

17 сд
3-13

ISSN 1028-6861

ЗАВОДСКАЯ
ЛАБОРАТОРИЯ
ДИАГНОСТИКА МАТЕРИАЛОВ

2

2015
ФЕВРАЛЬ

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

№2 ТОМ 81
2015

Основан в январе 1932 г., Москва
Учредитель: ООО Издательство "ТЕСТ-ЗЛ"

Адрес редакции:

119991, Москва, ГСП-1,
Ленинский пр-т, 49,
ИМЕТ им. А. А. Байкова, Редакция
журнала "Заводская лаборатория.
Диагностика материалов®".

Тел./факс: (499) 135-62-75,
тел.: (499) 135-96-56

**Внимание! В сети Интернет
представлен новый сайт:
<http://www.zldm.ru>;
E-mail: zavlabor@imet.ac.ru**

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

© 2015 ООО Издательство «ТЕСТ-ЗЛ»,
«Заводская лаборатория.
Диагностика материалов»
Перепечатка материалов журнала «Заводская
лаборатория. Диагностика материалов»
допускается только с письменного
разрешения редакции.
При цитировании ссылка обязательна.

**ЗАВОДСКАЯ®
ЛАБОРАТОРИЯ
ДИАГНОСТИКА МАТЕРИАЛОВ**

Логотип "Заводская лаборатория. Диагностика
материалов®" является зарегистрированной тор-
говой маркой ООО "ТЕСТ-ЗЛ". Все права охраня-
ются законом.

СОДЕРЖАНИЕ

АНАЛИЗ ВЕЩЕСТВА

- Каргин И. Д., Соколова Л. С., Пирогов А. В., Шпигун О. А.** Определение антибиотиков тетрациклинового ряда в молоке методом высокоэффективной жидкостной хроматографии с послеклоночной реакцией и флуориметрическим детектированием 5
- Ясныгина Т. А., Маркова М. Е., Рассказов С. В., Пахомова Н. Н.** Определение редкоземельных элементов, Y, Zr, Nb, Hf, Ta, Th в стандартных образцах серии ДВ методом масс-спектрометрии с индуктивно-связанной плазмой 10
- Суранов Г. И., Латышев А. А., Карманова О. М., Васильев В. В.** Исследование процесса катодного наводороживания образцов и состава проб выделяющегося газа 20
- Нагиев Х. Д., Алиева Р. А., Алекберов Дж. А., Чырагов Ф. М., Гюллярли У. А.** Спектрофотометрическое определение висмута (III) с 2,3,4-тригидрокси-4'-сульфоазобензолом 25

ИССЛЕДОВАНИЕ СТРУКТУРЫ И СВОЙСТВ

ФИЗИЧЕСКИЕ МЕТОДЫ ИССЛЕДОВАНИЯ И КОНТРОЛЯ

- Комаров В. Ю., Подберезская Н. В., Каменева М. Ю., Смоленцев А. И., Козеева Л. П., Лавров А. Н.** Выявление кристаллографических особенностей высококислородных кобальтатов $YBaCo_4O_{8.4}$ с использованием метода реконструкции обратного пространства 28
- Титов В. И., Тарасенко Л. В., Уткина А. Н., Шалькевич А. Б.** Фазовый анализ новой композиции высокопрочной конструкционной стали 35
- Гареев А. Р., Малинкин Д. А., Борисова А. Г., Колесников С. А.** Исследование взаимодействия на границе раздела фаз стержневых армирующих наполнителей и полимерной матрицы 3D углепластика «Грани» 40

МЕХАНИКА МАТЕРИАЛОВ: ПРОЧНОСТЬ, РЕСУРС, БЕЗОПАСНОСТЬ

- Гладштейн В. И.** Удельные затраты энергии на образование и рост трещины в роторной стали ЭИ-415 на разных стадиях ползучести 46
- Донсков А. С., Карманов В. В.** Исследование области локального упругопластического контакта при относительном скольжении двух упругих тел, ограниченных нелинейными поверхностями 53
- Обмен опытом
- Щербаков Ю. М., Фролов В. Н., Скачков Ю. А.** Определение предела выносливости пластмасс при испытаниях в режиме задаваемых деформаций . . 61

ОЦЕНКА СООТВЕТСТВИЯ. АККРЕДИТАЦИЯ ЛАБОРАТОРИЙ

- Немец А. М., Лебедева Н. В., Петров С. Н., Барахтин Б. К.** Состояние и перспективы развития Центра коллективного пользования ФГУП «ЦНИИ КМ «Прометей» 65
- Гундобин Н. В., Титов В. И., Пчелкин А. И.** Стандартные образцы состава жаропрочных никелевых сплавов системы Ni – Al – Co – Mo – Nb – Ta – Cr – W – Re для спектрально анализа 71

CONTENTS

ANALYSIS OF MATERIALS

- Kargin I. D., Sokolova L. S., Pirogov A. V., and Shpigun O. A.** Determination of Antibiotics from Tetracycline Series in Milk Using HPLC with Post-Column Reaction and Fluorescence Detection. 5
- Yasnygina T. A., Markova M. E., Rasskazov S. V., and Pakhomova N. N.** Determination of Rare-Earth Elements Y, Zr, Nb, Hf, Ta, Th in Reference Specimens from DB Series Using Inductively Coupled Plasma Mass-Spectrometry (ICP-MS) 10
- Suranov G. I., Latyshev A. A., Karmanova O. M., and Vasil'ev V. V.** Study of the Process of Cathode Hydrogenation of the Samples and Composition of Evolved Gas. 20
- Nagiev Kh. D., Alieva R. A., Alekberov Dzh. A., Chyragov F. M., and Gyullyarli U. A.** Spectrophotometric Determination of Bismuth (III) with 2,3,4-Trihydroxy-4'-Sulfoazobenzene 25

TESTING OF STRUCTURE AND PARAMETERS

PHYSICAL METHODS OF TESTING AND QUALITY CONTROL

- Komarov V. Yu., Podberezskaya N. V., Kameneva M. Yu., Smolentsev A. I., Kozeeva L. P., and Lavrov A. N.** Revealing the Crystallographic Features of High-Oxygen Cobalts YBaCo₄O_{8.4} Using Recovery of the Reciprocal Space 28
- Titov V. I., Tarasenko L. V., Utkina A. N., and Shalkevich A. B.** Phase Physico-Chemical Analysis in the Development of Novel Composition of High-Strength Structural Steel for Aviation-related Applications 35
- Gareev A. R., Malinkin D. A., Borisova A. G., and Kolesnikov S. A.** Study of the Interaction at the Interface of Core Reinforcing Fillers and the Polymer Matrix of 3D Carbon Fiber "Grani" 40

MECHANICAL TESTING METHODS

- Gladshstein V. I.** Specific Energy of Crack Formation and Growth in Rotor Steel EI-415 at Different Creep Stages. 46
- Donskov A. S. and Karmanov V. V.** Study of the Area of Local Elastoplastic Contact upon Relative Sliding of Two Elastic Bodies Bounded with Nonlinear Surfaces 53
- Exchange of Experience
- Shcherbakov Yu. M., Frolov V. N., and Skachkov Yu. A.** Plastics Endurance Determination in Tests in Conditions of Constant Deformation 61

CERTIFICATION OF MATERIALS AND ACCREDITATION OF LABORATORIES

- Nemets A. M., Lebedeva N. V., Petrov S. N., and Barakhtin B. K.** Current State and Prospects of Developing the Center of Multiple Access (CMA) of the Federal State Unitary Enterprise Central Research Institute for Structural Materials "Prometey" 65
- Gundobin N. V., Titov V. I., and Pchelkin A. I.** Reference Standards of Nickel Superalloys of the System Ni – Al – Co – Mo – Nb – Ta – Cr – W – Re for Spectral Analysis. 71

ABSTRACTS

- UDC 543.544.5
Determination of Antibiotics from Tetracycline Series in Milk Using HPLC with Post-Column Reaction and Fluorescence Detection
 Kargin I. D., Sokolova L. S., Pirogov A. V., Shpigun O. A.
 Conditions of sensitive and selective chromatographic determination of tetracycline complexes with Mg²⁺ in milk using HPLC with post-column reaction and fluorescence detection are specified. The fluorescence intensity of chelates is shown to be up to 1.8 times higher in microemulsions compared to that in water-acetonitrile medium. The detection limits are 5.8 and 25 ng/ml for tetracycline, oxytetracycline and doxycycline, respectively.
Keywords: antibiotics; tetracyclines; microemulsions, HPLC.
- UDC 543.42:543.51
Determination of Rare-Earth Elements Y, Zr, Nb, Hf, Ta, Th in Reference Specimens from DB Series Using Inductively Coupled Plasma Mass-Spectrometry (ICP-MS)
 Yasnygina T. A., Markova M. E., Rasskazov S. V., Pakhomova N. N.
 To refine the acceptance data we redetermine Y, Zr, Nb, Hf, Ta, Th in four Far East magmatic rocks: GSO 4317–88 meimechit enriched with olivine; GSO 4319–88 hornblende hyaloandesite; GSO 4323–88 biotitic trachyrhyolite; GSO 4322–88 greisenized granite. International reference samples JGb-1, JA-1, JG-1a (GSJ), and DNC-1 (USGS) are used as standards for comparison. Sample preparation: acidic decomposition using microwave furnace and fusion with lithium metaborate. Average results and standard deviations (for 4–5 parallel measurements) are presented and compared with certified and valuation data.
Keywords: certified reference samples of magmatic rocks; DV (Far East) series; rare-earth elements; inductively coupled plasma mass-spectrometry (ICP-MS).
- UDC 620.193.46:546.11
Study of the Process of Cathode Hydrogenation of the Samples and Composition of Evolved Gas
 Suranov G. I., Latyshev A. A., Karmanova O. M., Vasil'ev V. V.
 Experimental data on the composition of the gas evolved upon electrolytic hydrogenation of cast iron, zinc and iron samples are presented. Hydrogen interaction with the carbon samples results in formation of methane and other hydrocarbons (ethane-hexane). The hydrogen-charged samples of transformer iron exhibit significantly reduced content of silicon released in the deposit, precipitation, and electrolyte.
Keywords: electrolytic hydrogenation; gas samples; specimens; residue; cast iron; zinc; iron; hydrogen; methane; hydrocarbons; silicon.
- UDC 543.4:542.61:546.87
Spectrophotometric Determination of Bismuth (III) with 2,3,4-Trihydroxy-4'-sulfoazobenzene
 Nagiev Kh. D., Alieva R. A., Alekberov Dzh. A., Chyragov F. M., Gyullyarli U. A.
 Complexation of bismuth (III) with 2,3,4-trihydroxy-4'-sulfoazobenzene in presence and in the absence of nonionic surfactant — Triton X-114 — is studied. An acidic environment (pH 2.5–3.0) promotes formation of ternary complex with a maximum light absorption at λ = 464 nm. Optimal conditions of complex formation are specified and the basic spectrophotometric characteristics of binary and ternary complexes of bismuth (III) are calculated. A new sensitive and highly selective method for determination of bismuth in pharmaceuticals and in standard samples of copper-based alloys is developed.
Keywords: bismuth; spectrophotometry; 2,3,4-trihydroxy-4'-sulfoazobenzene; Triton X-114.
- UDC 548.3.12.30.736:546.73.431.669
Revealing the Crystallographic Features of High-Oxygen Cobalts YBaCo₄O_{8.4} Using Recovery of the Reciprocal Space
 Komarov V. Yu., Podberezskaya N. V., Kameneva M. Yu., Smolentsev A. I., Kozeeva L. P., Lavrov A. N.

The use of a standard methodology of reduction and visualization in primary experimental data processing in the x-ray diffraction study of oxygenated single crystals YBaCoO_{7+x} ($x = 1, 4$), topical for routine x-ray diffraction analysis is discussed. Substantiated determination of the parameters of the orthorhombic unit cell of oxygenated crystals revealed their correlation with the parameters of the parental hexagonal YBaCoO_{7+x} ($x = 0$) crystal. Additional crystallographic features undetectable earlier without analysis of the diffraction intensity distribution in reciprocal space are determined. Major oxygen-induced structural changes that occur in the basal plane (where the domains of the orthorhombic phase are formed according to the law of pseudotrigonal non-merohedric twinning) are not attributed to violation of the layer sequence along c axis.

Keywords: yttrium and barium cobaltate; reciprocal space; superstructure modulation; non-merohedric twinning.

UDC 544.01.544.02

Phase Physico-Chemical Analysis in the Development of Novel Composition of High-Strength Structural Steel for Aviation-related Applications

Titov V. I., Tarasenko L. V., Utkina A. N., Shalkevich A. B.

A core type of carbide intended for developing high-carbon wear-resistant structural steel without thermochemical treatment is specified. Tentative intervals of alloying high carbon structural steel for gear manufacturing are recommended with allowance for the matrix phase composition after hardening.

Keywords: phase physico-chemical analysis; carbide phase; steel; gears; chemical heat treatment.

UDC 691.175.3

Study of the Interaction at the Interface of Core Reinforcing Fillers and the Polymer Matrix of 3D Carbon Fiber "Grani"

Gareev A. R., Malinkin D. A., Borisova A. G., and Kolesnikov S. A.

We present a technological approach to assembling multidimensional structures comprising rods of circular cross section obtained from synthetic fiber by pultrusion which provides implementation of various reinforcement schemes to produce composite materials with the desired properties of spatial anisotropy. The specificity of this technique consists in dividing operations of pultrusion formation of carbon fiber microstructure in the bulk of reinforcing rod and subsequent superposing of the reinforcing cage with a polymeric composition forming the matrix of the material. Thus, the initial wetting of filament surfaces and their engaging into interface formation occurs in contact with a functional polymer used for rod manufacture. Current technology of the reinforcing rod manufacture is based on the use of aqueous solution of polyvinyl alcohol. We demonstrate the possibility of using low-viscosity epoxy compounds as an alternative to polyvinyl alcohol (PVA). The problems attributed to the choice of a binder responsible for interaction at the interface between the matrix and surface of the rod and the impact of different types of polymers used for rod manufacture on the matrix properties are considered. Practical significance of the study consists in analysis of the possibility of improving the strength properties of the composite with allowance for the technological potentiality of the method of assembling the reinforcing rod structures.

Keywords: bulk-reinforced polymer composite material; 3D reinforced carbon plastic; interaction at the interface; microstructure of the carbon fiber; strength implementation; rod reinforcing filler.

UDC 620.191.33

Specific Energy of Crack Formation and Growth in Rotor Steel EI-415 at Different Creep Stages

Gladshstein V. I.

A technique for determination of the specific fracture energy from the largest crack opening corresponding to completing of the each of three stages of creep is presented. Crack development in the samples was monitored indirectly by crack opening in testing. We developed criteria and signs that can be used to associate the certain stages of creep with corresponding phases of cracks development. Temporal boundaries of the main creep stages are determined using a statistical evaluation of the characteristic features of the kinetics of crack opening. The onset of crack growth determined from the curve of crack opening is compared with that calculated from the ratio of the lengths of the cracks formed in three notches present in the sample. Calculation of the crack resistance is based on the consideration of components of specific fracture energy using standard recommen-

dations for static loading at moderate temperatures. The values of J -integral and conventional value of the stress intensity are assessed from the degree of notch opening at the onset of crack growth as well as the crack length in the fracture. Parameters of the fracture toughness dependence on the temperature and time of loading, constants of the kinetic equation of cracks development are determined. The results of the crack resistance evaluation in cylindrical samples are compared to the data previously obtained on ST-1 samples.

Keywords: crack resistance; metal; creep; sample; notch; crack opening; stress intensity factor; J -integral; rotor.

UDC 620.178.152:539.214

Study of the Area of Local Elastoplastic Contact upon Relative Sliding of Two Elastic Bodies Bounded with Nonlinear Surfaces

Donskov A. S., Karmanov V. V.

A solution of the elastoplastic contact problem of relative sliding of two elastic bodies bounded with nonlinear surfaces is presented. It is shown that mathematical modeling using variable parameters of elasticity provides determination of the law of changing geometric characteristics of the contact area in different conditions of contact interaction of bodies. The developed mathematical model of contact interaction adequately reflects the physical nature of the process of elastoplastic strain of the counterbody by sliding indenter.

Keywords: elastic-plastic half-space; effective form of the indenter; Hertz problem; deformation; stresses; plastic flow; variable parameters of the elasticity; parameters of the contact area.

UDC 539.3/6:669.1/2

Current State and Prospects of Developing the Center of Multiple Access (CMA) of the Federal State Unitary Enterprise Central Research Institute for Structural Materials "Prometey"

Nemets A. M., Lebedeva N. V., Petrov S. N., Barakhtin B. K.

Arrangement and main focus area of the Center of Multiple Access (CMA) of the Federal State Unitary Enterprise Central Research Institute for Structural Materials "Prometey" are described. We present a wide range of available equipment and the results of several research and scientific-methodical studies.

Keywords: center of multiple access; electron microprobe and light microscopy; x-ray analysis; chemical-analytical studies; standard and precision mechanical tests.

UDC 543.423.1;543.632.495

Reference Standards of Nickel Superalloys of the System Ni – Al – Co – Mo – Nb – Ta – Cr – W – Re for Spectral Analysis

Gundobin N. V., Titov V. I., Pchelkin A. I.

We developed and certified the reference standards (SS) of nickel superalloys for spectral analysis in accordance with the standards of the Russian Federation [1, 2]. An SS set consists of five types of the samples (5 chemistries compositions — 5 fusions). The SS material is first melted in a vacuum induction furnace VIAM-2002 and then the ingots are remelted in UVNK furnace of directional crystallisation to get the samples homogeneous in chemical composition that are then used in manufacturing the standard cylindrical SS samples: diameter 40 mm, height 35 mm. The validation analysis of SS with determination of the mass fraction of the elements and evaluation of the measurement uncertainty is carried out proceeding from the data base of Federal State Unitary Enterprise «VIAM» and results of inter-laboratory experiment (ILE), obtained in various spectroscopic and chemical procedures at: LTD SPO «TsNIITMash» and GU IMET UrO RAS. The results of studying the chemical composition of SS formed the base for certification of the reference samples of heat-resistant nickel superalloys for spectral analysis of SSE category (standard samples of the enterprise containing instructions for their applications).

Keywords: standard (reference) samples; chemical composition; technology of melting SS material; spectral and chemical analysis of the alloys; mass fraction of the elements; certification; calibration curve; the homogeneity of SS material; convergence; reproducibility; standard (middle quadrature) deviation.