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ЗАВОДСКАЯ ЛАБОРАТОРИЯ

ДИАГНОСТИКА МАТЕРИАЛОВ

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

СОДЕРЖАНИЕ

КОЛОНКА РЕДКОЛЛЕГИИ

Карпов Ю. А. С Новым 2015 годом! 5

АНАЛИЗ ВЕЩЕСТВА

Болотник Т. А., Смоленков А. Д., Ярцев С. Д., Шпигун О. А. Применение
метода газовой хромато-масс-спектрометрии с предварительной дисперсион-
ной жидкостно-жидкостной микроэкстракцией для определения низких содер-
жаний ракетных керосинов в воде. 6

Петрова Ю. С., Неудачина Л. К., Пестов А. В., Яременко Д. А. Сорбцион-
но-атомно-абсорбционное определение меди в природных и питьевых водах с
предварительным концентрированием сорбентом на основе N-2-сульфоэтил-
хитозана. 11

Дударев В. И., Филатова Е. Г., Дударева Г. Н., Климова О. В., Минае-
ва Л. А., Рандин О. И. Сорбционное концентрирование тяжелых металлов и
определение никеля в производственных растворах. 16

Солманов П. С., Максимов Н. М. Определение микроколичеств азота в
сырье и продуктах нефтепереработки модифицированным микрометодом
Кьельдаля. 23

ИССЛЕДОВАНИЕ СТРУКТУРЫ И СВОЙСТВ

ФИЗИЧЕСКИЕ МЕТОДЫ ИССЛЕДОВАНИЯ И КОНТРОЛЯ

Антонова А. О., Савелова Т. И. Исследование влияния статистической зави-
симости элементов выборки при EBSD измерениях на погрешность вычисле-
ния функции распределения ориентаций. 26

Иванова В. А., Хапеева В. В. Определение истираемости литейного кокса 31

Разумовский А. Ю., Чернов М. А., Василенко А. П., Лошкарев И. Д., Тру-
ханов Е. М. Методические возможности двухкristальных рентгеновских
дифрактометров 34

Головкин Б. Г. Универсальный метод построения фазовых диаграмм с лю-
бым числом компонентов 40

МЕХАНИКА МАТЕРИАЛОВ: ПРОЧНОСТЬ, РЕСУРС, БЕЗОПАСНОСТЬ

Мостовой Г. Е., Фирсова Т. Д. Особенности механических свойств базальто-
вых волокон 44

Шиян А. В., Котречко С. А., Мешков Ю. Я., Сорока Е. Ф. Оптимизация
свойств прочности и пластичности конструкционных сталей с использовани-
ем критерия механической стабильности. 50

Шефер Л. А., Ерпалов А. В., Валеев Д. Х. Обобщенная диаграмма уста-
лости материалов при действии различных случайных, гармонических и по-
лигармонических процессов. 58

МАТЕМАТИЧЕСКИЕ МЕТОДЫ ИССЛЕДОВАНИЯ

Аверченков В. И., Шарапов М. Г., Гулаков В. К., Гулаков К. В. Двухуров-
невая модель выбора состава покрытия сварочных электродов 63

Каламбет Ю. А., Мальцев С. А., Козьмин Ю. П. Доверительные интервалы
метода взвешенных наименьших квадратов и стратегия градуировки 69

CONTENTS

EDITORIAL COLUMN

- Karpov Yu. A. Happy New Year! 5

ANALYSIS OF MATERIALS

- Bolotnik T. A., Smolenkov A. D., Yartsev S. D., Shpigun O. A.** Application of the Method of Gas Chromatography-Mass Spectrometry with a Pre-Dispersive Liquid-Liquid Microextraction to Determination of Low Rocket Kerosene Content in Water 6

- Petrova Yu. S., Neudachina L. K., Pestov A. V., Yaremenko D. A.** Sorption-Atomic-Absorption Determination of Copper in Natural and Drinking Waters with Preconcentration by the Sorbent Based on N-2-Sulfoethyl Chitosan. 11

- Dudarev V. I., Filatova E. G., Dudareva G. N., Klimova O. V., Minaeva L. A., Randin O. I.** Sorption Concentration of Heavy Metals and Determination of Nickel in Industrial Solutions 16

- Solmanov P. S., Maksimov N. M.** Determination of trace amounts of nitrogen in raw and refined products using a modified Kjeldahl micromethod 23

TESTING OF STRUCTURE AND PARAMETERS

PHYSICAL METHODS OF TESTING AND QUALITY CONTROL

- Antonova A. O., Savelova T. I.** Study of the Effect of Statistical Dependence of the Sample Elements upon EBSD Measurements on the Orientation Distribution Function Calculation Error 26

- Ivanova V. A., Khapeeva V. V.** Determination of the Abrasion Foundry Coke Strength 31

- Razumovsky A. Yu., Chernov M. A., Vasilenko A. P., Loshkarev I. D., Trukhanov E. M.** Methodological (Procedural) Capabilities of a Double Crystal X-Ray Diffractometer 34

- Golovkin B. G.** Universal Method of Constructing Phase Diagrams with an Arbitrary Number of Components 40

MECHANICAL TESTING METHODS

- Mostovoi G. E., Firsova T. D.** Features of the Mechanical Properties of Basalt Fibers 44

- Shiyan A. V., Kotrechko S. A., Meshkov Yu. Ya., Soroka E. F.** Optimization of the Properties of Strength and Plasticity of Structural Steels Using the Criterion of Mechanical Stability 50

- Shefer L. A., Erpalov A. V., Valeev D. Kh.** Generalized Fatigue Diagram Under the Impact of Various Random, Harmonic and Polyharmonic Processes 58

MATHEMATICAL TESTING METHODS

- Averchenkov V. I., Sharapov M. G., Gulakov V. K., Gulakov K. V.** The Two-Level Model for Choosing the Composition of Welding Electrode Coatings 63

- Kalambet Yu. A., Maltsev S. A., Kozmin Yu. P.** Confidence Intervals for Weighted Least Squares Technique and Calibration Strategy 69

ABSTRACTS

UDC 543.383.2

Application of the Method of Gas Chromatography-Mass Spectrometry with a Pre-Dispersive Liquid-Liquid Microextraction to Determination of Low Rocket Kerosene Content in Water

Bolotnik T. A., Smolenkov A. D., Yartsev S. D., Shpigun O. A.

A sensitive technique of rocket kerosene determination in water comprising dispersive liquid-liquid microextraction of analytes and their subsequent separation and determination by gas chromatography-mass spectrometry in the mode of chromatogram registration for the selected ions (m/z : 67, 81, 85, 95, 136, 137, 174, 183 and 193) is developed. The effect of the nature, amount of extractant and dispersant agents, salt adding and time of extraction on the efficiency of analyte extraction is studied. The detectable concentrations range within 0.005 – 0.05 mg/liter. The detection limits ($S/N=3$) for kerosene RG-1 and T-1 are 0.0015 and 0.0022 mg/liter respectively. Intra-day and inter-day precision of the results of measurements for the studied concentration ranges from 16 to 9% ($n=3$), and from 20 to 12% ($n=5$), respectively.

Keywords: rocket kerosene; gas chromatography; mass spectrometry; extraction; dispersive liquid-liquid extraction.

UDC 546.56:543.42.062

Sorption-Atomic-Absorption Determination of Copper in Natural and Drinking Waters with Preconcentration by the Sorbent Based on N-2-Sulfoethyl Chitosan

Petrova Yu. S., Neudachina L. K., Pestov A. V., Yaremenko D. A.

Sorption of metal ions by a cross-linking N-2-sulfoethyl chitosan with a substitution degree 0.5 (SEC 0.5) is studied in dynamic conditions. It is shown that SEC 0.5 selectively extracts copper (II) and silver (I) ions from ammonium acetate buffer solution containing a number of transition and alkaline earth metal ions. The aforementioned metal ions are quantitatively desorbed from SEC 0.5 surface with 0.1 mole/dm³ nitric acid. The developed technique of sorption-atomic-absorption determination of copper in natural and drinking water with preconcentration by SEC 0.5 under dynamic conditions exhibits high selectivity and provides determination of copper (II) within a concentration range of 0.001 – 0.1 mg/dm³.

Keywords: sorption dynamics; determination; selectivity; atomic absorption spectrometry; modified chitosan; copper; silver.

UDC 543.544

Sorption Concentration of Heavy Metals and Determination of Nickel in Industrial Solutions

Dudarev V. I., Filatova E. G., Dudareva G. N., Klimova O. V., Minaeva L. A., Randin O. I.

The adsorption activity of carbon sorbents with respect to nickel (II) ions is studied to specify optimal conditions of adsorption and calculate the characteristic parameters of the process. A technique of concentration and determination of nickel in industrial solutions is developed. The relative standard deviation in the determination of a few hundredths of mg/dm³ is 0.08. The possibility of sorption preconcentration and separation of a number of heavy metal ions using carbon sorbents is demonstrated.

Keywords: sorption concentration; heavy metal ions; carbon sorbents; nickel.

UDC 543.632.45

Determination of Trace Amounts of Nitrogen in Raw and Refined Products Using a Modified Kjeldahl Micromethod

Solmanov P. S., Maksimov N. M.

A three-stage (selective concentration of nitrogen compounds, mineralization, indophenol photocolometry) method is developed for determination of trace nitrogen in refinery products using a modified Kjeldahl method. Replacing the Nessler method for indophenol photocolometric determination provides elimination of the time-consuming stage of ammonia distillation after mineralization thus increasing the sensitivity of the method.

Keywords: nitrogen determination; Kjeldahl method; petroleum fuels and lubricants.

UDC 539.26.01

Study of the Effect of Statistical Dependence of the Sample Elements upon EBSD Measurements on the Orientation Distribution Function Calculation Error

Antonova A. O., Savelova T. I.

Simulation of statistically dependent orientations is carried out using specialized Monte Carlo method for the central normal distribution (CND). The re-

sults of statistical modeling, smoothed by nuclear techniques are presented in the form of three-dimensional surfaces. Quantitative characteristics of orientation distribution function (ODF): shift in the coordinate of ODF maximum, increase in the value of maximum deviation from the exact ODF in L_1 and L_2 metrics are compared. A hypothesis of the coincidence of the exact and model density distribution of orientations is tested using χ^2 criterion for pole figures (PF).

Keywords: EBSD method; specialized Monte Carlo method; orientation distribution function (ODF); statistical dependence orientations.

UDC 621.745

Determination of the Abrasion Foundry Coke Strength

Ivanova V. A., Khapeeva V. V.

A technique of foundry coke abrasion testing is described. The results of studying the effect of strength, size and macrostructure of the foundry coke on the fracture capability under abrasive loads are presented.

Keywords: foundry coke; abrasion test; strength; macrostructure.

UDC 54.07

Methodological (Procedural) Capabilities of a Double Crystal X-Ray Diffractometer

Razumovsky A. Yu., Chernov M. A., Vasilenko A. P., Loshkarev I. D., Trukhanov E. M.

Procedural capabilities of a double crystal x-ray diffractometer (Radicon R&D Center Ltd.) designed for study and control of single crystals and thin film systems using different diffraction methods including high resolution procedures are considered. A double crystal automated x-ray diffractometer DSO-1T with a large "sample to detector" spacing and a narrow slit in front of the detector provides high quality mapping in a reciprocal space of heteroepitaxial structures which is shown for GaAs/Si heterosystem. Double crystal PDP attachment to a DRON-3 diffractometer has five motorized axes, including XZ-table with a travel range $200 \times 200 \text{ mm}^2$. This attachment provides mapping of large plates by diffraction reflection curves, e.g., silicon structures on sapphire. DSO-2P vertical 3-axes double crystal x-ray diffractometer intended for study and control of single crystals up to 101 mm in diameter and 100 mm in height is equipped with a motorized attachment for linear displacement of the detector by 100 mm and a motorized rotary slit switch in front of the detector. The diffractometer provides measurements at extremely large Bragg angle, automatic measurement of crystal orientation, precise measurements of the lattice constant, and control of structural imperfection. A built-in writer of user macros provides a flexibility of the software developed at Radicon R&D Center for x-ray diffractometers.

Keywords: double crystal diffractometer; high resolution; reciprocal space mapping; lattice constant; diffraction reflection curve.

UDC 541.123.7

Universal Method of Constructing Phase Diagrams with an Arbitrary Number of Components

Golovkin B. G.

A method of constructing phase diagrams with an arbitrary number N of components which provides unique determination of figurative point of the multicomponent phase on the diagram is developed. The inverse problem is non-unique, however, determination of the composition becomes possible, if the content of $N - 3$ components is known.

Keywords: phase analysis; phase diagrams; multi-component systems.

UDC 620.172.22:677.5

Features of the Mechanical Properties of Basalt Fibers

Mostovoi G. E., Firsova T. D.

The results of the tensile tests of continuous basalt fiber (CBF) obtained in an experimental furnace are presented. A possibility of significant linear interpretation of the scale dependence of the mechanical characteristics on the fiber diameter is demonstrated within a diameter range of $7.2 - 21.6 \mu\text{m}$: the correlation coefficients are, respectively, for the tensile strength $r(\sigma_a, d) = (-0.70)$, for the ultimate strain $r(\varepsilon_{i0}, d) = (-0.48)$, for the elastic modulus $r(E, d) = (-0.44)$. The differences observed in the scale dependences are attributed to the special features of radial anisotropy of the fiber structure and to the different impact of defects on the mechanical characteristics depending on their location in the radial direction. Those factors also affect the correlation between the mechanical properties of CBF: correlation between the yield strength and ultimate strain is 0.88, between the tensile strength and modulus of elasticity is +0.35 (a significant deviation), while correlation between the modulus of elasticity and the ultimate strain are virtually absent $r(E, \varepsilon_{i0}) = -0.02$. Strain diagram of studied CBF, unlike glass and carbon fibers, are characterized with broken lines, which is the inflection point $(0.4 - 0.6)\sigma_a$. The relationships thus considered provide optimization of the process of high-strength CBF production.

Keywords: heat resistance; basalt fibers; composite materials; multifilament yarn; spun node; filaments; correlation coefficient.

UDC 620.17:620.162.2

Optimization of the Properties of Strength and Plasticity of Structural Steels Using the Criterion of Mechanical Stability

Shiyan A. V., Kotrechko S. A., Meshkov Yu. Ya., Soroka E. F.

Correlation of the properties "plasticity - strength - mechanical stability" of structural steels is shown to have a systemic character, and the relationships connecting the plasticity (ψ_0) and strength ($\sigma_{0.2}$) at a constant mechanical stability K_{ms} can be described by parabolic dependences of having two types of extrema, i.e., maximum and minimum. A generalized diagram of the relationship between the properties "plasticity - strength - mechanical stability" is plotted and equations are derived that reflect the structure and properties of the relationship. Using a generalized diagram we determined the intervals of the optimized values of the plasticity and mechanical stability, characterizing the highest levels of the quality for structural steels of different strength. The invariance of the obtained relationships that determine fixed K_{ms} levels to different combinations of strength and plastic properties of the studied materials, modes of their heat treatment, and test temperatures is demonstrated.

Keywords: mechanical stability; optimization of the properties; generalized diagram; optimization curve; optimal combination of the properties.

UDC 620.178.3

Generalized Fatigue Diagram Under the Impact of Various Random, Harmonic and Polyharmonic Processes

Shefer L. A., Erpalov A. V., Valeev D. Kh.

Parameters characterizing different operation loading processes of harmonic, polyharmonic, and accidental type are considered. A technique is developed which provides prediction of the fatigue resistance for the operational processes from a generalized diagram using test data obtained under harmonic loading. This eliminates the necessity of harmonic schematization of random processes using known techniques that result in significant errors in assessing the lifetime.

Keywords: fatigue testing of materials; fatigue curve; generalized fatigue diagram.

UDC 519.24

The Two-Level Model for Choosing the Composition of Welding Electrode Coatings

Averchenkov V. I., Sharapov M. G., Gulakov V. K., Gulakov K. V.

The problems of modeling multivariate dependencies on the basis of experimental data using the methods of cognitive modeling are considered. A method based on neural network approach is developed to construct a regression model of the dependence of the strength characteristics of welded joints on the composition of the electrode coating. The results of neural network modeling of the toughness function are presented.

Keywords: cognitive modeling; artificial neural networks; welding materials; neural network approximation; neural network modeling.

UDC 519.24

Confidence Intervals for Weighted Least Squares Technique and Calibration Strategy

Kalambet Yu. A., Maltsev S. A., Kozmin Yu. P.

We propose to use the calibration strategy based on the method of weighted least squares and a priori information about the measurement system and method of analysis. The strategy allows for the lowest possible detection limits and determinations, as well as for proper assessing of the prediction error of the analyte content from the magnitude of the analytical signal. A priori information including the dependence of the measurement variance on the magnitude of the analytical signal is obtained at stage of validation of the analytical procedure.

Method of least squares is used most often to approximate the dependencies in analytical chemistry, photography, physics, economics, and the expected measurement errors are explicitly or implicitly assumed to be the same for all the points. Allowance for the inequality of measurement errors (heteroscedasticity) leads to a variant of the method of weighted least squares, however, the theory of developing the confidence intervals in this case has not yet found wide practical application. To implement the calibration strategy we develop a model program capable of calculating the profile of the dispersion curve and providing plotting of the calibration curves with allowance for heteroscedasticity of the errors.

Keywords: least squares; ordinary least squares (OLS); weighted least squares (WLS); weighted regression; confidence interval; confidence band; heteroscedasticity; calibration.