, iar înregistrarea datelor a început îndată ce greutatea era eliberată. În toate cele 3 experimente înregistrarea datelor a fost efectuată la frecvența de 100 Hz. Pe urmă calculatorul construia dependența „Forță-Timp” pentru fiecare experiment. Aceasta ne-a permis să vizualizăm diferența dintre cele trei arcuri. Calculatorul utilizat în experiment are o capacitate foarte utilă de a genera funcții pentru grafice. Acest lucru poate fi făcut în scopul de a studia oscilații armonice ideale. Funcția generată din grafic este incontinuuă și poate fi utilizată pentru continuarea cercetărilor în domeniul bătilor, care sunt calculate automat și simulate la calculator. Datorită echipamentului precis de înregistrare a datelor erorile umane pot fi minimizezate, ceea ce duce la erori foarte mici în calcule.

Cuvinte-cheie: oscilator armonic, senzor, precizie, simulare.

INTRODUCTION

The following article studies real and ideal harmonic oscillators. With the help of the experimental equipment I was able to record accurate data, do some research and visualise the difference between ideal and real harmonic oscillators.

To understand the concept of harmonic oscillators, let's see their official definition. A

harmonic oscillator is a system that, when displaced from its equilibrium position, experiences a restoring force, F , proportional to the displacement, x . This can be put into the following equation:

$$\vec{F} = -k\vec{x} \quad (1)$$

This formula is derived from Newton's second law of motion.

IDEAL HARMONIC OSCILLATORS

For simple (or ideal) harmonic oscillators the damping force is taken as 0, so the system only depends on the mass of the oscillating object and the distance from the point where $x = 0$. With this in mind Newton’s second law of motion takes the following form:

$$F = ma = m \frac{d^2x}{dt^2} = m\ddot{x} = -kx \tag{2}$$

By resolving the equation we are able to derive a function describing the motion of the oscillator:

$$x(t) = A\cos(\omega t + \varphi) \tag{3}$$

$$\omega = \sqrt{\frac{k}{m}} = \frac{2\pi}{T}$$

The oscillator’s motion is repeating continuously with amplitude A. The period of one oscillation can also be found from the following equation:

$$T = 2\pi\sqrt{\frac{m}{k}} \tag{4}$$

Or, if the frequency of the oscillations is known, the time would simply be equal to the inverse of the frequency:

$$T = \frac{1}{f} \tag{5}$$

The position of the oscillator also depends on the phase, φ , which determines the starting point on the sine wave. Ideal harmonic oscillations continue forever with the same amplitude due to the fact that damping is not present.

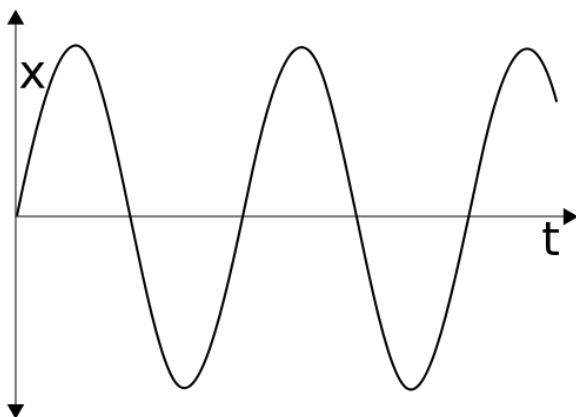


Fig. 1. A simple $x(t)$ graph describing the motion of an ideal harmonic oscillator

REAL HARMONIC OSCILLATORS

But in reality it is not possible to find ideal harmonic oscillators, since there will always be a friction force. Due to this force the velocity will decrease in proportion to the friction force acting on it. This relation can be shown in the following equation:

$$F_f = -cv, \tag{6}$$

where c is the viscous damping coefficient.

So the balanced equation with all the forces included will look like this:

$$F = F_{ext} - kx - cv, \tag{7}$$

where F_{ext} is the external force applied on the system.

Since friction can’t be reduced to 0, in reality only such oscillators can exist, which gives them their name, “real oscillators”.

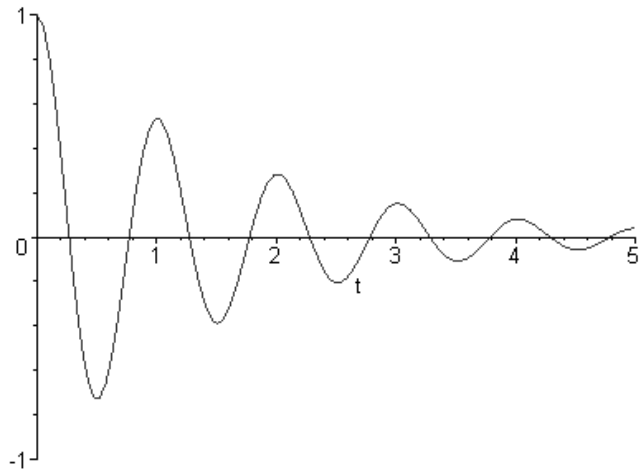


Fig. 2. An underdamped real harmonic oscillator gradually slowing down with time

The best way to illustrate a real harmonic oscillator would be a simple setup of a weight, attached to a fixed spring, oscillating up and down.

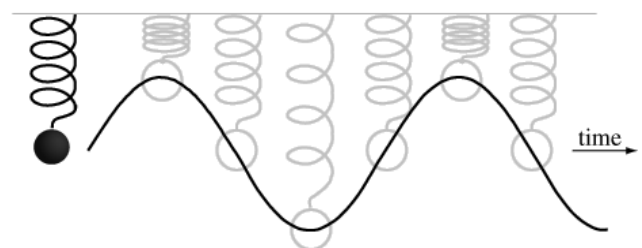


Fig. 3. An illustration of a harmonic oscillator in motion

EQUIPMENT

To do experiments with harmonic oscillators we used two different springs and weights, along with a force sensor and a computer. The spring was attached on the force sensor, and the weight was hung on the spring. The computer used in the experiment was the “Pasco SPARK science learning system”, and the force sensor was the “Pasco PASPORT”.

Pasco SPARK Science Learning System:

SPARK Science Learning System is an all-in-one mobile device that seamlessly integrates the power of probeware with inquiry-based content and assessment. With its large, full-color display, finger-touch navigation and completely intuitive data collection and analysis capabilities, SPARK completely redefines the concept of easy-to-use, so the focus remains on the learning of science.

- Full color, large screen for easy viewing;
- Finger-touch navigation – no stylus, ever!
- Simple two-button design;
- Temperature and voltage sensors included;
- Able to connect with a variety of sensors to work with all fields of science;
- More than 60 free guided inquiry SPARK labs pre-installed.

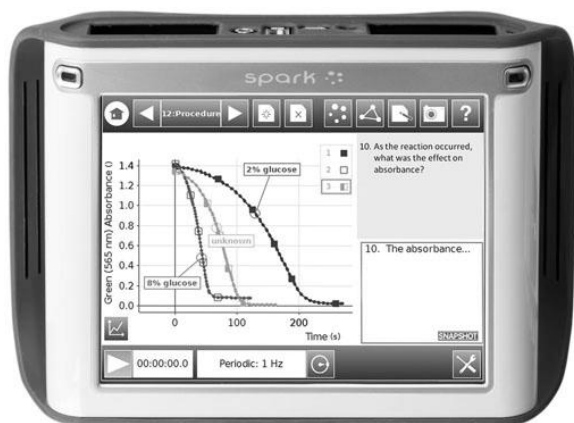


Fig. 4. The Pasco SPARK science learning system

Pasco PASPORT force sensor:

The study of force is critical to many science explorations. An accurate and rugged sensor will ensure your students get the most out of their force experiments. An overload stop in the force beam and a polycarbonate plastic case protect the unit from student abuse.

- Easy to zero - just press the button and the zero setting is stored digitally. No confusing manipulations of data are necessary.
- Measures forces in the direction of the sensor - side forces are minimized.
- Able to connect with the Pasco SPARK Science Learning System.
- Range: ± 50 N.
- Accuracy: 1 %.
- Resolution: 0.03 N.
- Maximum data collecting frequency: 1000 Hz.



Fig. 5. The Pasco PASPORT force sensor

EXPERIMENTAL SETUP

The experimental setup is fairly simple and does not require a lot of time to prepare. On an elevated spot, the force sensor is hung. A spring with a weight on it is placed on the hook of the force sensor. The sensor is then connected to the virtual laboratory.

The computer is programmed to generate a Force-Time graph as soon as the measurements start. Before performing the experiment the force sensor is set to zero to make it easier to perform calculations and observations. As soon as everything is ready, the weight is elevated at a height and released to bounce on the spring.

The recording process is started at the same time. After a few seconds of bouncing, the graph can already be seen and analysed.

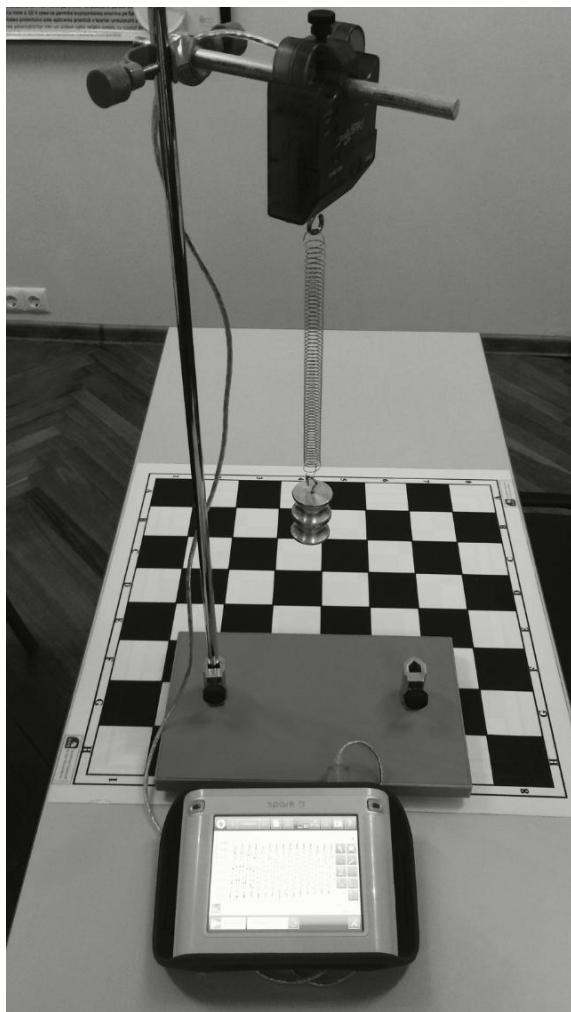


Fig. 6. The experimental setup in action

EXPERIMENT

After preparing the stand with all the equipment put in place and turned on, we started with a spring with a very low damping coefficient and a small weight. We hung the weight on the spring and reset the force sensor using the reset button on it, so that the spring hanging with the weight on it would be 0 N. Then we carefully lifted the weight to a height of a few centimetres, and released it.

At the same time on the computer we began the data collection at a rate of 100 Hz. The graph was set to be a Force-Time graph. After leaving the system oscillating for a few seconds the graph was ready.

The graph proves that the oscillator is damped only very slightly, since the force amplitude is practically staying the same. Of course, if the experiment was long enough, a decrease in motion would be clearly visible. In 7 seconds 12 oscillations occurred.

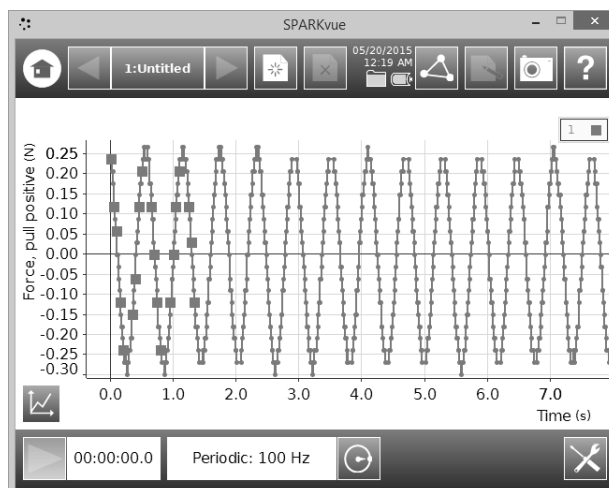


Fig. 7. The resultant graph of an almost perfect real harmonic oscillator

Using this data we can calculate $T = \frac{7}{12} \approx 0.58s$.

From such a graph it is very easy to calculate the time of one oscillation. Thanks to the precise measurements made by the laboratory we can clearly see the amount of oscillations and time.

To compare the resultant graph to ideal harmonic oscillations a function has to be created from the graph. Luckily, the virtual laboratory has such a function built into it. Since we already know that harmonic oscillators behave in a sinusoidal pattern, a sine fit function would fit the graph.

The function is generated using average values of the graph, and the overlapping of the function and graph show how much the real oscillator is different from an ideal one.

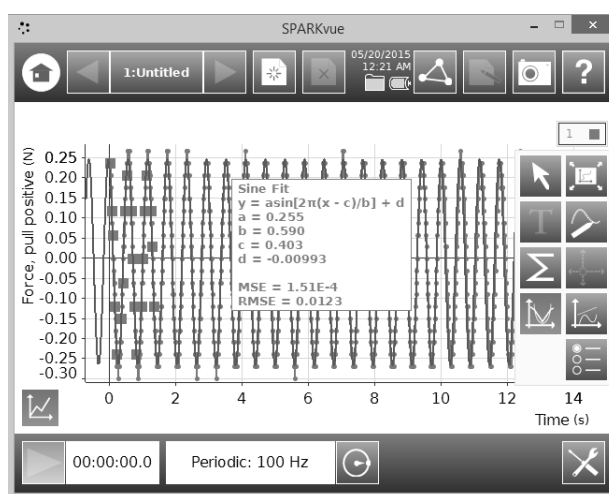


Fig. 8. Graph with a Sine Fit function overlapping it

This image again proves that the oscillations were practically perfect, since the function almost perfectly overlaps the graph.

After getting this result, we decided to redo the experiment with a different, fairly damped spring and larger weight. So we removed the previously installed spring and weight and installed the new ones. Again, we reset the force sensor to 0 N, saved the previous graph and opened a new, fresh one.

Already a few seconds after we released the weight we could clearly see that the damping was pretty strong and the oscillations were gradually slowing down. In a very short time the oscillations faded almost completely.

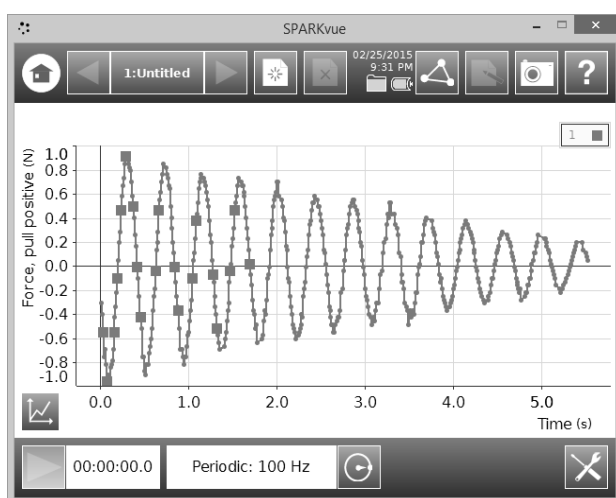


Fig. 9. The resultant graph of an underdamped mechanical oscillator

In this graph it's clearly visible how the oscillator was gradually slowing down throughout the experiment due to the damping.

After generating a Sine Fit function for the second graph it was clearly visible how much the oscillations were being slowed down by the damping of the system.

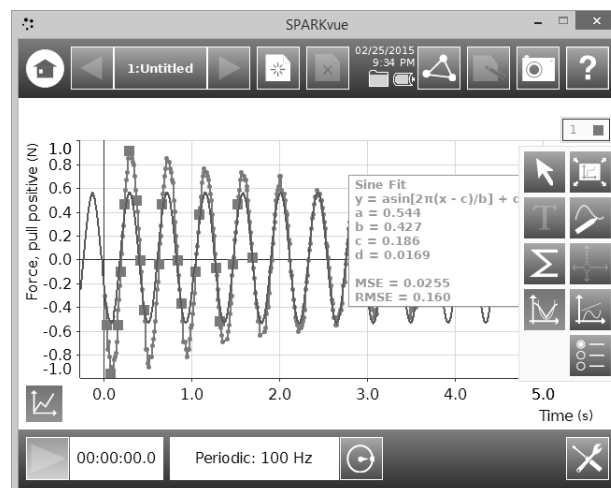


Fig. 10. Sine Fit function generated from average values of the graph

Since the average values were taken to generate an ideal harmonic oscillator function, its initial values differ from the graph. But nevertheless, observing this clearly shows the effect of damping on a harmonic oscillator.

CONCLUSIONS

Harmonic oscillations are a very interesting field of study and research. It's definitely fun to play around with them while performing calculations and measurements. In this experiment we observed real harmonic oscillators and compared them to their ideal counterpart.

Thanks to the great equipment it was very easy to explore this subject of study and to do precise calculations.

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FABRICATION OF TiO₂ NANOTUBULAR MEMBRANES OPENED FROM BOTH ENDS BY ELECTROCHEMICAL ANODIZATION TECHNIQUE

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Titanium dioxide is one of the most studied materials nowadays. TiO₂ nanotubular membranes find their applications in solar energy conversion, hydrogen sensing, catalysis for decomposition of organic materials, in biomedical applications, etc. In this paper we propose an electrochemical cost-effective method for obtaining both-ends opened nanotubular membranes. The method implies several stages: the first anodization step for a short period of time followed by removal of the formed oxide, the second anodization for growing the membrane, and a final stage of detaching the membrane from the metal surface. By choosing the appropriate parameters for each stage we can fabricate nanotubular membrane with large specific surface and identical nanotube sizes.

Keywords: titanium dioxide, free-standing nanotubular membrane, electrochemical anodization.

Bioxid de titan este unul dintre cele mai studiate materiale în prezent. Membranele nanotubulare de TiO₂ își găsesc aplicabilitate în conversia energiei solare, senzori de hidrogen, cataliză pentru descompunerea materialelor organice, biomedicină, etc. În această lucrare noi propunem o metodă electrochimică rentabilă pentru obținerea membranelor nanotubulare cu ambele capete deschise. Metoda presupune mai multe etape: prima etapă de anodizare pentru o perioadă scurtă de timp, urmată de îndepărtarea oxidului format, a doua etapă de anodizare pentru creșterea membranei și o etapă finală de desprindere a membranei de pe suprafața metalului. Prin alegerea parametrilor corespunzătoare pentru fiecare etapă se poate fabrica membrane nanotubulare cu suprafață specifică mare și dimensiuni identice nanotuburilor.

Cuvinte-cheie: bioxid de titan, membrană nanotubulară separată, anodizare electrochimică.

I. INTRODUCTION

Titanium dioxide is a wide band gap semiconductor, with high chemical stability and unique functional properties.

In recent years, more and more attention has been paid to TiO₂ nanotubes due to their advantages over TiO₂ thin films, like larger surface-to-volume ratio and unidirectional electrical channel with fewer grain boundaries. Today, anodic oxidation appears to be the distinguishing alternative due to its simplicity, low cost, self-ordering process and easily controllable nanotube morphology. Without special treatment, the TiO₂ nanotubes prepared by anodization are generally attached to the Ti substrate and their bottom ends are closed. One end opened membranes are quite limited in applications, and an eventual opening process for the bottom end is absolutely necessary.

In order to open the bottom end of the membranes many attempts have been performed, like ultrasonic splitting [1], selective dissolution of the metallic Ti

substrate, and selective dissolution of the amorphous layer between nanotube array and Ti substrate [2].

In spite of considerable research efforts, the bottom ends of the nanotubes remain partially closed, or the membrane is being destroyed. Chemical etching of the nanotube bottoms in acid vapors is an often used method for this goal. In this paper, we propose a simple way of obtaining both ends opened membranes using a single anodization step. By this way, bottom end opening process in hazardous acid vapors will be avoided, the second end of the membrane being opened during the fabrication process, detaching already both ends opened membrane.

The used electrolyte consists of a mixture of etilenglycol, ethanol and NH₄F, with a ratio of 50:2:1. The nanotube's growing process is initiated under anodic voltage increase from 0 to 120 V at a rate of 1 V/s and then maintained for 2 h [3]. At the end, a higher voltage is applied to detach the membrane from the Ti substrate.

II. EXPERIMENTAL PART

For nanotubular TiO_2 membranes fabrication Ti foils with a thickness of 250 μm and purity of 99.7 % were subjected to anodic oxidation. At the first step, the Ti foil was degreased in acetone and isopropanol in ultrasound bath and then rinsed in distilled water followed by drying in nitrogen atmosphere.

The electrochemical anodization process occurs by interaction of electrolyte with the surface of the Ti foil (99.7 % purity) under the influence of electrical field.

The electrolytic cell consists of a system with two electrodes. As electrolyte we used a mixture of ethanol, NH_4F and etilenglycol. The Ti foil is connected in the circuit as anode, and a platinum mesh electrode is used as cathode. During the anodization process, the electrolyte is mechanically mixed by a magnetic stirrer. Temperature control is secured by using a cryostat.

The schematic representation of the set-up for TiO_2 membrane fabrication is illustrated in fig. 1.

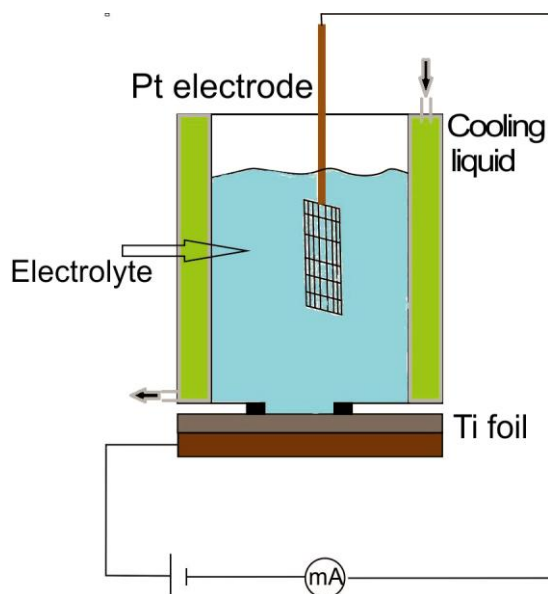


Fig. 1. Schematic view of the set-up used for TiO_2 tubular membrane fabrication

From the electrical source we apply a growing potential with a rate of 1V/s from 0 to 120 V and maintained for 2 h. In order to detach the membrane from the Ti substrate, a voltage as high as 100 V was applied for 20 s.

III. RESULTS

Nanotubular structure formation under anodic oxidation process results after Ti ions migrates from the regions between pores to the surface. At high voltage of oxidation, electrical field value is high enough to be able to mobilize the ions, and as a result ions migration leads to voids formation between pores, which contribute to the tubular structure formation [4].

TiO_2 nanotubes formation process (fig. 2) is influenced by many factors as electrolyte composition and concentration, electrolyte temperature, oxidation time, electrical voltage applied during the growing process. Nanotubes formation process by anodic oxidation technique occurs in several stages. First, the Ti foil was degreased in acetone and isopropanol in ultrasound bath, rinsed in distilled water followed by drying in nitrogen atmosphere. After this process, a first anodization step for 15 min at 120 V was applied and then etching of this oxide was realized to obtain a textural surface. The second anodization step took place for 2 h in the same conditions. The final stage, consisting of detaching the membrane from the metal surface, occurs when a 220 V potential is applied for 20 s.

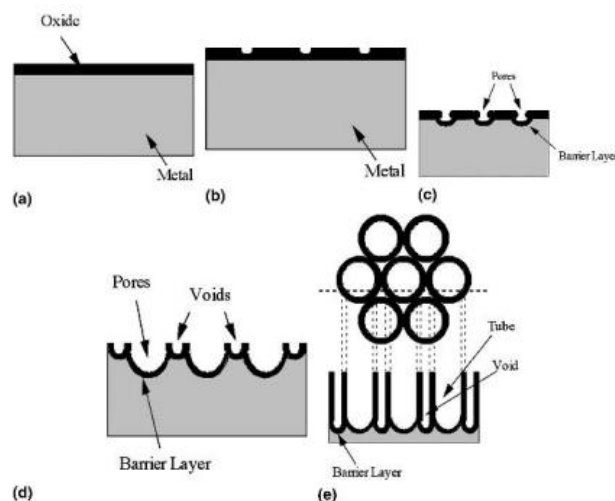


Fig 2. Schematic illustration of TiO_2 nanotubes formation process: (a) oxide continuous layer formation on top of the Ti foil surface; (b) voids formation in the oxide layer after the voltage is applied; (c) voids growing in pores; (d) oxidation and dissolution of the metallic area between pores in the presence of external electrical field; (e) nanotubes formation [5]

Fig. 3 illustrates SEM images of nanotubular membranes obtained as a result of electrochemical anodic etching. Electrolyte concentration plays an important role in the membrane detaching from the substrate. For instance, fig. 3a shows that barrier layer is still present on the bottom end of nanotubular membrane. By increasing the oxidation time, it is possible to reach thickness of membranes as high as 50 μm and even more without any modifications in the nanotube walls thickness or diameter. Also, the process temperature has an important role. In particular, we observed that at temperatures of about 30 $^{\circ}\text{C}$ the growth process of TiO_2 nanotubular membranes occurs well, but increasing the temperature to 40 $^{\circ}\text{C}$,

the surface of the membranes becomes not homogeneous, the nanotubes being separated, and nanotube etching occurs (fig. 3c). In the fig. 3d one can observe the membrane surface which was in contact with the electrolyte, and in the fig. 3e it is shown the opposite side of the membrane after optimizing process parameters, which had the barrier layer oxide. The membrane was detached from the barrier layer oxide by applying a high voltage after the growth process finished. Increasing the voltage, the nanotube walls become thinner and, at a certain time point, the membrane has no connections with the metal substrate, being released in the electrolyte solution.

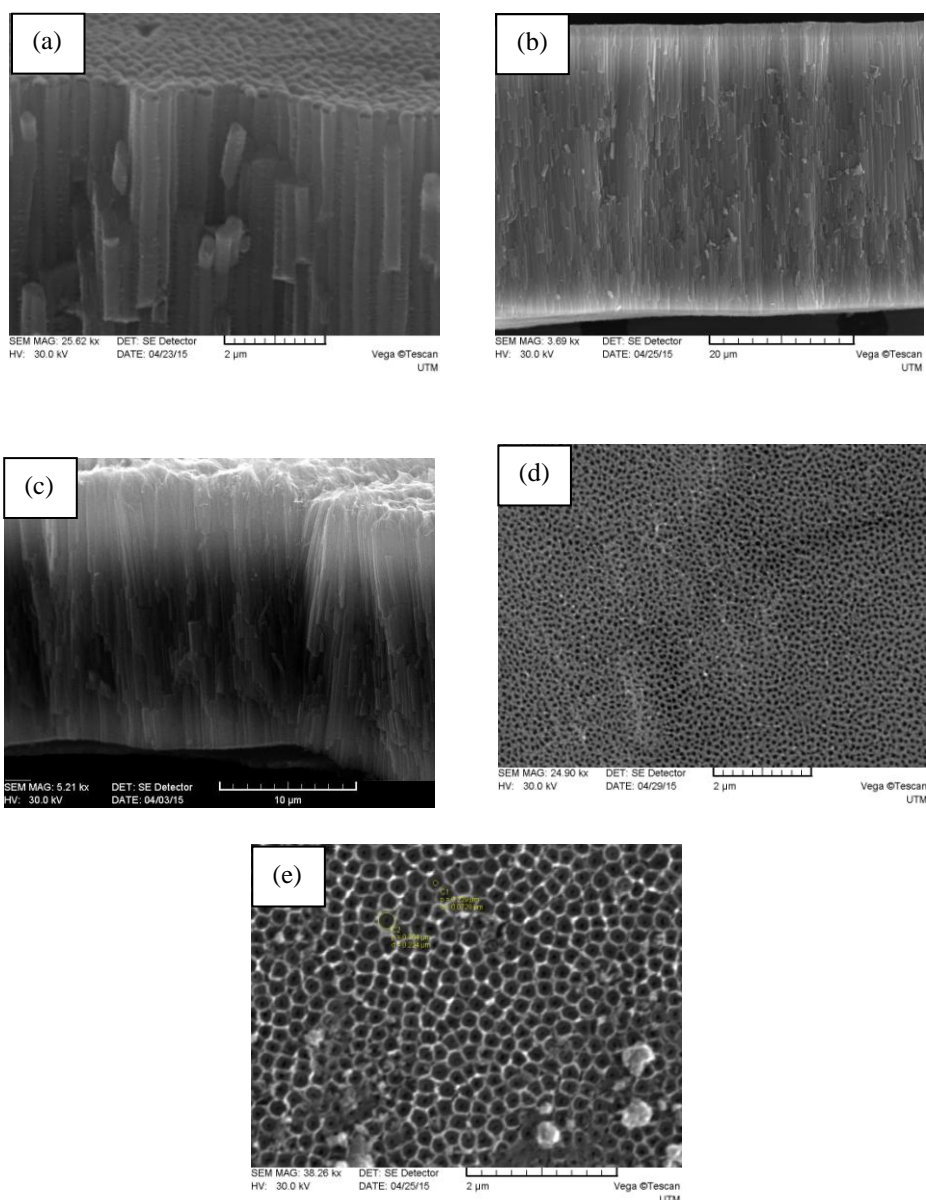


Fig. 3. SEM images of nanotubular membranes obtained without using ethanol in electrolyte composition (a); setting the electrolyte temperature at 30 $^{\circ}\text{C}$ (b) and 40 $^{\circ}\text{C}$ (c); top (d) and bottom (e) surfaces of a membrane after optimizing parameters

From the fig. 3d and 3e it is possible to see that the nanotubes diameter at the bottom side of the membrane is about 70 nm for the interior diameter and around 200 nm for the exterior one. We suggest that this is due to the gradient concentration of F^- ions inside the nanotube. To avoid this phenomenon it is proposed for the future experiments to increase the stirring rate during the growth process.

CONCLUSIONS

In this work, we demonstrated a cost effective method for obtaining both ends opened TiO_2 nanotubular membranes. An important advantage of the method is that an organic electrolyte is used for Ti foil oxidation instead of using hard acids. Opening the bottom end of the nanotubular TiO_2 membranes in the same technological process is both time- and cost- effective, making TiO_2 nanotubular membranes more accessible for future applications as templates.

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INPUT PARAMETERS INFLUENCE ON PARTS HEIGHT DEVIATION OBTAINED THROUGH BLOCK DRAWING

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The cold plastic working continue to develop compared to machining by molding or hot deformation. A number of advantages, both technical and economic, can be obtained by the plastic deformation. The experimental researches were carried out for three materials: aluminum, brass and copper. There are presented the influence of the number of whirls, of the discharge energy and of the discharge voltage on the deviation from the height of the part for different thicknesses of the sample. In case of aluminum there is a significant decrease of the deviation from height if the sample is of 0.15 mm thickness and a reduced decrease when using material with a thickness higher than 0.15 mm. When the brass material was used the increase of discharge energy leads to a significant decrease in height deviation of the parts. At different values of discharge energy the increase of the number of whirls leads to the almost comparable decreases of the deviation when the thickness was 0.15 mm and 0.5 mm. In the case of copper the increase of the discharge energy leads to the decrease of height deviation of the part with comparable values for the two thicknesses of the used material. The same observation can be drawn in case of the influence of the number of whirls on the deviation from the part's height.

Keywords: parameter, height, deviation, part.

Prelucrarea plastică la rece continuă sa se dezvolte, comparativ cu prelucrarea prin turnarea sau deformare la cald. Deformarea plastică poate duce la obținerea unor avantaje atât tehnice cât și economice. Cercetările experimentale au fost efectuate pentru trei materiale: aluminiu, alamă și cupru. Sunt prezentate influența numărului de spire asupra abaterii de la înălțime a piesei, influența energiei de descărcare asupra abaterii de la înălțime a piesei și influența tensiunii de descărcare asupra abaterii de la înălțime a piesei pentru diferite grosimi ale semifabricatului. În cazul aluminiului se înregistrează o scădere semnificativă a abaterii de la înălțime în cazul în care grosimea semifabricatului este de 0,15 mm și o scădere mai puțin intensă atunci când se utilizează un material de grosime mai mare de 0,15 mm. În cazul în care a fost utilizat materialul alamă creșterea energiei de descărcare conduce la o scădere semnificativă a abaterii de la înălțime a piesei. La diferite valori ale energiei de descărcare, creșterea numărului de spire a dus la scăderi aproape comparabile ale abaterii în cazul în care grosimea este de 0,15 mm și 0,5 mm. În cazul cuprului creșterea energiei de descărcare duce la scăderea abaterii de la înălțime a piesei, cu valori comparabile pentru cele două grosimi ale materialului folosit. Aceeași observație poate fi trasă și în cazul influenței numărului de spire asupra abaterii de la înălțime a piesei.

Cuvinte-cheie: parametru, înălțime, abatere, piesă.

INTRODUCTION

The electromagnetic forming process is based on the use of a concentrated source of energy which releases the workpiece. At the moment, due to increased accumulation of practical and theoretical research results, there are various construction-tool devices that allow high concentrations and thus electromagnetic energy flow distorting a certain areas of the piece. Magnetic shaping applications were extended peak areas such as automotive, civil engineering, aviation and space, etc.

The process is based on the electromagnetic induction phenomenon. According to this phenomenon, when a coil is driven by an electric current $i(t)$, that varies with the frequency f , creates in the space around it a magnetic field $B(t)$ of the same

frequency. If this field is inserted inside a massive piece (wire) made of a material with good electrical conductivity, the eddy currents are induced therein (also called Foucault currents) which have the same frequency f but meaning less opposite current $i(t)$. They are located close to the sample surface because of the skin effect. This effect was first described by Lamb (1883) for the case of circular conductors and was generalized to conductors of any shape by Heaviside (1951) [1].

For in-depth study of the phenomena of the electromagnetic field deformation process was necessary to know, by specialists of the mechanics of a dynamic process created high power electrical circuits, in which the parameters vary during the process. It should be noted that during deformation the blank is to induce an electromotive force in the coil, the

inductor, which also can lead to a reduction in the transfer of energy from the magnetic field formed for the track is formed [2, 3].

In processing equipments through the electromagnetic field, as noted above, energy is accumulated in the capacitor. When high voltage switch closure occurs discharge the capacitor. It will therefore be a discharge of

energy that will be transferred in a very short time the coil-inducer. From here, through the electromagnetic field the energy passes to the workpiece, which will change shape due to the pressure exerted by it.

The way energy is split capacitors (energy stock) during its transfer, is suggestively shown in fig. 1.

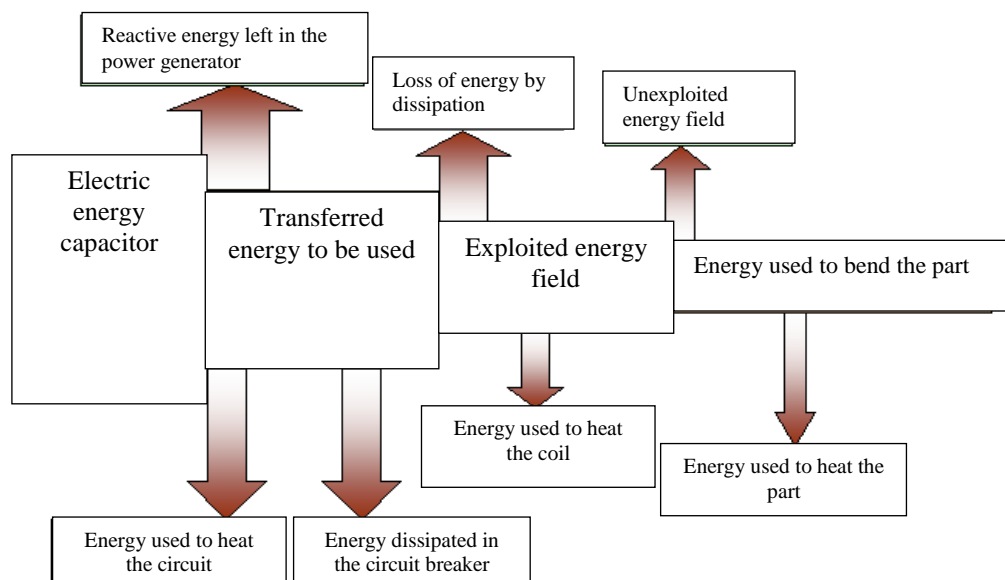


Fig. 1. Transfer of energy in electromagnetic energy systems [1]

The main parameters that influence the processing system in electromagnetic field are: electrical conductivity; deformed plate thickness; deformed shape and size of parts and mechanical characteristics of machined parts [4, 5].

METHOD AND RESEARCH EQUIPMENT

As input parameters that influence the process of forming through the electromagnetic field was chosen the following: discharge energy E ; number of whirls N of the coil; thickness of the sample.

As process output parameters chosen to study the following parameters: relative elongation at drawing; workpiece thickness after deformation at drawing; firing the piece at the height of the block.

Starting from choosing to study the three input parameters chosen to organize a full 2^3 factorial experiment.

Inductor coil construction is shown in fig. 2.

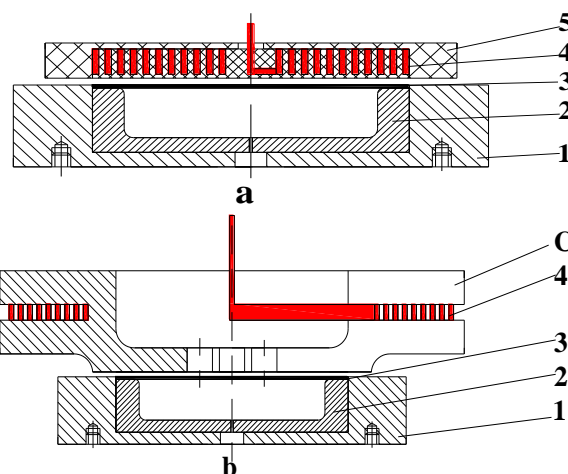


Fig. 2. Shape of induction coils used in the experimental research: a) induction coil without concentrator; b) induction coil with field concentrator:
1 - support plate; 2 - active plate; 3 - workpiece; 4 - inductor; 5 - insulated plate; C - field concentrator

Based on the theoretical consideration, coil form with Archimedes concentrator spiral field (fig. 2a) was designed and built in three variants (fig. 3a), which have the same outer diameter ($D=100$ mm), but different steps which led to the different lengths of the conductor winding and the coil of the field

concentrator (fig. 2b) has been designed and made in a single embodiment (fig. 3b), the outer diameter $D=200$ mm, and the diameter of the active surface of 120 mm.

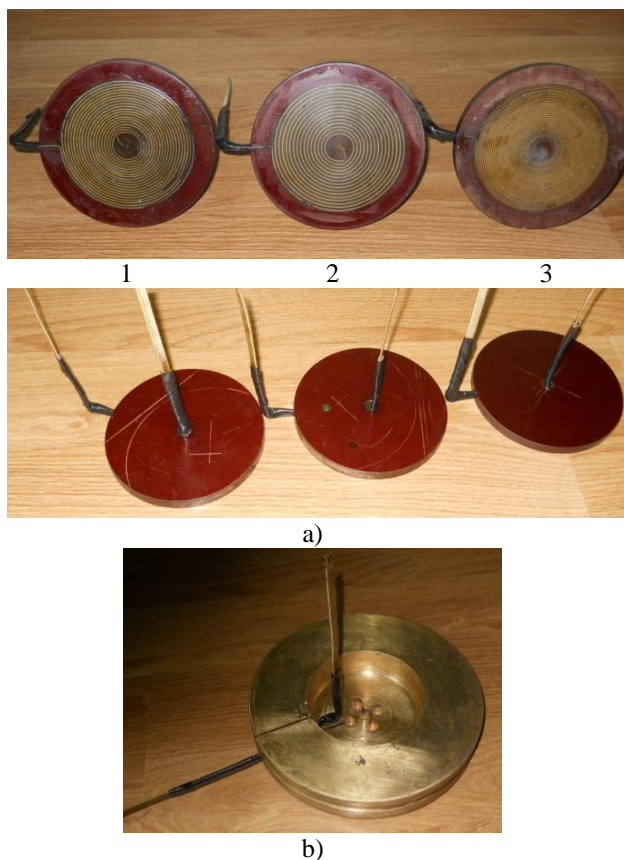


Fig. 3. Induction coils built for experiments:
a) induction coil without concentrator; b) induction coil with field concentrator

Based on the principle of electromagnetic field for processing is required mold to shape the sample. For this purpose it was necessary to concept, design and implementation of a device similar to a conventional molds, mechanical processing principle. Besides the device itself is a stamping die that punch is made of high intensity magnetic field generated by a coil charge.

Designed mold with inductor without concentrator for semiflat deformation is shown in fig. 4. In this construction there is a base plate 1, a support plate 2 on which is mounted the active plate 3, which will shape the plan sample S_f . "Stamp" is created through the coil 10 is placed on top of the sample in such a way that the whole field created by it interfering with the sample. Coil made of electrically conductive material is fixed in epoxy 4, considered a good insulating material with good mechanical strength. It is fixed to a plate

5 made of insulating material (textolit), the whole assembly being fixed to a top plate 7. The movable part centering is ensured by means of two guiding columns 8.

After positioning the sample in the working position, the mold is closed by using of two nuts 9, which can be screwed into the threaded portions on the ends of the guide columns.

For adjusting the distance between the coil 10 and the sample S_f between the bushing 6 and the coil holder plate 11 is inserted additions of different thicknesses.

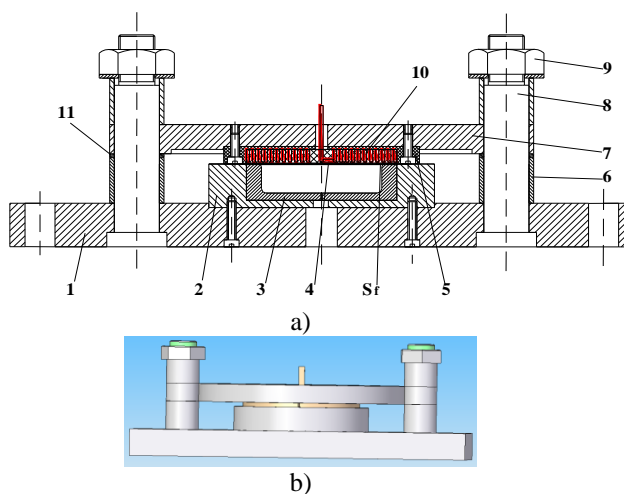


Fig. 4. a) Layout of the working mould with inductor, no concentrator for planar deformation of blanks;
b) 3D view of the work mould:
1 - base plate; 2 - support plate; 3 - active plate;
4 - epoxy resin; 5 - insulated plate; 6 - bush; 7 - top plate; 8 - guide column; 9 - nut; 10 - inductor;
11 - surplus

It should be noted that the induction coil with field concentrator is fitted with an insulating material between the field concentrator and plates 7 and 5 (rubber, lacquer, epoxy resin, etc.). Its purpose is to contain the electromagnetic field of the concentrator thus increasing process efficiency.

RESULTS AND DISCUSSIONS

In the case of aluminum experimental conditions and results of stamping parts are shown in table 1. Fig. 5 shows the influence of the number of whirls on the height part deviation.

Table 1

Experimental conditions and results obtained in stamping aluminum parts

Experimental conditions	Part code							
	IIIA1	IIIA2	IIIA3	IIIA4	IIIA5	IIIA6	IIIA7	IIIA8
Thickness g, [mm]	0,15	0,3	0,15	0,3	0,15	0,3	0,15	0,3
Discharge energy, [J]	150	150	150	150	600	600	600	600
Whirls number of coil	16	16	26	26	16	16	26	26
Deviation from the height of the piece, [mm]	0,2	0,1	0,13	0	*	*	*	*

*irrelevant

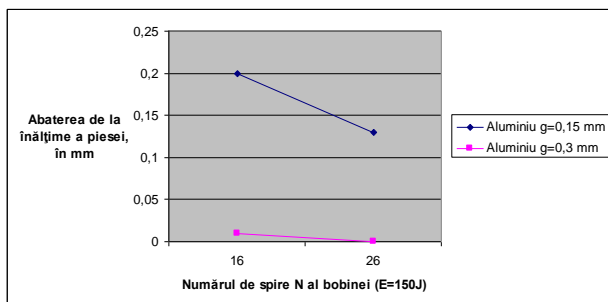


Fig. 5. Influence of the number of whirls on the deviation from the height of the piece

In the case of aluminum increasing the number of whirls N at an energy of 150 J and a thickness of 0.3 mm for insignificant sample result in a decrease in the deviation from the height of the piece. When using a thickness of 0.15 mm occurs significant precious drop of this offense.

Moving to the study of the second material, brass, experimental conditions and main results obtained are presented in table 2 and the influence of discharge power on the deviation of height for different thicknesses is shown in fig. 6 and fig. 7.

Fig. 8 and fig. 9 show influence the number of whirls at different values of discharge energy.

In the case of brass material with a thickness of 0.5 mm and 0.15 mm but increase energy discharge from a total of 16 whirls leads to a significant decrease in height deviation parts. At different values of discharge energy, 150 J and 600 J, increasing the number of whirls resulted in decreases almost comparable offenses if a thickness of 0.15 mm and a thickness of 0.5 mm in the case.

Table 2

Experimental conditions and results obtained from brass stamping parts

Experimental conditions	Part code							
	IIIB1	IIIB2	IIIB3	IIIB4	IIIB5	IIIB6	IIIB7	IIIB8
Thickness g, [mm]	0,15	0,5	0,15	0,5	0,15	0,5	0,15	0,5
Discharge energy, [J]	150	150	150	150	600	600	600	600
Whirls number of coil	16	16	26	26	16	16	26	26
Deviation from the height of the piece, [mm]	2,67	2,15	2,11	2,92	0,61	0,43	0,38	0,29

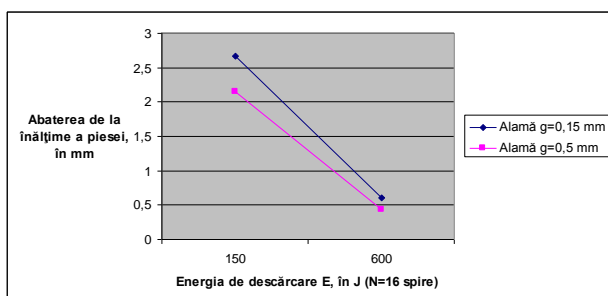


Fig. 6. Influence of discharge power on the deviation from the height of the piece

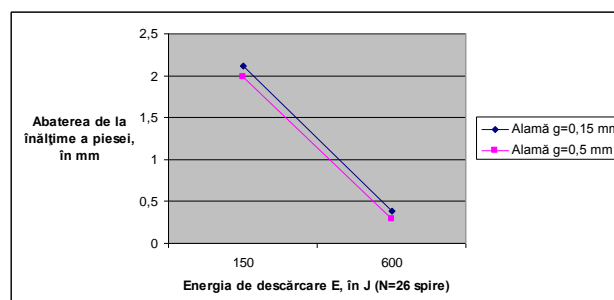


Fig. 7. Influence of discharge power on the deviation from the height of the piece

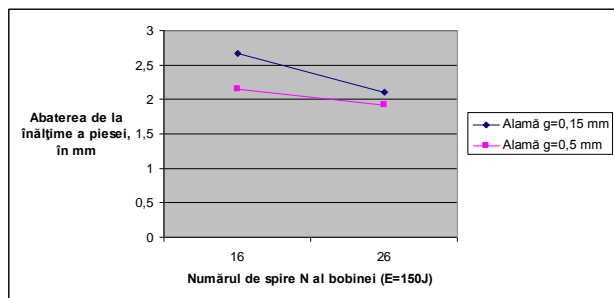


Fig. 8. Influence of the number of whirls on the deviation from the height of the piece

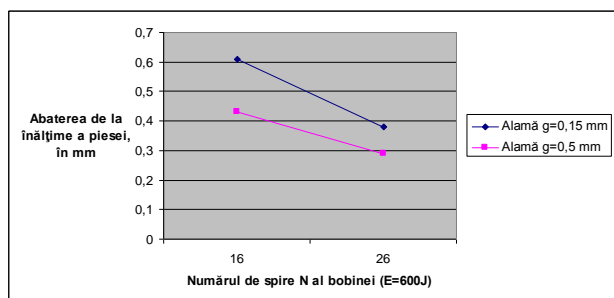


Fig. 9. Influence of the number of whirls on the deviation from the height of the piece

For copper discharge energy increase leads to decrease deviation from the height of the piece with comparable values for the two thicknesses of 0.25 mm and 0.4 mm used. The same observation can be drawn and where the influence of the number of whirls on the deviation from the height of the piece respectively influence discharge energy.

Experimental conditions and results for copper are shown in table 3 and fig. 10 shows the influence of discharge power on the deviation from the height of the piece obtained. Influence of the number of whirls on the deviation from the height of the piece is shown in fig. 11.

Table 3

Experimental conditions and results obtained on copper stamping parts

Experimental conditions	Part code							
	IIC1	IIC2	IIC3	IIC4	IIC5	IIC6	IIC7	IIC8
Thickness g, [mm]	0,25	0,4	0,25	0,4	0,25	0,4	0,25	0,4
Discharge energy, [J]	150	150	150	150	600	600	600	600
Whirls number of coil	16	16	26	26	16	16	26	26
Deviation from the height of the piece, [mm]	0,15	0,2	0	0,1	0	0,1	*	0

*irrelevant

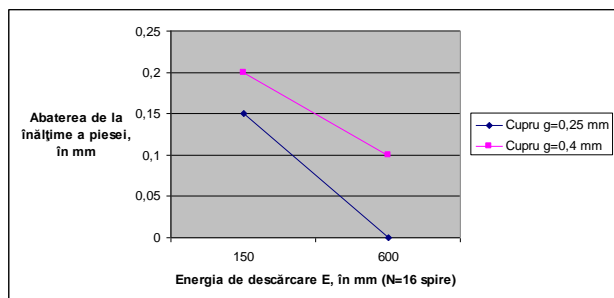


Fig. 10. Influence of discharge power on the deviation from the height of the piece

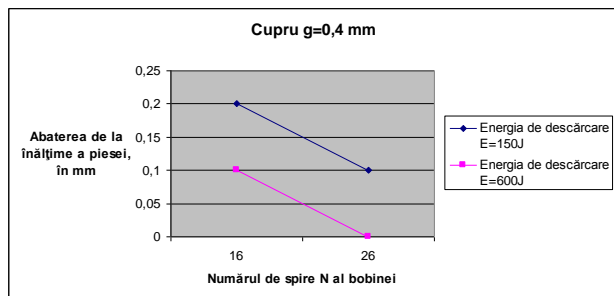


Fig. 11. Influence of the number of whirls on the deviation from the height of the piece

CONCLUSIONS

In case of aluminum is a significant decrease of the deviation from height if a sample with 0.15 mm thickness and a decrease in reduced when using a 0.15 mm thick of material. In case of brass material discharge energy increase leads to a significant decrease in the deviation from the height of parts. At different values of discharge energy, 150 J and 600 J, increasing the number of whirls resulted in decreases almost comparable offense so if a thickness of 0.15 mm and a thickness of 0.5 mm in the case. In the case of copper discharge energy increase leads to decrease deviation from height part with comparable values for the two thicknesses of material used, the 0.25 mm and 0.4 mm. The same observation can be drawn and in the influence of the number of whirls on the deviation from the height of the piece for the two values of the discharge energy of 150 J and 600 J.

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ВЗАИМОДЕЙСТВИЕ МЕТАЛЛОВ И СПЛАВОВ С ГАЗОВЫМИ СРЕДАМИ ПОД ДЕЙСТВИЕМ ИСКРОВЫХ РАЗРЯДОВ

Ч. 2. ФИЗИКО-ХИМИЧЕСКИЙ АНАЛИЗ И ИНТЕРПРЕТАЦИЯ РЕЗУЛЬТАТОВ

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В работе представлены результаты экспериментальных исследований взаимодействия металлов и их сплавов с газовыми средами под действием искровых разрядов. Исходя из результатов физико-химического анализа, установлено, что на процесс введения в искровом разряде легирующих примесей в металлы и сплавы, находящиеся в различном структурно-фазовом состоянии, большое влияние оказывает газовая среда, в которой осуществляется обработка. Это необходимо учитывать, поскольку состав, а, следовательно, и свойства поверхностного слоя, как правило, отличаются от требуемых.

Ключевые слова: искровой разряд, металл, газовая среда, легирующий элемент.

În lucrare sînt prezentate rezultatele cercetărilor experimentale privind interacțiunea metalelor și aliajelor lor cu mediile gazoase sub acțiunea descărcărilor prin scînteii electrice. Pe baza rezultatelor analizei fizico-chimice s-a stabilit că asupra procesului de introducere la descărcarea prin scînteie a elementelor de aliene în metale și aliaje, care se află în stări structural-fazice diferite, influențează în mare măsură mediul gazos, în care se efectuează prelucrarea. Acest lucru trebuie luat în considerare deoarece conținutul și, respectiv, proprietățile stratului de suprafață, de obicei, diferă de cele necesare.

Cuvinte-cheie: descărcărea prin scînteii electrice, metal, mediul gazos, elementul de aliene.

This paper presents results of experimental investigations on the interaction of metals and their alloys with the gaseous media by the action of spark discharges. Based on the results of physico-chemical analysis, it was found that on the process of introduction at the spark discharge of alloying elements in metals and alloys, which are in different structural-phase state, the great influence has the gaseous medium in which the processing is performed. This should be considered because the composition and, consequently, the properties of the surface layer are usually different from the desired.

Keywords: spark discharge, metal, gaseous medium, alloying elements.

3. РЕЗУЛЬТАТЫ (Продолжение. Начало в № 1, 2014)

Рассмотрим одновременное проникновение в основной металл и формирующееся покрытие атомов металла и газов воздуха на примере обработки железа молибденовым анодом. Анализ концентрационных профилей распределения меченых атомов молибдена ⁹⁹Mo в железе и в молибдене, проникающих в процессе ЭИЛ из предварительно нанесенного на железо слоя р/а изотопа, показал, что они не могут быть описаны простой экспоненциальной зависимостью, а

имеют сложный характер и характеризуются наличием по меньшей мере одного концентрационного максимума для каждой энергии разряда W , причем с увеличением значения W данные максимумы располагаются на все больших глубинах (рис. 1). Действительно, если при $W = 0,3$ Дж наибольшее значение концентрации находится почти на поверхности, то при $W = 6,4$ Дж смещение максимума C_{max} составляет приблизительно 15 мкм в глубь основного металла. Однако за пределами приповерхностного слоя зависимость $C = f(X)$ для всех значений энергии E можно аппроксимировать

экспоненциальной зависимостью от глубины как в основном металле, так и в материале покрытия. Кроме положения максимума с увеличением энергии также возрастают глубина проникновения атомов молибдена в железо и толщина образующегося покрытия (в $\sim 1,5$ и 2 раза соответственно). Аналогичные результаты получены при использовании меченых атомов железа, находящихся до начала обработки в виде нанесенного на образец слоя толщиной ~ 1 мкм. Смещение максимума концентрации меченых атомов железа в молибдене при $W = 6,4$ Дж также составляет около 15 мкм. Глубина проникновения атомов железа ^{55}Fe в железо и толщина образующегося молибденового покрытия увеличиваются в $1,7$ и $2,5$ раза соответственно при изменении W в ~ 20 раз.

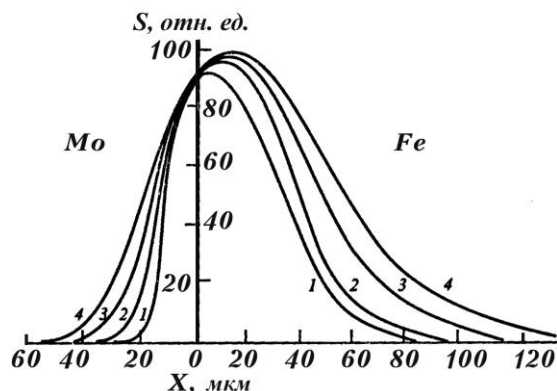


Рис. 1. Концентрационные профили распределения атомов молибдена ^{99}Mo в железе и молибдене после ЭИЛ с энергиями $0,3$ Дж (1), $0,9$ Дж (2), $3,1$ Дж (3), $6,4$ Дж (4)

Помимо взаимного проникновения металлов происходит введение в покрытие и подложку атомов среды. Так, использование активационной автордиографии показало присутствие азота, кислорода, аргона и следов углерода. Атомы инертного газа локализовались в порах диаметром до 1 мкм, находящихся в объеме зерен и в твердом растворе, а азот – преимущественно в виде твердого раствора и небольшого количества нитридов на границах зерен. Что же касается кислорода, то он был равномерно распределен в матрице. Интересно, что интерметаллидные фазы в диффузионной зоне отсутствуют. Однако при нанесении тонкого слоя молибдена на железо в зоне

протяженностью ~ 2000 нм от поверхности помимо твердых растворов было обнаружено наличие небольшого количества соединения $\text{Fe}_x\text{Mo}_{1-x}$. Его оказалось недостаточно для определения симметрии данного соединения и сопоставления с интерметаллидом Fe_2Mo (λ -фаза). При обработке по такому же режиму тонкопленочного (~ 1000 нм) исходного гальванического покрытия ^{57}Fe , находящегося на монокристаллическом молибдене, молибденовым анодом на поверхности возникает твердый раствор железа в молибдене и интерметаллическое соединение $\text{Fe}_x\text{Mo}_{1-x}$, где $x < 0,66$. На глубине ~ 1500 нм выявляется твердый раствор молибдена в α -Fe в виде фаз, размер которых достигает 15 нм.

С ростом энергии разряда увеличивается содержание легких элементов в железе и при $W = 15$ кДж оно становится сопоставимым с количеством атомов молибдена (рис. 2).

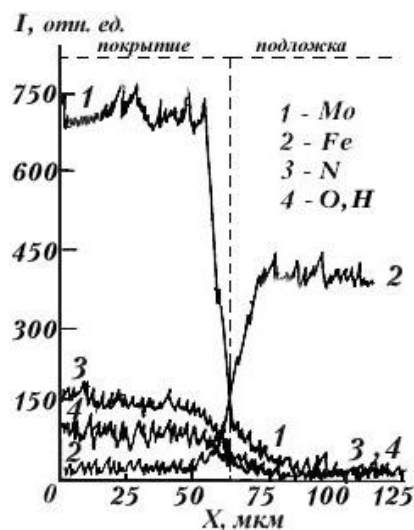


Рис. 2. Распределение элементов в покрытии и основном металле, 15 кДж, 1 мин/ cm^2 , 200 мкс. Оже-спектроскопия

Рассмотрим взаимодействие сталей (Ст.3, Ст.45, Ст.20) с материалом анода и газами воздуха. Рентгеноструктурный (табл. 1) и микрорентгеноспектральный анализы молибденовых покрытий на стали 3 показали, что под действием ЭИЛ возникает диффузионная зона с большим количеством фазовых составляющих.

Таблица 1

Влияние энергии разряда на перераспределение содержания фаз в молибденовом покрытии на стали Ст.3

Фазовый состав	Mo	α -Fe	α -Mo	Mo ₂ N	Mo ₂ C
Энергия (E, Дж)	Относительная концентрация, (C, %)				
0,3	100	20	10	< 1	< 1
0,9	100	4	12	10	< 1
3,1	100	0	15	15	< 1
6,4	100	0	15	15	1

Таблица 2

Фазовый состав молибденовых покрытий на стали Ст.20 при различных энергиях искрового разряда

0,3 Дж				0,9 Дж			
d/n, нм	(hkl)	Фаза	I, %	d/n, нм	(hkl)	Фаза	I, %
0,260	100	Mo ₂ C	1	0,260	100	Mo ₂ C	1
0,238	002	Mo ₂ C	1	0,238	002	Mo ₂ C	1
	111	Mo ₂ N			111	Mo ₂ N	
0,222	110	Mo	100	0,222	110	Mo	100
				0,206	200	Mo ₂ N	5
0,202	110	Fe	20	0,202	110	Fe	5
0,157	200	Mo	30	0,157	200	Mo	30
				0,146	220	Mo ₂ N	3
0,147	200	Fe	5				
0,128	211	Mo	50	0,128	211	Mo	50
				0,124	311	Mo ₂ N	2
0,117	211	Fe	10				
0,111	220	Mo	20	0,111	220	Mo	20
0,109	220	α -Mo	10	0,109	220	α -Mo	10
0,101	220	Fe	7,5				
0,990	310	Mo	50	0,990	310	Mo	50
0,930	310	α -Mo	10	0,930	310	α -Mo	10
0,911	222	Mo	40	0,911	222	Mo	40
3,14 Дж				6,4 Дж			
d/n, нм	(hkl)	Фаза	I, %	d/n, нм	(hkl)	Фаза	I, %
0,260	100	Mo ₂ C	1	0,260	100	Mo ₂ C	1
0,237	002	Mo ₂ C	1	0,237	002	Mo ₂ C	1
	111	Mo ₂ N			111	Mo ₂ N	
0,228	011	Mo ₂ C	2	0,228	011	Mo ₂ C	2
0,222	110	Mo	100	0,222	110	Mo	100
0,205	200	Mo ₂ N	6	0,205	200	Mo ₂ N	7
0,166			3	0,166			3
0,157	200	Mo	30	0,157	200	Mo	30
0,146	220	Mo ₂ N	4	0,146	220	Mo ₂ N	4
0,128	211	Mo	50	0,128	211	Mo	50
0,124	311	Mo ₂ N	2	0,124	311	Mo ₂ N	2
				0,119	211	α -Mo	1
0,111	220	Mo	20	0,111	220	Mo	20
0,109	220	α -Mo	15	0,109	220	α -Mo	15
0,990	310	Mo	50	0,990	310	Mo	50
0,930	310	α -Mo	15	0,930	310	α -Mo	25
0,911	222	Mo	40	0,911	222	Mo	40

Кроме исходных молибдена, твердого раствора углерода в железе и цементита (в несколько меньшем количестве в связи с его частичным распадом) в ней присутствуют твердые растворы замещения железа в молибдене и молибдена в железе,

небольшое количество твердых растворов внедрения азота и углерода в молибдене и азота в железе, фазы внедрения: нитриды и карбиды молибдена, а также следы нитридов железа (Fe₄N, Fe₂N), причем только при малых значениях энергии

разряда. С увеличением энергии разряда растёт толщина покрытия, о чём свидетельствует, в частности, уменьшение интенсивности дифракционных максимумов железа, и заметно возрастает содержание нитридов молибдена, в то время как количество карбидов изменяется незначительно (табл. 2). Это отличие может быть вызвано разными механизмами формирования фаз – появление азота в покрытии связано с его захватом из воздуха при ионизации молекул N_2 в плазме искрового разряда, тогда как поступление атомов углерода лимитировано его концентрацией в стали. Действительно, при обработке по тому же режиму стали 3, содержащей меньшее количество углерода, в диффузионной зоне только при значении $E = 6,4$ Дж можно увидеть следы Mo_2C .

При меньших энергиях дифракционные максимумы, соответствующие данной фазе, находятся на уровне фона. При взаимодействии молибдена с железом карбиды молибдена рентгеноструктурным методом не выявляются вообще. Что же касается отсутствия нитридов железа при обработке с энергией разряда, превышающей 0,3 Дж, то это связано с тем обстоятельством, что с увеличением толщины формирующегося покрытия атомы азота не успевают проникнуть за время обработки в основной металл в количестве, достаточном для образования нитридов. Содержание твёрдого раствора азота в железе при $E = 0,9$ Дж также становится незначительным, причем это имеет место даже при обработке молибденовым анодом железа, не содержащего атомов углерода, которые могут препятствовать проникновению атомов других элементов при импульсной обработке [1].

Отметим, что возрастание величины энергии разряда также приводит к большей гомогенизации всех образовавшихся фаз. Так, дифракционные максимумы на рентгенограммах становятся менее размытыми (рис. 3). Следует обратить внимание на тот факт, что в покрытии на всех исследуемых сталях и диффузионной зоне между покрытием и подложкой

отсутствуют оксиды молибдена и железа и твёрдые растворы кислорода в этих металлах при всех энергиях разряда.

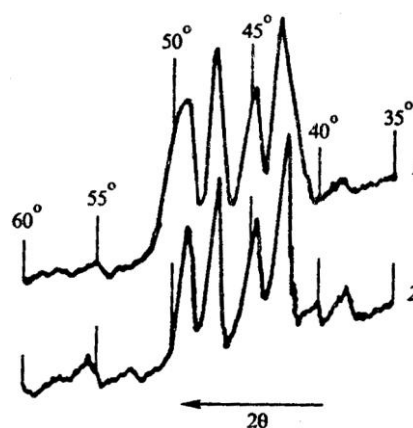


Рис. 3. Дифракционные максимумы плоскостей (100), (002), (011) соединения Mo_2C и (111) соединения Mo_2N при обработке стали 45 молибденовым анодом с энергиями 0,3 Дж (1) и 0,9 Дж (2)

При нанесении молибденового покрытия (молибденовый анод) на нержавеющую сталь, осуществляемом на воздухе, происходит не только насыщение приповерхностного слоя стали молибденом и атмосферными газами, но и перераспределение компонентов стали, причем содержание никеля и хрома в формирующемся покрытии даже несколько выше, чем в основном металле (рис. 4). По-видимому, это связано с появлением градиентов концентрации компонентов стали при формировании покрытия из молибдена, т.е. появления в каждый момент обработки металла, не содержащего хрома и никеля.

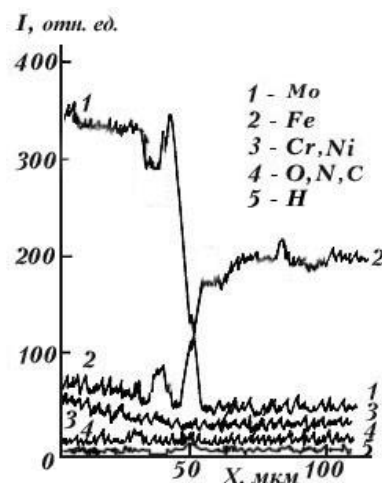


Рис. 4. Распределение элементов в молибденовом покрытии и стали X18H10T, 15 кДж, 1 мин/см², 200 мкс. Оже-спектроскопия с шагом 0,5 мкм

При нанесении железного покрытия (с помощью железного анода) на поликристаллический молибден возникают сходные фазы. Так, при энергии разряда $E = 6,4$ Дж появляются твердые растворы, нитриды железа и молибдена, интерметаллическое соединение с нарушенной стехиометрией $Fe_{1,9}Mo$ (рис. 5, б). Карбидные фазы не возникают, как это имело место при взаимодействии молибдена со сталями, поскольку количество углекислого газа в воздухе незначительно по сравнению с азотом, а других источников поступления атомов

углерода в покрытие и основной металл нет. Кроме того, на поверхности железного покрытия обнаруживаются атомы кислорода – порядка 12 ат. % кислорода, не связанного в окислы. Можно предположить, что кислород находится в виде пересыщенного твердого раствора в железе и в растворе молибдена в α -Fe. В основном металле, т. е. в молибдене, атомы кислорода не выявляются, как и в том случае, когда молибден был материалом покрытия, а железо подложкой.

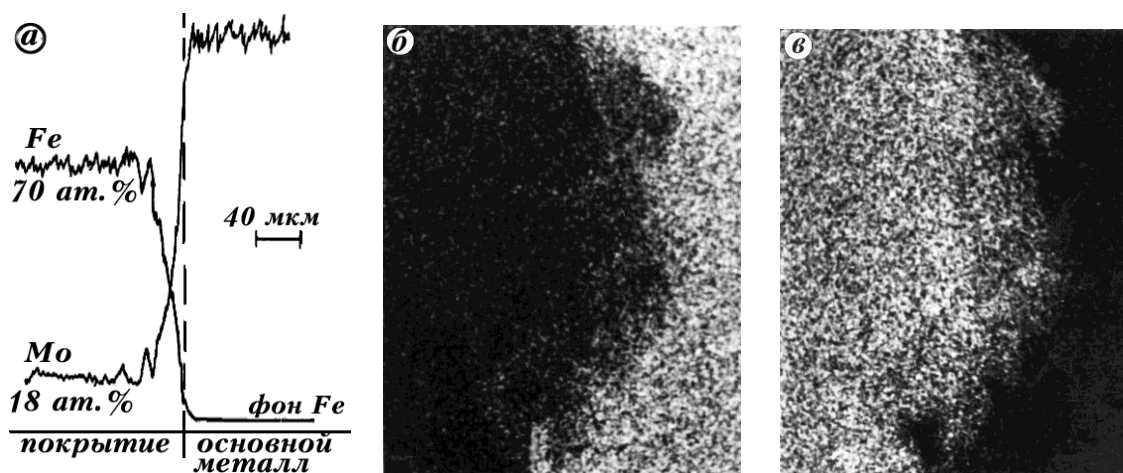


Рис. 5. Концентрационные кривые распределения молибдена в железе и железа в молибдене при ЭИЛ с энергией разряда 6,4 Дж (а), снятые в отраженных электронах железа (б) и молибдена (в), $\times 350$

Отметим, что интерметаллическое соединение образуется не на поверхности образующегося железного покрытия, а только в переходной зоне между покрытием и подложкой. Оно появляется в твердом растворе железа в молибдене и прорастает из него в молибденовую подложку на глубину до 30 мкм в виде иголок, расположенных по нормали к поверхности молибденового образца (рис. 6).

При нанесении молибдена ^{99}Mo (молибденовый анод) на образец из молибдена в различных средах было получено следующее. При $W = 6,4$ Дж (1 мин/см², 200 мкс, 0,5 мм) во всех используемых средах глубина проникновения меченых атомов в молибден составляет более 60 мкм, причем максимум концентрации находится в покрытии толщиной до 50 мкм на расстоянии от границы раздела 5–7 мкм. За пределами максимума имеет место экспоненциальная

зависимость радиоактивности молибдена от квадрата глубины проникновения в образец. В зависимости от среды, в которой горел разряд, был различный фазовый состав.

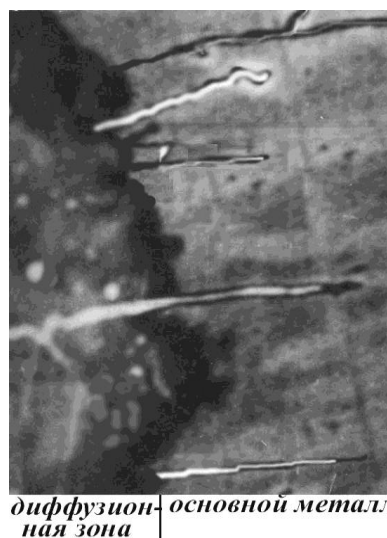


Рис. 6. Микроструктура монокристаллического молибдена с железным покрытием, $\times 600$

Помимо пересыщенного твердого раствора в молибдене соответствующих легких элементов (С, N, O) и инертных газов были также фазы внедрения, симметрия которых соответствовала образующимся в равновесных условиях, и газонаполненные поры:

Среда	N ₂	NH ₃	CO ₂	CH ₄	Ar	Kr
Фазы	Mo ₂ N	Mo ₂ N	Mo ₂ C	Mo ₂ C	Поры размером до 3 мкм	

Отметим, что ни твердого раствора водорода, ни гидридов не было обнаружено даже при использовании аммиака и метана, меченых по водороду. В равновесных условиях их образование в молибдене также затруднено [2, 3]. Что же касается атомов кислорода, то степень его насыщения в твердом растворе существенно выше, чем это имеет место для азота, углерода, аргона и криптона. Так, содержание кислорода доходит почти до 10%, в то время как концентрация других элементов не превышает 0,1 - 1 %.

Рассмотрим взаимодействия никеля с различными средами. Обработка никеля никелевым анодом в среде азота способствует образованию в матрице твердого раствора азота и нитрида Ni₃N, сохраняющего гексагональную симметрию и параметры решетки ($a = 0,2668$ нм, $c = 0,4294$ нм), характерные для этой фазы в равновесных условиях. Другие, возможные в данной системе нитриды Ni₄N, Ni₃N₂ и азид NiN₆, т.е. производное вещество, образующееся при взаимодействии никеля с азотистоводородной кислотой HN₃, обнаружены не были даже в тончайших приповерхностных слоях. На автордиограммах-репликах, полученных в излучении как никеля, так и азота видны равномерно распределенные в матрице одиночные восстановленные микрокристаллы бромистого серебра и их скопления, локализованные преимущественно вблизи границ зерен. Причем из-за низкой растворимости азота в никеле ($2 \cdot 10^{-4}$ ат.% в равновесных условиях) преобладает фаза внедрения. Тем не менее, в результате воздействия искровыми разрядами, подобно тому, что наблюдается при ионной бомбардировке в тлеющем разряде, происходит увеличение

растворимости азота в никеле в твердой фазе, и максимальное содержание никеля в твердом растворе в приповерхностном слое достигает 0,1 ат.%. Концентрация азота в кристаллической решетке никеля резко уменьшается с глубиной и на расстоянии от поверхности ~ 50 мкм перестает фиксироваться рентгеновскими и радиоизотопными методами. Что же касается фазы Ni₃N, то стехиометрические соотношения не изменятся, но уменьшается количество нитридов и на глубине до 40 мкм они не выявляются. Анализ автордиограмм, полученных при различных параметрах обработки ($W \sim 0,1 - 6,4$ Дж, $l_{\text{МЭП}} \sim 0 - 2$ мм, $\tau \sim 1 - 3$ мин/см²), показал, что внутри зерна, как правило, располагается твердый раствор азот в никеле, а нитриды находятся в металле в основном вдоль границ зерен (рис. 7).



Рис. 7. Распределение азота ¹³N в никеле после действия искровых разрядов в среде азота, $l_{\text{МЭП}} = 0,5$ мм, $W = 2,7$ Дж, $\tau_1 = 200$ мкс, $\tau = 1$ мин/см², $\times 4800$

При обработке никеля на воздухе никелевым анодом в твердых растворах внедрения помимо азота присутствуют атомы кислорода, углерода (поступающие в небольшом количестве из CO₂) и аргона (по данным активационной автордиографии), в то же время окислы отсутствуют даже на поверхности (в слое ~ 200 нм). Поскольку кислород, азот, углерод и аргон в твердом растворе определяли различными методами, их соотношение определить было невозможно.

Обработка никеля никелевым анодом в среде углекислого газа CO₂, меченого по углероду ¹⁴C, приводит к образованию в диффузионной зоне на протяжении 50 – 75 мкм при энергии импульса от 0,9 до 6,4 Дж концентрационного профиля с переменной

концентрацией углерода, в приповерхностном слое до 5 мкм - пересыщенного твердого раствора внедрения с максимальным содержанием углерода ~ 3 %. В равновесных условиях максимальная концентрация углерода в никеле при 1326°C не превышает 2,7 ат.% [4].

Аналогичные результаты (значения максимальных концентрации на поверхности - 3,05 ат.% и глубины проникновения - 78 - 80 мкм) получены при электроискровой обработке никеля в среде метана, меченого по углероду. Исследование взаимодействия никеля с метаном, меченым по углероду, при энергиях разрядов от 0,08 до 0,12 Дж показало, что образуется твердый раствор с максимальной концентрацией в приповерхностном слое ~ 1,8 - 1,9 ат.%. Величина параметра решетки доходит до 0,3534 нм (в исходном состоянии - 0,3524 нм), при этом, несмотря на столь значительное увеличение параметра, решетка остается гранецентрированной. Фазы внедрения Ni₃C, как и при изотермическом отжиге обнаружены не были. Этот карбид, как известно, можно получить при нагреве путем реакции $3Ni + 2CO = Ni_3C + CO_2 \uparrow$ [5, 6].

Как показали результаты электронномикроскопической автордиографии, при использовании меченых атомов криптона ⁸⁵Kr, смешанных с аргоном, в диффузионной зоне появляются мелкодисперсные ~ 1 - 3 мкм газонаполненные поры (рис. 8), количество и размер которых резко спадает с глубиной. На расстоянии до 20 мкм от поверхности наблюдаются считанные поры, тогда как твердый раствор переменной концентрации инертного газа в никеле простирается на расстояние более 40 мкм.

Тем не менее, наличие атомов инертных газов практически не сказывается на механических характеристиках металла, начиная от глубин ~ 10 - 15 мкм в зависимости от параметров обработки. Ближе к поверхности износостойкость еще меньше, чем при обработке в чистом азоте.

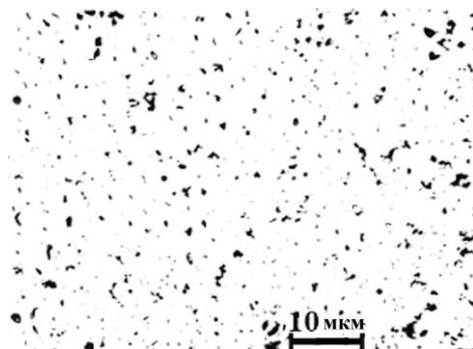


Рис. 8. Автордиограмма-реплика поверхности никеля после ЭИЛ в смеси ⁴⁰Ag и ⁸⁵Kr, 6,4 Дж, 100 мкс

Рассмотрим особенности фазообразования при электроискровом легировании титана. При взаимодействии титана с титановым анодом в среде азота на поверхности образца возникает рентгенографически выявляемое небольшое количество нитрида титана Ti₂N (ϵ -фазы) с тетрагональной симметрией ($a = 0,4943$ нм, $c = 0,3036$ нм), причем с увеличением длительности обработки заметно возрастает содержание ϵ -фазы, в то время как значения параметров решетки изменились слабо. Автордиограммы поверхности титана и двухфазного титанового сплава BT 20, меченых атомами титана ⁴⁴Ti, показали образование нитридов на границах зерен и фаз. За пределами слоя нитридов согласно данным активационной автордиографии с помощью изотопа ¹³N и вторичной ионной масс-спектропии уменьшение концентрации азота происходит по экспоненте (рис. 9).

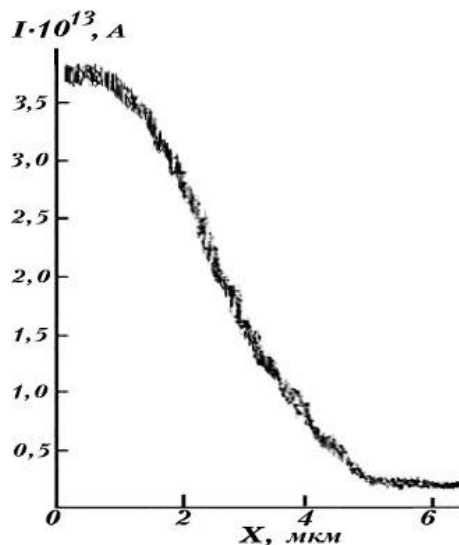


Рис. 9. Распределение азота, поступающего из среды, по глубине диффузионной зоны титана при обработке титановым анодом ($E_{и}=0,08$ Дж, $\tau_{и}=200$ мкс, $\tau = 10$ с, $l_{МП} = 0,5$ мм)

Отметим, что увеличение продолжительности обработки в 6 раз при прочих равных условиях способствует проникновению атомов азота в титан на глубину до 30 мкм, т.е. почти в 5 раз возрастает протяженность диффузионной зоны. Распределение в приповерхностных слоях формирующегося титанового покрытия на титане кислорода и углерода показано на рис. 10.

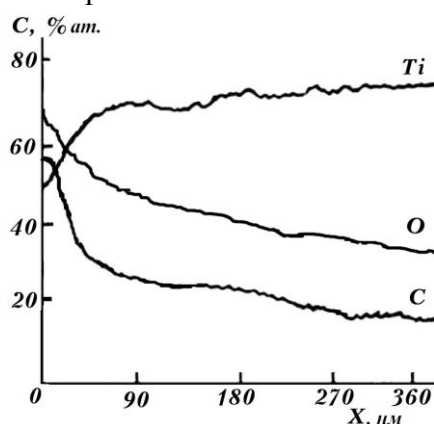


Рис. 10. Распределение кислорода и углерода, поступающих из среды, по глубине диффузионной зоны титана при обработке титановым анодом ($E_H=0,08$ Дж, $\tau_H=200$ мкс, $\tau=10$ с, $l_{MЭП}=0,5$ мм)

При одновременном насыщении поверхности титана никелем (материал анода или покрытие на титане) с азотом (среда), согласно данным рентгеноструктурного анализа, в зоне взаимодействия возникают фазы в следующем порядке: нитрид никеля; твердый раствор азота и титана в никеле и, наконец, твердый раствор обеих легирующих элементов (металла и неметалла) в титане (рис. 11). При этом микротвердость H_μ изменяется таким образом, что повторяет форму концентрационного профиля распределения легирующих элементов в титановом сплаве, а в приповерхностном слое H_μ достигает ~ 1100 кГ/мм², т.е. увеличивается в ~ 2 и 3 раза по сравнению с никелем и титаном соответственно. С ростом энергии искрового разряда и длительности обработки увеличивается глубина проникновения в титан никеля и азота, концентрационный профиль и распределение H_μ спадает с глубиной более полого, но максимальные значения

концентрации и микротвердости практически не изменяются. Трибологические испытания показали, что в тонком приповерхностном слое (до $X \cong 5-7$ мкм) стойкость к истиранию незначительна. Затем на протяжении 10-25 мкм износостойкость практически не изменяется и является максимальной для каждого режима ЭИЛ. На больших глубинах износостойкость резко падает.

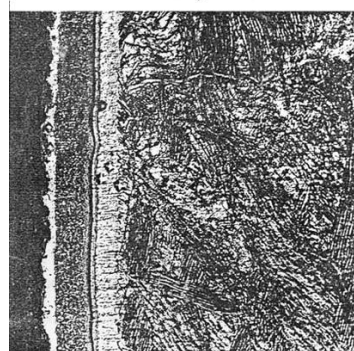
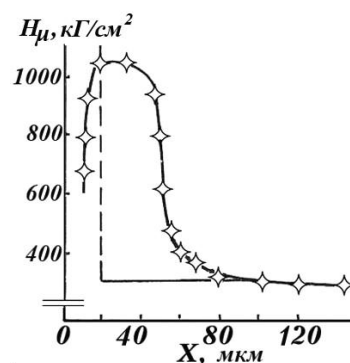


Рис. 11. Изменение микротвердости по глубине диффузионной зоны после ЭИЛ ($W=0,9$ Дж, $\tau_H=200$ мкс)

Рассмотрим взаимодействие с азотом и углеродом титана, на поверхности которого в исходном состоянии находится многокомпонентное покрытие, состоящее из твердого раствора фосфора в никеле и высокодисперсной упрочняющей фазы фосфида никеля Ni_3P (рис. 12).

Как видно из рисунка, микронапряжения, возникающие в пересыщенном твердом растворе фосфора в никеле после химического осаждения, настолько велики, что все рентгеновские рефлексы сливаются в одну чрезвычайно уширенную линию. Однако после часового стабилизирующего отжига форма дифракционных максимумов изменяется, и на рентгенограммах появляются линии, соответствующие твердому раствору фосфора в никеле и фосфиду никеля.

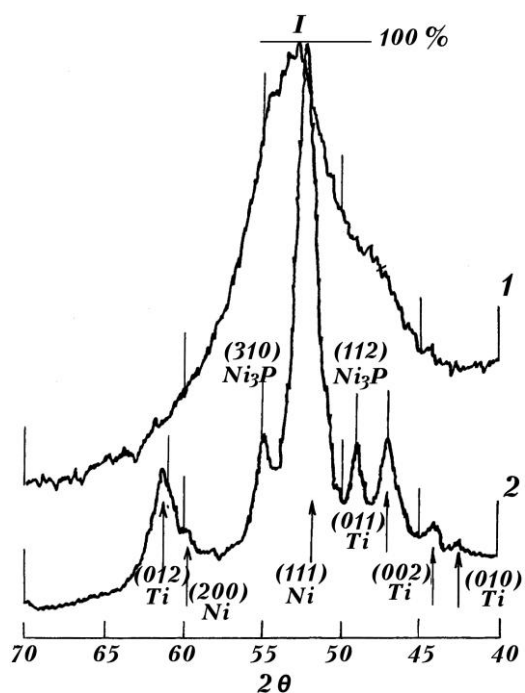


Рис. 12. Рентгенограммы образцов с никель-фосфорным покрытием на титане в исходном состоянии (1) и после часового отжига при 573 К (2), Со-излучение

Из рис. 13, на котором представлены концентрационные кривые после ЭИЛ никелевым анодом в среде азота, следует, что глубина проникновения (X) ^{63}Ni в титане как с исходным покрытием, так и без него увеличивается с ростом энергии разряда от до величин 80 - 120 мкм.

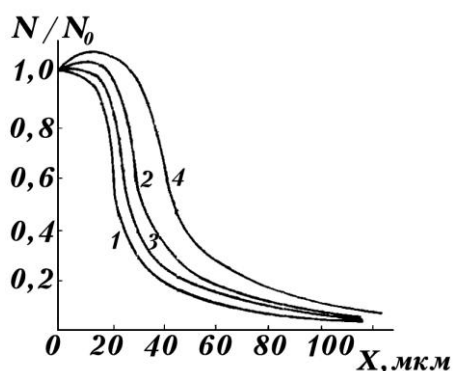


Рис. 13. Концентрационные кривые распределения ^{63}Ni в титановом сплаве: 1-без исходного покрытия при $W=0,9$ Дж, 2-3,14 Дж, 3 - с исходным покрытием при 0,9 Дж, 4 - 3,14 Дж при обработке в среде азота

Несколько иная форма начального участка концентрационной кривой (с появлением максимума) наблюдается на образцах с покрытием, что, по-видимому, связано с влиянием на перераспределение атомов наличия границы покрытие -

подложка. Следует отметить, что изменение фазового состава исходного покрытия в результате отжига не оказывает заметного влияния на форму концентрационного профиля. Анализ формы концентрационных профилей за пределами максимума показывает, что наилучшим образом изменение концентрации может быть описано экспоненциальной зависимостью от глубины. Плавное изменение концентрации никеля в титане с глубиной свидетельствует в определенной степени об отсутствии интерметаллических фаз, образующихся в системе Ni - Ti при температурах выше 673 К [2]. Это обстоятельство может свидетельствовать о том, что перенос вещества (Ni - Ti) за пределами тонкого приповерхностного слоя (до 20 мкм) происходит при $T < 673$ К, что обусловлено быстрым отводом тепла при данном способе обработки. Это указывает на диффузионный характер процесса переноса вещества при ЭИЛ. Однако наблюдаемые глубины проникновения существенно превышают таковые для диффузии в стационарных условиях даже при предплавильных температурах [7] и являются соизмеримыми с эффектами, возникающими при других видах импульсной обработки в твердой фазе [8].

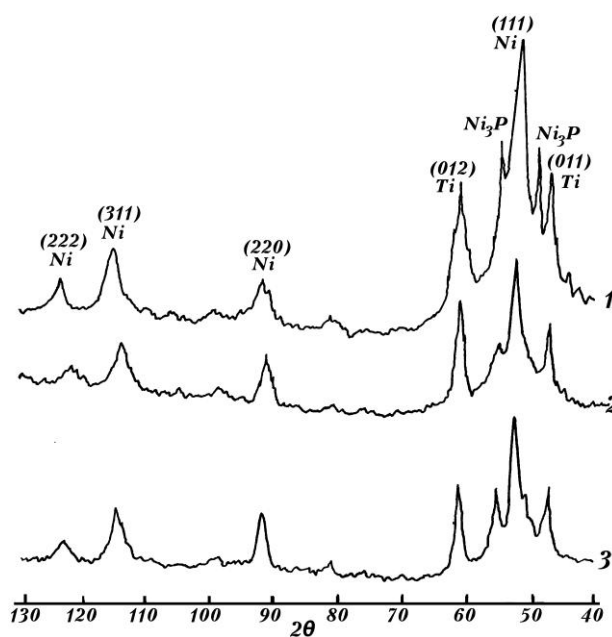


Рис. 14. Рентгенограммы образцов титана с никель-фосфорным покрытием после отжига (1), после легирования никелем в среде CO_2 с энергиями 0,9 Дж (2) и 3,14 Дж (3), Со-излучение

Изучение особенностей взаимодействия титана с никель-фосфорным покрытием с легирующими элементами (N, C) в процессе ЭИЛ анодами из никеля, хрома, сплава на их основе ВЖЛ с легирующими элементами (N, C) в процессе ЭИЛ показало, что фазовый состав образующихся слоев не зависит от того, были ли в исходном покрытии пересыщенный твердый раствор или двухфазное состояние (рис. 14, кривая 1, рис. 15, кривая 1). Из этого следует, что выход избыточного фосфора из кристаллической решетки никеля происходит в течение процесса ЭИЛ, то есть за несколько секунд, в то время как при изотермических отжигах на это требуется не менее 1 часа. Следовательно, как и для атомов легирующих примесей, для атомов основного материала имеет место увеличение их подвижности. Таким образом, отмеченное ранее отсутствие влияния состояния покрытия на распределение атомов по глубине проникновения связано с ускоренным изменением фазового состава в исходном покрытии под действием ЭИЛ.

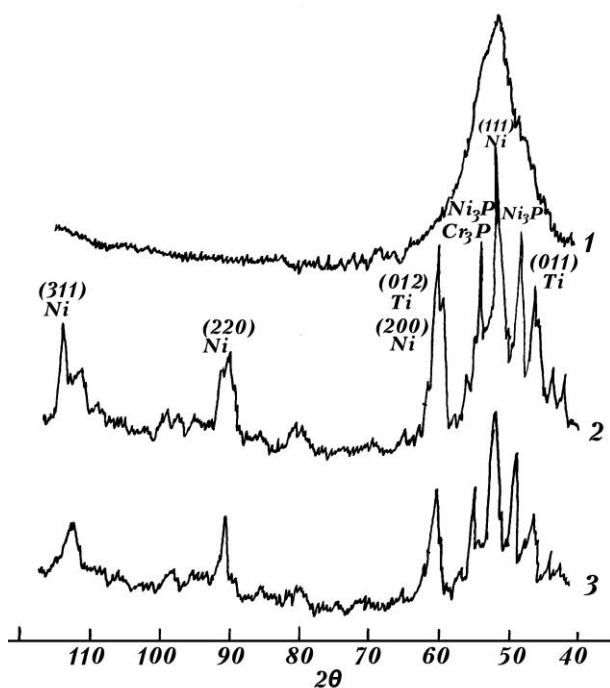


Рис. 15. Рентгенограмма титанового образца с никель-фосфорным покрытием в исходном состоянии (1) после легирования хромом в среде азота с энергиями 0,9 Дж (2) и 3,1 Дж (3), Со-излучение

При введении углерода из среды возникают твердый раствор углерода и титана в уже существующем растворе фосфора в никеле, раствор углерода и титана в никеле и часть атомов углерода и титана из подложки попадает в фосфид никеля. Новые фазы внедрения не появляются в покрытии и диффузионной зоне.

При легировании никелем в среде азота вследствие взаимной диффузии никеля и титана в прилегающих к покрытию слоях образца происходит их взаимное растворение и образование нитридов никеля Ni_3N и титана TiN . Что же касается упрочняющей фазы фосфида никеля, наблюдается частичный распад Ni_3P и связывание фосфора в твердый раствор с никелем, поступающим в покрытие из анода. Кроме того, часть атомов азота попадает как в твердые растворы, так и растворяется в фосфиде никеля.

Легирование хромоникелевым сплавом ВЖЛ приводит к аналогичному результату - исчезновению всех рефлексов, кроме соответствующих раствору легирующих элементов и титана в никеле. В то же время при введении атомов хрома в зоне взаимодействия остается некоторое количество соединения Ni_3P . Об этом свидетельствует соотношение интенсивностей линии (200) в K_α и K_β - излучении, превышающее 1:13, как это следует в отсутствие наложения другой фазы. По-видимому, хром частично растворяется в твердом растворе никеля с фосфором, а некоторое его количество проникает в соединение Ni_3P ($a = 0,9012$ нм, $c = 0,4422$ нм), растворяясь в нем или вытесняя из него атомы никеля с образованием соединения Cr_3P , обладающего той же симметрией (ОЦТ) и несколько отличающегося параметрами кристаллической решетки от фосфида никеля. Для фосфида хрома Cr_3P значения параметров решетки составляют $a = 0,9185$ нм, $c = 0,4560$ нм.

При легировании углеродом и азотом (угольный анод, азотная среда) образуются твердые растворы $NiTi-C$ и $Ti-N$, а также карбиды и нитриды Ti_2N титана и нитриды никеля Ni_3N . При этом сохраняется

симметрия образовавшихся фаз, но нарушается стехиометрические соотношения в фазах внедрения – во всех случаях наблюдается избыток металлов.

Отметим, что, как и при взаимодействии железа и молибдена, при нанесении взаимодействия титана с никель-фосфорным исходным покрытием с азотом и углекислым газом при увеличении энергии разряда возрастает гомогенность твердых растворов внедрения титана и никеля (кривые 2 и 3 на рис. 14, 15). Аналогичный результат гомогенизации фаз имеет место при использовании анодов, изготовленных из никель-хромового сплава ВЖЛ и хрома, как при обработке в среде азота, так и на воздухе.

ВЫВОДЫ

Таким образом, с помощью комплекса различных методов физико-химического анализа установлено, что на процесс введения в искровом разряде легирующих примесей в металлы и сплавы, находящиеся в различном структурно-фазовом состоянии, всегда накладывается проникновение газов среды, в которой осуществляется обработка. Это необходимо учитывать, поскольку состав, а, следовательно, и свойства поверхностного слоя, как правило, отличаются от требуемых. И это можно использовать для создания необходимых состава и свойств изделий, учитывая, что в данном обзоре рассмотрены многие конструкционные материалы и промышленные режимы обработки искровыми разрядами.

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(297X210 mm). Parametrii paginii: 25 - stînga (Left), 20 - sus (Top), 20 - jos (Bottom), 15 - dreapta (Right), 12,5 - antet (Header), 0 - subantet (Footer), 2 coloane. Dimensiunile fontului de imprimare - 12 points. Aliniatela - 1 cm. Spațiul dintre liniile (Line Spacing) aceluiasi paragraf, inclusiv titlul lucrării și informațiile despre autori - un interval. Se va accepta cu trecerea cuvintelor dintr-un rînd în altul. Ultima pagină, în limita posibilităților, va fi completă.

2.4. Structura articolului

CZU se va situa în partea stîngă a paginii.

Titlul se va da complet, maximum 3 rînduri, pe toată lățimea paginii (12 points, BOLD, CENTER, ALL CAPS).

Informațiile despre autori se vor da cu aldine, în limba în care este scrisă lucrarea, în următoarea consecutivitate: Numele și Prenumele autorului, afilierea. Dacă coautorii lucrării sînt angajații aceleiași instituții, denumirea ei se va da o singură dată. Pentru corespondența se indică e-mail-ul unuia din autori, însemnat cu asterix.

Rezumatul va cuprinde descrierea succintă a obiectului, metodelor și rezultatelor cercetării în limbile română și engleză și nu va depăși 10 rînduri. Mărimea caracterilor - 10 points. Cuvîntul „Rezumat” nu se va indica.

Cuvintele-cheie vor fi indicate după rezumatul articolului în limbile originalului, română și engleză.

Introducerea va reflecta stadiul actual al cercetărilor în domeniu. În caz de necesitate, va cuprinde o scurtă analiză istorică. Introducerea se va încheia cu expunerea scopului lucrării.

Conținutul lucrării va include expunerea metodicii cercetării (experimentală sau teoretică), obiectul cercetării, echipamentul, metodele de măsurare și de observare, precizia și erorile metodicii experimentului. Se vor indica rezultatele obținute și analiza lor. Nu se va admite repetarea datelor în tabele, desene și texte.

În concluzii se va expune succint esența cercetării efectuate, relievăndu-se importanța și gradul de noutate a rezultatelor obținute.

Titlul fiecărui paragraf se va evidenția cu aldine. Titlurile de capitol vor fi separate de textul curent printr-un spațiu.

În fața textului fiecare titlu de subcapitol cu doi indici se lasă un spațiu liber de un rînd. Aliniatela se vor marca prin introducerea unui „<Tab>”. Pentru scoaterea în relief a unor concepte se vor folosi aldinele (**fără subliniere**).

Tabele se vor numerota cu cifre arabe în partea stîngă (de ex.: „Tabelul 1”), după care, în același rînd, va urma denumirea și tabelul propriu-zis. Tabelele vor fi separate de textul curent printr-un spațiu. Toate liniile ce formează coroaiașul tabelului vor avea aceeași grosime (1 points). În tabela textuală cifrele se vor scrie cu fontul 10 points, normal. Dacă textul va conține un singur tabel, acesta nu se va numerota.

Ilustrațiile (figurile, schemele, diagramele, fotografiile etc.) se vor prezintă în alb-negru, inserate în textul de bază sau pe foi aparte. Toate figurile se vor numerota cu cifre arabe (în ordinea apariției lor în lucrare), după care se va da legenda lor. Toate semnele sau marcările ilustrate se vor defini în legendă. În cazul mărimilor fizice, se vor indica unitățile de măsură. Dacă lucrarea va conține o singură figură, ea nu se va numerota. Figurile vor fi separate de textul curent printr-un spațiu. Fotografiile introduse în text se vor scana cu o rezoluție de minim 300 dpi (preferabil 600 dpi) și se vor prelucra pentru un contrast bun.

Nu se admite lipirea fotografiilor sau desenelor pe foi separate. Adnotările de pe figuri se vor face în cifre sau litere cu înălțimea caracterelor echivalentă fontului 10 points. Legenda se va culege cu 10 points.

Formule matematice. Toate formulele matematice se vor scrie, **ÎN MOD OBLIGATORIU**, cu editorul de ecuații din procesorul de texte Microsoft Word for Windows'95/, 97/, 98/, 2000, (Version 6.0/Version 7.0, 2000) italice, centrat, prin culegerea fiecăreia din rînd nou. Exigențele corespunzătoare vor urma imediat după formulă și se vor introduce prin „unde”, respectîndu-se ordinea semnelor din ecuație sau relație. Dacă textul va conține mai multe ecuații sau relații, acestea se vor numerota cu cifre arabe la sfîrșitul rîndului, în partea dreaptă a coloanei. După descifrarea simbolului-literă, se va pune virgula, apoi se va indică unitatea de măsură.

Unitățile de măsură ale mărimilor fizice se vor prezenta în sistemul internațional de unități (SI).

Bibliografia Termenul „Bibliografie” va fi separat de textul curent prin spațiu. În text, referințele se vor insera prin cifre încadrate între croșete, de exemplu: [2], [5-7], și se vor prezenta la sfârșitul articolului într-o listă aparte, în ordinea apariției lor în text, în conformitate cu cerințele CNAA al Republicii Moldova. Referințele bibliografice se vor da în limba originalului. Nu se vor accepta referințe la surse nepublicate.

III. OBSERVAȚII FINALE

Informația despre autori și rezumatele în alte limbi decât originalul se vor plasa după

bibliografie. Conținutul rezumatului expus în trei limbi va fi identic.

Materialul cules se va prezenta în format electronic prin e-mail la adresa secretarului științific (alexandr.ozhegov@yahoo.com) sau la adresa redactorului-șef al revistei (pavel.topala@gmail.com), precum și într-un exemplar printat (cu contrastul bun) semnat de toți autorii (după bibliografie).

Pentru relații suplimentare se va indica adresa, numărul de telefon și e-mail-ul unuia dintre autori.

Articolele care nu vor corespunde cerințelor expuse, normelor limbii și stilului vor fi respinse.

Materialele prezentate la redacție nu se vor restitui autorului.

Prezentat la redacție la DD MMMM YYYY

GUIDELINES FOR SUBMISSION OF SCIENTIFIC PAPERS TO THE JOURNAL „PHYSICS AND TECHNICS: PROCESSES, MODELS, EXPERIMENTS”

Name_ author P.^{1*}, Name_ author P.², Name_ author P.[”]

¹Full address of the first author’s institution

²Full address of the second author’s institution

[”]Full address of the next author’s institution

* e-mail: adresa e-mail a autorului pentru corespondență

Abstract: (max 10 lines) The sleep of reason produces monsters, the sleep of reason produces monsters, the sleep of reason produces monsters, the sleep of reason produces monsters, the sleep of reason produces monsters, the sleep of reason produces monsters, the sleep of reason produces monsters, the sleep of reason produces monsters, the sleep of reason produces monsters, the sleep of reason produces monsters, the sleep of reason produces monsters, the sleep of reason produces monsters, Francisco José de Goya y Lucientes.

Keywords: (please, list 5-6 keywords).

Rezumat: (maximum 10 rînduri) Somnul rațiunii naște monștri, somnul rațiunii naște monștri, somnul rațiunii naște monștri, somnul rațiunii naște monștri, somnul rațiunii naște monștri, somnul rațiunii naște monștri, somnul rațiunii naște monștri, somnul rațiunii naște monștri, somnul rațiunii naște monștri, somnul rațiunii naște monștri, somnul rațiunii naște monștri, somnul rațiunii naște monștri, Francisco José de Goya y Lucientes.

Cuvinte-cheie: (introduceți 5-6 cuvinte-cheie).

I. THE JOURNAL SCOPE

The submitted materials should reflect recent achievements of the Alecu Russo Balti Sate University departments and research laboratories and of educational and research institutions from the Republic of Moldova and abroad.

The journal publishes scientific articles related to the following research areas:

- radio wave propagation in various media;
- development of numerically-analogical electronic devices;
- development of lasers and their application in material processing technologies;
- the study of external factor influence on the physical properties of the substance;
- classical and non-conventional technologies of material processing.

II. REQUIREMENTS FOR ARTICLE SUBMISSION

2.1. General arrangements

Papers that have been published in other editions are not accepted. Articles should be accompanied by an extract from the minutes of the Chair/Laboratory/Scientific Seminar sitting

where the papers had been discussed and recommended for publication. The submitted papers should be subject to the review of qualified specialists in the field, appointed by the editorial board.

2.2. Paper format and rules for submission

A person may be the author and / or coauthor of at most two articles. The volume of an article should not exceed, as a rule, 7 pages.

Each article should include:

- UDC;
- the title in original language, Romanian and English;
- information about the author/authors in original language, Romanian and English;
- the abstract in Romanian and English;
- the keywords in Romanian and English;
- the article itself;
- the bibliography.

2.3. Use of electronic support

Materials should be edited in **Microsoft Word, Times New Roman** font and page size should be A4 (297X210 mm). Page parameters: 25 - Left, 20 - Top, 20 - Bottom, 15 - Right, 12.5 - Header, 0 - Footer, 2

columns. Font size Print - 12 points. Paragraphs - 1 cm. Line Spacing within the same paragraph including the title and information about the author – one interval. Transference of words from one line to another is accepted. It is desirable that the last page should be complete.

2.4. Structure of the article

UDC should be placed on the left side of the page.

The title should be complete, up to 3 lines, on the full width of the page (12 points, BOLD, CENTER, ALL CAPS).

The information about the authors should be given in bold in the language of the written paper in the following sequence: author's surname and first name (full), affiliation. If the co-authors are employees at the same institution, its name should be given only once.

The abstract should include a brief description of the subject matter, methods and research findings and should not exceed 10 lines. Font size - 10 points. The word "Abstract" should not be typed.

The keywords will be listed after the abstract in original language, Romanian and English.

The introduction should reflect the current state of research in the field. If need be, it will include a brief historical analysis. The introduction should end with an account of the paper aims.

The main text should include an account of the research methodology (experimental or theoretical), subject of research, equipment, measurement and observation methods, the precision and errors of the experiment methodology. It should include the results and their analysis. It is not allowed to repeat data in tables, drawings and texts.

The conclusion should briefly expound on the essence of conducted research highlighting the importance and degree of the novelty of results.

The title of each paragraph should be in bold. A space should be left between chapter titles and the text.

One line space is left before the text of each two index sub-chapter title. Paragraphs should be marked by the introduction of a

"<Tab>". Bold type (no underlying) should be used to emphasize certain concepts.

The tables should be numbered with Arabic numerals on the left (e.g.: "Table 1"); this should be followed in the same line by the title and the table itself. The tables should be separated from the current text by one space. All the lines that form the table welding should have the same thickness (1 point). The font of the figures in the text of the table should be normal, 10 points. If the text contains a single table, it should not be numbered.

The illustrations (figures, charts, diagrams, photos, etc...) should be black and white, inserted in the main text or on separate sheets. All figures should be numbered with Arabic numerals (in order of their appearance in the paper) after which they should be explained. All signs and markings should be defined in the explanatory text. If there are physical dimensions, the measurement units should be indicated. In case the work contains only one figure it should not be numbered. The figures should be separated from the current text by a space. The photographs included in the text should be scanned at a minimum of 300 dpi (preferably 600 dpi) resolution and should be processed for a good contrast.

It is not allowed to stick photos or drawings on separate sheets. The notes on figures should be in numbers or letters with similar 10 point font characters. The explanatory text should be typed with the same character size - 10 points.

Mathematical formulas. **It is obligatory** to write all mathematical formulas using the equation editor of Microsoft Word processor for Windows 95/97/98/2000, (Version 6.0 /, Version 7.0, 2000) in italics, centered, each one should be typed beginning with a new line. The corresponding requirements should immediately follow the formula beginning with "where" and observing the order of signs in the equation or relation. If the text contains more equations or relations, they should be numbered with Arabic numerals at the end of the line on the right side of the column. A comma should be used after the letter - symbol is deciphered; the measurement unit should be indicated.

The measurement units of physical dimensions should be presented through the International System of Units (SI).

The bibliography. The term "*Bibliography*" should be separated from the text by space. In the text, the references should be inserted by numbers enclosed in square brackets, e.g. [2], [5-7], and should come at the end of the article in a separate list in order of their appearance in the text, in concordance with requirements submitted by NCAA of the Republic of Moldova. The references should be given in the source language. References to unpublished sources are not allowed.

III. CONCLUDING REMARKS

The information about authors and the abstracts in languages other than the source one

should be placed after the bibliography. The content of the abstract presented in three languages should be identical.

The typed material should be presented both in electronic format send by e-mail (on the scientific secretary address alexandr.ozhegov@yahoo.com or on the responsible editor's address pavel.topala@gmail.com) and in a printed copy (with good contrast) signed by all authors (after the Bibliography).

One of the authors' address, telephone number and E-mail should be indicated for additional information.

The articles that do not meet the described requirements, the norms of language and style will be rejected.

The materials submitted to the editorial board will not be returned to the author.

Prezentat la redacție la DD MMMM YYYY