Si-containing diamond-like carbon coatings to improve the wear resistance of solid ceramic end mills

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Abstract. The study's purpose is to determine the rational Si content in the DLC-Si external layer of (CrAlSi)N/DLC-Si coatings deposited on α/β-SiAlON+TiN ceramics. The influence of Si content on physical and mechanical properties of coatings such as microhardness, modulus of elasticity, and friction coefficient, and wear resistance of solid ceramic end mills in machining nickel alloy NiCr20TiAl is determined.

1. Introduction

Heat resistant nickel alloys such as NiCr20TiAl, NiCr19FeNiMo, NiCr17Fe7TiAl, and others are used to manufacture precision turbine blades which are the principal elements of aircraft components. Solid end mills are the most popular and functional tool for machining aircraft parts at high cutting speeds. Tool ceramics allow significantly higher cutting speeds of up to 350 – 600 m/min than tungsten carbide. These speeds correspond to cutting temperatures at which heat-resistant nickel alloys become ductile, and milling is realized at less cutting force [1, 2]. Therefore, leading cutting tool companies such as Kennametal, Mitsubishi Materials, Iscar, Sandvik, et al. are now producing solid ceramic end mills (SCEM) based on sintered α/β-SiAlON ceramic with various additives. MSUT «STANKIN» researches SCEM to improve their volumetric and surface properties in recent years [3–5].

One approach, which has proved its effectiveness, is to deposit coatings based on complex nitrides and diamond-like carbon structures on SCEM. The authors of the paper experimentally proved in previous studies that two-layer coating such as (CrAlSi)N/DLC up to 1.5 times increases the wear resistance of the α/β-SiAlON+TiN ceramic end mills and reduces the surface roughness of the machined workpiece. The coating deposition is based on the original technological approach of the Swiss company PLATIT. The technology involves vacuum-arc deposition of a nitride sublayer (CrAlSi)N and subsequent formation of an external diamond-like carbon (DLC) layer by plasma chemical gas-phase deposition by initiating a chemical reaction and decomposition of acetylene, nitrogen, argon and tetramethyldisilane gas mixture components. The (CrAlSi)N/DLC two-layer coating technology and conditions are fully described in the previous works [6, 7].

The selection of (CrAlSi)N as the sublayer material for the DLC layer functioning is explained below. This three-component nitride is different from traditional nitride films and is a nanocomposite. The nitride deposition does not result in the complete mixing of all components but the formation of two phases, such as AlCrN nanocrystals embedded in the amorphous SiN matrix, which provides a high level of interatomic bonding strength [8–10]. The introduction of silicon into the coating reduces the internal residual stress at the "ceramic - DLC layer" interface. The formation of secondary oxide-
based phases by the (CrAlSi)N layer components at high cutting temperatures slows down the tribochemical reactions at the tool contact surfaces. The capacity of improving the serviceability of the external DLC layer during high-speed machining of nickel alloys with SCEM with the introduction of silicon into its composition was not entirely experimentally investigated. Increased thermal loads are known to adversely affect pure carbon DLC layers' physical and mechanical properties [11, 12].

The purpose of this study is to determine the rational Si content of the external coating layer (CrAlSi)N/DLC-Si deposited on α/β-SiAlON+TiN SCEM. Spark plasma sintering technology was used for sintering the ceramic workpieces. The study evaluated the effect of Si content on the physical and mechanical properties of the DLC layer and the wear resistance of SCEM when machining NiCr20TiAl nickel alloy workpieces.

2. Research on the structure of (CrAlSi)N/DLC-Si coatings
The cutting edges of the α/β-SiAlON+TiN SCEM of 10 mm in diameter used in the experiments and the SEM imaging (CrAlSi)N/DLC-Si coating surface are shown in figure 1 (a). It is noticeable that the surface of the external DLC layer of the coating has a specific topography in the form of interconnected spherical segments. Figure 2 (b) shows the SEM images of the microstructure of a (CrAlSi)N/DLC-Si coated ceramic sample obtained using a VEGA3 LMH scanning electron microscope.

![Figure 1](image1.png)

**Figure 1.** The cutting edges of the α/β-SiAlON+TiN SCEM: SEM images of the (CrAlSi)N/DLC-Si coating surface (a) and the microstructure of the ceramic sample with the coating (b).

A transmission microscopy technique was used to study the coating structure in more detail, involving the preparation of special lamellas of the ceramic sample with coating. Figure 2 shows a TEM image obtained using a JEM-2100F JEOL microscope illustrating the (CrAlSi)N/DLC-Si coating structure. The (CrAlSi)N sublayer has a columnar structure, and the DLC-Si external layer has an amorphous structure, the elements of which are not detected by microscopy. The total thickness of the deposited coatings is 3.6 ± 0.1 µm, including 1.7 µm thickness of the (CrAlSi)N sublayer and 1.9 µm thickness of the external DLC-Si layer.

The Si content of the external DLC-Si coating was varied by adjusting the volume flow rate of the reaction gas tetramethylsilane, which is supplied to the vacuum chamber by the multi-channel gas inlet system (each channel is controlled by a separated gas flow meter). The volume flows of the other gases - acetylene, argon and nitrogen were unchanged during the experiments, with values of 232, 156, and 30 cm³/min, respectively. The quantitative elemental composition of (CrAlSi)N/DLC-Si coating samples, the external layer of which was formed at different tetramethylsilane volume flow rates, was obtained by EDX analysis [13]. Figure 3(a) shows the dependence of the Si content in the DLC layer on the volume flow rate of the tetramethylsilane. Figure 3(b) shows the distribution of elements, including Si, over the cross-section of the (CrAlSi)N/DLC-Si coating obtained by EDX analysis.
Figure 2. TEM image of (CrAlSi)N/DLC-Si coating: diffraction pattern (a), structure of two-layer coating (b), and structure of external DLC-Si layer (c).

Figure 3. Dependence of Si content in the DLC-Si layer on the tetramethylsilane volume flow (a) and the element distribution along the coating (CrAlSi)N/DLC-Si obtained by EDX analysis (b).

3. Research on the physical and mechanical properties of (CrAlSi)N/DLC-Si coatings
The Si content of the (CrAlSi)N/DLC-Si external coating layer influence on the physical and mechanical properties was investigated. The nanoindentation method with a diamond indenter (Berkowitz trihedral pyramid) was realized on an SCEM instrument Nano Hardness Tester. The load value was selected based on the requirement that the penetration depth of the indenter should not exceed 15% of the DLC-Si film thickness (in the experiment, the penetration depth of the indenter was 0.4 µm). Table 1 shows the evaluation results for the micro-hardness and modulus of elasticity of (CrAlSi)N/DLC-Si coating at different Si contents in the external coating layer.

| №  | Samples                  | Si content of the DLC layer (%) | Load (mN) | Microhardness $H$ (GPa) | Modulus of elasticity $E$ (GPa) |
|----|--------------------------|---------------------------------|-----------|-------------------------|--------------------------------|
| 1  | $\alpha/\beta$-SiAlON+TiN with (CrAlSi)N/DLC-Si coating | 0.8                              | 4.0       | 26 ± 3                  | 238 ± 8                        |
| 2  |                          | 3.0                              |           | 24 ± 3                  | 201 ± 10                       |
| 3  |                          | 5.0                              |           | 22 ± 2                  | 185 ± 10                       |
| 4  | (CrAlSi)N/DLC-Si coating | 7.0                              |           | 19 ± 2                  | 171 ± 8                        |
| 5  |                          | 10.0                             |           | 15 ± 3                  | 136 ± 14                       |

The influence of the Si content of the DLC-Si layer on the coefficient of friction (COF) at different thermal impacts (20°C and 800°C) on ceramic specimens is shown in figure 4. The tests were performed on an Anton Paar TriTec TNT tribometer through the ball-on-disk method at a load of 1.0 N and a sliding speed of 10 cm/s.
4. Wear resistance of solid ceramic end mills with (CrAlSi)N/DLC-Si coatings

Wear resistance tests of SCEM with (CrAlSi)N/DLC-Si coatings were performed using a CTX beta 1250TC DMG milling center at \( V = 376.8 \text{ m/min} \) and feed rate \( S = 0.031 \text{ mm/tooth} \). A cylindrical workpiece end face from NiCr20TiAl nickel alloy (hardness 34 HRC, strength 1150 MPa) was used for the tests. The critical wear area of the mill tooth of 0.4 mm was accepted as the criterion for loss of tool life. Thus, wear resistance was determined as cutting time to critical wear, monitored visually with a Stereo Discovery V12 ZEISS optical microscope with a measuring accuracy of 0.05 mm. Table 2 shows the results of the experiments.

| № | Samples of SCEM | Si content of the DLC layer (%) | Tooth wear value (mm) for different machining times (min) | Wear resistance (min) |
|---|----------------|--------------------------------|----------------------------------------------------------|----------------------|
| 1 | \( \alpha/\beta \)-SiAlON+TiN | - | 0.21 0.35 0.44 - - | 5.0 |
| 2 | | 0.8 | 0.1 0.25 0.32 0.41 - | 7.7 |
| 3 | \( \alpha/\beta \)-SiAlON+TiN | 3.0 | 0.1 0.21 0.29 0.37 0.43 | 8.9 |
| 4 | with coating | 5.0 | 0.11 0.22 0.30 0.39 0.46 | 8.1 |
| 5 | (CrAlSi)N/DLC-Si | 7.0 | 0.18 0.28 0.38 0.44 - | 6.8 |
| 6 | | 10.0 | 0.18 0.32 0.4 - - | 6.0 |
5. Results and discussion

Figure 3 demonstrates that the dependence of the Si content in the DLC-Si layer is linear and increases monotonically with an increasing volume flow rate of the tetramethylsilane in the gas mixture. Variation of the Si content of the DLC-Si layer markedly influences the microhardness and modulus of elasticity of the (CrAlSi)N/DLC-Si coating: the micro-hardness $H$ and the modulus of elasticity $E$ decrease with increasing Si content. The samples with a 3 and 5% Si content in the DLC-Si layer showed a slightly higher plasticity index $H/E$ (table 1).

The Si content of the DLC-Si layer has a significant influence on the COF at different temperatures. The COF of uncoated ceramics increases quite rapidly at 20°C over time, whereas DLC-Si films exhibit reduced COF values throughout the test cycle (figure 4, a). In particular, the COF was invariably at 0.1 for the sample with the lowest Si content. As the Si content of the DLC-Si layer increases, the COF of the ceramic specimens increases. Uncoated samples under heating conditions showed an unstable behavior when the COF changes discontinuously and reaches a value greater than 0.9 at the test end (figure 4, b). DLC-Si films enormously change the frictional interaction conditions when heated: the COF is low and varies in the range 0.09 – 0.3 (depending on Si content) for a long time; the COF starts to increase intensively for all DLC-Si variants only after a distance of 150 m. The DLC-Si layers with Si content of 3 and 5% had the lowest COF value. It can be assumed that these layers rationally combine the advantages of DLC films with the high-temperature resistance and lubricity of Si-based compounds.

SCEM testing results show a positive influence of (CrAlSi)N/DLC-Si coatings on tool wear resistance (table 2). Depending on the composition of the DLC-Si external layer, the operating time to wear of 0.4 mm increased by 1.2 - 1.7 times, and SCEM with a DLC-Si external layer with 3% Si content achieved its maximum durability.

6. Conclusions

The conducted complex of experimental researches allows concluding that the Si addition to the composition of DLC films formed by plasma chemical gas-phase deposition allows influencing their basic physical and mechanical properties. An efficient and technically simple way of varying the Si content in the DLC layer is to control the volume flow of the tetramethylsilane in the gas mixture.

The deposition of (CrAlSi)N/DLC-Si coatings on α/β-SiAlON+TiN SCEM when cutting NiCr20TiAl-type nickel alloys positively influences and increases the wear resistance by 1.2 – 1.7 times. The best solution is a Si content of 3% in the external DLC-Si layer, which is achieved at the tetramethylsilane volume flow rate of 6 cm³/min.

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