ISSN 1028-6861

ЗАВОДСКАЯ ЛАБОРАТОРИЯ

ДИАГНОСТИКА МАТЕРИАЛОВ

1

2016 ЯНВАРЬ

INDUSTRIAL LABORATORY. DIAGNOSTICS OF MATERIALS

ЗАВОДСКАЯ ЛАБОРАТОРИЯ
ДИАГНОСТИКА МАТЕРИАЛОВ

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

№1 ТОМ 82 2016

Основан в январе 1932 г.

СОДЕРЖАНИЕ

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Журнал включен в список изданий, рекомендованных ВАК при защите докторских диссертаций.

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ABSTRACTS

UDC 543.421

Determination of Sodium in High-Purity Graphite by High Resolution Continuum Source Electro-Thermal Atomic Absorption Spectrometry (HRCS-ETAAS)

Eskina V. V., Dalnova O. A., Tursunov L. Kh., Baranovskaya V. B., Karpov Yu. A.

Analytical capabilities of electrothermal atomic absorption spectrometry with a continuum spectrum source (HRCS-ETAAS) are analyzed in determination of sodium impurity in high-purity graphite powder. Various calibration procedures used for sodium determination are considered. A specific technique is used to plot the calibration curve of the analyte: argon dilution of the atomic vapor obtained upon introduction of the aqueous comparison solution (ICP Trace Metals in Drinking Water Standard in 2% HNO_3 + Tr HF (High-Purity Standards, USA)) into the atomizer. Conditions of preliminary thermal treatment of of high-purity graphite powder and sodium atomization program are specified. The limit of sodium detection using HRCS-ETAAS is determined (2.6×10^{-4} ng). The accuracy of sodium determination by HRCS-ETAAS is proved in weight variation measurements.

Keywords: high resolution continuum source electro-thermal atomic absorption spectrometry(HRCS-ETAAS); high-purity graphite.

UDC 543.427.4:662.613.122:543.62:669.292.3

XRF Analysis of Vanadium Slag After Borate Fusion

Volkov A. I., Osipov K. B., Zhdanov P. A., Seregin A. N., Bol'shov M. A.

The problems of chemical analysis of complex comnpounds are considered in case study of vanadium slag. Data on phase and chemical composition, problems of sampling and sample preparation as well as currently used methods of chemical analysis of vanadium slag are analyzed. The physicochemical processes occurring during fusion of the slag samples with lithium borate are considered. Optimal conditions are specified in the experiments on fusing glass samples of vanadium slag using lithium tetraborate, carbonate and nitrate. Conditions for measuring the intensity of analytical lines are specified with allowance for the intensity, spectral cross-over, and optimum x-ray fluorescence excitation. The metrological characteristics of the developed technique of x-ray fluorescence analysis of the vanadium slag samples are listed. The accuracy of chemical analysis data is confirmed in anlysis of state standard samples of vanadium, converter and phosphorous-bearing slag, and vanadium pentoxide. The developed method of analysis is applied to determination of the composition of vanadium slag and cinder.

Keywords: x-ray fluorescence analysis; vanadium slag; sample fusion; lithium borates; flux; converter slag; vanadium oxide (V); slags of metallurgical production.

UDC 543.51

Layer-to-layer Analysis of Crystal Lithium Tantalate Using MS-ICP and Laser Ablation

Drogobuzhskaya S. V., Shcherbina O. B., Novikov A. I.

Z-slice (perpendicular to the crystallographic axis *Z*) of $LiTaO_3$ single-crystal modified by the method of Vapor Transport Equilibration (LT VTE) is studied to determine a change in Li/Ta ratio. Data of layer analysis of LT using mass spectrometry with inductively coupled plasma with laser ablation (LA MS ICP) along with XRS and Raman spectroscopy data allowed us to confirm the presence of the areas with a thickness from tens to hundreds of microns having different stoichiometric composition and define more accurately their boundaries which is rather important for further formation of domain structures with the purpose of creating integrated optic devices.

Keywords: lithium tantalate, laser ablation, mass spectrometry; inductively coupled plasma; Raman spectroscopy; LT VTE.

UDC 543.423:546.59

Advanced Preparation of Geological Samples for Atomic Emission Determination of Gold

Pakhomova V. V., Shvetsov V. A., Pakhomov V. A., Belavina O. A.

A procedure is developed for preparation of geological samples for atomic-emission determination of gold. The procedure first consists in crushing the material of geological samples up to a grain size of 1 mm, then the sample mass is reduced to 0.5 – 1.0 kg and, finally, an analytical sample weighing 40 – 60 g is picked-off and triturated to a grain size of 0.071 mm. Sub-sample is taken without preagitation.

Keywords: sample preparation; atomic emission analysis; analytical sample; sub-sample; gold; determination; geological samples.

UDC 544.08,54.084,542.08

Some Aspects of the Glass Transition Temperature Determination by Dynamic Mechanical Analysis of the Samples After Hydrothermal Aging

Khaskov M. A.

The impact of measurement conditions of the dynamic mechanical analysis (DMA) and calculation procedures on the value of glass transition temperature (T_g) after hydrothermal aging (HTA)of the samples is considered. It was shown that T_g value calculated from the maximum of the damping factor, especially under low heating rates and medium moisture saturation, depends preferably on the irreversible decomposition of the polymer matrix and slightly depends on HTA conditions that can be attributed to high H_2O desorption rate during measurements. The T_g value, obtained from the extrapolated onset of the elastic modulus decline, reflects more reliably a decrease of the glass transition temperature after hydrothermal aging except the case of low heating rate. We believe that the optimal heating rate (5 K/min) which provides a reliable determination of T_g value by DMA of the samples after hydrothermal aging ensures the absence of high temperature gradient inside the samples and staves off substantial drying of the sample during measurements.

Keywords: dynamic mechanical analysis; hydrothermal aging; glass transition temperature; measurement conditions.

UDC 620.18

Study of the Structural Changes upon Plastic Strain of Steel 20

Kim V. A., Bashkov O. V., Popkova A. A.

A technique of color segmentation of the microstructure image as an element of computer metallography which provides powerful capabilities of the structural analysis is presented. Case study of structural changes of steel 20 upon plastic deformation demonstrated the practical application of color segmentation which provides ranking of the microstructural objects by their structure-energy state.

Keywords: microstructure; computer metallography; color segmentation; boundary density; defects of the crystal structure; free energy; chemical potential.

UDC 621.941.01

Using the Capabilities of the Combined Ultrasonic Treatment to Achieve the Desired Quality Parameters of the Surface Layer of the Parts Made of Titanium Alloys

Kiselev E. S., Blagovskiy O. V.

The results of studying the phase composition and residual stresses in the surface layer of the workpieces made of alpha-beta titanium alloy after their combined treatment by turning and ultrasonic hardmetal burnishing are presented. Control of the quality parameters is performed using modern equipment and nondestructive procedures. Data has shown that change in the elements of combined treatment significantly affect the value of the residual stress and ratio of α-/β-titanium in the surface layer, whereas the processing itself appeared to be an effective tool for attaining the desired parameters of the quality. The rate of the workpiece rotation has an opposite effect on the residual stresses and phase composition of the surface layer of the examined samples that can be attributed to the formation of TiFe and $TiFe_2$ intermetallic phases.

Keywords: titanium alloy; phase composition; residual stresses; surface layer; machining, ultrasonic treatment.

UDC 677.016.41+519.673

Digital Technologies for Express Colorimetric Assessment of the Dyed Surfaces

Tretyakova A. E., Safonov V. V., Trufanova O. Yu.

Current equipment (e.g. spectrophotometers for measuring transparent environments and solid bodies in visible spectrum range) and specially developed software provide a great bulk of information about coloristic parameters: reflection or absorption spectra, trichromatic coefficients, color coordinates in various color systems, purity, color tone, color saturation, etc. However in case of multiple serial measurements only primary data on the measured color, e.g., the color coordinates or color saturation/hue are required. In this case the use of available and mobile equipment and ordinary graphic editor operating in one of the known color systems, e.g., Adobe Photoshop appeared sufficient to meet the goals. We present the results of using Adobe Photoshop and scanner HP Scanjet 5470c for scanning dyed pieces and subsequent calculation of a number of coloristic parameters.

Keywords: color; color coordinates; color measurement.

UDC 620.179.16:620.179.143

Early Diagnosis of Damage and Fracture of Composite Materials Using Brittle Tenso-Sensitive Coatings and Acoustic Emission

Matvienko Yu. G., Vasil'ev I. E., Pankov A. V., Trusevich M. A.

Integrated use of acoustic emission and brittle oxide strain-sensitive coatings at early stages of deformation of damage and fracture zones in the specimens of polymeric composite materials (PCM) may be the most effective method of nondestructive defect detection and strength assessment of modern composite materials. An external load applied to the tested structure forms a crack pattern in brittle tenso-sensitive coatings (TSC), which reflects the actual distribution of the maximum primary stress not only in the zones of stress concentration at the surface but also in the vicinity of the local defects. Proceeding from the resulted crack patterns and refined characteristics of tenso-sensitivity of the brittle coating we may estimate the distribution of the maximum principal stress (strain). The limitations of the method of brittle coatings are attributed, first, to the location of tenso-sensitive coatings (in the zones that are technologically accessible for deposition) and, second, by the necessity of remote monitoring of the crack formation. To solve the aforementioned shortcomings and automate monitoring of the crack formation in a tenso-sensitive coating, we propose to use the method of brittle coatings in combination with acoustic emission (AE) and video recording. The developed integrated procedure provides a good opportunity to determine zones of the most probable fracture and the rate of specimen degradation during the entire period of structure testing, starting with 10 – 15% of the critical load, long before the beginning of the active phase of degradation of the PCM structure. The method ensures an accurate determnination of the area of forthcoming destruction of the sample and provides determination of the field distribution of the main principal strains in the area of TSC cracking and their quantitative assesment.

Keywords: brittle tenso-sensitive coating; crack; acoustic emission; AE pulse; descriptor; fracture.

UDC 620.178.3:539.431

Determination of the Coefficients of the Crack Growth Rate Equation upon Cyclic Load

Savkin A. N., Andronik A. V., Koraddy R.

No general procedure for determination of Paris coefficients is available nowadays. An approach providing determination of the intermediate values of the crack growth rates on $da/dN - dK$ fatigue fracture curve in each test is developed. The effect of the crack closure on the crack growth rate is demonstrated and the apmlitute of the effective stress intensity factor (SIF) is obtained. Prospects of developing the proposed procedure are discussed, for example, to determine the region of stable crack growth on $da/dN - dK$ curve leading to the fracture.

Keywords: Paris curve; $da/dN - dK$ curve; fatigue fracture diagram, curve approximation; crack closure estimation.

UDC 620.192.25

Determination of the Crack Resistance of Ultrafine-Grained Materials by Chevron-Notched Test of Small-Size Specimens

Derugin E. E., Lepov V. V.

The crack resistance behavior of ultrafine-grained commercial titanium VT1-0, titanium alloy VT6, welding steel 12GBA, and Fe – Ni steel obtained under severe plastic strain is determined in testing small-size specimens. The specific fracture energy is used as a main crack-resistant characteristic of the material. When testing small-size specimens with Chevron notch we propose a new characteristic of the fracture toughness — the relative displacement of the points of load application which is not correlated to a change in the compliance of the sample resulted from the crack development. Change of the specific fracture energy upon loading the samples with Chevron notch is also considered.

Keywords: ultrafine-grained structure; commercial titanium; welding steel; severe plastic deformation; Chevron-notched specimen; crack resistance; toughness; crack tip.

UDC 519.28

Hypothesis of Exponentiality: Methodological Aspect

Grigoriev Yu. D.

Different approaches used in testing statistical hypotheses are considered to discuss some methodological aspects of testing exponential hypothesis. Considered examples demonstrate that different exponential tests based on the same data can lead to conflicting results thus underlining the importance of such concomitant factors as the engineering data analysis, comparative properties of the power and asymptotic efficiency of different exponentiality tests. Methodological features of statistical modeling procedure are discussed including the limitations of the procedure regarding the possibility of drawing general conclusions proceeding from the particular results.

Keywords: hypothesis of exponentiality; statistics for the test; statistical modelling; power of tests; asymptotically optimal efficiency; methodological principles.

UDC 519.2

Estimation of Joint Reliability Function by Censored Observations

Abdushukurov A. A.

Paper is devoted to constructing of estimators of multivariate survival function under variable censoring of observations from the right. Using functionals of product, exponential and power types we proposed and studied three types of the estimates of two dimensional survival function in the general model of dependent and variable censoring from the right. The estimates are generalized analogues of theirs one dimensional variants. This type of observations was never considered by other authors. In particular, for estimators we prove the nearness, strong uniform consistency and weak convergence to the Gaussian process.

Keywords: reliability function; random censoring; integral-product; martingale; Gaussian field.