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# ТРИБОЛОГИЧЕСКИЕ СВОЙСТВА СЛОИСТЫХ ДВОЙНЫХ ГИДРОКСИДОВ РАЗЛИЧНОГО СОСТАВА

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***В данной работе были синтезированы порошки карбонатных форм слоистых двойных гидроксидов (СДГ), содержащие различные двойные и тройные комбинации соединений Zn, Cu, Cr и Al, взятых в соотношениях Zn:Al=2:1, Zn:Cr=2:1, Cu:Zn:Al=1:1:1 и Cu:Cr=3:1, позволяющих получить химически стабильные продукты. Полученные порошки были исследованы методами рентгеновского фазового анализа и, после высокоэнергетического помола, использованы в качестве добавок (3 масс.%) для приготовления смазочных композиций на основе универсального базового масла И-20А. Трибохимическое поведение полученных смазочных композиций было изучено с использованием машины трения. Исследовалось изменение химического состава трущихся поверхностей стали, величина износа и нагрузка сваривания, а также кинетика изменения коэффициента трения и температуры смазочной композиции в процессе трения. Показано, что добавки всех видов исследованных слоистых двойных гидроксидов существенно улучшают весь комплекс трибологических характеристик модельной смазочной композиции, оказывая комплексное влияние на ее антифрикционные, противоизносные и противозадирные свойства. При этом, вклад в увеличение конкретных трибологических характеристик определяется химическим составом частиц слоистых двойных гидроксидов. Присутствие Zn в наибольшей степени способствует уменьшению коэффициента трения, Cu увеличивает противоизносные свойства поверхности, а Al обеспечивает увеличение нагрузки сваривания трущихся поверхностей стали. Обсуждается механизм трибохимических процессов, протекающих в исследованных системах. Показано, что достигаемый эффект обеспечивается наноразмерным характером частиц слоистых двойных гидроксидов, а также возможностью их встраивания в дефекты поверхности стали и химического взаимодействия с ее поверхностными слоями.***

**Ключевые слова:** слоистые двойные гидроксиды, смазочные композиции, трибохимиическое поведение, трибологические свойства

# TRIBOLOGICAL PROPERTIES OF LAYERED DOUBLE HYDROXIDES OF VARIOUS COMPOSITIONS

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***Some carbonate forms of powdered layered double hydroxides (LDH) containing various double and ternary combinations of Zn, Cu, Cr and Al compounds were produced. The molar ratios of: Zn:Al = 2:1, Zn:Cr = 2: 1, Cu:Zn: Al = 1:1:1 and Cu:Cr = 3:1 were selected to obtain chemically stable products. The resulting substances were investigated by XRD methods and, after a high-energy grinding, were used as additives (3 wt%) to produce lubricating compositions based on I-20Abase oil. The tribochemical behavior of the obtained lubricants was studied using a friction machine. A change in the chemical composition of the rubbing steel surfaces, wear and welding load value, as well as the kinetics of the friction coefficient and temperature of the lubricants were measured during the testing process. It is shown that all the types of investigated LDH's additives significantly improve the complex of tribological characteristics of the model lubricant composition (antifriction, antiwear and extreme pressure properties). The contribution in specific tribologic characteristics is determined by the chemical composition of the LDH's particles. A presence of Zn mostcontributes decreased friction, Cu increased wear resistance, and Al provides increased welding loads. The mechanism of tribochemical processes occurring in the investigated systems is discussed. It is shown that the achieved effect is provided by nanoscale nature of LDH particles, as well as possibility of their incorporation into the steel surface micro-cracks and chemical interaction with the steel surface layers.***

**Key words:** layered double hydroxides, lubricating compositions, tribochemical behavior, tribology properties

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## INTRODUCTION

The tribology properties of lubricants represent fundamental factors ensuring long life service of machine assemblies and as well as low energy consumption influencing energy dissipation related to friction. To improve lubricating properties of greases, the particles of solid lubricants (antifriction, antiwear additives) of various chemical compositions [1-3] are widely used, i.e. graphite and molybdenum disulfide [4, 5] having a layered crystalline structure are most popular admixtures of this group. However, increased requirements to the tribology properties of antifriction additives favor a search of new solid lubricants characterized with enhanced complex of different tribology properties. In this regard, the main attention is paid to the development of lubricating additives that can withstand extreme loads and ensure minimal wear in the machines and mechanisms [6, 7].

Layered double hydroxides [8] (LDHs) represent one of perspective groups of new lubricating materials. These substances have hydrotalcite-like anionic clay structures with the chemical composition corresponding the formula $[M(II)_{1-x}M(III)_x(OH)_2]^{x+}[A^{n-}_{x/n}\cdot yH_2O]^{x-}$, where M(II) and M(III) are divalent and trivalent metal cations, respectively, and $A^{n-}$ is an n-valence anion. These compounds have a layered crystalline structure similar to $MoS_2$, with wide variations depending a nature of cations and M(II) / M(III) molar ratio, as well as a type of anion [9]. One of the advantages, among other layered materials, the LDH's could be formed with a large number of possible combinations of metals M(II) and M(III) and metal-anion combinations due to relatively simple experimental methods of synthesis based on “green” chemistry. The LDH's can form highly dispersed powdered (submicro- and nanoscale) systems having layered structure which strongly influences friction and wear processes due to potential tribochemical behavior and forming of different protective films onto worn surface. For example, the addition of Co/Al-$CO_3$ LDH particles to the basic oil contributed to a decrease in friction by 49% and a decrease in wear scar diameter by 33,1% [10], Mg/Al-$NO_3$ nanopowders intercalated with dodecanoic acid reduce friction by 23% [11]. Similar results were demonstrated by other types of LDH's, such as Zn/Al, Zn/Mg/Al, Ni/Al, Mg/Al/Ce, etc. carbonate systems [12-14]. In any case, LDH particles contribute to the improvement of various tribology properties of lubricating compositions however, the published data indicates that their tribology behavior strongly depends on the chemical composition [13].

In this regard, it is necessary to note that copper-containing LDH systems are not studied as lubricating additives yet, in spite of a great potential for application in this area as layered materials containing an effective cladding element [15- 17].

Taking into account above mentions comments, the aim of this work was to produce the Cu and Zn containing LDH's of various chemical compositions and carry out a comparative study of tribology properties of the obtained lubricating systems and standard basic oil in order to specify a mechanism of the processes taking place in the surface layers of a worn metal surface. In particular, the LDH's related to the carbonate systems of Cu/Cr, Zn/Cr, Zn/Al and Cu/Zn/Al were synthesized and investigated, Cr and Al were selected as additional components of the LHD compositions taking into account that the 1st positively influences antiwear behavior as well as the 2nd potentially may affect a value of critical loading [18-21].

## EXPERIMENTAL PART

To obtain the powders of the selected layered double hydroxides, the following chemical reagents were used as raw materials: $Zn(NO_3)_2{\cdot}6H_2O$ (chemically pure grade) GOST 5106-77, $Al(NO_3)_3{\cdot}9H_2O$ (analytical grade) GOST 3757-75, $Cr(NO_3)_3{\cdot}9H_2O$ (analytical grade) GOST 4471-78, $Cu(NO_3)_2{\cdot}3H_2O$ (analytical grade) TU 2622-003-62931140-2015, KOH (chem. h.) GOST 24363-80, $K_2CO_3$ GOST 10690-73.

The LDH powders were synthesized by the standard coprecipitation method maintaining a constant pH value (pH = 10) [22]. The following $Me^{2+}$ and $Me^{3+}$combinations characterized with a molar ratio of: Zn:Al=2:1, Zn:Cr=2:1, Cu:Zn:Al=1:1:1, Cu:Cr=3:1, were chosen for the selected systems: taking into account the results of some previous experimental works on synthesis of stable LDH systems [23].

To produce the carbonate forms of layered double hydroxides, 2.5 M aqueous solutions of KOH and $K_2CO_3$ were mixed in a molar ratio of 6:1 and added to the 1M mixed solutions of the corresponding nitrates of the $Me^{2+}$ and $Me^{3+}$ metals. The addition of mixed KOH and $K_2CO_3$ aqueous solution was carried out at a rate of 2 ml/min until obtaining pH = 10 in the reaction medium. After that, the resulting dispersions were subjected to the thermal treatment at 90 °C for 5 h. The obtained precipitate was filtrated and washed with distilled water. Furthermore, the precipitate was dried at 60 °C and grounded in the vibration mill (Fritsch PULVERISETTE 0) for 0,5 h to produce highly dispersed powder.

The phase composition of the obtained products was determined using an ARLX'TRA diffractometer (CuKα source $\lambda = 0.15439$ nm) in the 2Θ angle range from 5 to 600.The size of particles in the powders was estimated using the formula of Scherrer:

$$L_c = \frac{0.89\lambda}{\beta_{006}\cdot cos\theta_{006}},$$

Where $L_c$ is a length of t crystals, λ is a wavelength of X-rays, $\beta_{006}$ is the width of the reflection at half of maximum, obtained by reflection from the plane (006); $\theta = 23.48^0$ is an angle of the reflection for the corresponding plane.

The synthesized LDH powders were dispersed in the basic industrial oil of the I-20A trademark (GOST 20799-88) using a vibration mill (Fritsch PULVERISETTE 0). The content of powder fillers in the lubricating compositions was of 3 wt.% in accordance to the overall practice of such testing investigations [24]. The tribology properties of the prepared lubrication compositions were studied using a standard friction machine consisting of a clip-roller tribo-interface (Fig. 1) made of GCr15 bearing steel. The tests were carried out at a load of 360 N with a rotation speed of 800 rpm. Values of the friction coefficient and temperature of the lubricating composition were recorded. An area of wear spots formed onto the surface of the testing rollers was examined using an Explorer Aspex FEI scanning electron microscope and taken as a characteristic of wear resistance. A minimum load at which the rubbing surfaces adhered to each other was considered as a welding load factor.

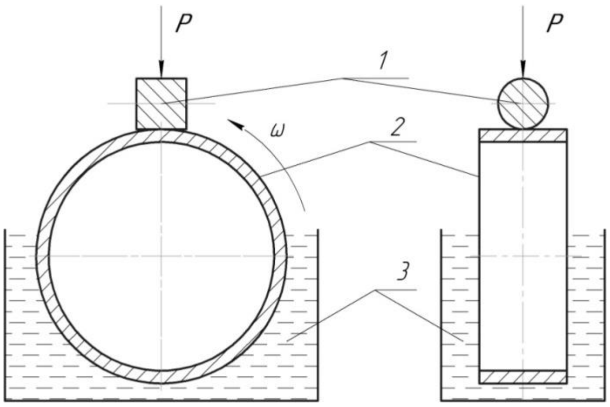

Fig. 1. Friction machine diagram. 1 - roller, 2 - clip, 3 - lubricant
Рис. 1. Схема машины трения. 1 - ролик, 2 - обойма, 3 - смазка

## RESULTS AND DISCUSSION

X-ray diffraction patterns of the synthesized LDH powders are shown in Fig. 2. All the main diffraction reflections are in a good agreement with the characteristics of the hexagonal phase of LDH's

(JCPDS-ICDD 37-630). The Zn/Cr system is characterized by low-intensity reflections, which indicates an almost amorphous structure of LDH. The Cu/Cr system is characterized by broad low-intensity reflections corresponding to the LDH phase, as well as reflections corresponding to copper oxide. An appearance of the CuO impurity in the phase Cu/Cr LDH can be explained by the cooperative effect of Jahn-Teller [25]. However, the Zn and Al containing LDH systems (Zn/Al and Cu/Zn/Al) are characterized by narrow and intense X-ray reflections, indicating a high degree of crystallinity of the materials produced. On the other hand, in accordance with the XRD data, the precipitate obtained using the Cu/Cr raw materials mixture has practically amorphous structure.

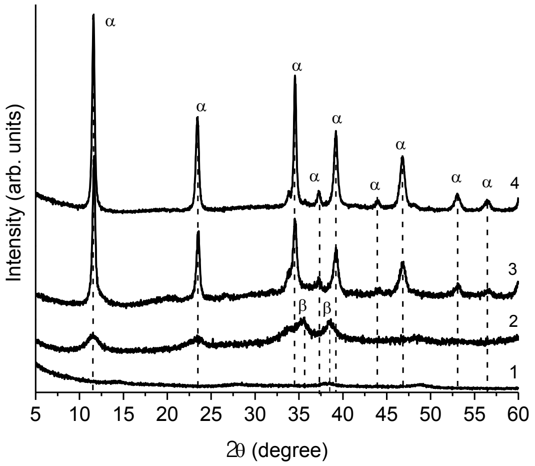

Fig. 2. XRD patterns of LDH powders of various chemical compositions. (1): LDH(Zn/Cr); (2): LDH(Cu/Cr); (3):LDH(Cu/Zn/Al); (4): LDH(Zn/Al); α: LDH; β: CuO

Рис. 2. Рентгенограммы порошков СДГ различного химического состава. (1): СДГ(Zn/Cr); (2): СДГ(Cu/Cr); (3): СДГ(Cu/Zn/Al); (4): СДГ (Zn/Al); α: СДГ; β: CuO

An average size (length) of the synthesized LDH crystals based on the analysis of XRD patterns of the synthesized and investigated products using the Scherrer formula is reported in Table 1. The Zn and Al containing LHD powders (Zn/Al and Cu/Zn/Al) have a size more than 20 nm, where as a size of Cr containing LHD's (Cu/Cr and Zn/Cr system) is so small that the particles of these products only contain a few tens of planes (may be less, for the Cu/Cr system), as a result XRD reflections are so wide that it is difficult to distinguish them from the background and to calculate their real size.

Fig. 3 shows the kinetic curves for the friction coefficient for pure basic oil and for the same containing 3 wt. % of different LDH additives. Each of the LDH additives reduced a value of the friction coefficient in comparison with the basic oil. Nevertheless, the level of friction reduction depended on chemical composition of LDH's powder (Table 1). The greatest reduction in friction (37%) was achieved with the Cu free admixture of the Zn/Cr-$CO_3$ LDH system. Copper containing LDH's (Cu/Zn/Al-$CO_3$ and Cu/Cr-$CO_3$) reduced the friction coefficient by 26 and 23%, respectively, while the system of Zn/Al-$CO_3$ reduced the coefficient of friction by 32%.

As can be seen, a stable value of the friction coefficient is established after 15-20 min of testing and is associated with a lapping of tribo-couplings as wall as separation of agglomerated LDH particles [26].

***Table 1***

**Average size (D) of LDH crystals of various synthesized compositions**

***Таблица 1.* Средний размер кристаллитов СДГ различного химического состава**

| Additive | D, nm |
|---|---|
| LDH (Zn/Al) | 20.9 |
| LDH (Cu/Zn/Al) | 20.4 |
| LDH (Cu/Cr) | 9.1 |
| LDH (Zn/Cr) | Could not be calculated |

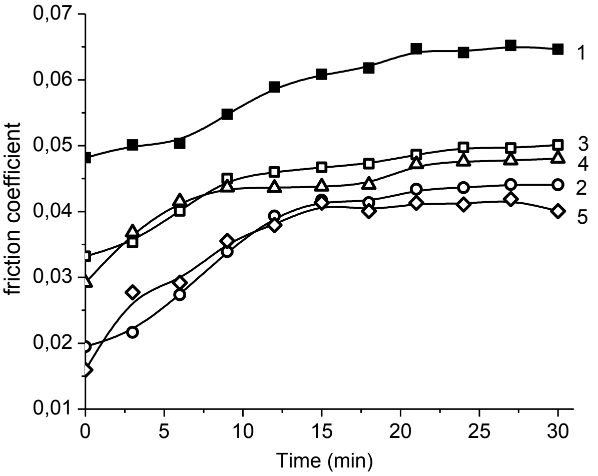

Fig. 3. Friction coefficient kinetics for the investigated lubricating compositions based on different LHD's at a load of 360N. The systematic measurement error of the friction coefficient value is of ±0.002

Рис. 3. Кинетические зависимости коэффициента трения для исследуемых смазочных композиций на основе различных СДГ при нагрузке 360 Н. Систематическая погрешность измерения значения коэффициента трения составляет ± 0,002

Fig. 4 shows a change of the temperature for different lubricating compositions during the tribo-testing process. It is known that up to 95% of the work of friction forces is spent on heating of directly contacting friction surfaces [27]. As you can see, the admixtures of LDH powders related to all the investigated dimetal systems (Zn/Cr, Zn/Al, Cu/Cr) decreased a value of the steady-state temperature of the lubricating composition indicating reduced friction. At the same time, a use of the Cu/Zn/Al-$CO_3$ LDH additive promoted increased temperature of the lubricant composition in spite of significant decrease in the friction (Fig. 1). Such phenomena can be explained by the abundant deposition of powders on the friction surface and further interaction of the powders with the friction surface. As a result, modified layers are formed that prevent heat removal due to their lower thermal conductivity than that of pure metal [28]. Therefore, the self-heating temperature for such systems is higher than for oils without additives [28]. On the other hand, LDH particles (Cu/Zn/Al) can contribute to an increase in the thermal conductivity of the lubricant composition as a whole [29], which leads to a more efficient heat removal from the friction zone.

Values of the welding load and wear spot are a obtained during the tribology testing procedures with different lubrication compositions are reported in table 2.

The most significant effect on the antiwear properties of lubricants is exerted by the LDH's of the (Zn/Al) and (Cu/Zn/Al) system, which may be associated with a presence of a well-developed crystalline layered structure of these substances. At the same time, the (Zn/Al) LDH system more intensively influences extreme pressure properties (welding load value); whereas, an introduction of Cu in this system (Cu/Zn/Al LDH) promotes maximum wear resistance, although it leads to slight increase in friction and some decline of a welding load value. This effect can be associated with the known fact of a stronger cladding effect of copper in comparison with zinc [30]. The best reduction of friction at low loads but, at the same time, low tribological efficiency at high loads, which were recognized for the compositions based on (Zn/Cr) and (Cu/Cr) LDH's, could be associated with a very low scale of particles forming these products. It is possible to propose that their particles with a size lower of 10-15 nm at low loads can easy penetrate into a friction zone and exhibit there a good lubricating effect: however, at high loads, a thin protective film, formed by nanoparticles may be easy destroyed [31].

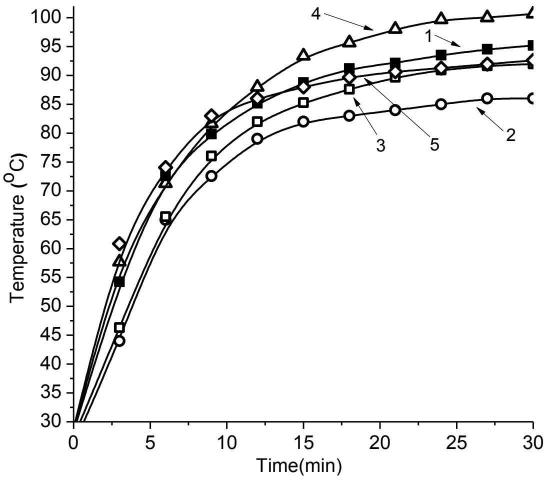

Fig. 4. Change in the temperature of lubricating compositions based on different LHD's at a load of 360 N. The systematic error in the temperature measurement is of ± 1 °C. (1 – base oil; 2 – LDH(Zn/Al); 3 – LDH(Cu/Cr); 4 – LDH(Cu/Zn/Al); 5 – LDH(Zn/Cr)

Рис. 4. Изменение температуры смазочных композиций на основе различных СДГ при нагрузке 360 Н. Систематическая погрешность измерения температуры составляет ± 1 ° С. (1 - базовое масло; 2 - СДГ(Zn / Al); 3 - СДГ(Cu / Cr); 4 - СДГ(Cu / Zn / Al); 5 - СДГ(Zn / Cr)

***Table 2***

**Influence of the nature of lubricating additive on tribology properties of the investigated LDH's based lubricating compositions**

***Таблица 2*. Влияние природы смазочных добавок на трибологические свойства исследуемых смазочных композиций на основе СДГ**

| Additivetype | Change of the friction coefficient. % | Change of the lubricating composition temperature. % | Welding load, N | Wear scar area, mm$^2$ |
|---|---|---|---|---|
| None | 100 | 100 | 580 | 11.8 |
| LDH (Zn/Cr) | -37 | -4.4 | 1260 | 10.6 |
| LDH(Zn/Al) | -32 | -9.7 | 1600 | 8.0 |
| LDH(Cu/Zn/Al) | -26 | +6.1 | 1440 | 6.2 |
| LDH(Cu/Cr) | -23 | -3.4 | 1050 | 8.3 |

Thus, the tribological properties of the investigated LDH's are determined mainly by their layered structure, where the LDH layers are linked by weak molecular bonds, which are easily broken under the action of a shear force, which allows them to easily slide relative to each other. During friction, the LDH particles are able to absorb on the contacting surfaces, thereby preventing direct contact of tribo-couplings and filling irregularities on the friction surface. As a result, the antiwear and loading properties of lubricants are increased. With a sufficient accumulation of energy on the friction surface, the adsorbed particles can form a protective film with improved micro hardness and smoothness.

Aluminum, transferred into the friction surfaces of steel, is known for its ability to form a strong oxide film, which leads to an increase in the hardness and strength of the friction surface, and so it also increases resistance to oxidation at high temperatures, which contributes to an increase in the welding load [32]. An introduction of copper into the composition of LDH can significantly improve the antiwear properties of this group of additives due to a well-known cladding effect. Copper-containing compounds are able to easily form protective films due to their high ductility and removal of the surface oxide layer due to abrasion of friction surfaces, preventing direct contact between surfaces and having low shear resistance, which subsequently leads to a decrease in friction and wear.

In any case, multicomponent LDH systems formed by nanoscale particles characterized by high mobility and chemical reactivity, ability to form thin film coating onto the steel surface as well as alloys in the surface layers of steel have to be considered as a very perspective kind of lubricating additives, first of all, due to a wide possibility to vary their chemical composition and tribochemical behavior promoting various synergetic effects.

## CONCLUSIONS

The obtained experimental results allow making the following main conclusions:

1) Layered double hydroxides (LDH's) can be considered as promising additives due to their complex influence on tribology behave or of lubricating compositions.

2) The chemical composition LDH's is a main factor which influences on tribology characteristics of lubricants based thereon: Zn promotes antifriction properties (reduced friction coefficient), Cu favors antiwear behave or (reduced wear scar area), Al provides increased antis cuff properties (enhanced welding load).

4) Nanoscale size, high mobility and chemical reactivity of the LDH particles, as well as ability to form thin film coating onto the steel surface and alloys in the surface layers of steel, support improved tribology characteristics of lubricating compositions.

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