ЗАВОДСКАЯ ЛАБОРАТОРИЯ

ДИАГНОСТИКА МАТЕРИАЛОВ

СОДЕРЖАНИЕ

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CONTENTS

ANALYSIS OF MATERIALS

TESTING OF STRUCTURE AND PARAMETERS

PHYSICAL METHODS OF TESTING AND QUALITY CONTROL

MECHANICAL TESTING METHODS

MATHEMATICAL TESTING METHODS

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ABSTRACTS

UDC 543.6:546

Determination of Rhodium in the Environment Using Catalytic Method from Sulfarsazene Oxidation with Periodate

Khomutova E. G., Karachevtsev F. N., Yakimovich P. V., Zhitenko L. P.

A technique of sample decomposition and rhodium determination in the environment using catalytic method from sulfarsazene oxidation with periodate is developed. The accuracy of the results is confirmed by an independent procedure of rhodium determination using mass spectrometry with inductively coupled plasma. The content of rhodium in the samples of street dust (Moscow) ranges within 0.01 – 0.04 g/ton depending on the place of sampling. The standard deviation of the repeatability of the results of rhodium determination is below 0.05.

Keywords: rhodium; catalytic kinetic method of analysis; urban environment.

UDC 543.34:545.5

Gas-Sensing Electrodes in Ionometric Analysis of Natural and Man-Made Objects. Determination of Ammonium Ion in Water

Bebeshko G. I., Nesterina E. M.

Principle of operation of gas-sensing cells containing ion-selective electrodes for determination of gases or corresponding ions in solutions is described. Prospects of using cells in ionometric analysis of various objects are demonstrated. Analytical characteristics of gas-sensing cells, different types of gas-permeable membranes, compositions of internal electrolyte are discussed. An original gas sensing cell with ammonium-selective electrode for ammonium-ion determination in water is developed. The developed cell containing air space as a gas-permeable membrane is durable and rather simple in design. Conditions of ammonia static gas extraction and ionometric determination are specified. Statistical data processing proved high selectivity, correctness and repeatability of the developed procedure of ammonium-ion determination in mineralized water of complex composition.

Keywords: gas sensing electrodes; gas permeable membranes; gas generation and extraction; NH_4-metric cell; determination of ammonium-ion; water of complex composition.

UDC 543.544.33

Chromato-Mass-Spectrometric Determination of Polycyclic Aromatic Hydrocarbons and Their Chloro- and Nitro-Derivatives in Soil

Belinskaya E. A., Zykova G. V., Semenov S. Yu., Finakov G. G.

High sensitive and selective comprehensive method of determining polycyclic aromatic hydrocarbons (PAH) and their chloro - and nitro-derivatives in soil is presented. The method consists in their extraction from the soil, removal of the compounds interfering with the determination, chromatographic separation of PAH into three groups of compounds, concentration and chromato-mass-spectrometric determination. The determinable concentrations of PAH chlorine- and nitro-derivatives in soil range within 0.1 – 100 µg/kg. The developed method used in analysis of soils of different functional zones of Moscow revealed the areas with excessive concentrations of benzo[a]pyrene (66% of analyzed samples) and provided determination of spatial distribution of PAH in Moscow.

Keywords: polycyclic aromatic hydrocarbons; chloro- and nitro-derivatives of polycyclic aromatic hydrocarbons; soil analysis; extraction; gas chromatography-mass spectrometry.

Simulation of Phase Transformations upon Welding of Alloyed Steels

Kurkin A. S., Makarov E. L., Kurkin A. B., Rubtsov D. E., Rubtsov M. E.

A computer-oriented mathematical model of the austenite decomposition in alloyed steel in conditions of welding thermal cycle is presented. It is shown that each diffusion phase transformation can be described using four parameters: two limits of the temperature range of transformation and two parameters of the Avraami equation. Martensitic transformation can be described using three parameters. A procedure for determination of all required time-temperature parameters of the model obtaining from the chemical composition of steel is described. Regression models of the temperature intervals and kinetic parameters of ferrite-pearlite, bainite and martensite transformations are obtained on the basis of literature data. It is shown that shortcomings of existing visual methods of analysis of the final phase composition of steels are the reasons for inaccuracy and inconsistency of published transformation diagrams. The hardness of the sample which correlates with a number of other mechanical properties of the material is thus chosen as the most objective and reproducible criterion of final phase composition. To check the developed model we compare experimental data and the results of hardness calculation for 140 alloyed steels after their cooling at different values of the cooling rate.

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Keywords: phase transformations; alloyed steel; computer simulation; model identification.

UDC 620.193.25;669.24.621.777

Electrochemical Method of Nondestructive Testing of the Propensity of Metal Parts of NPP Equipment to Inter-Crystallite Corrosion

Safonov I. A., Kharina I. L., Korneev A. E.

Standard GOST-regulated method is used to develop a procedure of testing stainless steels (the basic material of operating equipment of nuclear power plants) for inter-crystallite corrosion resistance (ICCR). The methodology includes the methods of surface preparation and environment control, the choice of anode etching parameters and conditions of metallographic evaluation of ICCR. The reliability of the results is confirmed by the data of comparative testing of 08KH18N10T steel specimens after sensitizing and austenite conditioning according to the developed standard (AMU GOST 6032–2003) procedures. The proposed approach to ICCR assessing directly upon viewing the surface is advantages compared to the method of "replicas." The developed procedure is used to study corrosion of the internal surfaces of the main circulating pumps MCP-195-M at Kalinin and Balakovo NPP to analyze the possibility of extending their lifetime after a 20-year period of their service.

Keywords: austenitic stainless steel; intercrystalline corrosion; metallographic method.

UDC 620.179.18

Study of the Grain and Dislocation Structure of Nanostructured Mechanically Alloyed Model Carbon Steels Using CMWP-Method

Volkov V. A., Elkin I. A., Chulkina A. A.

Different methods of x-ray diffraction data analysis are compared in a case study of mechanically alloyed powder steels $Fe_{95.5}C_{4.5}$, $Fe_{90.6}Si_5C_{4.4}$, $Fe_{92.5}Cr_3C_{4.5}$ (Fe — 4.3 at.% (1 wt.%) C alloyed with Si and Cr). It is shown that conventional techniques of x-ray analysis, e.g., Williamson-Hall method, are not suitable for studying the structure of nanostructured steels. The grain and dislocation structures of those materials are studied using CWMP full-profile method. The average size of the blocks of coherent scattering, their distribution in size, dislocation density and their characteristics are studied as a function of the type of alloying elements and temperature of annealing. A bimodal grain structure is formed upon annealing of mechanically alloyed steels due to selective growth of grains. The dislocation density and evolution of the dislocation structure upon annealing strongly depend on the type of alloying element.

Keywords: x-ray diffraction; dislocation structure; nanomaterials; steels; grain size; microstrains.

UDC 66.017:544.16:546.541.1

AES Control of Changes in the Steel Structure upon Annealing

Molchan N. V., Fertikov V. I.

A spectroscopic method of steel structure control is developed. Atomic emission spectroscopy (AES) is used to study the effect of the material structure on the analytical signal in analysis of Kh6VF die tool steel. Analysis of a series of samples taken from the same specimen of a saw blade revealed statistically different results obtained for the same sample before and after 20-min heat treatment at a temperature of 800°C and subsequent cooling in the furnace. The analysis is carried out on an AtomComp 81 and ARL 4460 devices with excitation by the high-voltage spark in equal conditions. The ratio of the line intensity of the analyte to that of the main component of the sample (Fe_{2599}) is used in calculations. Assessment of the reliability of differences is performed using contrast of means.

Keywords: atomic emission spectral analysis; steel; structure.

UDC 620.179.16:620.179.143

Testing of the Method of Cluster Analysis of the Arrays of Acoustic Emission Pulses in Case Study of Glass Granulate Heap Formation

Makhutov N. A., Vasil'ev I. E., Ivanov V. I., Elizarov S. V., Chernov D. V.

Testing methods of cluster analysis and classification of the recorded arrays of pulses of acoustic emission (AE) is used to detect crack-induced AE signals in the brittle layer of tenso-sensitive coating and also to reveal different phases upon transition from the regular to avalanche process of damage accumulation in the physical simulation model. Such phases of damage accumulation are simulated for the process of glass granulate cone formation and then detected using AE control system synchronized with a camera of high speed movie recording.

Keywords: acoustic emission; acoustic emission pulse; descriptor; record; glass granulate.

UDC 620.178:620.172.22

Study of the Hardness and Modulus of Elasticity of Ferrite Using Kinetic Indentation Method

Vorob'ev R. A., Litovchenko V. N., Dubinskii V. N.

Method of kinetic indentation at a micro level is used to determine the mechanical properties of ferrite in steel 10. Indentation is carried out within a micro range at the following parameters: the upper limit of the test load is 2 N, depth limit of indentation is 9 μm, and the curvature radius R of a tetrahedral Vickers indenter is 0.22 μm. Indentation load varied from 10 to 2000 mN and exposure time at a constant load from 10 to 60 sec. The dependences of the mechanical characteristics obtained upon kinetic indentation — the modulus of elasticity, Vickers hardness and kinetic hardness — on the indentation load and hold time under the load are derived. Study is carried out to improve the methodology of kinetic indentation, reduce the divergence of the results and reveal the factors affecting determination of the mechanical properties.

Keywords: mechanical properties; heat treatment; low-carbon steel; indentation; hardness; modulus of elasticity; microstructure.

UDC 620.191.32

Stress Intensity Factors for a Cylindrical Specimen with Annular Crack

Dobrovol'skii D. S.

Durability of the structural element up to the crack onset depends on the local stresses in the notch area, whereas subsequent survivability of the element prior to final fracture is determined by stress intensity factor (SIF). It is advisable to obtain local stresses in the notch and SIF using common analytical solutions. In this regard, we derived the expressions for SIF in case of small and deep annular cracks and consider them as very sharp notches in conditions of tension, bending or torsion of a rod proceeding from G. Neuber classical solutions for small and deep notches. Dimensional classification of small and deep annular cracks is presented. Estimation of the errors revealed good agreement between the proposed relationships and corresponding data of finite element method.

Keywords: specimen; shallow and deep annular cracks; stress concentration factors; estimation of errors.

UDC 519.24

Three Main Results of the Mathematical Theory of Classification

Orlov A. I.

The mathematical theory of classification is rather diverse, it contains a large number of approaches, models, methods, and algorithms. We distinguish three main results: the best method of diagnosis (discriminant analysis), an adequate indicator of the quality of algorithm of discriminant analysis, and the statement about stopping after a finite number of steps of iterative algorithms of cluster analysis. Proceeding from the Neyman — Pearson Lemma it is shown that the optimal method of diagnosis exists and is expressed through the probability density function corresponding to the classes. If the probability density is unknown, the non-parametric estimators of the training samples should be used. "The probability (or share) the correct classification (diagnosis)" is often used as a quality indicator of the diagnostic algorithm — the more the better. Is shown that a widespread use of this indicator is unreasonable and the other "predictive power" indicator obtained by conversion in the model of linear discriminant analysis is offered. Stopping after a finite number of steps of iterative algorithms of cluster analysis is demonstrated on an example of k-means. In our opinion, the results make a fundamental contribution to the theory of classification and each expert should be informed about the obtained conclusions for further developing and application of the theory of classification.

Keywords: mathematical theory of classification; mathematical statistics; applied statistics; diagnostics; discriminant analysis; Neyman – Pearson Lemma; indicator of the quality of diagnostic algorithm; probability of correct classification; predictive power; cluster analysis; stopping of the iterative algorithm; k-means.

UDC 519.24:543.429.23–42.062[1]H

Verification of the Normality of Distribution and Independence of the Results of Quantitative Analysis by NMR ^{1}H of High Resolution at Incomplete Resolution of Signals and Their Wide Groups

Smirnov M. B.

It is shown that in the method of NMR ^{1}H of high resolution for wide groups of signals at a low noise level, regardless their overlapping, the consecutive measurements performed during short periods of time are not independent. Getting data that can be used for statistical processing requires spectra recording with an interval of more than 1 h. For overlapping groups of signals only of the distributions of the measurement errors H_β è H_γ are compatible with the hypothesis of normality. For other variables and for the error of measuring integrated intensity of the narrow signal allocated against the background of the "hump,", the hypothesis of normality is rejected at a significance level less than 0.01.

Keywords: NMR ^{1}H; distribution of the measurement errors; independence of the measurements; oil.