This article deals with the new method for producing solid lubricating coatings on steel tools by thermo-hydrochemical treatment (THCT). The results of investigation of tribological properties of the coatings fabricated on high carbon steel of Y8 grade (0.8 wt.% C) using THCT method are presented. The composition of the medium and the temperature and the time parameters of THCT process were optimized by the friction coefficient of solid lubricating coatings. The diagrams of “parameters of process—property” were constructed using mathematical models. Processing by optimal THCT regime of Y8 steel in hydrosol medium based on TiO₂ - MoO₃ makes it possible under the condition of lubrication absence to decrease the friction coefficient of steel surface by 8.3 times as compared with untreated.

KEYWORDS: Thermo-hydrochemical treatment, solid lubricating coating, tool steel

1 INTRODUCTION

The problem of increasing the service life of steel tools, machining accessories, and machine parts remains relevant as before. There are various hardening methods of solving it. A lot of scientific researches are carried out on the development of wear-resistant ceramic coatings obtained by PVD (physical vapor deposition) methods, CVD (chemical vapor deposition), spraying, and TCHT (thermochemical heat treatment) in vacuum, which are activated by unconventional sources of heating (plasma, laser, electron beam, etc.). However, these hardening methods have a number of disadvantages, the main ones of which are (1) high temperatures of processes, which lead to deformation of products and weakening of the initial matrix; (2) low productivity and complexity of processes; (3) large labor intensiveness and energy capacity of processes; (4) environmental degradation and harmful energy effect on human health; (5) high cost of equipment and components to be applied; etc. [Hocking 1989], [Cavaleiro 2006], [Shmatov 2018]. So the process of thermo-hydrochemical treatment (THCT) is of the greatest scientific and practical interest, because it is distinguished by efficiency and high productivity, it makes it possible to fabricate coatings based on any ceramic materials, it is applicable to operation-ready products of various steels and alloys, and it slightly changes their initial sizes, shape, and structure [Shmatov 1998], [Shmatov 2014], [Shmatov 2016].

THCT is intended to chemically deposit on the surfaces of various materials antifriction solid lubricating coatings, which possess spare capacities in rugged and catastrophic operating conditions of tools and machinery [Shmatov 2014]. The coatings acquire solid lubricating properties in the following cases: (a) when they are fabricated of materials with layered polycrystalline structure (graphite, sulfides, etc.); (b) when they are formed on the basis of nanostructured refractory and ultrahard materials; (c) the Bernal theory is implemented, according to which any solid body acquires the properties of fluid if the crystal lattice contains more than 10% of vacancies; (d) the Rebinder effect is implemented, which leads to the plasticization of surface layer and creation of a positive gradient of mechanical properties in the friction zone; (e) the Kirkendall effect is implemented, which leads to selective dissolution of alloying elements from alloy owing to the differences in their electrochemical potentials, resulting in a quasi-liquid film being formed, which reduces friction coefficient and frictional heating [Polzer 1983].

THCT is the easiest and most universal method of fabricating solid lubricating coatings. Using the method, one can fabricate nanostructured coatings based on oxides, sulfides, carbides, diamond, carbon, and other antifriction materials [Shmatov 2014]. When fabricating such coatings, the Rebinder effect is implemented owing to introduction into an aqueous-dispersion medium of surface-active materials (SAM). The fabricated nanostructured coatings have superplasticity, and they facilitate the gap of adhesive joints in the friction zone; at the same time, the hardness of nanomaterials of metals and refractory compounds increases by 2–3 times [Cavaleiro 2006]. According to vacancy-diffusion and adhesion-deformation mechanisms of friction, increasing the wear resistance of solids is achieved by a combination of the above-mentioned effects: high hardness of its surface and low strength of adhesive bond [Polzer 1983].

Since in most cases as a result of THCT, the initial structure is preserved (is not weakened), and final dimensions and shape of products hardly change, these coatings can be deposited to ready-to-use tools and machine components. On the other hand, under the conditions of intense operation of products when there is no lubricant in the friction zone or its supply is limited, the best way to reduce the friction of working parts of products is depositing solid lubricating coatings on them [Vityaz’ 2007], [Shmatov 2016].

From the above presented analysis, it follows that the THCT process has great prospects for its development, especially for tools with a small operating life. In this regard, special attention should be paid to unalloyed tool steels (of Y8, Y10, Y12, etc., grades), which owing to their low cost are widely used to fabricate various types of stamps.

The mathematical methods of planning experiments can render great assistance to the researcher when there is a
labor-intensive choice of optimal variant of THCT process of steels and alloys. These methods make it possible to obtain a maximum of information with minimum costs. In materials science, traditionally direct problems are solved when, on the basis of the minimum number of experiments conducted under the preset temperature and time conditions (according to the plan of experiments), the properties of the material are defined; then the mathematical models describing the influence of factors are constructed and using graphical interpretation the optimal parameters of the process are chosen [Novik 1971]. However, such an approach can not solve the whole set of problems arising when designing technology, as the operation of any technological system (in this case, it is the technology of THCT of Y8 steel in hydrosol medium based on TiO$_2$ - MoO$_3$) occurs under the conditions of constant random change of values of the system parameters under the influence of various external and internal destabilizing factors. Technological systems themselves as the design objects have a number of specific properties: multicriteriality, multiparametricity, stochasticity (spread of parameters), presence of nonlinear intrasystem bonds, etc. When studying, designing, and developing such objects, it is necessary to solve not only direct problems and inverse ones, where the researcher specifies a set of necessary properties of the material in advance and using a computer simulation finds optimal time-temperature parameters of the process. This methodological approach, which is called the multidimensional designed synthesis of technological system, is successfully applied when developing new technologies and materials [Vityaz’ 1971], [Shmatov 2014], [Shmatov 2016].

In connection with the aforementioned, the objective of this work was optimization and computer-aided design of THCT technology for fabricating on Y8 steel a solid lubricating coating based on TiO$_2$ - MoO$_3$ owing to application of the method of multidimensional design synthesis of technical objects, materials, and technologies.

2 METHODS

Y8 steel, which is widely used in practice, has been subjected to thermo-hydrochemical treatment. The THCT process itself was carried out by conducting two of operations: (a) hydrochemical treatment (HCT) of steel surface at the temperature of 96–100°C for 10–420 min in a specially prepared aqueous suspension based on dispersed oxides TiO$_2$ and MoO$_3$; (b) subsequent thermal treatment (TT) under heating in an oxidative medium to the temperature of 130–1050°C, holding for 20–30 min, and cooling in air. The aqueous suspension was previously prepared by special technology upon mixing nano- and ultradimensional (0.1–1 μm) grains of oxides with 4–8% of sulfur (which was used as surface-active material — SAM). The working composition with acidity pH 6–8 was considered ready. The desired acidity was set and maintained by dosed introduction of NH$_4$OH. When conducting HCT, the specimens were placed and stored in a bath with the ready work composition, heated to the temperature of the process. The surface of specimens was previously degreased in white spirit and pickled in a 5–10% sulfuric acid solution for 1–2 min. After each HCT operation, the specimens were washed in water. Isothermal storage of steel at temperatures up to 200°C was carried out in air, and at temperatures higher than 200°C, the storage was carried out in a protective medium.

To determining the tribotechnical characteristics of solid lubricating thermo-hydrochemical coatings with a microtribometer (Fig. 1), specimens with size of 10 × 10 mm were used.

Figure 1. Reciprocating microtribometer with maximum applied load of 1 N (manufactured by MPRI, Gomel, Belarus): (1) electromagnets of drive; (2) guides of bend; (3) table holder of specimen; (4) position sensor; (5) triboacoustical emission sensor; (6) incremental drive; (7) electromagnet of loading system; (8) lever; (9) load sensor; (10) head; (11) balance weights; (12) optical coupler; (13) friction force sensor.

When designing the technological process of THCT of Y8 tool steel, we applied the synthesis technologies of the method of multidimensional design synthesis of technological objects, materials, and technologies in the form of basic computer software—SINTEZ MK software [Vityaz’ 1971]. The proposed method makes it possible to operate not only with the mathematical criteria but also with the technical criteria of optimality. To implement the procedures of multidimensional design synthesis of the technological system when carrying out the THCT of Y8 steel, a set of new methods was used, the most significant of which are the following: the method of solving the inverse multicriterial problem; the method of computer selection of the technically optimal variant; the method of allocation of stability domains of the studied technological system in the multidimensional space of technological parameters; and the method of constructing a graphical representation of states of the technological system.

The method of multidimensional design synthesis of the technological system, unlike the traditional optimization method [Novik 1971], makes it possible to do the following:

1) to select the technically optimal variant which possesses the greatest resistance to the impact of destabilizing factors of manufacture;

2) simultaneously to solve the inverse multicriterial problems: to allocate in a space of the system the stable domains and select the technically optimal variant of the technological system in one of the stable domains when ensuring the desired level of the reproducibility of material properties;

3) to select in the space of technological parameters the domain of a steady state of the system in which the specified properties of the materials are achieved and stably reproduced simultaneously.
3 RESEARCH RESULTS

To design a new THCT process, Y8 steel was subjected to hydrochemical treatment in hydrosol containing TiO₂ - MoO₃ when heating to a temperature close to the boiling point for 50–70 min, and tempering was carried out at a temperature of 160–200°C. Under such conditions at THCT of Y8 steel, solid lubricating coatings based on TiO₂ - MoO₃ with the best antifriction properties are formed (Fig. 2).

![Figure 2](image)

**Figure 2.** Influence of parameters of THCT process on the friction coefficient of Y8 steel. THCT conditions are hydrosol for HCT based on TiO₂ - MoO₃: (a) HCT at T = 100 °C without tempering; (b) tempering at TT r = 0.5 h after HCT at T = 100 °C, r = 2 h.

Test conditions are dry sliding friction (without lubrication); friction pair is hardened Y8 steel (plane) — W15X15 steel (sphere 4 mm in diameter); load is 1 N; stroke length (track) is 3 mm, speed is 4 mm/s. 1 — Y8 steel (before treatment); HCT conditions: 2 — 30 min; 3 — 30 min; 4 — 60 min; 5 — 120 min; 6 — 240 min; 7 — 420 min; TT conditions: 8 — 100 °C; 9 — 200 °C; 10 — 250 °C; 11 — 300 °C; 12 — 350 °C; 13 — 400 °C; 14 — 450 °C; 15 — 500 °C; 16 — 550 °C; 17 — 600 °C; 18 — 650 °C; 19 — 700 °C; 20 — 870 °C; 21 — 1000 °C; 22 — 1050 °C.

In optimization of the process, only the temperature and time of hydrochemical treatment, the fractional content of the basic component of the chemically active medium, and the tempering temperature were varied. In the present work, the volume fraction and the morphology of initial particles of TiO₂ and MoO₃ were not taken into account since they affect the tribological properties of fabricated coatings very little, which is related to modification of all structural parameters of particles during hydrochemical nanodispersion up to hydrosol formation. The results of tribotechnical tests of the thermo-hydrochemical hardened Y8 steel obtained upon implementation of 8 experiments of the test plan [Novik 1971] are presented in Tab. 1.

**Table 1.** The results of investigation of the friction coefficient of coatings on Y8 steel fabricated with THCT in hydrosol based on TiO₂ - MoO₃

| Experiment number | Designation | Factors                                                                 |
|-------------------|-------------|-------------------------------------------------------------------------|
|                   |             | Hydrochemical treatment | Thermal treatment | Parameter of optimization |
|                   |             | Temperature, °C | Time, min | MoO₃ in oxides, % | Temperature, °C | Number of cycles, f |
| 1                 |             | 98 | 60 | 50 | 180 | 0.068 |
| 2                 |             | 2 | 10 | 10 | 60 | 0.072 |
| 3                 |             | 98 | 60 | 50 | 160 | 0.076 |
| 4                 |             | - | - | + | - | 0.070 |
| 5                 |             | + | + | - | + | 0.069 |
| 6                 |             | - | + | - | - | 0.075 |
| 7                 |             | + | - | - | - | 0.082 |
| 8                 |             | - | - | - | + | 0.073 |

On the basis of these data, the linear and nonlinear mathematical models describing the influence of temperature and time parameters and composition of active mixtures on the friction coefficient of THC of solid lubricating coatings based on TiO₂ - MoO₃ were calculated. However only the nonlinear multicriterial mathematical models of following form were recognized as adequate:

\[
Y₁ = 73,13 ∙ 10^{-3} - 0,75 ∙ 10^{-3} X₁ - 1,75 ∙ 10^{-3} X₂ - 3,0 ∙ 10^{-3} X₃ - 4,5 ∙ 10^{-3} X₄ + 7,5 ∙ 10^{-4} X₁X₃ - 7, 5 ∙ 10^{-4} X₂X₄ + 6,25 ∙ 10^{-3} X₃X₄ ,
\]

where \( Y₁ \) is the friction coefficient, \( X₁ \) is the temperature of hydrochemical treatment, \( X₂ \) is the time of hydrochemical treatment, \( X₃ \) is the MoO₃ percentage in oxides TiO₂ - MoO₃ mixture of hydrosol, and \( X₄ \) is the tempering temperature.

Owing to multicritieriality, stochasticity, and nonlinearity of “the THCT process of Y8 steel” technological system, the prediction of its behavior is complicated. The values of parameters of each actual object differ from the designed ones and are randomly distributed in a scatter band. As a consequence, there is no guarantee that all points of optimization of the actual system will be placed in the stable domain, i.e., it is not always possible to improve the material properties up to the desired its level. So that this does not happen, when designing the technological process, a certain system stability margin is provided, which makes it possible to avoid a degradation of the quality of system functioning by the criterion of reproducibility of material properties.

Under the traditional methodology of optimization of system parameters, the solutions of the problems of technological designing of the system are not entirely correct, as the processes are considered as deterministic ones, i.e., passing under adherence of precise values of technological system.
parameters. In fact, deterministic systems do not exist, as the values of parameters of actual technology systems always are random and the systems themselves are stochastic. The selection of a technically optimal variant of THCT of Y8 steel in hydrosol of TiO₂·MoO₃ was carried out with the method of computer-aided design of technology systems with the help of the specially developed SINTEZ MK software program, which is intended to solving nonlinear and stochastic tasks for providing for whole working capacity of technological systems.

The computer-aided design of THCT technology of Y8 steel using SINTEZ MK software was carried out in several stages. The results of the selection of the optimum variant of the studied technological systems are summarized in Tab. 2 and 3. While solving the inverse multicriterial problem, the desired limit values of properties of solid lubricating coatings fabricated with THCT of Y8 steel were set (Tab. 4). The manufacturing tolerance margins of technological system by input parameters are presented in Tab. 5. For graphical interpretation of the results obtained when solving the tasks of the research and design of THCT technology of the Y8 steel in hydrosol based on TiO₂·MoO₃, discrete portraits were constructed (Fig. 3).

| Name of process parameter | Rated value | Permissible scatter bands |
|---------------------------|-------------|--------------------------|
| Temperature of hydrochemical treatment, °C | 100 | 4 |
| Time of hydrochemical treatment, min | 67 | 9 |
| MoO₃ percentage in oxides mixture, % | 57 | 8 |
| Tempering temperature, °C | 189 | 22 |

Table 2. Optimal parameters of the THCT process of Y8 steel in hydrosol based TiO₂·MoO₃ and their scatter bands

| Name of optimal index of properties | Rated value | Scatter band |
|------------------------------------|-------------|--------------|
| Friction coefficient, f | 0.067 | 0.013 |

Table 3. Optimal indices of properties of solid lubricating coatings fabricated with THCT of Y8 steel in hydrosol based on TiO₂·MoO₃ and their scatter bands

| Name of desired index of properties | Minimum value | Maximum value |
|------------------------------------|---------------|---------------|
| Friction coefficient, f | 0.060 | 0.075 |

Table 4. Set limits of properties of solid lubricating coatings fabricated with THCT of Y8 steel in hydrosol based on TiO₂·MoO₃

Figure 3. Discrete portraits of virtual space of technological system of THCT process of Y8 steel in hydrosol based on TiO₂·MoO₃:
- – variant of the system providing the set properties of Y8 steel; – variant of the system not providing the set properties of Y8 steel.
The allocation of stable domains in a multidimensional space of states is an important stage in the selection of a technically optimal variant of the system. The friction coefficient with desired level of 0.060–0.075 is used as the optimization criterion (see Tab. 4). It is seen from Fig. 3 that the THCT technology of the Y8 steel is implemented with high properties only when the system gets into the stable domain marked with dark dots. Overrunning of one or more parameters of the process beyond the stable domains into the domains marked with light dots is evidence that the material with the set properties in this case will not be fabricated.

As a result of solution of the problem of design of THCT technology of the Y8 steel in the hydrosol based on TiO₂ MoO₃, the actual indices of its main tribotechnical property (Tab. 6), 100% reproducibility of which is achieved upon exact compliance of the process parameters within manufacturing tolerances (see Tab. 5), were stated. The results of tests confirmed this too (Fig. 4).

For any tool of which the place of contact with a machinable workpiece changes over time [Bel’skii 1984], it is important to have minimum and invariable values of the friction coefficient during the whole operating period. The thermo-hydrochemical coatings correspond to these demands; and this is their advantages against PVD diamond coating [Vityaz’ 2007].

### 4 APPLICATION OF RESEARCH RESULTS

The manufacturing testing results revealed that the THCT using aqueous-dispersion oxide and carbon compositions makes it possible to increase the resistance of various types of steel tools by 1.3–8 times as compared with an unprocessed tool (Tab. 7).

### 5 CONCLUSIONS

(1) The mathematical simulation and the computer-aided design of the THCT process of Y8 steel in a hydrosol medium of TiO₂ MoO₃ using computer technologies of the method of multidimensional design synthesis of technical objects, materials, and technologies were implemented. The technically optimal regimes for implementing the process in manufacture with guaranteed achievement of the set properties of steel products subjected to THCT were determined.

(2) The treatment of Y8 tool steel by the optimal THCT
regime makes it possible to considerably (by 8.3 times) reduce the friction coefficient of the steel surface under sliding conditions without lubrication.

(3) The simple and economical method of thermo-hydrochemical treatment of tool steels has been developed, the use of which makes it possible to increase the operational resistance of different types of steel tools by 1.3–8 times in comparison with the traditional used method.

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CONTACTS:
Dr. Alexander Shmatov, docent, Ph.D.
Belarusian National Technical University
Faculty of Mechanical Engineering
Nezavisimosti ave., 65, 220013 Minsk, Belarus
Tel.: +375 29 3777 357, +375 44 777 9213
e-mail: shmatovalexander@gmail.com