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ЕЖЕМЕСЯЧНЫЙ НАУЧНО-ТЕХНИЧЕСКИЙ ЖУРНАЛ ПО АНАЛИТИЧЕСКОЙ ХИМИИ, ФИЗИЧЕСКИМ, МАТЕМАТИЧЕСКИМ И МЕХАНИЧЕСКИМ МЕТОДАМ ИССЛЕДОВАНИЯ, А ТАКЖЕ СЕРТИФИКАЦИИ МАТЕРИАЛОВ

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ABSTRACTS

UDC 543.544.45

The Main Source and Compensation of the Random Component of Errors in Determination of the Areas of Chromatographic Peaks
Zenkevich I. G. and Prokof'ev D. V.

Scattering of chromatographic peak areas is the main source of the uncertainty of chromatographic quantitation. Scattering is attributed to several factors but non-controlled partial losses of the samples injected into chromatographic column is the most important reason among them. However, it is shown that peak areas of the same components (including the solvent) of the same samples correlate well with each other, which gives ground for replacing the absolute peak areas of target analytes by their ratios to peak areas of additional (other than internal) standards, e.g., solvents contained in the samples can be used as additional standards, due to the absence of principal restrictions on their chemical nature.

The advantages of the procedure are considered on the example of the method of internal standard. Similar approach can be used in the methods of standard addition and absolute calibration. The uncertainty of the results (the ratio of the variation coefficients of the absolute and relative peak areas) decreases 5–30 times depending on the scatter of peak areas determined experimentally.

Keywords: chromatographic quantitation; uncertainty; additional standards; solvent as additional standard.

UDC 543.552

Voltammetric Determination of Cholesterol in Food*Derina K. V., Korotkova E. I., Dorozhko E. V., Voronova O. A., and Chulkova I. V.*

We report on a cholesterol electrochemical behavior on chemically modified carbon-containing electrode. All the experiments are carried out on a voltammetric workstation. The dependence of the cholesterol electrooxidation signal on pH, electrochemical concentration (potential and time) and scan rate is demonstrated. Working conditions (pH = 6.86, $v = 40$ mV/sec) of cholesterol determination in model media are specified. The linear dependence of cholesterol electrooxidation current at a potential of +1.06 V on the cholesterol concentration is observed up to $50 \mu\text{mol}/\text{dm}^3$. The detection limit is $0.07 \mu\text{mol}/\text{dm}^3$. Results of comparative determination of cholesterol in food using voltammetric and spectrophotometric procedures are presented.

Keywords: cholesterol; modifier; bicyclic bisureas; voltammetry; spectrophotometry; food.

UDC 543.4.546.3

Synthesis of ZnO-Based Thin Films Doped with Ga/In and Determination of Their Structure Using X-Ray Spectroscopy and Inductively Coupled Plasma Mass Spectrometry*Filatova D. G., Vorob'eva N. A., Rumyantseva M. N., Baranovskaya V. B., Baranchikov A. E., Ivanov V. K., and Gas'kov A. M.*

An approach to studying the composition of synthesized zinc-based thin films doped with In and Ga using local X-ray spectroscopy (LXS) and inductively coupled plasma mass spectrometry (ICP-MS) is developed. The dependence of the content and distribution of the additives in a sample on conditions of synthesis on rotating substrates is derived. The results of ICP-MS determination of predominant impurities in the solutions of films are used for validation of the results obtained by LXS without sample preparation. It is shown that dopant impurities at a level of less than 1 % at. may be determined only using inductively coupled plasma mass spectrometry.

Keywords: advanced materials; thin films; inductively coupled plasma mass spectrometry; X-ray spectroscopy; zinc; gallium; indium.

UDC 543.423

Preconcentration of Toxic Elements on Carbon Nanotubes for Atomic Spectroscopic Methods of Environmental Analysis*Grazhulene S. S., Telegin G. F., Zolotareva N. I., Redkin A. N., and Milnikova Z. K.*

A possibility of using carbon nanotubes (CNT) (obtained at different temperatures of synthesis and functionalized in oxidative treatment with concentrated HNO_3) for extraction of toxic elements from aqueous media and their subsequent determination is studied. Sorption of Be, Bi, Cd, Cr, and Pb ions from aqueous solutions is studied as a function of their concentration, pH of the solution, temperature and modification time and compared with the same sorption characteristics of activated carbon. Optimal conditions of preconcentration are specified for further analysis of the concentrate both as a solid phase isolated from the column (using arc atomic emission spectroscopy (AES)) and as an eluate (using atomic absorption analysis (AAA)). The determination limits at a preconcentration coefficient $C_{\text{conc}} = 300$ range within $1 \times 10^{-8} - 3 \times 10^{-7} \%$ wt. (AES) and $0.1 - 0.5 \text{ ng}/\text{ml}$ (AAA), respectively. Accuracy of the determinations is proved in spiking tests and by comparison of AAA and AES data. The obtained results can be used in the analysis and purification of different process media and for environmental protection as well.

Keywords: sorption; carbon nanotubes; ions of toxic elements; flame atomization; atomic absorption analysis; arc atomic emission spectrometry.

UDC 621.762

The Effect of Heat Treatment on the Strength and Wear Resistance of T15K6 Hard Alloy*Bogoduhov S. I., Kozik E. S., and Svidenko E. V.*

The results of studying operational properties of T15K6 hard alloy subjected to heat treatment in salt baths of different composition are presented. Heating temperatures of

quenching (both in 120 oil and in air) and tempering are 1050 ± 100 and 400 ± 200 °C, respectively. Cutters with quick-change carbide inserts and rods (GOST 3882–74) are used in the experiments. Vickers hardness, cutting-induced wear, fine structure parameters, microstructure and surface fracture of the alloy are studied as a function of heat treatment modes. The microstructure of the material after heat treatment is studied on a μ Vizo-MET-221 metallographic microvisor and Jeol JCM-6000 scanning electron microscope. Tensile strength is determined in bend tests of carbide rods on a IR 5047-50-10 universal tension testing machine. Analysis of the degree of structure imperfection, stress state of the carbide phase and determination of phase composition is performed using an MD-10 microdiffractometer.

Keywords: hard alloy; heat treatment; hardness; wear; x-ray structure analysis; microstructure.

UDC 54.084

Determination of the Grain-Size Distribution of Zirconia Based Powders by the Methods of Static Laser Scattering and Optical Microscopy

Mironov R. A., Zabezhaylov M. O., Yakushkina V. S., and Rusin M. Yu.

Dispersion conditions of zirconia doped with 3 % mol yttrium oxide are specified. Grain-size distribution of the power particles is determined using static laser scattering and optical microscopy. Electrophoresis with titration is used for measuring the dependence of zeta-potential of zirconia suspension on pH value. The effect of the acidity on the stability of suspension during measurement is considered. It is shown that stable suspension can be obtained by shifting pH value of dispersion media both to acidic or alkali regions and also by adding surface active substances (e.g., Dolapix CE 64).

Keywords: static laser scattering; particle size distribution; isoelectric point of zirconia; stabilization of suspensions.

UDC 621.762:620.22

Study of the Properties of Fe-P Powder Materials Using Static Magnetic Measurements

Kem A. Yu. and Kitaev V. V.

The use of the methods of powder metallurgy (PM) appeared promising in manufacturing core pieces and electromagnets working in medium and strong static magnetic fields thus significantly improving the efficiency of production of magnetic circuits of electrical machines made of soft magnetic powder materials. One of the problems attributed to assessing the quality of soft magnetic powder materials consists in the lack of reliable methods for measuring the magnetic properties of materials obtained by PM-technology. We propose a method for measuring the basic magnetization curve $B(H)$, magnetic hysteresis loop, as well as magnetic parameters of powder materials (initial μ_{in} and maximum μ_{max} magnetic permeability, remanence B_r and coercivity H_{cA}). The method is advantageous for the possibility of real-time measurements of multi-stage changes of the magnetic flux (using electronic microwebermeter F-191). Coercivity force H_{cA} is determined using extrapolation of the dependence of the width of the hysteresis loop for ring samples on the value of maximum magnetizing field. Measurements are carried out at successively increasing magnetizing field. The tested method of measurements provide determination of the magnetic properties of powder samples and features of formation of the magnetic properties of Fe-P powder soft magnetic materials attributed to the prehistory of their production which results in the porosity and defect structure of the material.

Keywords: powder metallurgy; soft magnetic material; measurement technique; magnetic properties; hysteresis loop; magnetic permeability; magnetic flux density; coercive force; structural defects; porosity.

UDC 544.013/014:544.016

A Method of Accompanying Signal for Determination of the Chemical Composition of Multicomponent Phases

Golovkin B. G.

A general method for determination of the chemical composition of multicomponent phase in equilibrium solid-phase mixtures without their isolation is developed. The method is based on the dependence of any of intensive or extensive characteristics of the system (accompanying signal: reflection intensities, lines of Raman and IR spectra, thermal effects, etc.) on the composition of test samples of the system under study. For example, to determine the unknown phase X ($A_p B_q C_z$, wherein $0 \leq x < y, z, x + y + z \leq 1$, and $z = 1 - x - y$) of a ternary (three-component) system, it suffices to prepare three equilibrium samples $P - A_p B_q C_{1-p-q-r}$, $Q - A_q B_r C_{1-q-r-g}$, and $R - A_r B_g C_{1-r-g-h}$. The intensity of the signal accompanying the reaction in sample P :

$$pA + fB + (1 - p - f)C \rightarrow a(A_p B_q C_{1-p-q-r}) + (p - ax)A + (f - ay)B$$

is $I_p = aI$. Expressions for I_q and I_r follow from the reaction equations in samples Q and R . Solution of the system of the equations thus obtained provides determination of the unknown values x, y , and z . Thus, for n -component systems it is necessary to prepare n samples, measure the intensity of accompanying signal for each sample and carry out similar calculations.

Keywords: phase analysis; multi-component system; method of accompanying signal.

UDC 620.179.18:539.42:531.7.08

Determination of the Parameters of Two-Parameter Mechanics of Fracture Along the Crack Front Using Data of Digital Image Correlation

Chernyatin A. S., Matvienko Yu. G., and Lopez-Crespo P.

A method of mathematical processing and algorithm of numerical adjustment of experimental fields of displacements on a specimen surface in the vicinity of the crack

tip is proposed taking into account displacement of the body as a rigid unit upon loading and actual position of the crack tip. The initial experimental displacement fields around the tip of the fatigue crack in a compact specimen are determined by the method of digital image correlation. Taking a compact specimen as an example we demonstrate the possibility of successful using of this approach to obtain the distribution of the stress intensity factors and nonsingular T -stresses along the front of a spatial crack.

Keywords: compact specimen; stress intensity factor; T -stress; experimental and numerical method; digital image correlation method; mathematical data processing; minimization problem; finite element method.

UDC 620.1:621.311.001.57:51–74

Simultaneous Measurement of the Strain and Temperature Using Fibre Bragg Grating-Based Sensors (a generalizing article)

Makhsidov V. V., Shiyonok A. M., Ioshin D. V., and Reznikov V. A.

Fibre sensing elements based on Bragg gratings (FBG) form a promising basis for developing sensors used for determination of the deformation in measuring systems, in particular, for built-in control of different designs. A possibility of their use in the structure of polymeric composite material intended for aircraft industry. The operation principle of the FBG is based on a change of the lattice constant — periodic structure of the refractive index of the optic fiber core — which can entail also thermal expansion (compression) of the fiber. Therefore, bearing in mind the goal of built-in control, it is important to understand what is the reason for optical fiber deformation in the site of FBG location - mechanical loading or change in temperature conditions. We systematize different approaches to consideration of temperature changes occurred upon strain measurements using FBG and present the main results thus gained. Moreover, we describe possible pathways of implementing each approach, provide data on the accuracy of strain and temperature measurements and present a structural design of the sensor.

Keywords: fibre optic sensor; fibre Bragg grating (FBG); deformation; polymer composite material (PCM); built-in control.

UDC 620.169:621.646.1

Evaluation of Stress-Strain State of the Valves Taking into Account the Operating Parameters on the Example of Wedge Gate Valves ZKL2 300-25

Zakirnichnaya M. M., Kul'sharipov I. M., and Chernova A. Yu.

The effect of technological flow parameters on the stress-strain state of structural elements of wedge gate valves is considered in the case study of ZKL2 300-25 using ABAQUS software package. Numerical calculations are performed with allowance for the temperature of liquid (water, mazut and petrol) flowing through the valve in given range of the flow velocities. The main parameters affecting the life-time of the structural elements of valves are determined. An algorithm of integrated approach to evaluation of the durability of wedge gate valves is developed. The algorithm can be recommended for institutions developing design specifications and estimates regarding repair, reconstruction, expansion and technical re-equipment of hazardous industrial facilities.

Keywords: wedge gate valve; stress-strain state; remaining lifetime; ABAQUS; FE-SAFE.

UDC 519.24

Application of the Statistical Approach to Construction of Direct and Inverse Characteristics of the Object

Skibitskii N. V.

A problem of determining direct and inverse static characteristics of the object with interference present is considered. It is shown that solution of the problem in the framework of the statistical approach often ignore the existence of various sources and triggers of model interferences, which leads to substantial distortion of the estimates of errors and formation of inadequate conversion response. The types and sources of errors in determination of the static characteristic and conditions under which the use of statistical approach is valid are considered.

Keywords: static characteristic; experiment; distribution law; errors in variables.

UDC 519.2

Energy Criterion for Assessing Potential Qualitative Characteristics of Measurement Experiment in Conditions of Correlated Monitoring Noise

Ausiannikau A. V.

A generalized energy criterion which makes possible to assess the potential quality of a measuring experiment and link the accuracy of the experiment (the lower boundary of the error estimate variance) and time of achieving the accuracy in a certain class of distributions of correlated monitoring noise is determined. The use of energy criterion is considered for measuring experiment conducted in a discrete or continuous observation time. It is shown that regardless of the time of the experiment the resulting expressions have internal unity. The expressions of the energy criterion are derived which provide correlation between the parameters of the measuring experiment: duration of measurements; time constant of correlated observation noise; conventional signal-to-noise ratio and desired accuracy of measurements. The relations thus obtained can be used in theoretical and applied calculations if the correlation function of the monitoring noise exhibits exponential or close to exponential character.

Keywords: measuring experiment; energy criterion; correlation function; correlation interval; potential quality.