

ЖУРНАЛУ **85** ЛЕТ

ЗАВОДСКАЯ
ЛАБОРАТОРИЯ
ДИАГНОСТИКА МАТЕРИАЛОВ

1

часть I

2017

ЯНВАРЬ

№ 1 ^{часть I} ТОМ 83
2017

Основан в январе 1932 г.

Адрес редакции:

119334 Москва, Ленинский пр-т, 49,
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Журнал включен в список изданий,
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лаборатория. Диагностика материалов», 2017

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**ЗАВОДСКАЯ
ЛАБОРАТОРИЯ**
ДИАГНОСТИКА МАТЕРИАЛОВ

ЗАВОДСКАЯ ЛАБОРАТОРИЯ

ДИАГНОСТИКА МАТЕРИАЛОВ

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

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Analytical Control is an Integral Part of the Diagnostics of Materials

Karpov Yu. A. and Baranovskaya V. B.

Analytical control is an applied segment of analytical chemistry, including selection of the representative sample, determination of the chemical composition (elemental, phase, molecular, isotopic, etc.), evaluation of the metrological characteristics of the results obtained, and conclusion about the conformance of the objects under study to the specified requirements. Those basic components of analytical control are described in the article, along with such important aspects as standardization and accreditation of analytical laboratories. Special attention is paid to the methodology as the main component of analytical control, i.e., to the metrological characteristics and metrological support of analysis.

Keywords: analytical control; metrology of chemical analysis; certification of the methods of analysis; accreditation

UDC 543.51:543.621

Optimization of Conditions of Laser Sampling and ICP-MS Analysis

Khvostikov V. A., Karandashev V. K., and Burmii Zh. P.

The influence of laser radiation parameters on the sensitivity coefficients of relative sensitivity of element determination in analysis of different materials using ICP-MS method combined with laser ablation is studied. The mechanism of laser ablation is shown to change from thermal to "phase explosion" in the samples of silicon glass, basalt glass and polymetal sulfide as the laser power density increases above 2×10^{10} W/cm². Coefficients of the relative sensitivity of trace elements determination against matrix elements are shown to change by 3 – 5% when the laser power density varies within the range of 5×10^9 – 1.5×10^{11} W/cm². For most of the trace elements the coefficients of relative sensitivity of their determination in different matrices thus considered differ by 10 – 15 %, however, for some elements the observed difference is rather large being 1.5 – 2 times and more. A comparative study of two modes of ablation [(i) laser beam scanning along the sample surface and (ii) ablation "in the point"] demonstrated that the sensitivity of element determination is 2 – 3 times higher for the first mode, whereas the coefficients of relative sensitivity of determination being equal for both modes.

Keywords: laser ablation; inductively coupled plasma mass spectrometry (ICP-MS)

UDC 543.542.054

On the Specificity of Sorption Preconcentration of Ru on the Hyper-Crosslinked Polystyrene Sorbents in Analysis of Rock Samples Using Inductively Coupled Plasma – Mass Spectrometry

Dubenskiy A. S., Iakurnova E. D., Seregina I. F., Pavlova L. A., Tsyurupa M. P., Davankov V. A., and Bolshov M. A.

New sorption systems based on hyper-crosslinked polystyrenes "HP" and "Stirosorb" are developed for Ru concentration from final solutions after rock decomposition. Aromatic amines (N-methylbenzylamine (MBA), N,N-dimethylbenzylamine (DMBA), N,N-dibenzylmethylamine (DBMA)) and aliphatic tributylamine (TBA) are used for the first time for Ru extraction from the solutions via reverse-phase mechanism. The reproducible 60% extraction of Ru from a model solution can be achieved using sorbent system Stirisorb-514, reagent TBA and eluent 1 M HCl in ethanol. An important role of solid sample processing in analysis of CRM (Certified Reference Materials) of ultrabasic and basic rock samples is demonstrated. Reproducible quantitative (100%) extraction of Ru using the aforementioned system is achieved only via NiS fire-assay of a solid rock sample followed by the matte digestion in aq. reg. and final dilution in HCl_{conc}. The reasons for remarkable difference in the efficiency of Ru extraction from model solutions and final solutions after solid rock samples digestion are investigated. The interference effect of Ni upon MS determination of Ru by ⁹⁹Ru is studied in a wide range of Ni concentration (20 – 2000 µg/liter). The interference effect of Ni on the signal of Ru is considered negligible. The obtained results proved the possibility of using developed scheme for determination of Ru in rock samples. The developed extraction scheme is validated in analysis of CRMs: GPt-5, GPt-6 and SARM-7. The results of Ru determination in three CRM using the developed scheme match the certified values within the range of experimental errors.

Keywords: ruthenium; sorption preconcentration; hyper-crosslinked polystyrene; reversed-phase sorbents; tributylamine; ultrabasic rocks; NiS fire-assay; inductively coupled plasma-mass spectrometry.

UDC 543.062:546.55/.59

Current State and Problems in Determination of High Silver Content in Alloys and Articles (Review)

Goldshtrakh M. A. and Zhitenko L. P.

Current methods used in the laboratories and procedures included into international and national standards as well as publications regarding determination of high silver content (from several percent to 99.9 %) in alloys, compounds and articles that cover a period of 2010 – 2015 are reviewed. An emphasis is made on the publications in which the accuracy of considered methods are evaluated.

Keywords: silver alloys; silver articles; artefacts of cultural heritage; methods of silver determination; gravimetry; potentiometric titration; atomic spectrometry; non-destructive analysis.

UDC 538.915

Electrical Properties of the High-Quality Synthetic Boron-Doped Single Crystal Diamonds and Schottky Barrier Diodes on Their Base

Bormashov V. S., Tarelkin S. A., Buga S. G., Volkov A. P., Golovanov A. V., Kuznetsov M. S., Kornilov N. V., Teteruk D. V., Terentiev S. A., and Blank V. D.

Temperature dependencies of the resistivity and Hall coefficient in high-quality boron-doped synthetic diamond single crystals grown by HPHT method with different boron content are investigated. Acceptor concentration is varied in the range of $2 \times 10^{15} - 3 \times 10^{17} \text{ cm}^{-3}$ in (001) cut plates via changing the boron content in the growth mixture in the range of 0.0004 – 0.04 at.%. Thin rectangular plates with uniform boron content and free of extended structural defects are subjected to laser cutting after x-ray topography and UV luminescence mapping. Donor and acceptor densities in each sample are calculated from the data of Hall effect and current-voltage characteristics. The concentrations correlate with the boron content in the growth mixture. The minimum donors to acceptors compensation ratio less than 1% is observed in crystals grown in the growth mixture containing 0.002 at.% boron, while any change in the boron content increases the ratio. Samples grown at aforementioned boron concentration exhibit the maximum charge carrier mobility: $2200 \text{ cm}^2/(\text{V} \cdot \text{sec})$ at $T = 300 \text{ K}$ and $7200 \text{ cm}^2/(\text{V} \cdot \text{sec})$ at $T = 180 \text{ K}$. The phonon scattering of holes dominates over the entire temperature range 180 – 800 K, and scattering on the point defects, such as neutral and ionized impurity atoms is negligible. The diamond crystals grown from a mixture containing from 0.0005 to 0.002 at.% boron exhibiting excellent quality and lattice scattering mechanism can be considered reference semiconductor materials.

Keywords: semiconductor diamond; electrical properties; Schottky diode.

UDC 539.26:519.21

Methods of Recovering Orientation Distribution Function Using Texture Components with a Circular Normal Distribution

Ivanova T. M. and Serebryany V. N.

Two methods of recovering orientation distribution function (ODF) based on experimental pole figures for materials with low symmetry of the sample (medial section of hot-pressed strips of magnesium alloy Mg – 4.5 % Nd) are compared: (i) texture component method with normal circular distributions on SO(3) with different scattering and (ii) superposition of a large number of normal distributions with the same small scattering. Both methods lead to a similar ODF. The texture component method which is low sensitive to the errors of pole figure measurement relies on a nonlinear optimization with a complicated selection of initial approximation of the model parameters whereas the second method is more sensitive to the errors of measurements, but easy to use.

Keywords: orientation distribution function; method of texture components; method of superposition of a large number of circular normal distributions; pole figure; magnesium alloy Mg – 4.5 % Nd.

UDC 620.179

Special Features of Ultrasound Propagation as a Basis for Nondestructive Testing of Laminar Composite Materials

Samokrutov A. A. and Shevaldukin V. G.

The speed of longitudinal ultrasonic wave propagation in laminar composite materials depends on the inclination of the trajectory of their propagation to the surface of the layers. We present a procedure of measuring the speed of wave propagation using ultrasonic piezo transducers with a dry point contact. It is shown that the speed of wave propagation in the material monotonically grows with the angle of trajectory deflection from normal to the surface according to the law close to the dependence of the ellipsis radius on the angle between the radius and semiminor axis. The obtained data should be considered in developing the algorithms of visualizing the internal structure of composite materials.

Keywords: composite materials; carbon-fiber reinforced plastics; glass-fiber-reinforced plastics; ultrasonic waves; longitudinal waves; transducers with the dry point contact; wave propagation speed; echo method; antenna array.

UDC 621.4:539.4

Integral System for Computational -Experimental Determination of Conditions and Parameters of the Limit States

Makhtov N. A.

The role of strength characteristics and resource in the maintenance of safety control and protectability of items and facilities is analyzed. It is demonstrated that loaded and deformation criteria and state equations on their base including characteristics of the mechanical properties of materials provide amply description of attaining limit state conditions as well as nucleation and development of the fracture. Introduction of risk criteria in the set of equations which contain the characteristics of strength and resource as key parameters provide their use as a universal approach to determination of the danger level and operational hazard of the object.

Keywords: strength; resource; fracture; state equations; criteria; mechanical properties; limit states; risk; safety; accidents; disasters.

UDC 620.169:621.172.2:531.781

Methods for Analysis of the Fields of Residual Stresses in Spatial Structural Components

Makhtov N. A. and Razumovskii I. A.

A brief review of the methods of studying residual technological stresses (RS) in structural components and specimens is presented. Opportunities and fields of efficient use of non-destructive control in RS analysis are considered. Particular attention is paid to experimental-computational methods of studying two- and three-dimensional non-uniform RS fields present in spatial structures. The methods are based on interpretation of experimental data as an inverse problem of the theory of elasticity. To get a great bulk of experimental data thus required, i.e., deformation responses, resulting from different notches of different configurations, it is recommended to use optical-digital methods of recording the fields of displacement.

Keywords: residual stresses; stress-strain state; finite element method; electro-optic digital speckle interferometry; Mechanics of deformable solids.

UDC 620.191.33.001.57

Modeling of the Crack Growth Kinetics in the Surface Layers of Materials

Matvienko Yu. G.

Methods and results of physico-mathematical modeling of the fracture processes at different scale levels under various (including extreme) loading conditions are considered. The crack kinetics and crack trajectories in surface layers of the material under cyclic loading, rolling and sliding friction, indentation and edge chipping of the specimen are analyzed. It is shown that the combined methods of experimental and numerical modeling are very promising for studying fracture processes. The criterion of average stress in the crack tip is proposed to predict the crack trajectory in the case of mixed loading mode.

Keywords: physical-mathematical modeling; surface cracks; scale-structural levels.

UDC 620.178.151

Distribution of Stresses and Strains in a Deformed Volume of Metal upon Indentation of a Spherical Indenter

Matyunin V. M., Kazantsev A. G., and Marchenkov A. Yu.

Hardness method and method of finite elements are used to study distribution of stresses and strains in the deformed metal under the impression produced by indentation of a spherical indenter with different degree of loading. It is shown that those methods complement each other and their combined use provides more information about the values of stress and strain in different zones of the deformed volume of the metal.

Keywords: spherical indenter; indentation; stress intensity; strain intensity; hydrostatic core; strained metal; hardness; finite elements method.

UDC 519.2

Development of the Methods of Mathematical Research (2006 – 2015)

Orlov A. I.

The research subjects and quality of publications presented in the section “Methods of mathematical research” of our journal over the last ten year period (2006 – 2015) are analyzed. Mosaic of separate publications (123 articles of 114 authors) develops into a coherent picture of the research and practical recommendations. Along with publications on the subjects, common to the section, we consider the works in applied statistics, i.e., statistics of random variables, multivariate statistical analysis, statistics of time series and non-numeric data. Since the vast majority of these publications refer to statistics of non-numeric data, we highlighted three mainstreams — measurement theory, problems of classification, and statistics of interval data. Research in design of experiments is most common for the section. We also published the articles on expert estimates, forecasting, decision making theory, software of mathematical research methods. Theoretical results of rather high level and a great number of important applied problems thus solved made journal a unique publishing platform for mathematical methods research — nothing like that in Russia.

Keywords: mathematical research methods; mathematical statistics; applied statistics; statistics of random variables; multivariate statistical analysis; time series; statistics of non-numeric data; measurement theory; the classification problems; statistics of interval data; design of experiments; expert estimates; forecasting; decision theory; software.

UDC 519.28

Construction of Direct and Inverse Static Characteristics of the Objects by Interval Data

Skibitskiy N. V.

An approach to the construction of static characteristics of the systems based on experimental data is considered. It is based on the assumption that experimental errors are limited in value. A procedure for interval data processing which allows determination of the guaranteed range of the uncertainty of the static characteristic is proposed. The developed approach, in contrast to the statistical one, allows taking into account any *a priori* information about the errors in both input and output variables including the results of duplicate tests, information about the absolute and relative errors, rounding errors and also expert information.

Keywords: statistical characteristic; errors of variables; interval uncertainty; experiment.