Analysis and rationale for the use of coating microhardness as a controlled parameter

I I Yagafarov
Ufa State Aviation Technical University, 12 K. Marx Str., Ufa, 450008, Russia
E-mail: ilyagafarov@gmail.com

Abstract. The article is concerned with the possibility to use coating microhardness as a parameter of standardization. The TiN coatings were deposited by vacuum arc plasma. A series of experiments was carried out to establish the patterns for the microhardness to change, subject to specific deposition regimes. The article presents the conditions for coating microhardness to be taken as a parameter of standardization. It provides the microhardness ranges necessary to monitor the quality of coatings.

1. Introduction
There is a number of factors that determine service properties of workpieces with vacuum arc plasma coatings [1–5]. They include substrate roughness, coating thickness, surface microhardness, coating adhesion, and residual stresses in a coating. However, design documentation specifies solely surface roughness (Ra) and coating thickness (H+h), which is not enough to establish a qualitative correlation between operating conditions of coating deposition and service properties of coated workpieces. Thus, coating deposition by vacuum arc plasma is becoming increasingly complicated, taking into account the purpose of a workpiece and the requirements for its surface.

Coating microhardness and thickness as well as surface finish designate such properties as wear resistance, antiadhesivity, and corrosion resistance. Besides, coating microhardness can be easily measured. Thus, measuring coating microhardness will increase the efficiency of surface quality control.

The data provided in the scientific and technical literature [2] show the interrelation of the obtained coating microhardness and the nitrogen content in a coating (figure 1).

Thus, when depositing coatings by vacuum arc plasma, measuring the microhardness enables to control the chemical composition of coatings.

The paper is aimed at assessing the use of coating microhardness to control the quality of vacuum arc plasma coatings on the basis of experimental studies, analysis of technical documentation and scientific publications.

2. Experimental approach
The TiN coatings were deposited on upgraded HHB 6.6-I1 and URM3 installations. The microhardness was measured using a Nanovea tester. The studies were carried out to establish the relationships between the load applied on the indenter, and to determine the thickness when measuring the microhardness. The microhardness was measured following a standard method in accordance with [6]. Each measurement was repeated 10 times and for each point the microhardness H50 was...
calculated as an arithmetic average of 10 measurements with an estimated sputtering field against the relation: \( \Delta H = H_{\text{max}} - H_{\text{min}} \). To measure the microhardness, flat samples of \( \varnothing 15 \text{ mm} \) and a thickness of \( h = 10 \text{ mm} \) with a surface roughness of \( Ra = 0.05 \mu m \) were used.

\[ \begin{align*}
\text{Figure 1.} \quad & \text{TiN coating microhardness (■) and nitrogen content in coatings (△) vs nitrogen pressure in a vacuum chamber [2].} \\
\text{Figure 2.} \quad & \text{TiN coating microhardness vs applied load. 13KH11N2V2MF-SH substrate.} \\
& \text{Spray mode: } I_s = 180 \text{ A}; U_s = 200 \text{ V}; T = 420 \degree \text{C}; p = 10^{-1} \text{ Pa}; \quad \Delta - h = 10 \mu m; \quad \bullet - h = 5 \mu m; \quad \Delta - h = 3 \mu m.
\end{align*} \]
It is evident that the increased load results in a decrease in the actual microhardness, which is associated with an increase in the substrate hardness impact and the probability of coating puncture. To obtain the objective data as regards the microhardness of coatings with a thickness of \( h \geq 10 \mu m \), it is advisable to apply a load of \( P = 0.5 \) N. The microhardness of less thicker coatings is measured at a given load with a significant error. A series of experiments were conducted to determine the effect of coating thickness on microhardness. The results are shown in figure 3.

Thus, the correlation between the measured microhardness and the coating thickness is complex – steeply increasing, with a fracture and stabilization for a certain value of the coating hardness. Moreover, the stabilization point depends on the load applied on the indenter. To measure the microhardness of the coating with a thickness of \( h \leq 5 \mu m \), it should not exceed \( P = 0.2 \) H. The maximum load on the indenter is determined by an increase in the substrate hardness impact. According to [6], the depth of indentation should not exceed 10% of the coating thickness.

![Figure 3](image)

**Figure 3.** TiN coating microhardness vs thickness. Substrate 13KH11N2V2MF–SH. Spray mode: \( I_s = 180 \) A; \( U_s = 200 \) V; \( T = 420^\circ \)C; \( p = 10^{-1} \) Pa; \( \bullet \) – \( P = 0.2 \) N; \( \Delta \) – \( P = 0.5 \) N; \( \Delta \) – \( P = 1 \) N.

The effect of the location area of samples on microhardness are shown in figure 4.

The maximum microhardness of the coating was found at \( R_{loc}/R_c \approx 1 \). Moving away from the source of particles and increasing the ratio bring about a microhardness decrease. This is due to a decrease in coating thickness when the distance from the cathode grows. The magnitude and error of the microhardness is determined by the thickness and roughness of the coating, which corresponds to [1]. Subject to the distance from the cathode, the microhardness varies in an extremely wide range from 4 to 37 GPa. To reduce the spread in microhardness, it is advisable to locate workpieces in an area at a ratio \( R_{loc}/R_c \leq 3 \), with the microhardness to be in the range of 28–37 GPa at a distance of 200 mm from the cathode, 25–30 GPa at 300 mm, 21–25 GPa at 400 mm.

To establish the main relationships between operating conditions and substrate materials and the microhardness of the coatings, the samples were prepared from EI961 SH and ZHS6 UD-ID steels and VT18 alloy, which had various surface finishes (grinding, turning, polishing). The operating conditions were changed in the following range: \( I_s = 130–280 \) A, \( T_{subs} = 300–900^\circ \)C, \( p = 1.33 \times 10^{-3}–6 \times 10^{-1} \) Pa. The results of the experiments are given in table 1 and figures 5–6.
Table 1. Correlation between microhardness of coatings and operating conditions and types of pretreatment for different substrates.

| Substrate    | Operating condition | Type of treatment | Microhardness | \( (H_{\text{max}} - H_{\text{min}})/2 \) |
|--------------|---------------------|-------------------|---------------|------------------------------------------|
| EI961 SH     | 200 \( \times 10^{-4} \) 50 400 polish. | | 46.35 | 2.92 |
|              | 200 \( \times 10^{-3} \) 175 600 polish. | | 44.28 | 3.99 |
|              | 130 \( \times 10^{-3} \) 200 600 grind. | | 13.10 | 0.58 |
|              | 130 \( \times 10^{-3} \) 200 600 polish. | | 15.77 | 0.00 |
|              | 200 \( \times 10^{-2} \) 190 600 grind. | | 23.88 | 2.76 |
|              | 200 \( \times 10^{-2} \) 190 600 polish. | | 30.78 | 1.85 |
|              | 280 \( \times 10^{-3} \) 60 600 polish. | | 51.56 | 4.74 |
|              | 280 \( \times 10^{-3} \) 60 600 grind. | | 17.80 | 0.84 |
| ZHS6 UD-ID   | 200 \( \times 10^{-3} \) 50 90 polish. | | 42.97 | 2.94 |
|              | 130 \( \times 10^{-3} \) 200 50 polish. | | 55.81 | 4.24 |
|              | 200 \( \times 10^{-3} \) 50 400 polish. | | 38.48 | 2.80 |
|              | 200 \( \times 10^{-3} \) 175 600 grind. | | 55.85 | 4.25 |
|              | 200 \( \times 10^{-3} \) 175 600 polish. | | 39.89 | 2.78 |
|              | 200 \( \times 10^{-3} \) 200 700 grind. | | 46.06 | 3.38 |
|              | 200 \( \times 10^{-3} \) 200 700 polish. | | 47.25 | 3.60 |
|              | 280 \( \times 10^{-3} \) 60 600 polish. | | 41.28 | 2.80 |
|              | 280 \( \times 10^{-3} \) 60 600 polish. | | 55.85 | 4.25 |
|              | 200 \( \times 10^{-3} \) 200 300 grind. | | 48.09 | 3.98 |
|              | 200 \( \times 10^{-3} \) 20 300 polish. | | 39.89 | 2.78 |
| VT18         | 200 \( \times 10^{-3} \) 20 300 polish. | | 46.06 | 3.38 |
|              | 200 \( \times 10^{-3} \) 20 300 polish. | | 57.93 | 4.74 |
|              | 200 \( \times 10^{-3} \) 175 600 polish. | | 46.49 | 3.80 |
|              | 200 \( \times 10^{-3} \) 200 700 polish. | | 46.06 | 3.38 |
|              | 200 \( \times 10^{-3} \) 300 800 polish. | | 31.89 | 1.91 |
|              | 130 \( \times 10^{-3} \) 200 600 polish. | | 23.79 | 1.95 |
|              | 200 \( \times 10^{-3} \) 300 400 polish. | | 39.89 | 2.78 |
| ZHS6K        | 200 \( \times 10^{-3} \) 200 700 polish. | | 32.82 | 2.50 |
|              | 200 \( \times 10^{-3} \) 300 800 polish. | | 39.89 | 2.78 |
|              | 200 \( \times 10^{-3} \) 190 600 polish. | | 22.67 | 1.00 |
|              | 200 \( \times 10^{-3} \) 320 900 polish. | | 26.69 | 2.30 |

The microhardness of substrates was found to be as follows:

- EI961 SH \( H_{50} = (3.16 \pm 0.13) \) GPa;
- ZHS6 UD-ID \( H_{50} = (4.6 \pm 0.14) \) GPa;
- VT18 \( H_{50} = (3.34 \pm 0.22) \) GPa;
- ZHS6K \( H_{50} = (4.53 \pm 0.15) \) GPa.

Analysis of the results suggests that the scatter of measurements to define the microhardness of the surface after deposition does not exceed \( \pm 5 \) GPa. The type of pretreatment significantly influences the actual microhardness of the coating. The magnitude and measurement error of the microhardness is determined by the combination of two factors: the surface roughness (height of asperities) and the depth of indentation. Thus, the minimum magnitudes of the load applied on the indenter when measuring the microhardness are determined by the roughness of the coating in terms of reducing the spread of measurements.
Figure 4. TiN coating microhardness subject to the ratio of the location radius of a sample to cathode radius at different distances from the cathode; ■ – $L = 200$ mm; ○ – $L = 300$ mm; △ – $L = 400$ mm. Load – 150 mN.

The correlation between the TiN coating microhardness and the arc current has an increasing character for all materials. With an increase in the arc current of the vaporizer, the number of vaporized metal ions and the growth rate of the coating increase, which leads to an increase in the density of dislocations in the coating being formed and, as a consequence, an increase in the microhardness. The most significant increase in microhardness occurs when the current changes to 200 A (figure 5). The measurement error does not depend on the magnitude of the arc current of the vaporizer and amounts to ±3.5 GPa.

Figure 5. TiN coating microhardness vs arc current. Deposition mode: $T_s = 600^\circ$C; $I_{m.s.} = 200$ A; $p = 0.13$ Pa; ○ – EI961; ■ – VT18; △ – ZHS6.
The microhardness of the coatings with the increased substrate temperature (increase $U_s$) monotonously decreases, while the measurement error of the microhardness does not change depending on the magnitude of the voltage on the substrate and amounts to $\pm 5$ GPa.

Figure 6. TiN coating microhardness vs temperature in a substrate. Deposition mode: $I_d = 200$ A; $I_{m.s} = 200$ A; $p = 0.13$ Pa; ○ – VT18; ▲ – ZHS6

The most significant change in the microhardness from 3500 to 2500 MPa is observed when the temperature increases from 500 to 800°C.

The correlation between the microhardness and gas pressure is shown in figure 7.

Figure 7. TiN coating microhardness vs gas pressure. Deposition mode: $I_d = 200$ A; $I_{m.s} = 180$ A; $U_s = 180$ V; $h_{coat} = 8–10$ µm. Substrate – EI961 SH.
The results show that in terms of the durability of the coating, the optimum deposition is that produced at $T_s \leq 500^\circ\text{C}$ and gas pressure $p \approx 8 \times 10^{-2}$ Pa. The results of the chemical study show that in these conditions the resulting coatings correspond to the TiN stoichiometric composition with a lattice parameter of 0.4244 with a nitrogen content of 50 at%.

Based on the obtained patterns for the load to affect the indenter, the thickness of the coating, the surface roughness before and after the deposition of the coating to affect the magnitude and scatter of the microhardness of the surface, several GOSTs [6] and scientific and technical data [1, 3], certain requirements have been established for the surface quality depending on the thickness of the coating (table 2).

**Table 2. Requirements for surface quality when depositing coatings by vacuum arc plasma.**

| Coating thickness, $h$ (μm) | Type of pre-treatment and surface roughness | Coating roughness $Ra$ (μm) | Magnitude of load on indenter, $P$ [H] |
|-----------------------------|------------------------------------------|-----------------------------|-------------------------------------|
| $\leq 5$                    | polishing (0.04)                        | 0.1                         | $\leq 0.2$                          |
| $5 < h < 10$                | grinding (0.2)                          | 0.4                         | $0.2 < P < 0.5$                     |
| $\geq 10$                   | grinding (0.63)                         | 0.8                         | $0.5 \leq P \leq 1$                 |

To reduce the roughness after the deposition of the coating, according to the recommendations [4, 5], it is reasonable to apply different plasma filtration systems and lower the arc currents in the vaporizer.

**4. Results and discussion**

The study shows that it is necessary to develop specific technical requirements, technological recommendations for design documentation to include the specifications for the coatings deposited by vacuum arc plasma. The microhardness of coatings should be applied as a controlled parameter of the surface quality of workpieces.

Based on the experimental data and normative and technical requirements, surface quality regulations have been developed for measuring the microhardness of a coating depending on its thickness.

The authors provide the patterns for the surface microhardness to change subject to specific deposition parameters. Being applied for the development of coating deposition technology, they will make it possible to control the properties of coatings.

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