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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 543.51,543.621

Highly Enriched Stable Isotopes in Large Scale Analysis of Rocks, Soils, Subsoils and Bottom Sediments Using Inductively Coupled Plasma Mass Spectrometry (ICP-MS)

Karandashev V. K., Khvostikov V. A., Nosenko S. Yu., and Burmii Zh. P.

Two methods of acid decomposition (in an open system and autoclave-cured decomposition) suitable for large scale analysis of rocks, soils, ground and bottom sediments using atomic emission (AE) and inductively coupled plasma mass spectrometry (ICP-MS) are developed. We propose a method of using domestic highly enriched stable isotopes, which, although with limitations, provides control of the dissolution phase of for each test sample.

Keywords: isotope dilution; mass spectrometry with inductively coupled plasma(ICP-MS); autoclave-cured decomposition; rocks; ground; soil; sediment.

UDC 543.544.54.07/08

Gas Chromatographic Determination of Some Derivatives of Methyl-Phosphonic Acid

Punkevich B. S., Tretyakova S. V., Sadovnikov S. V., Sosnov A. V., Kondrashov A. Ya., Stan'kov I. N., Kondratyev V. B., and Derevyagina I. D.

A technique of gas chromatographic determination of the derivatives of methyl-phosphonic acid: O-isopropylmethylfluorophosphonate (sarin), O-1,2,2-trimethylpropylmethylfluorophosphonate (soman), O-isobutyl-S-[2-(N,N-diethylamino)ethyl]methylthiophosphonate (Russian Vx, RVx), O-methyl-O'-isobutylmethylphosphonate (DAMF) is developed. The method suggests the use of a detector of thermal conductivity. The method of internal standard is used for quantitative calculations. The error of the methods is below 3%, time of analysis is 40 min.

Keywords: mass fraction; state standard sample; capillary gas chromatography; titrimetric methods; organophosphorus compounds; thermal conductivity detector; internal standard.

UDC 543.421

Electrothermal Atomic Absorption Determination of Gold in Iron-Containing Materials Following Dispersion Microextraction Concentration

Alemasova N. V., Babenko N. V., and Alemasova A. S.

To reduce the detection limit of gold determination by atomic absorption spectrometry with atomization (ETAAS) in iron-containing materials, we propose to use dispersion microextraction with chloroform of ion associates $AuCl_4^-$ with a cation of brilliant green. Optimal conditions of microextraction which provide 96% of gold recovery (two-fold extraction) are specified. The developed extraction-ETAAS procedure of gold determination is used in analysis of sedimentary rocks (shale pyrite) and oxidized (secondary) polymetallic ore containing 7 and 37% iron, respectively. The detection limit is 0.0002 mg/kg ;the relative standard deviation does not exceed 0.2.

Keywords: electrothermal atomic absorption spectrometry; gold; dispersion microextraction; iron-containing materials.

UDC 621.774.21,621.78.019.3

Determination of Optimal Temperature of Heating upon Local Normalizing of Welded Seam in Pipes Made of Low-Alloyed Steels

Tkachuk M. A., Bagmet O. A., and Stepanov P. P.

Processes that occur in heat affected zone (HAZ) upon heating and cooling of metal of welded joint upon local heat treatment (LHT) in normalizing mode are simulated. For cooling rates used in workshop conditions critical points are determined for steels of different chemical composition. Novel LHT modes are developed for different pipe steels capable of increasing the impact toughness due to formation of small austenitic grains after heating and uniform fine-grained ferrite-pearlite microstructure in welded joint after cooling. The results are tested and implemented in production of electric-welded tubes 114 – 530 mm in diameter (K50 – K60 pipe grades, according to API standard) at the JSC “Vyksa Steel Works.”

Keywords: electric-welded pipes; high-frequency welding; local heat treatment; welded joint; heat affected zone (HAZ); microstructure.

UDC 669-1

Study of the Structure and Phase Composition of a Dispersion-Hardened Titanium Super Alloy VT8-1 Using Transmission Electron Microscopy

Lukina E. A., Kashapov O. S., and Zavodov A. V.

The results of studying the structure and mechanical properties of VT8-1 alloy subjected to different heat treatment procedures are presented. The phase com-

position and morphology of the precipitating phases are determined using transmission electron microscopy. It is shown that temperature elevation at the first stage of annealing leads to an increase in the strength properties and fracture toughness due to increased dispersion of the secondary and tertiary precipitates of α -phase. The width and morphology of α -plates, and the ratio of primary and transformed (secondary) α -phase depends on the initial ($\alpha + \beta$) state, and hence on the selected processing temperature at the high temperature stage with respect to the temperature of polymorphic transformation. The formation of the complex of structural characteristics is also affected by the nature of the discharge and the size of the titanium silicides near the interphase boundaries.

Keywords: fracture toughness; thermal treatment; the α -phase; the β -phase; transmission electron microscopy; silicides; dispersion strengthening; solid solution strengthening.

UDC 537.226.621.317 cavity

Determination of Dielectric Permeability of Materials upon High Temperature Heating in Guide Cavity

Krylov V. P.

We specified an algorithm of determining the SHF dielectric permeability (DP) upon heating in a cylindrical guide cavity. It is shown that uneven heating and different thermal and physical conditions of measuring the inherent parameters of the cavity with and without the specimen are the main sources of errors in determination of dielectric permeability. The developed algorithm of DP calculation upon heating includes an integral expression for determination of the electric length of the cavity with allowance for unevenness of heating which provides improved accuracy in determination of the temperature dependence of dielectric permeability.

Keywords: superhigh frequency; dielectric properties; dielectric permeability measurements; guide cavity; high-temperature heating.

UDC 537.31.53.082–084

Determination of the Specific Conductivity of Aqueous Electrolyte Solutions

Alekseev A. N., Dovgopola A. V., Kovalov K. M., Lazarenko M. M., and Tkachev S. Yu.

A technique for determination of the specific conductivity of complex aqueous electrolyte solutions containing the admixtures is developed. Capillary cell of a contact type with three platinum electrodes and AC bridge R5083 ensure a wide frequency range (0.1 – 100) kHz of measurements with a relative accuracy of 0.01 %. Calculation of the electrical conductivity with allowance for the polarization impedance is performed using the method of the cell constant variation (CCV). A comparison between CCV results and data obtained by frequency method revealed that the discrepancy with the literary data does not exceed 4 %. Testing of method and measurements are carried out using reference KCl solutions 0.1 and 1 mol/kg in a temperature range of 20 – 70 °C.

Keywords: specific conductivity; electrolyte solutions with admixtures; method of the cell constant variation.

UDC 531.754.1:531.754.4

Measurements of the Apparent and Real Densities of Carbon-Carbon Composites and Structural Graphites

Ryashencev M. S. and Kolesnikov S. A.

We developed a method and a set of measuring devices for rapid determination of the apparent and real densities of structural graphites and carbon-carbon composites and compared the results with the corresponding data of conventional procedures. The values of the apparent density obtained on a device GeoPyc 1360 are higher than "standard" values that can be attributed to pseudoliquid environment thus used, whereas the values of real density obtained on AccuPyc 1340 are below standard which can be attributed to closed pores present in the specimen in case of non-destructive analysis. The divergence decreases as the degree of sample fragmentation increases.

Keywords: structural graphites; carbon-carbon composites; real density; apparent density; porosity; pseudoliquid environment.

UDC 620.178.3:620.193:669.14

Analysis of the Impact of Aqueous Media on the Cyclic Crack Resistance of Steels

Grin E. A.

Three physical models of crack growth in condition of corrosion fatigue failure of steels are developed: energy model; model of hydrogen embrittlement, and a model of local anodic dissolution of metal at the crack tip. Anodic dissolution model appeared most preferable for quantitative analysis and provided good agreement of the variants calculation with experimental results. A predominant role of the local anodic dissolution as the main mechanism activating the process of fatigue failure of middle-strength carbon and low alloyed steels in aqueous corrosive media is demonstrated.

Keywords: aqueous media; metal; physical model; electrochemical reaction; activation energy; hydrogen embrittlement; anodic dissolution; fatigue; corrosion crack resistance; steel; fracture mechanism.

UDC 620.179.17:53.07:[534.14+534.2]

Multi-Function Training Bench for Studying the Processes of Generation and Propagation of Acoustic and Emission Waves

Rastegaev I. A., Danyuk A. V., Merson D. L., Vinogradov A. Yu.

A design and algorithm of using certified multi-function education and research training bench are presented. The design of the bench provides simulation of a wide range of conventionally monitored items (vessels, pipelines, tubular furnaces, tanks, etc.) and main sources of acoustic emission (AE): cracks, leaks, corrosion damage induced by aggressive media, etc. AE simulator devices (AESD) are mounted on the bench via waveguides which provides acoustic communication and safety of the key elements even upon damage of the simulator. AESD are controlled using a loading device which provides setting of the onset and rate of AESD destruction. Design features of the bench also provide a possibility of repetitive simulation of a variety of situations that arise in-service and upon technical diagnostics of dangerous industrial objects at minimum costs. The bench can be used in research, training, certification of AE techniques and equipment, and nondestructive laboratory testing.

Keywords: acoustic emission; signal simulators; training bench; training of professionals; non-destructive testing.

UDC 620.178.162.4

Accelerated Corrosion Testing of Materials for Wear

Aseeva E. N., Bagmutov V. P., Parshev S. N., and Aseeva S. D.

The methodology of accelerated tests of materials for wear in a pair of the disc-plane and the disk-to-disk is considered. A distinctive feature of the proposed method of testing is the constancy of the contact area of the elements in the friction zone. A design of a portable installation developed to implement considered testing methodology is described. The results of comparative tests of steel samples with different carbon content hardened by electromechanical treatment for wear are presented. It is shown that the results of testing for wear resistance obtained according to the proposed procedure match with the data of standard testing method, the difference being no more than 9%. The duration of each sample testing is reduced ten times.

Keywords: wear; wear rate; friction coefficient; the surface layer; surface hardening; microhardness.

UDC 519.2

Limit Theorems and Monte Carlo Method

Orlov A. I.

The goal of mathematical statistics is development of the methods for data analysis aimed at solution of applied problems. Approaches to developing methods of data analysis change as time goes by. Hundred years ago it was assumed that distributions of the data are of a certain type, for example, normal distributions, and developed statistical theory on proceeding from that assumption. Limit theorems guided theoretical studies at the next stage. A term of "small sample" suggests a sample for which the conclusions based on the limit theorems can not be used. In each statistical problem there is a need to divide the final sample sizes into two classes — those for which you can apply the limit theorems, and those for which limit theorems can not be used because of the risk of incorrect conclusions. The Monte Carlo method is often used solve those problems. More complex problems arise when the properties of statistical procedures of data analysis are affected with various deviations from original assumptions. The Monte Carlo method also can be used to study those impacts. The basic — and not solved in a general way — problem of the studying the stability of conclusions in the presence of deviations from the parametric families of distributions is the problem of choosing some distributions to be used in modeling. We consider some examples of application of the Monte Carlo method relating to the activities of our research team and formulate the main unsolved problems.

Keywords: mathematical statistics; applied statistics; data analysis; limit theorems; Monte Carlo method; small sample; stability of conclusions; unsolved problems.

UDC 519.21

Monte Carlo Method: Accuracy of Asymptotic Solutions and Quality of Pseudorandom Number Generators

Grigor'ev Yu. D.

The goals of improving the efficiency of Monte Carlo calculations are considered. The key role is assigned to selection of a body of statistical tests (simulated random numbers) and to the quality of random number generators. Problems of implementation of the Monte Carlo algorithms attributed to the goal of increasing rate of convergence of asymptotic solutions to the true solutions are discussed.

Keywords: Monte Carlo method; effectiveness and difficulty of calculations; limit theorems; basic random number generators.