CONTENTS

ANALYSIS OF MATERIALS

TESTING OF STRUCTURE AND PARAMETERS

PHYSICAL METHODS OF TESTING AND QUALITY CONTROL

MECHANICAL TESTING METHODS

CERTIFICATION OF MATERIALS AND ACCREDITATION OF LABORATORIES

ABSTRACTS

UDC 543.054.2/.9

Determination of Semivolatile Hydrocarbon Fuels in Plants by Gas Chromatography-Mass Spectrometry with Headspace Analysis of the Extract

Bolotnik T. A., Jarcev S. D., Smolenkov A. D., Krechetov P. P., Shpigun O. A.

A new approach to determination of semi-volatile hydrocarbon fuels in plants grown on the soils contaminated by the aforementioned fuel is developed on the example of aviation kerosene. The method is based on ultrasonic methanol pre-extraction of the analyzed compounds followed by static headspace analysis and gas chromatography-mass spectrometry determination. Chromatograms were registered in selected ion monitoring mode (SIM), *m/z* 57 and 142. The calculated detection limit for aviation kerosene is 1 mg/kg of dry weight of green plants, the detectable concentrations range within 3 – 500 mg/kg of dry weight.

Keywords: aviation kerosene; gas chromatography; mass spectrometry; hydrocarbon fuels.

UDC 543.422.8:546.92

X-ray Spectral Determination of Gold in Geological Samples after Preconcentration Using Low Temperature Assay Melting

Simakov V. A., Isaev V. E.

The technology of gold extraction into the lead from the mixture with powder sample during the low-temperature alkaline melting is developed. The optimum mass ratio of the sample, lead and alkali, heating conditions, the temperature and duration of fusing that provide a satisfactory extraction of gold are specified. A low fusing temperature lightens the furnace design, reduces energy consumption and shortens the entire cycle from the beginning of fusing to loading of the next batch of crucibles. Simultaneous fusion of a large number of samples on a small area appeared possible due to the modular principle of furnace arrangement. The results of gold determination using x-ray fluorescence method and developed technology of concentration meet the regulatory requirements to the accuracy of determination.

Keywords: gold preconcentration; low-temperature fire assay; x-ray fluorescence method.

UDC 636.085.3:577.18:543

Identification and Determination of Mycotoxins and Pesticides in Feed from Single Sample by HPLC/TOF High Resolution Mass Spectrometry

Amelin V. G., Andoralov A. M., Timofeev A. A.

A method for simultaneous identification and determination of 25 mycotoxins and 170 pesticides and their metabolites in grain and vegetable-based animal feed for animals and birds from single sample using time of flight high-resolution mass spectrometry combined with high-performance liquid chromatography and easy express sample preparation is developed. A scheme for identification and determination of the detected analytes by the method of standard additive is proposed. The lower limit of determination is 1(500) μg/kg. The relative standard deviation of the test results does not exceed 0.1. The duration of the sample screening and quantification is 30 – 40 min and 1 – 1.5 h, respectively.

Keywords: mycotoxins; pesticides; feed for animals and birds; high performance liquid chromatography; high resolution time of flight mass spectrometry.

UDC 543.544.45

Determination of Formaldehyde in Polymethylene Naphthalene Sulphonates Using Gas-Liquid Chromatography

Zamuruev O. V., Petrovich O. M., Vovk A. I., Tyurina L. A., Dubyakov T. V.

A method of formaldehyde determination in polymethylene naphthalene sulphonates (PNS) using gas-liquid chromatography with trihexylborate as a derivatizing reagent is presented. The method is tested in analysis of commercial PNS specimens and approved in analysis of spiked samples. The limit of formaldehyde determination is 0.0005 % wt. whereas the accuracy does not exceed 10 %.

Keywords: gas-liquid chromatography; polymethylene naphthalene sulphonates; formaldehyde; trihexylborate; derivatization; extraction.

UDC 548.4

The Reason for Broadening of Bragg Diffraction Peaks Obtained from the Lapped Surfaces of Single-Crystal Silicon Plates

Pavlov V. F.

The reason for broadening of Bragg diffraction peaks (BP) and dependence of their integral width on the diffraction angle upon diffractometric shooting of the defective layers (DL) formed on the surface of plates upon cutting of monocrystalline silicon ingots and subsequent lapping is considered. To calculate the integral width of BP we used a simplified model of DL treated as a multilayered structure in which each layer consists of randomly linearly shifted single-crystal blocks. An equation is derived that links DL parameters with integral width of BP and the width of the total reflection area. BP are obtained from the surfaces of silicon plates (polished with KZM-50 (grain size 50 μm) and fine abrasive ÉBM-10 (grain size 10 μm)) having orientation (111), (110), (100) in reflection (111), (220), (400), (333), (440) of Cu$K\alpha_1$ and Mo$K\alpha_1$-radiation on a double-crystal goniometer in the scheme (n, $-n$). The BP analysis showed that equation is true for DL formed on the surfaces after their polishing with coarse-grained abrasive KZM-50. The validity of the derived formula for "thick" DL indicates to the formation of BP as a sum of diffraction peaks from a lim-

ited number of layers composed of non-deformed single-crystal blocks divorced from each other. For thin DL formed upon the surface treatment with fine-grained abrasive ÉBM-10 the formula is not applicable due to the increasing contribution of the diffraction angle to the diffraction peak of reflection from the monocrystalline matrix of the silicon substrate.
Keywords: semiconductor-grade silicon; lapping; defective layer; Bragg peak; integral width; block thickness.

UDC 620.179.14

Quality Control of Oil-and-Gas Pipe Heat Treatment Using Magnetic Structurescopy

Gorkunov E. S., Zadvorkin S. M., Putilova E. A., Bakunova A. A.

The goal of creating high efficient methods of nondestructive testing of the mechanical properties of thermally hardened steel pipes used in petroleum industry is still relevant. We have studied the effect of heat treatment parameters – hardening temperature (in a range of 764 – 980°C), cooling rate (different conditions of cooling in water, oil and air) and tempering temperature – on the structure, hardness and magnetic properties (coercive force, residual induction, maximum magnetic permeability, saturation magnetization) of the pipe steel 22CrMn2A, 30CrMoA, and 32Mn2. Magnetic parameters are measured both in conditions of closed magnetic circuit and on the model and full-scaled samples using attachable magnetic devises in laboratory conditions. Possibility of using the coercimetric method as an alternative to the durometric hardness measurements providing continuous in-process control of quenching underheating and tempering quality of steel pipes is demonstrated.
Keywords: pipe steels; quenching; tempering; non-destructive testing; coercitive force.

UDC 661.183.1

Thermoanalytical Study of Lithium Containing Sorbents

Rachkovskaya L. N., Chterzer N. V., Rachkovskii E. E., Kotlyarova A. A., Khasin A. A.

Sorbents modification by immobilization of active substances (compounds of lithium, silver, etc.) on their surface provides strengthening of their sanative properties in different pathologies. The sorbents deliver active substances to the area of therapeutic action and at the same time play the role of detoxicants, sorptioning different toxins on their surface and bringing them out naturally from the body. When selecting the technology of sorbent production, it is rather important to consider the effect of temperature on the modifying agent. Thermoanalytical study of lithium-containing sorbents obtained on the basis of thermo-activated aluminum hydroxide and lithium citrate showed that the synthesis technology significantly affects the modificator behavior at elevated temperatures: mixture heating results in dehydration of lithium citrate. The absence of CO and CO_2 (according to mass spectrometric analysis) after heating of the samples to 250°C proves that deep decomposition of the modificator on the surface does not occur.
Keywords: sorbents; modification; lithium citrate; thermoanalytical study.

UDC 669:004.94

Computational and Experimental Adjustment of the Databases for Computer Simulation of Cast Technologies

Ogorodnikova O. M., Martynenko S. V.

Results of the full-scale experiments on crystallization of test casting of 20GL steel used in manufacturing cast parts of the freight car bogies are presented. The data of the temperature measurements (thermocouples located within the mold) are used to solve the inverse problem of recovering thermophysical characteristics of mold materials in the heat conduction equation. The proposed calculation-experimental recovery procedure can be used for data replenishment of the material databases of specialized CAE software (Computer Aided Engineering) intended for simulation of foundry technologies. The method consists in a series of computational experiments, identical to full-scale ones thus bringing the calculated temperature curves close to the full-scale readings of thermocouples due to directional changes in the dynamic variables using Levenberg–Marquardt multivariate optimization.
Keywords: simulation; CAE; casting technologies; material database; Levenberg – Marquardt method.

UDC 621.315.592

Setup for Measuring the Galvanomagnetic Properties of Semiconducting Materials by Sample Rotation in the Field of Permanent Magnet

Golubyatnikov V. A., Grigor'ev F. I., Lysenko A. P., Strogankova N. I., Belov A. G., Kanevskii V. E.

A setup providing measuring of galvanomagnetic parameters of semiconductor materials using sample rotation in the field of the permanent magnet is developed and manufactured. Measurements are carried out at room temperature and at a temperature close to the boiling point of liquid nitrogen (when the sample is placed in liquid nitrogen vapor). The rotation of the specimen is effected by means of the control/display unit based on a microcontroller that activates a stepper motor. The direction of the current through the sample is changed by a switch. The measured signal is fed via the interface to a personal computer, recorded and processed using a special software. The developed setup is tested on a sample of *p*-germanium at 300 and 82 ± ± 3 K. It is shown that at the indicated temperatures the dependences of the measured signal on the angle of sample rotation are close to sinusoidal and cross the zero-axis at 0, 180 and 360° which proves the absence of hysteresis. The feasibility of the setup can be significantly extended at increased values of the magnetic field induction close to 1,4 T which can be attained by replacing of the core bits. Processes of sample rotation control, recording of the measured signal and data processing can be automated through the development of the corresponding software.
Keywords: galvanomagnetic measurements; permanent magnet; sample rotation in magnetic field.

UDC 620.19:531.781

Scientific and Methodical Bases of Flaw Inspection, Diagnostics and Monitoring of Conditions of the Materials and Engineering Systems

Makhutov N. A., Gadenin M. M., Ivanov V. V., Miodushevsky P. V.

Methodology of substantiation of safety, basic and remaining lifetime, reliability and fail-safety of the bearing parts of equipment is considered. The methodology is based on estimation of the stress-strain states from the results of diagnostics and monitoring of their current and limiting states taking into account the rate of service damageability accumulation. The possibility of determination of the used part of the resource, the remaining lifetime, fail-safety and reliability of heavy-duty parts of machines and structures is considered proceeding from the analysis of stress-strain states and changes in the properties of structural materials and defective factor. It is showed that integrated implementation of control, flaw inspection, preliminary engineering diagnostics, fracture diagnostics and general monitoring of the heavy-duty parts of the equipment opens new capabilities for resource and safety management of high risk objects.
Keywords: diagnostics; monitoring; status monitoring; resource; fail-safety; damages accumulation; limiting states; safety; risk; structural materials.

UDC 620.181:678.074

Thermal Cyclic Tests of Shrink Polymeric Products with the Shape Memory

Zachinjaev G. M., Kondratov A. P.

Improved device of the automated laboratory bench for thermomechanical testing of solid materials with «shape memory» is presented. It is shown that air heating in the heat chamber release the internal stress (attaining 1.5 – 2 MPa) in the samples of shrink polymer films, which cause the «shape memory» effect. Thermostating process under isometric conditions reduced stresses due to the relaxation processes occurring in the thermoplastic polymer with a linear structure. This relaxation process obeys the common rheology laws of viscoelastic systems. However, if the samples are cooled in the clamps of a standard test device, tension rises above the maximum and it does not have any theoretical explanation, and can be attributed to unaccounted interaction of the samples with clamps of the testing machine. We demontrate that the use of construction materials with different known linear coefficients of thermal expansion; choice of proper configuration and installation of additional elements for the clamps with adjustable length can eliminate the systematic errors of tension-measurements upon thermal cycling of shrink polymer films. Moreover, we present a research of unusual thermomechanical phenomena in polymeric materials.
Keywords: laboratory bench; compensation of thermal extension of details; thermomechanical testing; thermal cycling; internal tension; shrink polymer films.

UDC 620.172.251.222:621.315.55

Electroplastic Effect in Nanocrystalline and Amorphous Alloys

Stolyarov V. V.

The effect of structure-phase state of nanocrystalline and amorphous alloys and modes of pulse current on electroplastic effect is studied upon their tension. Reduction of the grain size up to nanoscale, occurrence of the second phases and amorphization in alloys leads to a decrease or full disappearance of the electroplastic effect (EPE). Introduction of current pulses in tensile tests of nanocrystalline alloys with reverse thermoelastic martensite transformation suppresses stress jumps downwards induced by EPE display and promotes upward stress jumps that are attributed to with shape memory effect (SME).
Keywords: electroplastic effect; shape memory effect; pulse current; nanostructure; amorphouse alloys.

UDC 001.891.53

Application of Laser Interferometry to Calibration of Variable Forces Transducers

Prilepko M. Yu.

Issues attributed to different calibration of variable force transducers (VFT) are considered. A kinematic model of the variable forces transducer operating in a harmonic oscillation mode is presented. The method of VFT calibration based on laser interferometry is developed. The developed method and device on their base provide calibration that meet real service conditions of VETs. Amplitude- and phase-frequency characteristics of variable forces transducers are determined using the method of laser interferometry in a range of $10 – 20 \times 10^3$ Hz.
Keywords: variable forces; conversion factor; damping; sinusoidal excitation; piezoactyuator; force transducer; test mass; elastic body; laser interferometer; calibration device.

UDC 665.63

Determination of the Fraction Oil Content Using an Automated Minidist Unit: Issues and Options

Kolyada S. A., Kirillova L. B., Kolyada A. S.

Erroneous belief of the Russian specialists that automatic apparatus are unsuitable for determination of the fraction oil content and can be used only in analysis of light petroleum products is considered. The reasons of that current statement are attributed, first to the errors present in the text of GOST 2177–99. We discuss possible reasons of erroneous results and offer recommendation in tuning automated equipment and determination of the fraction oil content.
Keywords: petroleum; fraction oil content; automatic apparatuses; manual apparatuses; state standard sample; onset of boiling; the ent of boiling; correction for a bulged out mercury column in glass thermometer; repeatability; reproducibility; GOST 2177–99; ASTM-D86-11.