Analysis of physical and chemical transformations during thermal spraying of coatings based on carbides of tungsten and chromium

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Abstract. The formation of structure and properties of thermal coatings is largely determined by the transformation intensity of sprayed materials at all stages of obtaining of coating. It is shown that during D – gun spraying, in order to ensure optimal conditions for leading transformations and phenomena that have a decisive influence on structure and properties of sprayed coatings, composition of combustible mixture, geometry and dimensions of barrel, conditions for introducing powder into the barrel and a unit dose of powder, spraying distance, timing of spraying cycle and other technological methods can be used. The possibility of transformation in powders of tungsten carbide and chromium under different methods of thermal spraying is analyzed and the structure and properties of coatings are obtained. The influence of the conditions of D – gun spraying on spraying distances, gas flow rates, type of powder, etc.; phase composition and properties of coatings is studied. The basic physicochemical transformations that accompany D – gun spraying of coatings of alloy WC – C, such as oxidation and reduction of tungsten carbide in high – temperature oxygen – containing media, interaction of composite coating components are considered problems of modelling of bonding of particles with a part surface during thermal spraying of coatings are considered.

1. Introduction

To improve the reliability and durability of elements and units of machines and mechanisms [1-3], it is reasonable to apply thermal spraying of coatings [4-6]. Due to the use of protective coatings it is possible to solve a number of problems that allow to reduce material and energy consumption of production and operation of machines, to increase their durability, develop and introduce new types of modern machinery [7-9]. Therefore, it is relevant to use protective coatings to increase the strength and reliability of parts and structures of railway rolling stock [10-12].

The thermal spraying of coating encompasses a group of technologies [13-15] and equipment [16-18] that provide heating and acceleration of dispersed particles of material by high – temperature streams of gas and subsequent collision of particles with surface of sprayed part. A significant advantage of thermal spraying method is the ability to control the composition, structure and, accordingly, properties of obtained materials and coatings, including through the use of various powder compositions [19-21]. However, technological systems of thermal spraying are quite complex [22-24], the process of obtaining of coating is accompanied by various effects on particles of initial material of coating as a result of interaction of particles with environment gas, the processes of
interaction of components of composite powders when heated, as well as the interaction of particles with part surface of and between themselves and the subsequent cooling [25–27].

Therefore, obtaining of coatings with high performance properties requires an understanding of the transformation processes with the material that sprayed by thermal spraying technology. Carbides of tungsten and chromium are used as a major component of many thermal sprayed coatings. Regularities and tendencies of change of structure of coatings $WC - Co$ and others, depending on complex of technological parameters of the thermal spraying technology have not been sufficiently studied. Under the action of high temperatures, oxidizing environment and shock interaction of particles of $WC$ with part surface, the transition of $WC \rightarrow W_C \rightarrow W$ is carried out. Intermetallides and double tungsten carbides are also found in coatings [28–30].

The objective of this paper is to make a comparative analysis of possible transformations in coatings based on carbides of tungsten and chromium by various methods of thermal spraying, as well as to study transformations occurring during thermal spraying of such materials.

2. Physico–chemical transformations during thermal spraying of coatings based on carbides of tungsten and chromium

The most widespread thermal spraying methods include gas flame, plasma and high speed methods.

At gas – flame spraying, acetylene – oxygen flame (or gases – acetylene substitutes) is used as a high – temperature heat source. The advantages of method are high productivity, locality of processing, a small influence on substrate, ability to apply coatings on products of large sizes, etc. The use of these technologies for spraying of carbides of tungsten and chromium is limited due to poor quality of obtained coatings.

Plasma spraying allows spraying of coatings of almost any type of material, although to optimize the process of spraying of powders with different melting points, some modification of plasmatron design may be required. During plasma spraying, tungsten monocarbide ($WC$) is decarbonised to tungsten sub-carbide ($W_C$), as well as metallic tungsten. The presence of oxygen in plasma stream contributes to generation of oxycarbides in a significant amount, which is undesirable for wear resistance of coatings. In addition, high porosity and lower hardness are major drawbacks of plasma coatings for some applications. High – speed processes of thermal spraying, that based on the use of mechanical activation, minimize the decomposition of carbide phase due to lower heat enthalpy and shorter duration of coating formation process. Higher particle velocities provide a number of advantages to coating, such as lower porosity, higher adhesion strength and hardness.

Among carbides the $WC$ has the highest wettability of metals which is used as bonding. This, along with its relatively high viscosity, makes $WC$ the most widely used carbide for sintered alloys. Cobalt is the most commonly used binder because it has excellent wetting and adhesive properties. $WC$ does not have a stable molten phase, but is easily converted to other phases when the carbon content is non – stoichiometric. Therefore, $WC$ is a non – technological material for treatment at high temperatures, which lead to oxidation / decarburization during thermal spraying, especially in plasma.

The high – velocity spraying methods make it possible to significantly expand the capabilities of traditional thermal spraying methods. Here, the residence time of particles in the high – temperature stream is reduced and their overheating is reduced and, accordingly, the oxidation of particles and, accordingly, the content of oxides in coatings are reduced. Increasing the kinetic energy of particles leads to an increase of coatings density and adhesive strength of coatings with the substrate. The characteristic features of coatings obtained by high – velocity spraying are low porosity (less than 1,5 % ) and preservation of chemical composition of original powder, as well as high adhesion strength (more than 50...70 MPa ). High – speed coating methods include D – gun spraying, high velocity plasma gas air spraying (HVPGA) and high velocity oxygen fuel spraying (HVOF). The particle velocity at spraying is: up to 1000 m/s at D – gun spraying, 500–1000 m/s at HVPGA spraying and up to 800 m/s at HVOF spraying.

High durability of thermal coatings of $WC - Co$ is ensured by combination of $WC$ as a solid component and cobalt as a plastic binder. In the case of spraying of $WC - Co$ coatings, high – speed
methods are better than other methods of thermal spraying, as the high velocities and lower temperatures of powder particles reduce the degree of decomposition of WC during spraying process, thus reducing the hardness and wear resistance. Powders of WC–Co with content of 6–7 % of cobalt obtained by various methods are used for spraying. To increase the resistance of coatings of WC–Co to corrosion, powders of WC–Co are alloyed by chromium, since Co–Cr matrix provides higher corrosion resistance compared to WC–Co materials. Chromium content in powders is 4–8 % (WC–10 % Co–4 % Cr; WC–6 % Co–8 % Cr). Depending on powder composition, type of equipment and the modes of spraying, microhardness of obtained WC–Co coatings is 6–12 MPa at D–gun spraying, 10–15 MPa at HVPGA and HVOF spraying. The porosity of such coatings obtained by high–speed spraying techniques does not typically exceed 1.5 %. In the structure of coating obtained by D–gun method, there are oxide layers (about 5 %).

The microhardness of coatings of WC–9 % Co–9 % Cr obtained by the HVOF and HVPGA method is 11.0–11.7 MPa; the microhardness of the D–gun coating is 8.5 GPa. Reduced (by about 3 GPa) microhardness of D–gun coating and presence of oxide layers in microstructure are associated with application of oxidizing environment of detonation products of oxygen–propane–butane mixture during spraying process, which leads to development of oxidation processes of sprayed material and as a result of partial carbon loss and oxidation of matrix material.

In the study of structure and properties of composite coatings based on carbides of tungsten and chromium as a source used following powders: 1. Mechanical standard mixtures of WC–8%Co powders (VK8); 2. Conglomerated powders of alloy WC–15 % of alloy NiCrSiB (VSNGN–85); 3. Conglomerated powders of alloy WC–25 % Co (VK25); 4. Conglomerated powders of Cr,C,Ni alloy (KHN15C); 5. Mechanical mixtures of powders of chromium carbides and nickel of KHN type.

The chemical composition of D–gun sprayed coatings of standard VK8 powder, depending on spraying distance, was studied by method of local X–ray spectroscopic microanalysis (Table 1). The change of carbon content of coatings may be related to both the dissociation and interaction of WC with oxygen and nitrogen that present in high temperature stream. The monocarbide WC melts with decomposition at 2600 °C. Sub–carbide W₂C is thermally resistant to 2750 °C, i.e. practically to melting point (2730±15 °C).

| Table 1. The chemical composition of D–gun sprayed coatings of VK8 powder (in mass, %). |
|-----------------------------------------|-----------|-----------|-----------|
| Spraying distance, mm                  | 50        | 100       | 150       |
| ---                                     |           |           |           |
| Co                                      | 9.3; 9.06; 9.22 | 9.05; 11.82 | 9.55     |
| C                                       | 3.05; 5.72; 5.98 | 2.5; 2.59   | 3.42; 3.37 |
| W                                       | 87.65; 85.22; 84.8 | 88.44; 85.59 | 87.03; 86.71 |

The influence of spraying distance, cost of working gas flow, powder and transport gas on phase composition of coatings of VK8 was studied (Tables 2, 3). At the consumption of acetylene 179 cm³/cycle (γ = O₂ / C₂H₂ = 1,07) the coatings are containing W, W₂C, WC, Co₃W₉. At the consumption of acetylene 183 cm³/cycle (γ = 1,04) coatings consist of W₂C, Co₃W₉C₄, Co₂W₅C, WC.

With excess in the gas phase of oxygen, the following reactions of its interaction with WC are thermodynamically possible [29]:

\[ WC + 2O₂ \rightleftharpoons WO₂ + CO \] (1)
\[
WC + 2O_2 \leftrightarrow WO_2 + CO; \\
WC + 2O_2 \leftrightarrow WO_2 + CO_2; \\
WC + 15/8O_2 \leftrightarrow 1/4W_4O_{11} + CO; \\
WC + 19/8O_2 \leftrightarrow 1/4W_4O_{11} + CO; \\
WC + 2O_2 \leftrightarrow WO_3 + CO; \\
WC + 5/2O_2 \leftrightarrow WO_3 + CO_2.
\]

Table 2. Spraying modes of coatings of VK8 powder.

| No | \(Q_{C,H_2}\) cm³/cycle | \(l, mm\) | \(n\) | \(t_s, \mu m\) | \(t_s, \mu m\) |
|----|-----------------|--------|-----|-------------|-------------|
| 1  | 200             | 120    | 30  | 0.5         | 16.7        |
| 2  | 183             | 120    | 80  | 0.56        | 7           |
| 3  | 179             | 120    | 50  | 0.42        | 8.4         |
| 4  | 163             | 150    | 80  | 1.04        | 13          |
| 5  | 179             | 150    | 50  | 0.71        | 14.2        |
| 6  | 183             | 150    | 30  | 0.41        | 13.7        |
| 7  | 183             | 180    | 30  | 0.33        | 11          |
| 8  | 200             | 180    | 50  | 0.31        | 6.2         |
| 9  | 163             | 180    | 50  | 0.34        | 6.8         |

Note: \(Q_{C,H_2}\) – consumption of acetylene per cycle; \(l\) – is the spraying distance; \(n\) – is the number of single spraying cycles; \(t_s\) – is the total thickness of spraying spot; \(t_s\) – is the thickness of a single sprayed spot.

Table 3. Phase composition and modulus of elasticity of D – gun sprayed coatings of VK8 powder at different spraying modes.

| \(N\) | Phases | \(E, MN/m^2\) |
|-------|--------|----------------|
| 1     | \(W, W_2C, WC, C_3W_2C\) | \(1.99\times10^5\) |
| 2     | \(W, W_2C, C_3W_2C WC\) | \(3.04\times10^5\) |
| 3     | \(W, W_2C, WC, C_{17}W_6\) | \(3.24\times10^5\) |
| 4     | \(W, W_2C, WC, C_{17}W_6\) | \(2.34\times10^5\) |
| 5     | \(W, WC, C_{17}W_6\) | \(3.44\times10^5\) |
| 6     | \(W, W_2C, WC, C_{3}W_2C\) | \(3.04\times10^5\) |
| 7     | \(W_2C, C_3W_2C, C_3W_2C\) | \(2.33\times10^5\) |
| 8     | \(W_2C, WC, C_3W_2C\) | \(2.61\times10^5\) |
The interaction reactions occurring with the formation of \( CO_2 \) are thermodynamically more likely than similar reactions with the formation of \( CO \). The most likely is the last reaction. The thin powder \( WC \) starts to oxidize rapidly at \( 500 - 520 \) °C, the sub-carbide \( W_2C \) at the same temperature is oxidized to \( WO_3 \) [30].

Tungsten carbide is decarburized at temperatures above \( 750 \) °C:

\[
WC + H_2O \leftrightarrow W + H_2 + CO(CO_2)
\]  

(8)

As water vapor content in gas environment increases the carbon losses increase sharply. Some of cobalt grains of output powder are in an oxidized state. Cobalt oxides can be completely recovered by hydrogen or carbon. At temperatures above \( 190 \) °C a reaction is possible:

\[
Co_3O_4 + H_2 \leftrightarrow Co + H_2O.
\]  

(9)

In temperature range of \( 200 - 400 \) °C:

\[
CoO + H_2 \leftrightarrow Co + H_2O
\]  

(10)

If soot (free carbon) is formed during combustion of combustible mixture, then if it is contacted with particles of oxidized cobalt at temperatures above \( 500 \) °C, a reaction is possible:

\[
2Co + 2C = 2Co + CO_2
\]  

(11)

The process of carbonation of tungsten is possible mainly due to content of carbon at gas environment. Carbidization reactions can be represented by following equations:

\[
2C + H_2 = C_2H_2;
\]  

(12)

\[
2W + C_2H_2 = 2WC + H_2;
\]  

(13)

\[
C + CO_2 = 2CO;
\]  

(14)

\[
2CO + W = WC + CO_2;
\]  

(15)

\[
2W + CH_4 = W_2C + 2H_2;
\]  

(16)

\[
W_2C + CH_4 = 2WC + 2H_2
\]  

(17)

\[
2W + 2CO = W_2C + CO_2
\]  

(18)

\[
W_2C + 2CO = WC + CO_2
\]  

(19)

The diffuse layer formed on particle surface of \( W \) consists of two phases – \( WC \) and \( W_2C \), and initially a \( WC \) phase is formed on the surface, from which carbon diffuses inside with formation of \( W_2C \) phase layer.

A similar mechanism is also manifested during decarburization of tungsten carbide particles. Initially, a \( W_2C \) phase is formed on surface into which carbon from the particle nucleus diffuses.

If the particles of \( WC \) and \( Co \) are in contact during movement, reaction at a temperature above \( 530 \) °C is possible:

\[
2CoO + WC = CO_2 + 2Co + W
\]  

(20)

At temperatures above \( 850 \) °C a reaction is possible:

\[
3CoO + 3WC = W_3Co_3C + CO + CO_2
\]  

(21)
It is also possible to dissolve of carbon in cobalt at direct contact of particles and through the gas phase at a temperature above 600 °C, eutectic melting of \( \text{Co} + \text{WC} \) at a temperature of 1300 – 1370 °C.

3. Discussion

Physico-chemical transformations in powder particles are due not only to composition of detonating mixture of gases, but also to a number of other technological factors (Table 2). First of all, it is necessary to take into account features of output placement in the barrel of a fresh charge – detonation mixture and portions of powder of sprayed material, which determine the nature of dynamic, thermal and chemical interaction of particles with combustion products and then with the surface of workpiece. This is evidenced by significant dependence of coatings hardness from consumption of acetylene (Figure 1).

![Figure 1](image)

**Figure 1.** The dependence of coatings hardness of powder VK8 from consumption of acetylene. Spraying distance, mm:

120 (1,2), 150 (3,4), 180 (5,6). Consumption of transporting gas, cm³/cycle: 35 (1,3,5), 45 (2,4,6).

For sintered hard alloys of \( \text{WC} – 8 \% \text{Co} \) the HV hardness at size of main mass of carbide grains of \( 1 – 2 \mu \text{m} \) is 12160 MPa, and at size of main mass of carbide grains of \( 2 – 5 \mu \text{m} \) is –10300 MPa. The \( \text{W}_2\text{C} \) sub-carbide has a slightly higher hardness compared to the \( \text{WC} \) monocarbide, but has a lower strength. As the consumption of acetylene decreases, the total consumption of fuel mixture for spraying cycle is also reduced, as well as powder cloud is moved to closed end of the barrel. This leads to a longer residence of powder in hot detonation products and the intensification of the \( \text{WC} \rightarrow \text{W}_2\text{C} \rightarrow \text{W} \) conversion. The decrease of coatings hardness with increasing consumption of acetylene to 200–218 cm³/cycle, associated with increase of content of free carbon coatings.

With increase of mass of a single dose of powder introduced into the barrel, and thus the thickness of single coating layer, the hardness of coatings decreases, their composition increases content of tungsten (Figure 2). The transformation phase of output of VK8 powder is significantly influenced by the type of transporting gas. The use of air intensifies the process of transformation \( \text{WC} \rightarrow \text{W}_2\text{C} \rightarrow \text{W} \).

Similar phase transformations are also observed in coating from conglomerated powder – VK25 (\( \text{WC} – 25 \% \text{Co} \)). In the output powder the main phases are monocarbide \( \text{WC} \) and \( \text{Co} \), there are a small amount of \( \text{W}_2\text{C} \) carbides. On the radiograph of coating, the major phases are \( \text{W}_2\text{C} \) sub-carbide, \( \text{Co} \), and a small amount of \( \text{WC} \) monocarbide is contained.
Figure 2. Dependence of coatings hardness of VK8 powder from thickness of a single layer.

Similar patterns are evident during spraying of coating from output conglomerated powder – VSNGN – 85 (WC – 15 % of alloy SNGN). The radiograph of output powder contains only lines of WC monocarbide. Two new phases appear in the coatings – $W_2C$ sub-carbide and $W$ tungsten. As the spraying distance increases, the relative intensity of $W_2C$ and $W$ lines increases, it means that the content of these phases increases.

The change of hardness and other characteristics of chromium carbide coatings is also associated with a change of intensity of transition of higher carbides to lower ones ($Cr_3C_2 \rightarrow Cr_7C_3$, $Cr_{23}C_6$), whose hardness is much lower than $Cr_3C_2$ carbide. The X-ray structural studies of phase composition of coatings from conglomerated powder – KHN15 (chromium carbides – 15% NiCr) were carried out. The output powder contains the $Cr_3C_2$, $Cr_7C_3$, $Cr_{23}C_6$, Ni, Cr phases.

At a spraying distance of 150 mm the coatings containing $Cr_7C_3$ phases (hexagonal), $Cr_3C_2$, $Cr_3Ni_2$, NiCr, small amount of $Cr_3C_3$ (triclinic) and $Cr_2C$, $Cr_7C_3$ (cubic), $Cr_{23}C_6$. When the spraying distance is increased to 200 – 250 mm that means the increase of residence time of powder particles in high – temperature stream, the lines of $Cr_2C$, $Cr_3Ni_2$, $Cr_7C_3$ (cubic), $Cr_{23}C_6$, are disappear and the traces of $Cr_2O_3$ oxide appear. From position of thermodynamic the most stable is higher chromium carbide – $Cr_3C_2$. By reducing of oxygen environment stability, carbides of VI group form a variety of $Cr_3C_2$, WC, $Mo_2C$, $Cr_7C_3$, $Cr_{23}C_6$.

Figure 3. The microstructure of coatings of KHN15S powder: a – in secondary electrons, $\times500$; b, c, d – in characteristic X-ray radiation: b – Fe $K\alpha$; c – Cr $K\alpha$; d – Ni $K\alpha$.

The microstructure of coating from KHN15S powder and contact area of coating – substrate was studied by the method of micro–X-ray spectroscopic microanalysis (Figure 3). The coating is characterized by a dense structure and a fairly uniform distribution of Cr and Ni. An increased concentration of Fe and Cr in the transition area between coating and substrate is observed.

4. Summary

- The structure and properties of thermal coatings are determined by nature and intensity of processes of physico–chemical transformations of powder particles and their interaction with
working environment at all stages of coating obtaining, including manufacture and preparation of powder. At D – gun spraying to provide optimal conditions for the flow of conductive transformations and phenomena, that have a decisive influence on structure and properties of sprayed coatings, the regulations of such parameters as composition of combustible mixture, geometry and dimensions of barrel, conditions of introduction of powder into the barrel, a single powder dose, spraying distance, timing of spraying cycle and other technological techniques can be applied.

- The basic physico–chemical transformations accompanying D – gun spraying of coatings from powders of WC – Co alloy are studied: oxidation and reduction of tungsten carbide in high – temperature oxygen environment, interaction of components of composite coating.
- The general principle of design of optimal operating technology of D – gun spraying of coatings is to provide a homogeneous heating conditions, composition of surrounding gas environment and same residence time of particles in spraying stream.
- To obtain coatings based on transition carbides with high adhesion and cohesion strength and preservation of initial phase composition, it is recommended to ensure their formation from solid particles heated to pre – melting temperatures, at collision velocities with substrate of 300 – 600 m/s and cooling velocities of single layers of molded coatings exceeding $5 \times 10^4$ K/s, that excluding carbide decomposition.
- The use of protective coatings should be recommended to increase the strength and reliability of parts and structures of railway rolling stock.

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