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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

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UDC 543.426  
**X-ray Fluorescence Analysis Using a Modified Method of Standard-Background in the System of Analytical Control of Solid Products in Metallurgical Manufacturing of Precious Metals**  
*Bakhtiarov A. V., Savel'ev S. K., Zaitsev V. A., Devitsina O. V., Chuprina S. N., Sapunov R. V.*  
 We present an original approach to XRF analysis of the products of precious metal manufacture in which multiple regression equations are replaced with a certain type of equation derived from theoretical and semi-empirical considerations. This reduces the number of calibration samples, significantly simplifies the calibration procedure and reduces the cost of analysis. The developed method of analytical control is successfully applied to the analysis of the products of metallurgical shop of the Copper Plant (Polar Division of the JSC «MMC «Norilsk Nickel»).

**Keywords:** X-ray fluorescence analysis; precious metals; modified method of standard-background.

UDC 543.42.062/546.289.4  
**Spectrophotometric Determination of Germanium in Coke, Coals and Plant Material**  
*Chmilenko T. S., Ivanitsa L. A., Chmilenko F. A.*  
 The use of salicylfluorone (polyelectrolyte) in spectrophotometric determina-tion of germanium in the process feedstock and plant material provides a 9-fold increase in the molar absorption coefficients of complexes compared to that formed with unmodified reagent. The linear dynamic range and detection limit for Ge are 2 – 73 and 0,5 ng/ml, respectively.

**Keywords:** determination; germanium; trihydroxyfluorones; polyelectroly-te; spectrophotometry; polyhexamethyleneguanidine chloride.

UDC 543.423.1  
**Determination of Sulfur in Rocks, Iron and Polymetallic Ores Using Atomic Emission Spectrometry with Inductively Coupled Plasma**  
*Simakov V. A., Vasil'ev G. A., Zybinskii A. M., Sul-khanov I. V.*  
 Study of the behavior of sulfide and sulfate forms upon fusion revealed that in rocks with a low content of sulphide sulfur determination of sulphate sulfur be-comes possible after decomposition of previously calcined sample by borate fu-sion. Incomplete removal of sulphide sulfur upon calcination can interfere with the determination of sulfate sulfur in polymetallic ores. It is shown that low-temperature fusion of the samples of iron and polymetallic ores with sodium peroxide provides sulfur confinement and simultaneous sulfur determination with the main ore elements. Methods of ICP-AES determination of sulphate sulfur in rocks and total sulfur in the ores of ferrous and non-ferrous metals are developed to meet the accuracy requirements specified for the methods used in assessing mineral reserves.

**Keywords:** sulfur; determination; ICP-AES; polymetallic and iron ores.

UDC 543.422; 422.8; 423  
**An Integrated Approach to Elemental Analysis of the Sample of Unknown Composition**  
*Zemlyankina A. S., Korkina D. A., Grinshtein I. L.*  
 Determination of trace concentrations in the samples of unknown composition is particular difficult, due to a significant influence of the sample matrix com-position on the impurities. Traditional methods of analysis appeared to be ineffect-ive due to general lack of information about the sample composition which inter-feres with the choice of the optimal method of decomposition and impedes taking into account their impact on the results of quantitative measurements. We consider a procedure of complex elemental analysis of the sample of un-known composition using x-ray fluorescence, atomic absorption and atomic emission analysis with inductively coupled plasma. Algorithm of analysis is chosen in such a way that makes use of the benefits and limitations of each method. The described approach allows for the most accurate and efficient identification of trace element concentrations for the sample of unknown composition.

**Keywords:** mineral raw; mineral wastes; sample of unknown composition; elemental analysis; modeling of standards; XRF; ICP-AES; AAS.

UDC 539.194  
**Features of the Defect Structure Reconstruction of Epitaxial Films CeO<sub>2</sub> and La<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub> in an Alternating Magnetic Field**  
*Chibirova F. Kh., Kotina G. V., Bovina E. A., Tarasova D. V., Khalilov V. R., Polisan A. A., Parkhomenko Yu. N.*  
 The paper reports on experimental results obtained by x-ray spectroscopy and atomic force microscopy (AFM), features of the restructuring of the defect structure in epitaxial cerium oxide CeO<sub>2</sub> and lanthanum zirconate La<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub>

during processing in an alternating magnetic field or magnetic structural processing (MSP). Investigated epitaxial films  $\text{CeO}_2$  and  $\text{La}_2\text{Zr}_2\text{O}_7$  are seed and barrier layers of buffer in the current-carrying element of the high temperature superconducting wires 2<sup>nd</sup> generation (HTS 2 wires). Changes in the crystalline structure of epitaxial films  $\text{CeO}_2$  and  $\text{La}_2\text{Zr}_2\text{O}_7$  are reflected in the x-ray diffraction patterns and AFM images of the films. Changes were also observed in the crystal structure of the alloy Ni – 5 % W of textured substrate of  $\text{CeO}_2/\text{La}_2\text{Zr}_2\text{O}_7$  buffer upon sample processing in magnetic field. The experimental results indicate the existence of complex and fast processes in the crystal structure of epitaxial  $\text{CeO}_2$  and  $\text{La}_2\text{Zr}_2\text{O}_7$  films and the alloy Ni – 5 % W at MSP which resulted in irreversible changes in the crystal structure or in existence of magnetic structure effect (MSE) [1 – 3] in those materials.

**Keywords:** x-ray spectroscopy; magnetic structural effect; magnetic structure processing; defect structure of crystalline material; epitaxial films  $\text{CeO}_2$  and  $\text{La}_2\text{Zr}_2\text{O}_7$ ; Ni – W alloy.

UDC 620.17:620.18:539.42

#### Determination of the Microstructure of 3D Carbon Reinforced Plastics «Grani» Using X-Ray Tomography

Gareev A. R., Danilov E. A., Pylaev A. E.,  
Elizav P. G., Kolesnikov S. A.

Nowadays, State Research Institute for Graphite-Based Structural Materials «NIIGrafit» develops different types of multidimensional carbon reinforced plastics (CRP) based on prefabricated rod structures. An increase in the strength characteristics of the composite filler entails the proper choice of a binder with optimal elastic and strength characteristics and low viscosity to ensure the homogeneity of the material. We managed to reduce the number of different methods of structure studying due to application of modern x-Ray computer tomography which appeared the most effective procedure for determination of the structure of 3D-carbon fiber reinforced composite. Analysis of the initial microstructure of the composite allowed us to optimize the regimes of composite matrix formation thus improving the homogeneity of the material. Study of the bulk microstructure of the composite material after static loading made it possible to estimate the efficiency of the joint effect of the filler and binder.

**Keywords:** 3D-reinforced polymer composite material; 3D-carbon fiber reinforced plastic (3D-CFRP); infusion, failure mechanism; CFRP microstructure.

UDC 536.2.023:519:669:699.86

#### Determination of Thermal Diffusivity of Materials by Numerical-Analytical Method

Sokolov A. K.

A simple numerical-analytical method used in analysis of experimental data for determination of the thermal diffusivity of materials is described. The formulas for calculating the thermal diffusivity upon heating during the time intervals corresponding to Fourier numbers  $0.05 < Fo < 0.12$ , are derived. The estimation of the complexity and accuracy of the determination of thermal diffusivity on the test temperature fields calculated by the method of finite differences revealed essential accuracy of the method for temperature fields at Fourier numbers  $Fo \geq 0.3$ . The potentiality of the method to improve the accuracy of calculations for  $Fo < 0.3$  is also demonstrated. The described method does not require a «rigid» compliance with standard boundary conditions: constancy of the environmental temperature, heat flow, and heat transfer coefficients which greatly simplifies the experimental setup for investigation and determination of the thermophysical properties of the materials.

**Keywords:** thermal diffusivity determination; solution of the inverse problem of heat conduction; numerical-analytical method.

UDC 54.03

#### The Effect of Purge Gas on a Glass Transition Temperature

Shimkin A. A.

The dependence of the glass transition temperatures determined by dynamic mechanical analysis on the purge gas nature is studied. The thermal properties of the purge gas are shown to have a significant effect on the results of measurements obtained at different heating rates whereas the extrapolated quasi-equilibrium glass transition temperature (according to ISO 6721–11) remains almost constant. It is shown that the choice of the maximum loss modulus as a characteristic glass transition temperature improves significantly the reproducibility of the measurement results.

**Keywords:** dynamical mechanical analysis; composites; loss modulus; purge gas; glass transition temperature.

UDC 620.171.3:539.4

#### The Use of Non-Local Criteria in Description of the Destruction of Brittle Materials with a Hole Under Biaxial Loading

Suknev S. V.

Tensile crack formation in a brittle material with a hole under biaxial loading is studied. Data of the experiments obtained on the samples of high-strength gypsum are compared with the results of calculations carried out according to the

mean stress and fictitious crack criteria. The above-mentioned criteria match the experimental data rather well and can be used to predict quasi-brittle fracture in varying conditions of loading.

**Keywords:** brittle fracture; scale effect; nonlocal fracture criteria; hole; stress concentration; biaxial loading.

UDC 620.193.1:620.178.14:621.165.51

#### Integrated Indentation Tests of Metal Nanocomposite Coatings

Kudryakov O. V., Varavka V. N.

The results of studying strength and erosive properties of ion-plasma layered coatings of different metal and ceramic (nitride) systems are presented. The total thickness of the coating attained several micrometers whereas the thickness of each layer did not exceed two or three tens of nanometer. Using different indentation schemes we gained data on the physical and mechanical properties that determined the coating strength. Multiparametric optimization allowed us to obtain a satisfactory correlation with the data of bench erosion tests. A possibility of accurate diagnostics of the erosion resistance of nanocomposite coatings proceeding from a set of physical and mechanical properties measured in the laboratory instead of that usually determined in difficult conditions of bench or field trials is demonstrated.

**Keywords:** wear-resistant coating; the structure and properties of coatings; nanocomposite materials; indentation; micro- and nanohardness; sclerometry; erosion resistance.

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#### Study of the Working Surface of the Tin Bronze Based Material Hardened with Ultrafine Powders of Natural Diamond

Safonova M. N., Fedotov A. A., Syromyatnikova A. S., Tarasov P. P.

The results of studying the effect of additives of ultrafine natural diamond powder on the physical and mechanical properties of the metal matrix of diamond tools are presented. It is shown that a powder content of ~1 – 3% is optimal for production of the diamond containing composite material (DCM). The carbon content in the metal matrix increases when the concentration of ultrafine diamond powder exceeds the desired level. It was found that natural diamond powders of two different degree of fineness present in DCM ensure high mechanical and tribological properties of the material.

**Keywords:** ultrafine natural diamond (UFND); diamond-containing composite material; composite; hardness; density; wear-resistance; durability; abrasive; tin bronze; diamond tool.

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#### Features of High-Temperature Hydrogen Corrosion of the Cylindrical Steel Pipe

Nazarov V. V.

Features of the fracture of steel cylindrical tube under the impact of hydrogen pressure in conditions of isothermal creep are specified. The equations describing an increase in the concentration and pressure of the products of chemical interaction of hydrogen and iron carbides of the solid are derived. It is shown that the high hydrogen corrosion significantly affects both the strength properties and fracture pattern on the cylindrical steel pipe.

**Keywords:** hydrogen corrosion; porous pressure; main crack.

UDC 519.24

#### On the Estimates of the Distribution Function in a Random Censorship Model

Abdushukurov A. A., Muradov R. S.

Nonparametric and semi-parametric estimates of the distribution function of multiplying, exponential and power structures in the model of independent random right censoring are considered. A comparative analysis of the estimates and generalization of the estimates for the model of dependent random censoring using Archimedean copula function are presented.

**Keywords:** random censoring; distribution function; integrated intensity; copula function.

UDC 519.24

#### Overhaul Policy Optimization

Smolyak S. A.

Optimization of the remedial maintenance policy is linked to the problem of used machinery and equipment valuation. We develop a model based on the general principles of asset valuation and non-traditional use of discounted cash flow method. The model is based on available commercial and technical information and takes into account both the scheduled overhauls and repairs attributed to random failures. The model provides calculation of the machine wear dependence on the age and working time of the equipment within the current repair interval and prompt well-thought-out choice between the overhaul and decommissioning of the equipment item.

**Keywords:** equipment; valuation; reliability; overhaul; service life; optimization.