ABSTRACT AND REFERENCES
MATERIALS SCIENCE

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INFLUENCE OF THE ALLOY COMPOSITION ON DETERMINING THE MILLESIMAL FINENESS OF GOLD BY XRAY FLUORESCENT AND ASSAY ANALYSIS (p. 6-18)

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The conducted studies revealed the influence of the methods of cupellation and the XFA on the accuracy of determining the millesimal fineness in the system of the assay control. The effect of the alloy composition of jewelry alloys on determining millesimal fineness was established. In particular, it was experimentally proved that nickel in combination with zinc as an integrated alloying component substantially changes the cupellation process of gold alloys. This leads to deviation of the millesimal fineness of a gold alloy, determined by cupellation compared with the X-ray fluorescent analysis (XFA) towards a decrease ranging from 0.10 to 0.15 %.

The research of gold alloys with the content of nickel with the help of the XFA revealed microalloying by modifiers and actively acting deoxidizing agents (indium, palladium, platinum, etc.). It was established that modifiers are used both separately and in complex, but they do not make a significant impact on determining the millesimal fineness.

A number of systemic and random errors in techniques, which affect the results of determining and are related to the technique of performing preparatory operations, as well as analyses operation, were detected. Based on the obtained results, the permanent temperature mode was developed and the procedure of the process of cupellation of gold alloys with the content of nickel was improved with a view to further improvement and metrological certification of the procedure.

It was proved that the use of the XFA method for control of millesimal fineness and the content of the component composition of modern gold-based pieces of jewelry and museum values (antiques) is possible not only as the method of screening. The method can replace the touchstone (with obvious advantages), and be an alternative to cupellation. Application of the XFA for current control of millesimal fineness and the content of an alloy of jewelry pieces is advisable to increase up to 30 % (against 2 % in accordance with the current legislation). Its use will make it possible to increase the number of analyzed samples without worsening the trading presentation of the jewelry pieces of a customer.

Keywords: gold alloys, alloy composition, X-ray fluorescence analysis, method of cupellation (assay analysis), millesimal fineness, assay control.

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A STUDY OF THE EFFECT OF TUNGSTATE IONS ON THE ELECTROCHROMIC PROPERTIES OF NI(OH)$_2$ FILMS (p. 18-24)

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Thin films of nickel hydroxide were prepared using the cathodic template method and were tested in different electrolytes. The electrolytes were 0.1 M KOH and 0.1 M KOH with the addition of 0.1, 0.3 and 1 mM K_2WO_4. The test revealed, that the presence of tungstate can have a significant effect on electrochemical and electrochromic characteristics of $Ni(OH)_2$ films. The initial sample, cycled in 0.1 M KOH showed different characteristics from those cycled in tungstate-containing electrolytes: significant difference between current densities of cathodic and anodic peaks and presence of the current plateau on the cyclic voltamperometry curve. However, the initial sample demonstrated the highest coloration degree of 74 %. On the other hand, the sample showed degradation of the coloration degree past initial growth.

The samples cycled in the tungstate-containing electrolyte showed better electrochemical characteristics – sharper cathodic and anodic peaks, with the lesser difference between peak values. The dynamics of the absolute coloration degree of the samples cycled in tungstate-containing electrolyte showed a constant increase. The sample tested in a solution with 1 mM tungstate had the lowest value of the absolute coloration degree – 60 %. For tungstate concentrations of 0.1 and 0.3 mM, the absolution coloration degree at the last cycle was 72 and 71 % respectively.

The samples tested in a solution with tungstate additive had a significantly lower bleaching time – 40–50 s in comparison to 360 s of the sample cycled in 0.1 M KOH.

A possible mechanism that explains such differences in behavior was proposed.

Keywords: electrochromism, intercalation, $Ni(OH)_2$, nickel hydroxide, tungstate, WO_4^{2-}, polyvinyl alcohol.

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INFLUENCE OF THE DURATION OF AGING THE SYSTEM Ti/Al_2O_3 IN A HYDROGEN ATMOSPHERE ON HYDROGEN SORPTION, ADHESION, TRIBOLOGY, AND ELECTRICAL CONDUCTIVITY OF THE FILM (p. 25-30)

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This paper reports results on the interaction between an aluminum oxide film, deposited on technically pure titanium of grade VT1-0 by the magnetron reactive sputtering method, and a hydrogen-containing atmosphere. Such a study is important in order to find protective coatings that would prevent the penetration of hydrogen inside a product. A given system aged in a hydrogen atmosphere in the interval of 1–4 hours at a pressure of $2 \cdot 10^5$ Pa (2 bar) and a temperature of 400 °C. We have acquired data on the distribution of hydrogen along a film thickness and its content in a thin-film system. It is shown that hydrogen diffuses into the film and builds up in it up to three hours, and only then it begins to penetrate the substrate. We have managed to increase aging duration in a hydrogen-containing environment and increase the temperature of heating up to the stage of film destruction. In the case of the starting film and after aging from 1 to 3 hours the adhesion force between a film and a substrate increases, apparently due to the formation of hydrogen bonds film-substrate. The adsorption of hydrogen atoms at the surface of the Al_2O_3 film is accompanied by an increase in its conductivity by not larger than 4 % with the increased time of aging. Such a change in the conductivity of the Al_2O_3 film can be explained based on the formation of a zone structure. Thin oxide films may possess continuous one-side conductivity, but in the case the film is thick (0.5 μm and above), it is not possible to argue about the one-side conductivity. The data acquired on the influence of aging duration in a hydrogen atmosphere indicate an increase in adhesive strength by almost 6 times within 3 hours and by 2.5 times in 4 hours. The determined coefficient of film friction increases by not larger than 2.5 times. By measuring the electrical conductivity of the film surface, it was found that it increases with an increase in the time of aging in a hydrogen atmosphere. This pattern is obviously linked to the creation of transitions of the p-n-type in the film of aluminum oxide at the expense of hydrogen ions.

Keywords: titanium of grade VT 1-0, method of magnetron sputtering, aluminum oxide, hydrogen atmosphere, adhesion, tribology, electrical conductivity of film surface.

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DEVELOPMENT OF FATIGUE TEST TECHNOLOGY OF SHEET AUTOMOBILE MATERIALS (p. 31-37)

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Ensuring the operability of the cars' parts and components is one of the most topical problems in the modern automotive industry. Most of the car parts are under cyclic loads leading to materials' destruction. Therefore, one of the important factors affecting the performance of products is the fatigue strength of the material. In this paper, the existing methods of fatigue tests are analyzed, their advantages and disadvantages are presented. The methodology of fatigue tests of sheet automobile materials was developed. The main idea of this methodology is that it enables to study the fatigue of sheet automobile materials based on single-plane pure bending. This scheme is very close to the conditions of the actual load of car body structural elements. The results of the study of fatigue strength obtained using this methodology allow studying the kinetics of the failure process, fixing the beginning of macrofailure, crack growth rate and, as a consequence, maintainability of the structure.

Comparative tests enable to determine the material that best meets the operating requirements and provides the reduction of the failure rate of the car metal structures.

In this paper, important characteristics of fatigue strength were obtained for a number of automobile structural steels 08kp and 20kp: service life to complete failure, fatigue limit, period to fatigue crack nucleation and rate of further propagation and, as a consequence, maintainability of the structure. So, for example, the number of cycles for 08kp steel to complete failure (262,000 cycles) and the period to fatigue crack nucleation (82,000 cycles) is greater, and the rate of further growth ($5.38 \cdot 10^{-5}$ mm/cycle) is lower than for 20kp steel (174,000, 68,000 cycles and 8.86.10-5 mm/cycle, correspondingly). Although these parameters were obtained at higher stress (265 MPa) for 08kp steel against only 235 MPa for 20kp steel. This explains the operating advantage of 08kp steel against 20kp steel in the process of car design.

The obtained data enable to prevent failure of structural elements and parts under cyclic loads at the stage of car maintenance, and as a consequence, to increase the car operation safety, and to reduce the cost of repair.

Keywords: fatigue tests, cyclic life, automobile structural materials, current sample deflection.

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ANALYSIS OF THE TECHNOLOGY TO MANUFACTURE A HIGHTEMPERATURE MICROSTRIP SUPERCONDUCTIVE DEVICE FOR THE ELECTROMAGNETIC PROTECTION OF RECEIVERS (p. 38-47)

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Technological features of the process of manufacturing a high-speed high-temperature superconducting microstrip protective device which can reduce in a picosecond period (the time of switching or operation speed) the incoming power from the antenna-feeder path and the power passing through it to a level safe for sensitive semiconductor elements of the receiver (preventing current destruction of p-n junction). The study enables determination of the features and conditions for the use of modern technological methods for creating a superconducting microstrip protective device taking into account influence of the substrate material, superconductor and contacts and the method of their connection on the switching properties of superconducting films of the proposed protective device. The switching properties of superconducting films include speed of phase transition of a film from a superconducting to a nonconducting state. To determine degree of material influence on switching properties, it was proposed to use the following: lattice parameter, thermal expansion coefficient of materials, degree of interaction of molecular structures of the contacting surfaces, probability of local defects on the surface (nonconducting zones). The study outlines basic conditions (methods of film deposition, applying a certain superconducting film (YBCO) on the chosen substrate) which should be met in order to create an operable protective device. The study results make it possible to assess the degree of influence of contact materials and the method of deposition (of both film on the substrate and contacts on the film) on microstructure and switching properties of the superconducting protective device. Such results can be used in synthesis of high-temperature superconducting devices for protecting receiver elements from current destruction of their p–n junctions.

Keywords: high-temperature superconducting film, magnetron spraying, laser spraying, substrate material, contacts on superconductors.

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INFLUENCE OF A MATERIAL AND THE TECHNOLOGICAL FACTORS ON IMPROVEMENT OF OPERATING PROPERTIES OF MACHINE PARTS BY RELIEFS AND FILM COATINGS (p. 48-47)

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A joint effect of preliminary cold plastic deformation and environmentally friendly lubricating substances of plant origin on the improvement of machinability of austenitic steels was considered. This extends the use of such steels as structural material in machine building. The influence of micro- and macroreliefs of machined parts both as technological and operational factors was explored. The former involves division of allowances during machining deep openings, and the latter involves the improvement of operational characteristics of the surface of a part. The influence of film coatings on an increase in durability of rolling bearings was studied. In particular, it was found that these coatings heal micro cracks from grinding of rolling bearings cases and bodies and increase the reliability of these crucial parts by increasing the level of hardness. It was shown that the curvature of the generatrix of deep openings should be decreased by changing the directions of major movements in the related operations. It was established that special attention should be paid to the heredity of machining.

The ways of improvement of machinability of austenitic steels were substantiated. This is the use of preliminary cold plastic deformation to execute a part of work of cutting and transferring paramagnetic status of steels into magnetic. It is also the use of environmentally friendly lubricants of plant origin during cutting. The dual purpose of the reliefs of the surface of a part – technological and operational – was established. The former is executed by a special broaching tool and is effective during machining deep openings. The purpose of the latter is to create labyrinths for lubricants and division of shells into separate elements. The mechanism of the action of vacuum film coating on reliability of rolling bearings, which contributes to healing micro cracks from the preliminary abrasive treatment and an increase in surface hardness, was studied. It was shown that a decrease in the height of curvilinearity and waviness of deep openings is achieved by the change in the main motion of the related operations.

Results of the research are the basis for creating technologies of critical machine parts in production.

Keywords: operational properties, rolling bearings, austenitic steels, regular reliefs, deep openings.

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ENRICHMENT ON BANGKA TIN SLAG'S TANTALUM AND NIOBIUM OXIDE CONTENTS THROUGH NON-FLUORIDE PROCESS (p. 56-64)

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This research explored how non-fluoride solutions including 8M NaOH, 0.8, 1.6 and 2.4 M H_2SO_4, and 0.1, 0.4 and 0.8 M $HClO_4$ increased the contents of tantalum and niobium oxide through leaching. Before leaching, Bangka tin slag (BTS) was characterized through XRF. The slag was then 900 ^oC-roasted, quenched, and dewatered. Next, BTS underwent a sieving process with size classifications of +100, –100+150, –150+200, –200+250, and –250 mesh. After that, the –200+250 mesh slag was leached with 8M NaOH. Then, the leached product was divided into two, one of which was 0.1, 0.4, and 0.8 M $HClO_4$-leached and the rest of which was leached with 0.8 M $HClO_4$ followed by 0, 0.8, 1.6, and 2.4 M H_2SO_4 at 25 ^oC within 2 hours. All the residues characterization used an XRF while that of filtrates used an AAS as well as an ICP-OES. The motives that drive this investigation are the deficit of tantalum supply and its status as one of the technology-critical elements. In addition to that, most of prior investigations enhanced the contents of tantalum and niobium oxide using fluoride acid while this study ventured non-fluoride solutions. The result shows that perchlorate acid followed by sulfuric acid leaching slightly enriches the tantalum and niobium contents. However, this method is the most effective among NaOH, $HCIO_4$, and $HCIO_4$ followed by H_2SO_4 leaching. This finding is a form of scientific effort to maintain the tantalum supply through utilizing worthless waste of tin smelting.

Keywords: leaching, tantalum niobium oxide (TNO), Bangka tin slag, NaOH, $HClO_4$.

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