Development of new flux cored wires based on the tungsten oxide for improvement of drill bits wear resistance

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Abstract. A new flux cored wire was developed for wear-resistant surfacing of machine parts in the mining and metallurgical industry, in which tungsten oxide WO₃ and substances containing reducing agents: carbon and silicon are used as fillers.

1. Introduction
Rational use of tungsten in the production of steel and alloys is one of the problems of today. The technology of the so-called direct reduction of tungsten from oxide raw materials, excluding the process of obtaining reduced tungsten and its derivatives, is of interest for practical application. Such a process is rationally organized at the arc discharge in the process of surfacing with flux cored wire, in which tungsten oxide and various reductants are used as a filler.

Tungsten flux cored wires have been widely used for surfacing steels with the highest wear resistance, in which reduced tungsten in the form of ferroalloys, alloying elements and metal powder of various degrees of purity are used as fillers. In addition, silicides and tungsten carbides can be used to impart certain performance properties. Therefore, the possibility of replacing reduced tungsten with oxide tungsten-containing raw material at least reduces the cost of the flux cored wire.

2. Methods of research
The chemical composition of the deposited metal was determined by the X-ray fluorescence method on XRF-1800 spectrometer and the atomic emission method on DFS-71 spectrometer. A metallographic study of microsections was carried out using OLYMPUS GX-51 optical microscope in a bright field at various magnifications. Grinding of samples was carried out according to GOST (State Standards) 23505-79. Samples were polished using a FORCIPOL2 grinding machine with sandpaper and a felt coating treated with a chrome-containing 2% solution. The etching technique was carried out according to GOST 25336-82. Etching was performed in a 2% solution of nitric acid (HNO₃). Etching time 0.5 min. Washing in cold running water. Grain score (grain size) is determined according to the scale of GOST 5639-82. Evaluation of non-metallic inclusions was made according to GOST 1778-70.

Surfacing was performed using flux produced from slag of manganese silicate production of the following chemical composition, wt.%: Al₂O₃ 6.91-9.62%, CaO 22.85-31.70 %, SiO₂ 46.46-48.16 %, FeO 0.27-0.81 %, MgO 6.48-7.92 %, MnO 8.01-8.43% , F 0.28-0.76%, Na₂O 0.26-0.36%, K₂O up to 0.62%, S 0.15-0.17 %, P 0.01 %. The ASAW 1250 tractor was used for surfacing. The surfacing modes were I = 430-490 A, U = 30-32 V, V = 26-28 m/h. The number of deposited layers is 5.

3. Results and discussion
The comparative thermodynamic evaluation of the reduction processes of tungsten oxide $WO_3$ with carbon and silicon in the arc discharge during surfacing with flux cored wire indicates the prospectivity of direct reduction.

The thermodynamic estimation of the probability of the following reactions was carried out:

\[
\begin{align*}
2/3WO_3 + 2C &= 2/3W + 2CO; \\
2/3WO_3 + C &= 2/3W + CO_2; \\
1/3WO_3 + CO &= 1/3W + CO_2; \\
2/3W + 2/3C &= 2/3WC; \\
2/3W + 1/3C &= 1/3W_2C; \\
2/3WO_3 + 5/3C &= 2/3WC + CO_2; \\
2/3WO_3 + 4/3C &= 1/3W_2C + CO_2; \\
1/4WO_3 + 5/4CO &= 1/4WC + CO_2; \\
2/7WO_3 + 8/7CO &= 1/7W_2C + CO_2; \\
2/3WO_3 + 8/3C &= 2/3WC + 2CO; \\
2/3WO_3 + 7/3C &= 1/3W_2C + 2CO; \\
2/3WO_3 + Si &= SiO_2 + 2/3W; \\
2/3WO_3 + 7/3Si &= SiO_2 + 2/3WSi_2; \\
2/3WO_3 + 21/15Si &= SiO_2 + 2/15W_5Si_3; \\
2/3W + 4/3Si &= 2/3Si_2; \\
2/3W + 2/5Si &= 2/15W_5Si_3; \\
2/3WO_3(s,f) + 2Si &= 2SiO + 2/3W; \\
2/3WO_3 + 10/3Si &= 2SiO + 2/3WSi_2; \\
2/3WO_3 + 36/15Si &= 2SiO + 2/15W_5Si_3; \\
WO_3(vap) + 3C &= W + 3CO; \\
2/3WO_3(g) + 2Si &= 2SiO + 2/3W.
\end{align*}
\] (1) (2) (3) (4) (5) (6) (7) (8) (9) (10) (11) (12) (13) (14) (15) (16) (17) (17a)

According to the nature of the chemical interaction between the components, all reactions can be divided into three types: (3), (8), (9) – reactions of indirect reduction of tungsten oxide $WO_3$ with carbon; (4), (5) and (15), (16) – reactions of the compound of tungsten with carbon and with silicon; the rest – direct reduction reactions of $WO_3$ with carbon and silicon. The oxide reduction reactions were recorded for 1 mol of oxygen $O_2$, and the reactions of tungsten compounds with carbon and silicon were recorded for 2/3 mol of tungsten W. The probability of reactions (1) – (19) was estimated using the standard Gibbs energy of reactions.

The necessary for the estimation of the oxygen and silicon reduction properties with respect to tungsten oxide $WO_3$ thermodynamic characteristics of reactions (1) – (19) in the standard conditions $\Delta_r H^\circ(T)$, $\Delta_r S^\circ(T)$, and then $\Delta_r G^\circ(T)$ were calculated by the known methods [1] in the temperature range 1500 – 3500 K according to the thermodynamic properties $[H^\circ(T), S^\circ(T), \Delta_r H^\circ(298.15 K)]$ reagents $WO_3$, $W$, $C$, $CO$, $CO_2$, $Si$, $SiO$, $SiO_2$ [2] and $WSi_2$, $W_5Si_3$ [3,4].

Thermodynamic properties of tungsten carbides WC and $W_2C$ depending on temperature in the form of tables adopted in [2, 3] were calculated additionally using the necessary initial data from reference books [4, 5]. The following states were selected as standard for reactant substances in the range of 1500–3500 K: $W$ (TV), $WO_3$ (TV, W) with a phase transition at 1745 K, $WC$ (TV), $C$ (TV), $CO$ (g), $CO_2$ (g), $WSi_2$ (TV, W) with a phase transition at 2433 K, $W_5Si_3$ (TV, W) with a phase transition at 2623 K, $Si$ (TV, W) with a phase transition at 1690 K, $SiO$ (g) $SiO_2$ (s, g) with a phase transition at 1996 K.

To assess the degree of influence on the thermodynamic properties of reactions of possible evaporation of tungsten oxide $WO_3$ in the arc, the thermodynamic characteristics of two reactions were calculated, in which the standard state of $WO_3(g)$ was chosen as the standard for the entire temperature range for tungsten oxide:
2/3WO₃(g) + 2C = 2/3W + 2CO;  \quad (1a)
2/3WO₃(g) + 2Si = 2/3W + 2SiO. \quad (17a)

The chemical composition and melting points of tungsten – silicon compounds were determined from the melting diagram of the W – Si system according to the data [6-8].

The standard Gibbs energies of the considered reactions (1) – (19) are given in Table 1 and in figure 1. Starting with a temperature of 2500 K and above, the graphs are grouped into three sectors. The first sector is the most likely reactions (11), (10), (1a), (1), (18), (17a), (19), (17) (in the order of decrease in their occurrence probability). These are reactions of direct reduction of WO₃ by carbon to metal and carbides with the formation of CO gas, reactions of direct reduction of WO₃ with silicon to metal and CO₂ according to carbon gasification reaction

\[ \frac{2}{3} \text{W}O_3 + \text{C} = \text{W} + \frac{2}{3} \text{CO} + \text{CO}_2 \]

The second sector consists of graphs \( \Delta_r G^\circ = f(T) \) of reactions with the average probability of occurrence (13), (6), (7), (2), (14), (15), (12). These are reactions of direct reduction of WO₃ by carbon and silicon with the formation of tungsten, carbides and tungsten silicides and higher oxides of reducing agents (CO₂, SiO₂) and reaction (15) of the formation of a silicide WSi₂ with a compound of tungsten and silicon. The third sector is the unlikely reactions (5), (4), (16), (3), (9), (8). These are the reactions of tungsten compounds with silicon and carbon with the formation of the known carbides of tungsten and silicide W₅Si₃, as well as the reaction of indirect reduction of WO₃ by carbon (II) oxide.

It can be noted that in the case of estimating the thermodynamic probability of reactions (3), (9), (8), indirect reduction of tungsten oxide with carbon, using the Gibbs energy of reactions \( \Delta_r G \), calculated from the Van’t Hoff isotherm equation taking into account the equilibrium partial pressures of CO and CO₂ according to carbon gasification reaction \( C + CO_2 = 2CO \), graphs \( \Delta_r G = f(T) \) of these reactions move to the second sector.

**Table 1. Standard Gibbs energies of reactions (1) – (19) depending on temperature.**

| Reaction | 1500 | 2000 | 2500 | 3000 | 3500 |
|----------|------|------|------|------|------|
| 1        | -172.230 | -326.830 | -469.260 | -607.390 | -742.270 |
| 1a       | -347.580 | -449.500 | -545.970 | -644.870 | -746.810 |
| 2        | -81.040 | -151.090 | -210.610 | -267.200 | -321.740 |
| 3        | 5.080 | 12.320 | 24.020 | 36.490 | 49.400 |
| 4        | -31.840 | -36.647 | -42.38 | -48.893 | -54.900 |
| 5        | -23.407 | -31.847 | -41.207 | -51.247 | -59.160 |
| 6        | -112.880 | -187.740 | -252.990 | -316.100 | -392.500 |
| 7        | -104.450 | -182.940 | -251.810 | -318.450 | -397.050 |
| 8        | 14.660 | 39.430 | 66.790 | 94.080 | -119.250 |
| 9        | 7.350 | 22.020 | 39.880 | 57.920 | -84.900 |
| 10       | -204.070 | -363.480 | -511.640 | -656.290 | -792.810 |
| 11       | -195.640 | -358.680 | -510.460 | -658.640 | -801.000 |
| 12       | -328.433 | -305.668 | -270.556 | -234.836 | -198.978 |
| 13       | -435.490 | -414.301 | -377.616 | -509.980 | -632.230 |
| 14       | -361.281 | -337.117 | -298.786 | -490.170 | -593.210 |
| 15       | -107.057 | -108.633 | -107.060 | -127.994 | -149.732 |
| 16       | -32.848 | -31.449 | -28.230 | -57.070 | -80.370 |
| 17       | -139.948 | -268.310 | -373.169 | -473.615 | -570.770 |
| 17a      | -315.291 | -392.631 | -457.241 | -520.749 | -583.703 |
| 18       | -247.005 | -376.943 | -480.229 | -601.610 | -720.502 |
| 19       | -172.796 | -299.759 | -401.399 | -505.911 | -609.540 |
Figure 1. Standard Gibbs energies of reactions (1) - (19) depending on temperature.

With a decrease in temperature, the temperature dependences $\Delta_rG^\circ = f(T)$ are combined into one region, in which a significant change in their relative location is observed. For example, at $T < 1500$ K, reactions (13), (14), (12) of WO$_3$ reduction by silicon with formation of a higher oxide of reducing agent SiO$_2$ become most probable.
To improve the quality of thermodynamic evaluation, it should be taken into account that tungsten produced according to reactions (1), (1a), (2), (3), (17), (17a), and tungsten as an initial material for reactions (4), (5), (15), (16), apparently, will be in the state of the dissolved component of the surfacing bath at temperature T. Thermodynamic assessment of the effect of such state on the properties of the reaction is produced according to the Van’t Hoff isotherm equation:

$$\Delta_r G = \Delta_r G^\circ \pm n R T \ln a_{[W]}$$  \hspace{1cm} (20)$$

where $a_{[W]}$ – the tungsten activity in the weld metal solution with respect to the standard state “pure solid tungsten at the melt temperature T”; $n$ is the stoichiometric coefficient before tungsten in the reaction equation. The “+” sign corresponds to reactions in which tungsten is a product and the sign “−” corresponds to reactions in which tungsten is the initial material.

To assess the trend, the second summand in equation (20) was calculated with $n = 2/3$ and a constant temperature of 2500 K (average temperature of the interval), which determines the degree of influence at various probable activities (table 1). The assessment of the trend indicates that the dissolution of tungsten increases the thermodynamic probability of reactions occurring with its formation: $\Delta_r G$ becomes more negative than $\Delta_r G^\circ$. And, on the contrary, it will further lower the probability of reactions (4), (5), (15), (16). To a greater degree, the change in $\Delta_r G$ compared to $\Delta_r G^\circ$ occurs at the minimum values of tungsten activity ($\approx \pm 96$ kJ).

At the same time, it can be stated that the analysis of the graphs in the figure shows that taking into account the dissolution of tungsten in the melt does not have a significant effect on their relative position. Each of the reactions will remain in its sector, as described above. Only reaction (2) is close to the border of the sector of the most likely reactions. Thus, we can conclude that the temperature of the melt and the arc during the surfacing, along with the composition of the flux cored wire, are factors that can affect the composition and, consequently, the service properties of the weld layer. In the system under consideration, at high (> 2500 K) melt temperatures the formation of tungsten [reactions (1), (1a), (17), (17a)], tungsten silicides [reactions (18), (19)], tungsten carbides [reactions (10), (11)] is probable. The course of reactions significantly changes the composition of the gas phase, but not the slag phase. At temperatures below 1500 K, the formation of tungsten silicides [reactions (13), (14)] and tungsten [reaction (12)] is most likely due to the reduction of WO$_3$ by silicon. In this case, the slag phase becomes more acidic due to the resulting SiO$_2$. However, it should be noted that this temperature range is below the melting point of WO$_3$ (1745 K). In the temperature range of 1500–2500 K (the shaded zone in the figure), a number of competing reactions occur, leading to the formation of both tungsten and its silicides and carbides in the metal melt.

The conducted thermodynamic analysis shows that the presence of carbon and silicon reducing agents in the flux cored wire used for surfacing, along with tungsten oxide WO$_3$, will necessarily lead to the formation of silicides and tungsten carbides, and possibly tungsten itself. Carbides can not be formed only at low (less than 1500 K) temperatures. During the surfacing process at low temperatures, an increase in the acidity of the slag phase is probable [9]. On the basis of the performed calculations, laboratory studies of new flux cored wire were carried out. As a reduced material, tungsten concentrate of grade KSh-4 was used according to GOST (State Standard) 213-83, produced by JSC AIR Mining Company with a content of 54% WO$_3$. Carbon-fluorine-containing dust from gas cleaning plant of aluminum production was used as a carbonaceous reducing agent, wt. %: Al$_2$O$_3$ = 21 – 43.27; F = 18–27; Na$_2$O = 8–13; K$_2$O = 0.4 – 6%; CaO = 0.7– 2.1; SiO$_2$ = 0.5–2.48; Fe$_2$O$_3$ = 2.1–2.3; C$_{tot}$ = 12.5–28.2, MnO = 0.03–0.9, MgO = 0.04–0.9, S = 0.09–0.46, P = 0.1–0.18.

The choice of this material was made on the basis of the results of previous studies, in which [10] it was shown that these carbon-fluoride powdered metallurgical production wastes makes it possible: 1) to remove hydrogen using fluorine-containing compounds (type Na$_3$AlF$_6$, CF$_x$ (1 ≥ x > 0) etc.) to form the gaseous compound HF; 2) to deoxidize the weld pool with carbon due to CO and CO$_2$, formed by the interaction of carbon fluoride CF$_x$ (1 ≥ x > 0) with oxygen dissolved in steel; 3) to increase the stability of the arc due to the elements that facilitate the ionization in the arc column –
potassium and sodium. Silicon powder KR-1 according to GOST 2169-69 Si ≥ 98% was used as a silicon reducing agent-filler of the flux cored wire.

Table 2 shows the investigated compositions of the charge of the flux cored wires with the use of carbon-fluorine-containing dust from gas cleaning aluminum production, as well as the fill factors of the manufactured wires. The selected ratio is based on stoichiometric calculations, taking into account the varying degrees of reduction by carbon and silicon.

| Table 2. Characteristics of flux-cored wires. |
|------------------------------------------------|
| Wire No. | Component, wt. % | Wire fill factor, % | Tungsten extraction rate, % | HRC |
|----------|----------------|-------------------|-----------------------------|-----|
| 11       | 75.76          | 24.24             | -                           | -   |
| 111      | 48.54          | 51.46             | -                           | -   |
| 112      | 31.85          | 68.15             | -                           | -   |
| 331      | 66.67          | 33.33             | -                           | -   |
| 441      | 25             | 75                | -                           | -   |
| 551      | -              | 50                | 50                          | -   |
| 661      | -              | 50                | -                           | 50  |
| 2        | 90.65          | 9.34              | 16.8                        | 100.0 |

1 – WO₃ concentrate, 2 – carbon-fluorine-containing dust-like wastes of metallurgical production, 3 – tungsten ore, 4 – “tails” after the enrichment of tungsten ores, 5 – KR-1 silicon powder.

The ratio in the wires is selected during carbon reduction: samples 551, 661 – by stoichiometric calculation for ore and “tails” after enrichment, samples: 11, 111, 112, 331, 441 – for concentrate with different ratio of carbon.

The chemical composition of the weld metal is given in table 3, the chemical composition of the slag obtained after the surfacing operation is shown in table 4.

| Table 3. The chemical composition of the weld metal. |
|---------------------------------------------------|
| No. | C | Si | Mn | Cr | Ni | Cu | Ti | W | V | Mo | Co |
|-----|---|----|----|----|----|----|----|---|---|----|----|
| 11  | 0.05 | 0.02       | 0.16 | 0.04 | 0.03 | 0.08 | 0.001 | 1.04 | 0.001 | 0.002 | 0.006 |
| 111 | 0.29 | 0.25       | 0.50 | 0.07 | 0.12 | 0.12 | 0.001 | 1.29 | 0.002 | 0.006 |
| 112 | 0.35 | 0.31       | 0.49 | 0.07 | 0.12 | 0.13 | 0.001 | 0.67 | 0.008 | 0.005 | 0.007 |
| 331 | 0.13 | 0.15       | 0.43 | 0.06 | 0.12 | 0.11 | 0.001 | 1.90 | 0.01 | 0.03 | 0.007 |
| 441 | 0.26 | 0.31       | 0.43 | 0.06 | 0.12 | 0.10 | 0.002 | 0.58 | 0.01 | 0.03 | 0.006 |
| 551 | 0.11 | 0.34       | 0.38 | 0.05 | 0.12 | 0.09 | 0.001 | 0.02 | 0.01 | 0.03 | 0.005 |
| 661 | 0.23 | 0.46       | 0.33 | 0.05 | 0.12 | 0.08 | 0.001 | 0.01 | 0.01 | 0.02 | 0.005 |
| 2   | 0.05 | 0.19       | 0.36 | 0.02 | 0.06 | 0.08 | 0.001 | 5.47 | 0.01 | 0.02 | 0.003 |

| Table 4. The chemical composition of the slag. |
|---------------------------------------------|
| No. | FeO | MnO | CaO | Al₂O₃ | SiO₂ | MgO | Na₂O | K₂O | F | C | S | P | WO₃ |
|-----|-----|-----|-----|-------|------|-----|------|------|---|---|---|---|-----|
| 111 | 2.82 | 3.64 | 14.24 | 28.40 | 18.86 | 18.29 | 0.62 | 0.09 | 11.37 | <0.1 | 0.047 | 0.018 | 0 |
| 112 | 2.97 | 3.41 | 14.48 | 28.95 | 19.42 | 18.92 | 0.63 | 0 | 11.86 | <0.1 | 0.048 | 0.017 | 0 |
| 331 | 3.39 | 4.03 | 13.63 | 27.44 | 18.11 | 17.80 | 0.67 | 0.01 | 11.71 | <0.12 | 0.05 | 0.02 | 0.07 |
| 441 | 2.76 | 3.31 | 14.04 | 27.33 | 18.26 | 17.92 | 0.65 | 0 | 11.67 | <0.12 | 0.05 | 0.02 | 0 |
| 551 | 2.22 | 3.00 | 13.77 | 26.90 | 16.87 | 16.46 | 0.52 | 0.04 | 10.39 | <0.12 | 0.03 | 0.02 | 0.001 |
| 661 | 3.02 | 2.87 | 13.02 | 31.21 | 19.30 | 17.04 | 1.11 | 0.20 | 11.67 | <0.12 | 0.07 | 0.02 | 0.001 |

Metallographic analysis of the weld metal revealed the following (figure 2): The structure of sample No. 11 is pearlite-ferritic (lamellar pearlite), the grain size is 3. There are inclusions of the type of globular oxides and single globular oxides (groups D and DS, respectively). These nonmetallic
inclusions are point (3-4 points) and line oxides (1 point). Sample 111 has a pronounced Widmannstatt structure with fine needles extending from the ferritic network located along the boundaries of the original austenitic grains. Grain size corresponds to 3 – 4 points.

Sample 112 has a Widmannstatt structure with fine needles extending from a ferritic network located along the boundaries of the original austenitic grains. Grain size corresponds to 4 points. Sample 331 – granular ferritic-carbide structure. The sizes of carbides are in the range of 1-5 μm. Sample 441 is a type of widmanstatten structure with a significant number of fine needles inside the grains. Grain size corresponds to 3 points. Sample 551 is a structure consisting mainly of ferrite with small areas of pearlite. Sample 661 has a Widmannstatt structure with massive needles and a thick ferritic network. Grain size corresponds to 3 points.

**Figure 2.** Microstructure of the weld metal.
Thus, studies showed the fundamental possibility for tungsten oxides reduction from ores by the carbon-containing reducing agent and tungsten carbides production in the deposited layer with sizes ranging from 1 to 5 μm.

With the introduction of silicon in the composition of the wire (sample 2) and the reduction by silicon, the following results were obtained: the grain size is 3-2. In the sample inclusions of globular oxides type and single globular oxides (groups D and DS, respectively) are observed. These non-metallic inclusions are point (4-5 points) and line oxides (1 point). The structure of this sample after etching is a pearlite-ferrite one (lamellar pearlite). Further studies were continued in the direction of replacing the reduced tungsten-containing components in the cored wires with tungsten-containing oxide concentrates. In the laboratory flux cored wires were produced (tables 5.6), and wire 721 was produced from dust obtained at gas cleaning plant of aluminum production and powders: KR-1 silicon according to GOST 2169-69, MR-0 manganese according to GOST 6008-82, PKhA-1M chromium according to TU 14-1-1474-75, VEL-1 vanadium according to TU 48-0533-71, PNK-115 nickel according to GOST 9722-97, PAP-1 aluminum according to GOST 5494-95, PVT tungsten according to TU 48-19-72-92, iron powder PZhV-1 according to GOST 9849-86; in wire 722 tungsten concentrate of grade KSh-4 was used instead of tungsten powder according to GOST 213-83 produced by OJSC Mining Company “AIR”.

| Wire No. | Wire fill factor, % | Tungsten extraction rate, % | HRC |
|---------|-------------------|----------------------------|-----|
| 721     | 19.307            | 72.24                      | 42  |
| 722     | 13.568            | 97.93                      | 42  |

The metallographic analysis of samples 721 and 722 revealed the following (figure 3):

In sample No. 721, non-deformable silicates are found which are mainly of points 1b, 2b, 2a rarely; point oxides are of 1a points. In sample No. 722, non-deformable silicates are found that are mainly of points 2b, 1b, 2a rarely; point oxides are of 1a points. The structure of sample No. 721 is a large-needle martensite (point 8) and residual austenite. The grain size of austenite on a scale of grain is No. 5. The structure of sample No. 722 is a large-needle martensite (point 7, 8) and residual austenite. The grain size of austenite on a grain scale is No. 5, 6.

| No. | Mass fraction of elements, % |
|-----|------------------------------|
|     | C    | Si    | Mn    | Cr    | Ni    | Cu    | Ti    | W    | V    | Mo   | Al   |
| 721 | 0.10 | 0.69  | 0.67  | 5.14  | 0.25  | 0.06  | 0.007 | 3.41 | 0.17 | 0.01 | 0.041 |
| 722 | 0.09 | 0.39  | 0.55  | 3.99  | 0.21  | 0.07  | 0.002 | 3.32 | 0.09 | -    | 0.021 |

The metallographic analysis of samples 721 and 722 revealed the following (figure 3):
4. Conclusions

The performed thermodynamic calculations and laboratory studies formed the basis of the developed flux cored wires from the charge mixture using tungsten oxide, designed for wear-resistant surfacing of machine parts in the coal mining and metallurgical industry, in particular drill bits. The charge composition of the flux cored wires is protected by the patents of the Russian Federation [11, 12]. The regression model of the effect of temperature on the specific electrical resistivity of rail steel E76KhF was build. Application of this regression model will help to optimize the technology of flash butt welding of rails.

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