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

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UDC 543.423.1

ICP-AES Analysis of Yttrium Oxide with Concentration of Impurities by Co-precipitation*Evdokimov I. I., Pimenov V. G.*

The feasibility of ICP-AES method in combination with impurity concentration by co-precipitation are studied to determine a broad range of impurities contained in Y_2O_3 at the level of 10^{-4} %wt. and less. A technique has been developed for analysis of pure Y_2O_3 by ICP-AES with preconcentration of the impurities from a nitrate solution of the sample by co-precipitation with micro amount of yttrium hydroxide. The detection limits (X_{lim}) for Al, Bi, Cr, Fe, Si, As, Be, Co, Cu, Ga, Ge, In, Mo, Pb, Sb, Se, Sn, Te, Ti, V, W, Er, Tm, Dy, Ho, Lu, Sc and Yb range within $10^{-7} - 10^{-4}$ %wt. depending on the element. The value of the total relative standard uncertainty of the results of analysis (X , the mass part of impurities) at $X \geq 10X_{\text{lim}}$ ranges from 0.05 to 0.15. The main merits of the developed rapid technique of analysis are the broad range of simultaneously determined impurities (28 elements), the use of available reagents (H_2O , HNO_3 , aqueous solution of NH_3), and lower labor intensity.

Keywords: yttrium oxide; inductively coupled plasma atomic emission spectrometry (ICP-AES); impurity preconcentration; co-precipitation.

UDC 669.234;543.245;543.427.4;53.089.68

Development of the Procedure of Analysis of Certified Reference Samples of Palladium-Tungsten Alloy*Lisienko M. D., Lutsak A. K., Anchutina E. A., Kostrikova T. P., Moricheva N. A., Mazaletskii A. G.*

Production of palladium-tungsten alloys at the JSC Yekaterinburg Non-Ferrous Metal Processing Plant (JSC EZOCM) entailed the necessity of control of their chemical composition. Until very recently the reverse complexometric titration with a visual indication of the equivalence point was used for palladium determination in palladium-tungsten alloys. Disadvantages of the method promote the development of new analytical procedures for analysis of palladium-tungsten alloy and reference materials for their metrological support. Certified reference materials (CRM) of palladium-tungsten alloys are developed at the central analytical laboratory of the JSC EZOCM to control the accuracy of analysis and calibration procedures. The developed CRM are used to validate new analytical procedures based on titrimetric and x-ray fluorescence analysis of palladium-tungsten alloys.

Keywords: palladium-tungsten alloys; complexometric titration; certified reference materials; X-ray fluorescence analysis; accuracy of measurements.

UDC 547.245.04;543.544(043);546.281.027*29

Study of the impurity profile of silane enriched with isotope ^{29}Si , using gas chromatography–mass spectrometry*Sozin A. Yu., Chernova O. Yu., Bulanov A. D.*

Gas chromatography-mass spectrometry is used to study the impurity profile of silane enriched with ^{29}Si to 99.9 % at. We managed to identify 32 impurity substances (hydrocarbons $\text{C}_3 - \text{C}_9$, alkylsilane, homologues of silane, siloxanes, and fluorotrisiloxanes) and first obtained mass spectra of Si_2H_6 , Si_3H_8 , *n*- Si_4H_{10} , iso- Si_4H_{10} , $\text{Si}_2\text{H}_6\text{O}$, $\text{Si}_3\text{H}_8\text{O}_2$, *n*- $\text{Si}_4\text{H}_{10}\text{O}_3$, iso- $\text{Si}_4\text{H}_{10}\text{O}_3$, $\text{Si}_3\text{H}_7\text{FO}_2$, SiH_3CH_3 , $\text{SiH}_3\text{C}_2\text{H}_5$, $(\text{SiH}_3)_2\text{CH}_2$, $\text{Si}_2\text{H}_5\text{CH}_3$, $\text{SiC}_3\text{H}_{10}$, enriched with ^{29}Si .

Keywords: gas chromatography-mass-spectrometry; identification; isotopically enriched silane; impurities; mass spectra.

UDC 543.054;543.424;546.98

Sorption-Photometric and Test-Determination of Palladium Using Silica Modified with Polyhexamethylene Guanidine and Nitroso-*R*-Salt*Didukh S. L., Maznyak N. V., Losev V. N.*

Sorbent based on silica successively modified with polyhexamethylene guanidine and nitroso-*R*-salt is developed for sorption-photometric and test determination of palladium. Intensively red colored Pd (II) complexes with nitroso-*R*-salt are formed on a sorbent surface during sorption. Diffuse reflectance spectra of the complexes peaked at 440 and 510 nm. The aforementioned effect formed a basis for developing the procedures of sorption-photometric and test determination of Pd. The limit of Pd detection is 0.06 μg per 0.1 g of the sorbent. Minimum quantity of Pd (II) that can be detected visually is 0.5 μg per 0.1 g of the sorbent. Calibration

curves are linear in the range of 0.1 – 30 µg per 0.1 g of the sorbent. The developed procedures are used for determination of palladium in washing water of palladation and in the solution obtained after opening of post-combustion catalyst.

Keywords: modified silica; polyhexamethylene guanidine; nitroso-*R*-salt; solid phase extraction; palladium; determination.

UDC 622.788:669.054.8

Development of a System of On-line Monitoring of the Agglomerate Chemical Composition

Masalimov A. V., Usherova E. V., Sharanov P. Yu., Usherov A. I., Ishmet'ev E. N., Necheporenko O. N.

A possibility of in-process on-line monitoring of the agglomerate chemical composition is studied using a CON-X 02 pipeline x-ray fluorescence analyzer. Relative error of Fe determination in the chemical composition of sinter is 0.30 % rel. at a content of 56 %; 2.33 % rel. for calcium oxide at a content of 6%; and 6.33 % rel. for basicity at a value of 1.45 units. We proposed and experimentally tested a model for calculating chemical composition of the agglomerate proceeding from the chemical composition of the components and their consumption for sinter production.

Keywords: agglomerate; x-ray fluorescence analysis; chemical composition; determination; iron; calcium; basicity.

UDC 538.971

Diagnostics of Multilayer Nanomaterials Using Methods of X-Ray and Electron Crystallography

Imamov R. M., Klechkovskaya V. V., Galiev G. B., Pushkarev S. S., Ganin G. V., Maltsev P. P.

Multilayer nanoheterostructures A³B⁵ on gallium arsenide and indium phosphide substrates – promising materials for microwave and optoelectronic products – are used to demonstrate the potentiality of x-ray and electron crystallography in diagnostics of nanomaterials. Development and application of the new approach based on a comprehensive study of the structure of experimental nanosamples (using a combination of electron and atomic force microscopy, electron diffraction, x-ray and photoluminescence techniques, as well as a comparative analysis of the data obtained enable us to refine a laboratory technology of growing A³B⁵ multi-layer nanoheterostructures; optimize conditions of the growth process and obtain experimental samples with predicted physical properties.

Keywords: x-ray diffractometry; x-ray reflectometry; electron microscopy; electron diffraction; atomic force microscopy; metamorphic buffer; superlattice; quantum well.

UDC 539.1:678.01

Study of Dielectric Properties in Region of Liquid-Solid Phase Transition

Alekseev A. N., Lazarenko M. M., Lazarenko M. V., Kovalev K. N., Tkachev S. Yu.

A method for determination of the complex permittivity of liquid systems which takes into consideration temperature-dependent change in geometrical parameters (thickness) of the sample and the effect of phase transition is developed. The method which provides measurement in wide range of temperatures (–190 ... 60 °C) at different frequencies (0.1 – 100 kHz) is used to calculate temperature dependences of the complex permittivity of ethanol. The obtained results match the literature data within 3 %.

Keywords: complex permittivity; liquid systems; phase transitions.

UDC 541.123:772.968

Determination of Thermodynamic Compatibility of Crystallizing Polymers and Plasticizers

Vasilyeva G. A., Fedoseev A. M., Ibragimov N. G., Afiatulloev E. H.

A method for determination of thermodynamic compatibility (TDC) of the components of a binary system “crystallizing polymer – plasticizer” is developed. The method is based on experimental determination of the melting point of the system using differential thermal analysis (DTA). TDC of the plasticizer and polymer is determined from the absolute value and sign of the energy interaction parameter which is obtained from the Flory equation that describes lowering of the melting point of the polymeric crystal with a solvent present. Computerized calculation of the energy interaction parameter from experimental data is carried out using the least squares technique. The experimental procedure includes preparation of “polymer – plasticizer” compositions: mechanical mixing of the components with the plasticizer content in a binary mixture ranging within 0 – 50% (in

10% increments) and studying of the compositions weighting about 0.2 g in a DTA device. The results of TDC determination of some known plasticizers with a crystallizing polymer (the crystallinity of 50 – 70 %) are presented.

Keywords: “polymer – plasticizer” system; thermodynamic compatibility; differential thermal analysis; Flory equation; the least squares technique.

UDC 620.178.151

Correlation of the Strain Values and Strengthening Parameters in Tension and Indentation Tests in a Plastic Region

Matyunin V. M., Marchenkov A. Yu.

Analytical dependences of the ultimate uniform strain and yield stress to ultimate stress ratio on the Meyer's coefficient *n* are obtained and exemplified in the study of carbon and low-alloyed perlitic steels using sloping light for measuring the indent diameter upon *n* determination. Meyer's law appeared to be incorrect and invalid for austenitic steels due to structure-phase transformations in the strained metal under the indent.

Keywords: spherical indenter; indentation test; strengthening parameter; ultimate uniform strain; yield strength; ultimate strength; metals diagnostics.

UDC 620.178.3

Evaluation of the Durability of the Elements Made of Polymer Composite Materials under Cyclic Irregular Loading

Pankov A. V.

A procedure providing evaluation of the durability of the structure elements made of polymer composite materials (PCM) under irregular loading in compression area is developed. A number of aspects of PCM damaging under cyclic loading are considered. The verification is made proceeding from available experimental data. The verification demonstrated the applicability of the procedure. Recommendations regarding the value of the procedure parameters are specified.

Keywords: strength; durability; fracture; hub; fracture of composites; irregular loading; method.

UDC 620.169:621.743.43

Stresses and Elastoplastic Strains in Rods with Annular (Circumferential) Cracks

Dobrovolskii D. S.

The service life of structural elements upon a variable or long-term loading consists of three stages: stage of durability (up to crack formation in the notch), stage of vitality, and stage of final destruction. To improve the reliability of forecasting safe operation it appeared expedient to measure and determine the stress-strain state in the notches and cracks using unitary analytical approaches. We developed a method of calculating fields of local stresses and elastoplastic strains in the net section of rods with annular cracks in conditions of tension, bending or torsion proceeding from G. Neuber elastic solutions for circular notches and energy method of evaluating stress concentration and elastoplastic strains in the notches. Acceptability of the method for engineering applications is proved in comparison of the obtained results and data of experimental and analytical method of N. A. Makhutov.

Keywords: rod; annular crack; diagram of the elastoplastic strain of material; solutions; assessment of the acceptability of the method.

UDC 620.171.2:539.52:539.374

Determination of the Rheological Parameters of Superplasticity from the Results of Bulge Tests

Enikeev F. U., Tulupova O. P., Ganieva V. R., Shmakov A. K., Kolesnikov A. V.

Method for determination the material constants of superplastic aluminum alloys from the results of bulge tests is developed. The influence of entry radius is taken into consideration when developing a simplified analytical model of the process under consideration based on main assumptions of thin shell theory. Finite element modeling of the process is implemented using ANSYS-code. The experimental data on superplastic bulge forming of the commercial aluminum alloy AMg6 are used to verify the suggested procedure. It is shown that the deviation of the experimentally measured forming duration from corresponding calculated model value does not exceed 0.3%.

Keywords: superplastic forming; inverse problem; rheology, ANSYS; thin shell theory; aluminum alloys.