Наведено результати експериментального дослідження густини розчинів ізопропілового спирту і наночастинок Al_2O_3. Отримані дані дозволили вивчити температурну і концентраційну залежності вивчених нанофлюїдів і розрахувати величину надлишкового мольного об'єму, а також величину гідродинамічного діаметра наночастинок. На підставі проведених досліджень запропонована нова методика прогнозування мольного об'єму нанофлюїдів, яка реалізує трифазну модель нанофлюїда

Ключові слова: нанофлюїд, густина наноізопропанола, мольна концентрація, гідродинамічний радіус, методика прогнозування, трифазна модель

Приведены результаты экспериментального исследования плотности растворов изопропилового спирта и наночастиц Al_2O_3. Полученные данные позволили изучить температурную и концентрационную зависимости изученных нанофлюидов и рассчитать величину избыточного мольного объема, а также величину гидродинамического диаметра наночастиц. На основании проведенных исследований предложена новая методика прогнозирования мольного объема нанофлюидов, которая реализует трехфазную модель нанофлюида

Ключевые слова: нанофлюид, плотность наноизопропанола, мольная концентрация, гидродинамический радиус, методика прогнозирования, трехфазная модель

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RESEARCH INTO THE INFLUENCE OF Al_2O_3 NANOPARTICLE ADMIXTURES ON THE MAGNITUDE OF ISOPROPANOL MOLAR VOLUME

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1. Introduction

Influence of nanoparticles on the thermophysical properties of substances attracts close attention of researchers in view of the possibilities of artificial regulation of properties of technically important liquids. Introduction of nanotechnologies in power engineering enables higher heat transfer coefficients in heat exchangers and efficiency of power equipment [1, 2]. However, application of nanotechnologies in power equipment is hampered by the lack of reliable information on various thermophysical properties of working bodies and coolants as well methods for their calculation.

2. Literature review and problem statement

A detailed analysis of research is given in works [3, 4]. As follows from the published reviews [5–9], the authors pay special attention to the study of the effect the nanoparticles exert on viscosity and thermal conductivity of base liquids. However, the matter of influence of nanoparticles on the density or molar volume of base fluids remains insufficiently studied.

The equations proposed in literature for calculating density of nanofluids are based usually on the principles of additivity. For example, the following simple but insufficiently confirmed by experimental data formula for calculating density of nanofluids was proposed in article [10]:

$$\rho_{nf} = \varphi_{np} \cdot \rho_{np} + (1 - \varphi_{np}) \cdot \rho_{mp}, \quad (1)$$

where φ_{np} is the volume fraction of nanoparticles in the base fluid, m^3/m^3; ρ_{np} is the nanoparticle material density, kg/m^3; ρ_{mp} is the base fluid density, kg/m^3.

There are only a few papers devoted to the experimental study of the effect of nanoparticles on the base fluid density. For example, an experimental study of the effect of carbon nanotubes on Toluol density was described in paper [5]. However, the authors did not show the mechanism of the carbon nanotube effect on density. In work [6], authors give approximate equations describing temperature dependences of density for a single thermodynamic system (polyester oil/

R143a/CuO nanoparticles). The authors of article [7] confined themselves to presenting experimental data on propanol/Al_2O_3 density at room temperature and the nanoparticle concentration range from 0 to 5 wt. %. The presented data indicate that admixtures of nanoparticles at a concentration of 5 % contribute to an increase in propanol density by 7.7 %. Authors of paper [8] checked applicability of the additive model for calculating density of such nanofluids as polyester oil/R143a/Al_2O_3 nanoparticles in a temperature range from 15 to 45 °C and concentration from 5.6 to 40 wt. %. Since this model did not provide an adequate description of the data obtained, the authors used additional correction factors in equation (1). The authors of article [9] reported their study results for density of various nanofluids: admixtures of Al_2O_3, Sb_2O_5, SnO_2, ZnO nanoparticles with their concentration from 1 to 10 vol. % in ethylene glycol/water solutions (60:40) at temperatures from 0 to 50 °C. At the first stage of experimental data processing, the additive model of density calculation was used. The study have shown that discrepancy between calculated and experimental data were as high as 7.5 % for the ethylene glycol/water/ZnO nanoparticle system, Therefore, a simple approximation equation was used at the second stage of processing experimental data which is impossible for other thermodynamic systems.

Thus, the conducted studies show the authors' disinterestedness in developing models for predicting nanofluid density. Moreover, the ultimate goal of the nanofluid density study has been reduced to a trivial problem of approximating the experimental data for the well-defined thermodynamic system.

The performed studies indicate that equation (1) does not provide sufficient accuracy for predicting density of nanofluids important for practical applications. At the same time, lack of reliable methods for predicting properties of nanofluids significantly hinders the progress in introduction of nanotechnologies in development of energy-efficient coolants and working bodies for refrigeration and power engineering.

Thus, the volume of experimental data on density of various nanofluids presented in literature does not ensure development of a procedure for calculating density of nanofluids. It should be stressed that only experimental data on the magnitude of the excess molar volume for various state parameters carry valuable information on the nanofluid structure.

3. The aim and tasks of the study

The study objective consisted in determining influence of Al_2O_3 nanoparticles on density of isopropyl alcohol in a wide range of state parameters.

To achieve the objective, the following tasks were set:

– perform an experimental study of density of a model isopropyl alcohol/Al_2O_3 nanoparticle nanofluid;

– determine the excess molar volume of nanofluid;

– based on the data obtained, compile a procedure for predicting molar volumes of nanofluids.

4. Materials and methods used in the studies of isopropanol/Al_2O_3 nanofluid

4. 1. Preparation of the object of study

Choice of the object of investigation was dictated by high stability of colloidal solutions of isopropyl alcohol/Al_2O_3 nanoparticles.

Nanofluid samples were prepared by the dilution of concentrated nanofluid (20±1 vol. % Al_2O_3, 50 nm, 702129 Aldrich, South Korea) with pure isopropyl alcohol (99.7 % purity, CAS 67-63-0, W292907 Aldrich, UK).

To study the effect of nanoparticles on density of the base liquid, studies were conducted to determine density of pure isopropyl alcohol and four nanofluids. The experiment was carried out at the following mass concentrations of nanoparticles in nanofluid: 0.92±0.053 %, 1.81±0.01 %, 4.01%± ±0.23 % and 6.65±0.38 %.

4. 2. Experimental procedure

Density was measured by a variable-volume pycnometer. Pycnometer with a range of measured volumes from 2.4 to 2.8 cm^3 was used for this purpose. In calibration of the pycnometer, deaerated distilled water was used, the density data of which are given in work [11]. The obtained values of the pycnometer volume depending on the liquid level in it, Δh, were approximated by the linear dependence $V(\Delta h)=2.15074+0.0028446\cdot\Delta h$ (cm^3). The standard uncertainty of the pycnometer volume calculated from this dependence was 0.0006 cm^3.

Weight of the pycnometer loaded with nanofluid and empty pycnometer was determined using GR-300 analytical balance (Japan); the uncertainty of measuring weight did not exceed 0.0004 g. The study was carried out in a liquid thermostat and temperature fluctuations did not exceed 0.03 K. Temperature was measured by Wika TR10-A platinum resistance thermometer (Germany); the extended uncertainty of the measured temperature did not exceed 0.1 K (at reliability of 95 %).

5. Results of the nanofluid density study

Measurements of nanofluid density were performed in the temperature range from 250 K to 315 K. The results of the density measurements are shown in Table 1 and in Fig. 1.

The extended uncertainty of the obtained nanofluid density values was ±0.0010 g/cm^3 (±0.13 %) at a coverage factor k=2 and confidence level p=0.95.

The excess molar volume of the isopropyl alcohol/Al_2O_3 nanoparticle solution under study was calculated from the obtained experimental data (Fig. 2):

$$\Delta V = V_{exp} - V_{add} = V_{exp} - \left[V_{np}x_{np} + V_{bf}\left(1-x_{np}\right)\right], \quad (2)$$

where V_{exp} is the molar volume of isopropyl alcohol/Al_2O_3 nanoparticle solutions determined during the experiment:

$$V_{exp} = \frac{M_{np}x_{np} + M_{bf}\left(1-x_{np}\right)}{\rho_{exp}}, \quad (3)$$

where V_{add} is the molar volume calculated from the additivity condition, cm^3/mol; V_{np} is the molar volume of the nanoparticle material taken equal to the molar volume of aluminum oxide in α-modification at 20 °C [12], cm^3/mol; x_{np} is the mole fraction of nanoparticles, mol/mol; V_{bf} is the molar volume of the base liquid (isopropyl alcohol) at the measurement temperature, cm^3/mol [13]; $M_{np,}$ M_{bf} are molar weights of solution components, g/mol; ρ_{exp} is the measured density of nanofluid, g/cm^3.

Table 1

Experimental data on the density of solutions of isopropyl alcohol/Al_2O_3 nanoparticles

No.	T, K	w, vol. %	x, mol. %	ρ, g/cm^3	ρ calculated by formula (1)
1	263.1	0.0	0.0	0.8109	0.8109
2	283.1	0.0	0.0	0.7953	0.7949
3	295.3	0.0	0.0	0.7843	0.7847
4	303.6	0.0	0.0	0.7775	0.7776
5	323.4	0.0	0.0	0.7601	0.7599
6	343.3	0.0	0.0	0.7410	0.7410
7	263.1	0.92	0.54	0.8169	0.8122
8	283.3	0.92	0.54	0.8007	0.7960
9	303.5	0.92	0.54	0.7835	0.7789
10	323.2	0.92	0.54	0.7657	0.7612
11	343.2	0.92	0.54	0.7467	0.7423
12	262.9	1.81	1.08	0.8275	0.8135
13	283.2	1.81	1.08	0.8114	0.7973
14	303.2	1.81	1.08	0.7931	0.7803
15	323.1	1.81	1.08	0.7751	0.7625
16	342.9	1.81	1.08	0.7558	0.7437
17	263.1	4.01	2.40	0.8438	0.8165
18	283.0	4.01	2.40	0.8278	0.8005
19	303.2	4.01	2.40	0.8093	0.7833
20	323.1	4.01	2.40	0.7912	0.7654
21	343.2	4.01	2.40	0.7716	0.7463
22	263.1	6.65	4.03	0.8599	0.8204
23	283.3	6.65	4.03	0.8436	0.8041
24	303.3	6.65	4.03	0.8260	0.7871
25	323.1	6.65	4.03	0.8079	0.7692
26	343.2	6.65	4.03	0.7881	0.7500

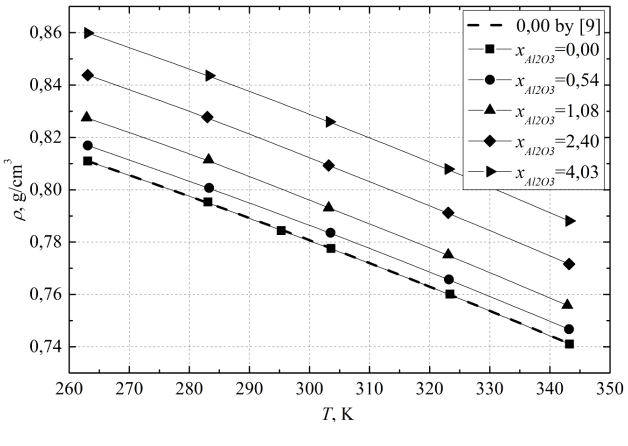

Fig. 1. Temperature dependence of density of isopropyl alcohol/Al_2O_3 nanoparticle solutions

Analysis of the obtained results shows that the excess molar volume of the investigated nanofluids depends on temperature (within the error of measuring the molar volume of isopropyl alcohol/Al_2O_3 nanoparticle solutions). There is also a nonlinear character of dependence of the excess molar volume on nanoparticle concentration. The obtained values of the excess molar volume were approximated by the following dependence:

$$\Delta V = -0{,}046292\left(1-\exp\left(-259{,}2052\cdot x\right)\right), \quad (4)$$

where x is the molar fraction of nanoparticles in isopropyl alcohol.

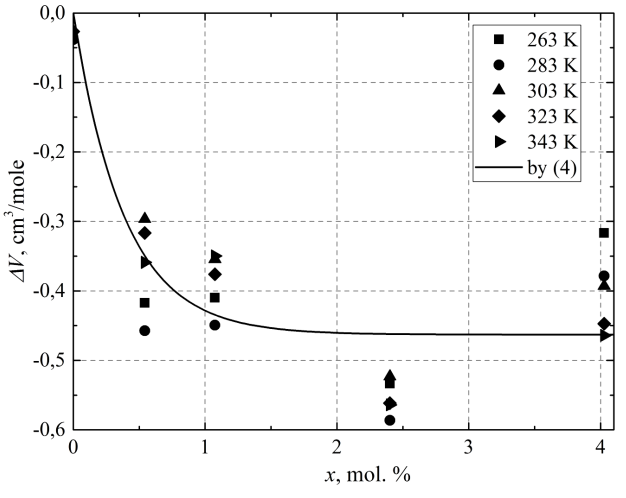

Fig. 2. Concentration dependence of the excess molar volume of isopropyl alcohol/Al_2O_3 nanoparticle solutions

Standard deviation, St (ΔV),

$$St(\Delta V)=\sqrt{\frac{\sum_{i=1}^{n}\left(\Delta V_i-\Delta V_{(3)}\right)^2}{n(n-1)}} \quad (5)$$

of the values of the excess molar volume for isopropyl alcohol/Al_2O_3 nanoparticle solutions, ΔV_i, obtained from equation (3) from the values calculated from approximation equation (4), $\Delta V_{(3)}$, was 0.02 cm^3/mol. This value was less than the uncertainty (0.1 cm^3/mol) of the obtained experimental data.

As it follows from information given in Fig. 3, deviations of the molar volume calculated using expression (1) from the experimental data approximated by equations (2) and (4) reach 4 cm^3/mol. These deviations repeatedly exceed uncertainty of the experimental data given in Table 1. On the contrary, calculation of the molar volume of the studied objects according to formulas (2) and (4) adequately reproduces the experimental information (Fig. 4).

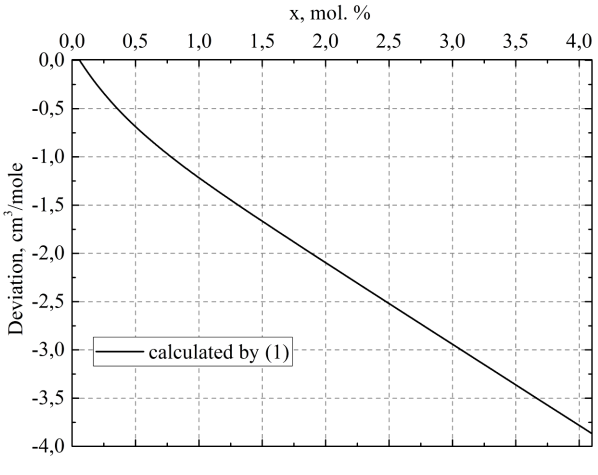

Fig. 3. Deviation of the values of the molar volume of isopropyl alcohol/Al_2O_3 nanoparticle solutions calculated by equation (1) from the data calculated from formulas (2) and (4)

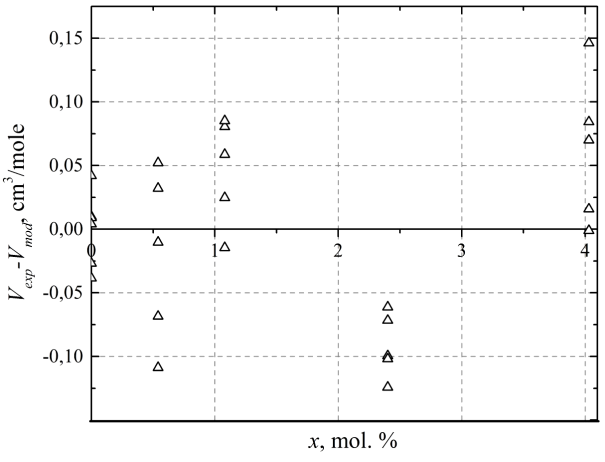

Fig. 4. Deviation of the experimental values of the molar volume of nanofluids (3) from those calculated by formulas (2), (4)

Thus, the conducted study shows that the nanofluid density cannot be calculated according to the additivity rules using concentration of nanoparticles, both in volume and mole fractions.

6. Modeling molar volume of nanofluids

The obtained data on the excess molar volume enable consideration of the possibility of using a three-phase model of nanofluids to predict their density. Within the framework of the proposed model, a nanofluid consists of the following phases:

– nanoparticles: a dispersed phase with thermophysical properties corresponding in the first approximation to the properties of the nanoparticle material;

– base liquid: dispersion medium with thermophysical properties corresponding to the properties of a pure substance at corresponding temperatures;

– surface layer of nanoparticles: the base fluid molecules adsorbed on the nanoparticle surface forming a phase with thermophysical properties (molar volume) different from those of the dispersion medium at the study parameters.

Fig. 5 shows a three-phase model for predicting density of nanofluids.

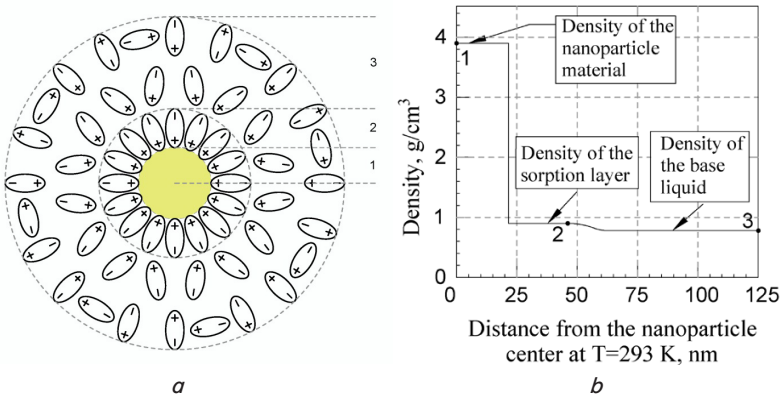

Fig. 5. A three-phase model for predicting density of nanofluids: *a* – micelle structure; *b* – dependence of the sorption layer density on the distance from the center of the nanoparticle

In his monograph [14], the author formulated the concept of closeness of the liquid properties at low temperatures to the properties of a solid phase. Within the framework of these concepts, the molar volume of the surface layer can be taken in the first approximation equal to the molar volume of the base liquid phase at the melting temperature.

According to these model approximations, the molar volume of the nanofluid will have the following form:

$$V = V_{np}x_{np} + V_{bf}\left(1 - x_{np} - x_{sl}\right) + V_{mp}x_{sl} =$$
$$= V_{np}x_{np} + V_{bf}\left(1 - x_{np}\right) + \left(V_{mp} - V_{bf}\right)x_{sl} =$$
$$= V_{add} + \left(V_{mp} - V_{bf}\right)x_{sl}, \quad (6)$$

where V is the molar volume of the nanofluid; x_{sl} is concentration of the base liquid in the surface layer expressed in mole fractions; V_{mp} is the molar volume of the base fluid in the surface layer.

Within the framework of the proposed model for calculating density of nanofluids, the mole fraction of the base liquid in the surface layer is:

$$x_{sl} = \frac{V - V_{add}}{V_{mp} - V_{bf}} = \frac{\Delta V}{V_{mp} - V_{bf}}. \quad (7)$$

The values of the molar concentration of the base liquid in the surface layer calculated according to the proposed model are given in Fig. 6.

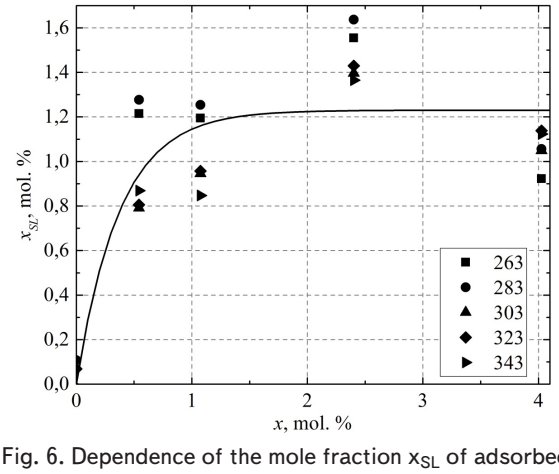

Fig. 6. Dependence of the mole fraction x_{SL} of adsorbed isopropanol molecules on the nanoparticle surface on their concentration in the base liquid

As it follows from the obtained data, the molar fraction of adsorbed molecules of the base liquid intensively increases only at small concentrations of nanoparticles in the base liquid (less than 0.02 molar fraction).

Assuming that Al_2O_3 nanoparticles (according to electron microscopy [15]) have shape close to a spherical shape, the surface layer will be a spherical layer of isopropanol molecules sorbed on the nanoparticle (Fig. 5). Based on this model, it is possible to calculate equivalent diameter of a nanoparticle with a surface layer of isopropyl alcohol around it (diameter of the adsorption layer):

$$D_{SL} = D_{np}\sqrt[3]{\left(\frac{x_{sl}}{x_{np}}\cdot\frac{V_{mp}}{V_{np}}+1\right)} = D_{np}\sqrt[3]{\left(\frac{x_{sl}}{x_{np}}\cdot\frac{\rho_{np}}{\rho_{mp}}\cdot\frac{M_{bf}}{M_{np}}+1\right)}, \quad (8)$$

where D_{np} is the equivalent diameter of nanoparticles, nm.

Results of the conducted study show that the equivalent diameter of Al_2O_3 nanoparticles in isopropyl alcohol essentially depends on composition and slightly depends on temperature. Equivalent diameter of the adsorption layer of isopropanol molecules on the surface of nanoparticles varies depending on concentration in a range from 80 to 55 nm. These data satisfactorily agree with the results of measuring hydrodynamic diameter of nanoparticles. Hydrodynamic diameter of nanoparticles was determined by one of the methods of dynamic light scattering, i. e. laser correlation spectroscopy of homodynation [16] with a wavelength of probe radiation of 532 nm (Fig. 7). In this case, the hydrodynamic diameter of the optical inhomogeneities was determined from their mobility found from the correlation functions of scattered light. In calculations, the particle diffusion coefficient was estimated as an Einsteinian one:

$$\frac{kT}{3\pi\eta r},$$

where T is the medium temperature; η is viscosity of the dispersive liquid; r is hydrodynamic radius of the dispersed particles.

Analysis of the information presented in Fig. 7 makes it possible to conclude that the method of dynamic light scattering states an increase in the multiscale heterogeneities of the cluster type with an increase in concentration of nanoparticles. The lifetime of such structural inhomogeneities is small but it is longer than the sampling time for constructing one correlation function of the scattered light intensity (of the order of seconds). In long-term monitoring, this leads to a very significant "scatter" of the experimental data on the value of the hydrodynamic diameter of the nanoparticles (Fig. 7). In addition, it can be stated that the probability of occurrence of structural inhomogeneities in the nanofluid increases with increase in the concentration of nanoparticles in the base fluid.

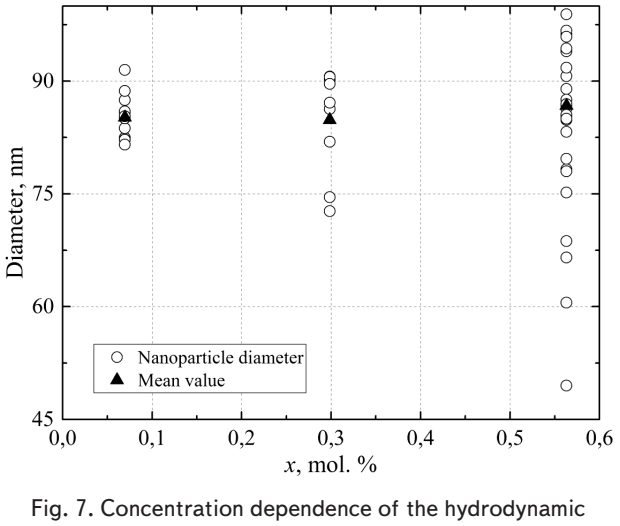

Fig. 7. Concentration dependence of the hydrodynamic diameter of Al_2O_3 nanoparticles

Despite the considerable data scatter, average values of D_{sl} are in good agreement with the values of the equivalent hydrodynamic diameter of nanoparticles for isopropyl alcohol/Al_2O_3 nanoparticle nanofluids calculated from formula (8) (Fig. 7).

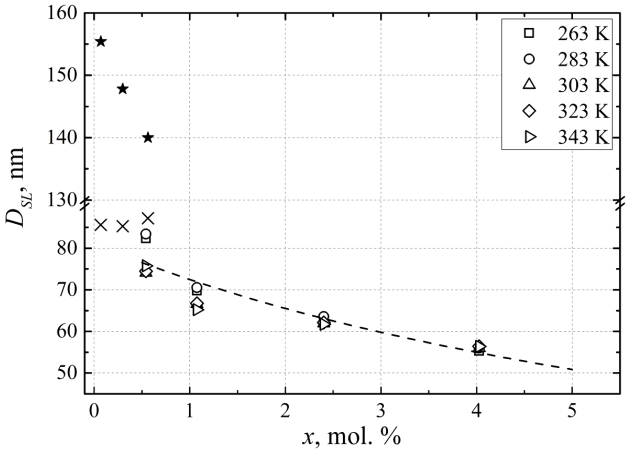

Fig. 8. Concentration dependence of the equivalent D_{SL} diameter of the adsorbed layer of isopropyl alcohol molecules on the surface of Al_2O_3 nanoparticles: ★ — diameter determined by spectroturbidimetry; × — diameter determined by the dynamic light scattering method; - - - — approximation of the calculated values of the nanoparticle diameter

As it follows from Fig. 8, the equivalent diameter of the adsorption layer of isopropanol molecules decreases with an increase in concentration of Al_2O_3 nanoparticles. This conclusion was confirmed by the results of measuring the hydrodynamic diameter by the dynamic scattering method. Appearance of different-scale cluster-type inhomogeneities in the isopropyl alcohol/Al_2O_3 nanoparticle solution results in appearance of submicron to microscopic formations. These formations can promote reduction of the amount of isopropyl alcohol molecules sorbed on nanoparticles. According to the proposed method for calculating molar volume of nanofluids, this effect determines the decrease in the equivalent diameter of nanoparticles. This factor must be taken into account when predicting density of nanofluids. In addition, the obtained information on thickness of the surface layer of nanofluids can be used in equations for predicting thermal conductivity within the framework of the models proposed in [17].

Fig. 8 also provides data on equivalent Al_2O_3 particle sizes which were obtained by spectroturbidimetry in the wavelength range from 400 to 700 nm. These data significantly differ from the values of the equivalent diameter of nanoparticles obtained from the information on the molar volume of nanofluids (7) and the D_{sl} values measured by the method of hydrodynamic light scattering. The authors explain discrepancy of their experimental data by the fact that when using different optical techniques, several different geometric parameters of optical inhomogeneities are measured. Dynamic scattering is determined by motion of the optical inhomogeneity and, consequently, by motion of the nanoparticle with its adsorbed layer. The spectro-turbidimetry method makes it possible to determine entire volume of inhomogeneities with a refractive index differing from the pure base liquid due to interaction with the nanoparticle. Thus, if the method of dynamic light scattering makes it possible to determine the equivalent diameter of granules (nanoparticle/sorbed layer of alcohol molecules), then the spectro-turbidimetry method

determines the already equivalent size of micelles (sorbed layer of alcohol molecules/diffuse layer of alcohol molecules).

Experimental data on the isopropyl alcohol/Al_2O_3 nanoparticle nanofluid density were obtained. From information shown in Fig. 1, it follows that an increase in concentration of Al_2O_3 nanoparticles contributes to an increase in density of isopropyl alcohol/Al_2O_3 nanoparticle nanofluids. It should be noted that the temperature dependences of the nanofluid density remain practically equidistant.

As it follows from the analysis of the excess molar volume of the studied isopropyl alcohol/Al_2O_3 nanoparticle solution (Fig. 2), the nanofluid density cannot be calculated according to additivity rules using nanoparticle concentrations in both volume and mole fractions. Thus, when calculating density of nanofluids, it is necessary to take into account presence of a layer of isopropanol molecules adsorbed around the nanoparticles. Due to its ordered structure, density of the adsorption phase is higher than the base liquid density. Data on the diameter of the adsorption layer obtained from the study of the excess molar volume are in good agreement with the data obtained by the method of dynamic light scattering. This result confirms validity of the proposed technique for calculating density of nanofluids.

It should be noted that the effect of adsorption phase formation around nanoparticles should be taken into account when developing models for calculating other thermophysical properties of nanofluids. This task will be addressed in subsequent publications of the authors.

8. Conclusions

1. Information on the effect of Al_2O_3 nanoparticles on isopropyl alcohol density was obtained. It was shown that the effect of nanoparticles on density is not additive.

2. A three-phase technique for predicting density of nanofluids has been developed. This technique takes into account presence of a sorbed layer of the base fluid molecules on the nanoparticle surface. As the studies show, density of the sorption phase is higher than density of isopropyl alcohol at the set-up parameters. Presence of a sorption layer of isopropyl alcohol molecules on the nanoparticle surface determines magnitude of the excess molar volume. This fact should be taken into account when simulating density of nanofluids.

3. It was shown that the value of hydrodynamic radius of nanoparticles obtained from the data on the molar volume of isopropyl alcohol/Al_2O_3 nanoparticle solutions agrees with the data obtained by dynamic light scattering within the error of determining the value.

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Наведені дослідження структури і властивостей покриттів, отриманих при мікродуговій обробці на магнієвому сплаві. Обробка проводилася при анодно-катодному режимі в лужному електроліті з різними домішками. Показана можливість формування кристалічних оксидних покриттів різного фазового складу (MgO, $MgAl_2O_4$, Mg_2Si_4, $Mg_3(PO_4)_2$) товщиною до 300 мкм, що мають високу адгезію з основою, гарні захисні властивості і високу твердість, яка досягає 6,6 ГПа

Ключові слова: структурна інженерія, мікродугове оксидування, магнієві сплави, фазовий склад, адгезійна міцність, твердість

Приведены исследования структуры и свойств покрытий, полученных при микродуговой обработке на магниевом сплаве. Обработка проводилась в анодно-катодном режиме в щелочном электролите с разными примесями. Показана возможность формирования кристаллических оксидных покрытий разного фазового состава (MgO, $MgAl_2O_4$, Mg_2Si_4, $Mg_3(PO_4)_2$), толщиной до 300 мкм, которые имеют высокую адгезию с основой, хорошие защитные свойства и высокую твердость, достигающую 6,6 ГПа

Ключевые слова: структурная инженерия, микродуговое оксидирование, магниевые сплавы, фазовый состав, адгезионная прочность, твердость

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INVESTIGATION OF THE INFLUENCE OF TECHNOLOGICAL CONDITIONS OF MICROARC OXIDATION OF MAGNESIUM ALLOYS ON THEIR STRUCTURAL STATE AND MECHANICAL PROPERTIES

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1. Introduction

The solution of the problem of improving special working properties, reliability and durability of functional elements, devices and machines can be achieved by targeted modifying the shape, structure and surface properties (or, more precisely, near-subsurface layer). This is the basis for the development of priority scientific practical direction in modern materials science – surface engineering [1]. Surface engineering combines methods of directed change in the physical and chemical properties of the surface layers of materials by deformation [2], modification [3], deposition of single-layered [4], multi-period [5], multi-element [6] coatings and protective layers by different combined methods [7]. Innovative character of the surface engineering development is determined by the fact that the main quality indicators of machines are reliability and performance efficiency. These parameters are mainly determined by the properties of the surface layers of parts and joints (endurance limit, corrosion resistance, wear resistance, coefficient of friction, contact stiffness, fit strength, tightness of joints, etc.) [8]. This can be achieve d by modifying the surface with high-performance methods. Surface modification is especially relevant for the lightweight materials (mainly based on aluminium, magnesium and titanium). The effectiveness of their use at present is very high not only in the traditional aerospace and