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## **DUPLEX TYPE COATINGS FROM Ti-Si-N SYSTEM APPLIED ON WC CUTTING PLATES**

**Keywords**

Duplex coatings, TiN/Si<sub>3</sub>N<sub>4</sub> layers, WC cutting plates.

**Abstract**

The observations of microstructure as well as the results of work test of duplex type coated WC cutting plates are presented. The buffer hard TiN layer was deposited using PUSK -83 Arc system, while the ultra-hard composite TiN/Si<sub>3</sub>N<sub>4</sub> layer was deposited using WM-50 planar magnetron system equipped with the TiSi<sub>4</sub> at. target. The transmission electron microscopy observations indicated that the buffer layer is a columnar, relatively coarse crystalline, while the top layer is characterised also by columnar microstructure but of much finer crystallites. It was confirmed that buffer layer is filling up WC cracks and other surface roughness presenting much better deposition condition for the ultra-hard top layer. The work test showed that duplex coating extends the plates life by 60%. Additionally, the cutting plates' lifetime was correlated with their hardness.

**Introduction**

The TiN coatings, characterised by a gold colour, high both in hardness and adhesion, was an industrial standard on high class cutting tools by the end of

XX century. The development of coatings for such applications went mostly either toward reaching for materials of higher hardness or improved high temperature resistance. Both of these aims could be achieved with multilayered coatings, but their deposition process is usually much more complicating and in consequence rises production costs. An alternative approach is based on producing ultra-hard composite coatings through co-deposition of TiN and Si<sub>3</sub>N<sub>4</sub> [1-3]. In this case, limiting the size of TiN crystallites with thin sheets of amorphous Si<sub>3</sub>N<sub>4</sub> resulted in the coating's improved mechanical properties, moving it into the class of ultra-hard materials [4-6].

Taking advantage of the possibility of using dense TiSi compacts, which production details were described elsewhere [7], allow to use the same, as for TiN coatings, the single target reactive magnetron sputtering method keeping the overall costs low. The transmission electron microscopy investigations and mechanical tests showed that coatings obtained with such targets are characterised both by nano-composite amorphous-crystalline TiN/Si<sub>3</sub>N<sub>4</sub> and improved hardness of up to ~27GPa [7-10]. The experiments were performed using range of steel substrates, i.e. stainless steel (316L), high speed steel (SW7M), indicated that these relatively thin coatings characterised by ~1µm critical thickness, perform better on materials of higher hardness.

The last results turned our interest toward applying the TiN/Si<sub>3</sub>N<sub>4</sub> composite in duplex coatings, which originally meant that the hard nitride coating was deposited on surface nitrided surface [11-18], but it later was also extended for other hybrid surface treatments forming layers with stepped hardness, like substituting surface nitriding with laser cladding [19], arc plating [20] or others [21]. The magnetron systems seems to be well suited for producing duplex coating, because it is possible to use them both for glow discharge nitriding and deposition of hard surface coating. However, glow discharge nitriding results in the formation of surface defects, which result adversely on that deposited on the hard top layer [11]. The glow discharged surface "pox marks" might be limited with lowering the temperature at which the process is executed, but it makes it too long for industrial practice.

Taking into account the above, and the positive results of extending the ceramic cutting plates lifetime with nitride coatings, it was decided to test the effectiveness of the duplex coatings of the magnetron TiN/Si<sub>3</sub>N<sub>4</sub> layer deposited on arc formed TiN on WC tungsten cutting plates. The test was performed by comparing the lifetime of duplex coated plates with those coated solely with arc TiN coating and finally with non-coated plates mounted on the same turret. The coating's microstructure was examined using transmission electron microscopy. The mechanical properties were assessed through micro-hardness measurements.

## 1. The substrates and coatings

The WC cutting plates (TPKN 1603PPR SM 25T/P25) were arc coated with  $\sim 3 \mu\text{m}$  TiN buffer layer and magnetron deposited  $\sim 1 \mu\text{m}$  TiN/Si<sub>3</sub>N<sub>4</sub> top layer. The TiN coated plates showed distinct gold colouring, while those topped with TiN/Si<sub>3</sub>N<sub>4</sub> were of brownish-violet (Fig.1). The details of arc and magnetron deposition conditions were described previously in [22-24] and [7], respectively.

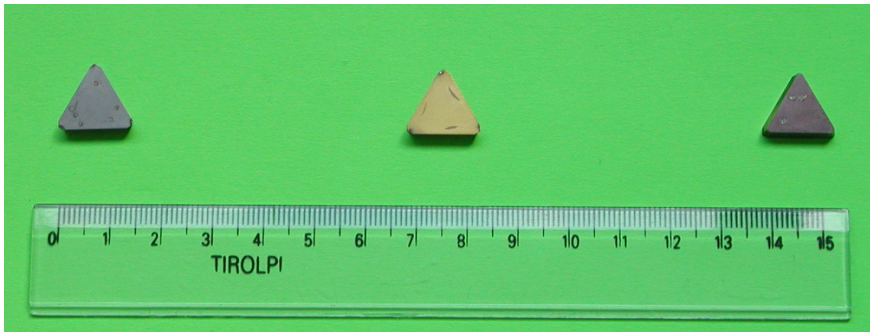


Fig. 1. The WC cutting plates in as received condition (a), arc TiN (b) and duplex (c) coated

## 2. Microstructure characterisation

The microstructure examinations were performed using Philips CM20 (200 kV) and TECNAI FEG (200kV) transmission electron microscopes. The thin foils presented the coating in *cross-section* were cut with a Quanta 3D-focused ion beam (FIB) system. It should be noted that the preparation of specimen for transmission microscopy observations from WC material is practically impossible with other techniques and at the limit of FIB. In the latter technique, in which platinum or tungsten are routinely used as masking strips due to their very low sputtering rate, it should be taken into account that cutting will take a long time and eventually will result in the removal of the mask as a consequence.

The microstructure observations of the arc TiN coated WC substrate plate performed with maximised mass contrast, i.e. without the objective aperture (Fig. 2a). This indicated that it is built of approximately equal fractions of both high mass and low mass blocky crystallites among which up to 5-10% vol. of vein like material is also of high mass contrast (dark at this image). The EDS measurements helped to prove that the described phases could be classed as WC carbides, TiC carbides and the veins as cobalt. The presence of up to 40% of TiC in the substrate should be an advantage in improving the adhesion of arc TiN coatings. It is significant that the WC plate surface is quite rough with frequent “wells” resulting from the carbides, which fell away during

compacting. Shallow cracks, especially around WC carbides closest to the surface were identified as well. Observations of the same specimen, but with maximised diffraction contrast (masking mass contrast), help to show that the arc TiN coating is characterised by a relatively coarse columnar microstructure (Fig.2b). The coating total thickness can be determined by relying on the remnants of the platinum masking bar (dark contrast) marked with a dark broken line in Fig. 2a.

The observations of the “duplex” coated substrates confirmed both the high roughness of the WC plates and cracks extending around the most top WC carbides (Fig. 3).

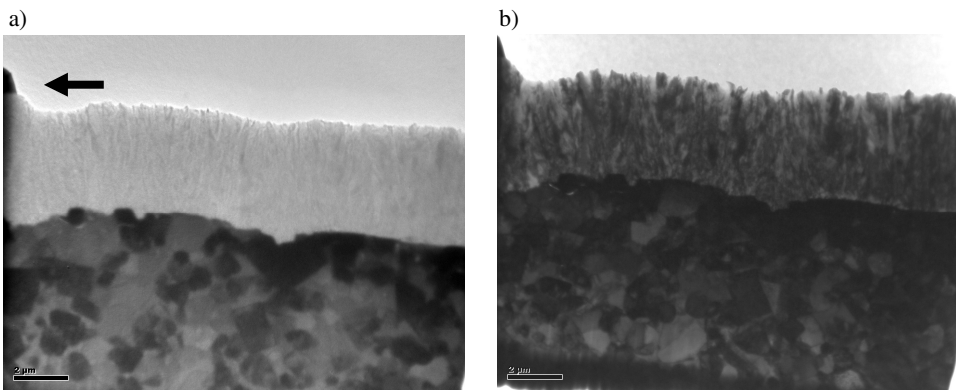


Fig. 2. The transmission microstructure presenting arc TiN coating on WC cutting plate by relying on mass contrast (a) and diffraction contrast (b) i.e. with the objective aperture out and in respectively. The arrow points toward the remnants of the platinum masking strip which was helpful in determining the total coating thickness

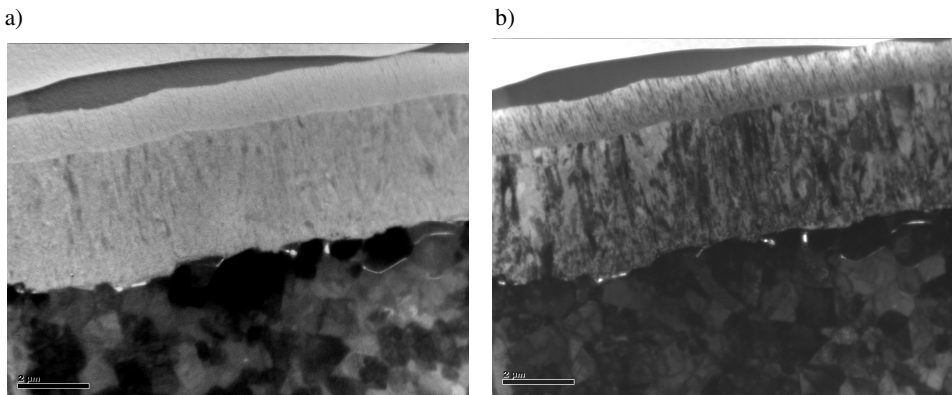


Fig. 3. The transmission microstructure presenting “duplex” coating of arc TiN buffer and magnetron TiN/Si<sub>3</sub>N<sub>4</sub> top layer on WC cutting plate by relying on mass contrast (a) and diffraction contrast (b). The rest of the platinum-masking strip is still attached to the top

The arc-TiN filled in all of the WC plate surface cavities and produced a much smoother base for the deposition of the TiN/Si<sub>3</sub>N<sub>4</sub> top layer. The observations with maximised diffraction contrast indicated that, while both layer are built of columnar crystallites, though the TiN/Si<sub>3</sub>N<sub>4</sub>, it is characterised by a much finer crystallite size.

### 3. Coatings hardness and Young modulus

The microhardness and Young modulus of WC cutting plates with arc-TiN, magnetron TiN/Si<sub>3</sub>N<sub>4</sub> and “duplex” arc-TiN topped with magnetron TiN/Si<sub>3</sub>N<sub>4</sub>, as well as for non-coated plates, were estimated using a CSM Micro-Combi-Tester equipped with a Vicker diamond indenter (ISO 14577-1 and PN-EN ISO 6507-1 Polish Norms). All measurements were performed at the load of  $P_{max} = 20\text{mN}$  kept for 5 seconds. Up to five measurements were performed for each coating.

The performed experiments indicated that the deposition of arc-TiN and magnetron TiN/Si<sub>3</sub>N<sub>4</sub> coatings on a WC-cutting plates raised the hardness from ~22 GPa up to 25 and 24 GPa, respectively (Fig. 4). The increase in Young modulus with respect to substrate showed only thicker coatings, i.e those exceeding 3  $\mu\text{m}$  rising from 350 up to 450 GPa.

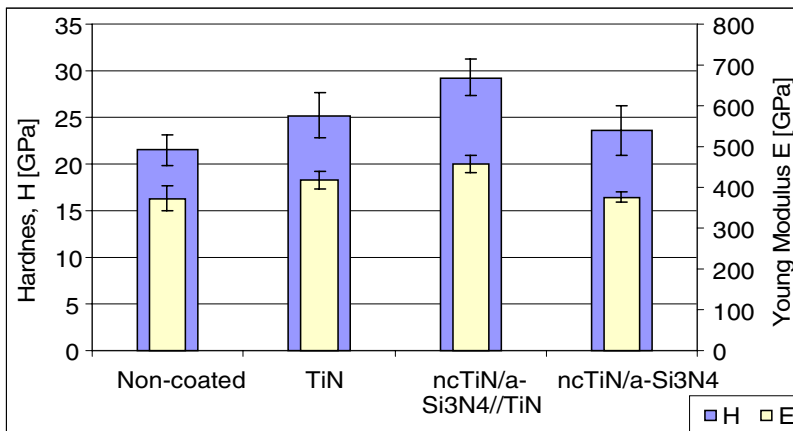


Fig. 4. Microhardness and Young modulus of coated and non-coated WC cutting plates

### 4. The work test on non-coated and coated WC cutting plates

The work test was performed on the WC cutting plates with the same type of coatings as in the case of micro-hardness measurements. The test was repeated three times.

The cutting plates were mounted on four holder head ( $Z-3 \Phi = 50$ ) of the vertical milling machine and acted against H13MNF steel. The experiment was performed at the following conditions: head revolution  $n=355$  rev./min., cutting speed  $V_c = 55.7$  m/min, milling speed  $V_f = 50$  mm/min,  $V_{f0} = 0.141$  mm/rev,  $V_{fz} = 0.047$  mm/cutting edge, depth of milling – 1 mm. The wear of edges was examined every 440 min and assessed using the “arbitrary blunting”  $VB = 0.6$  mm (above this value the edge wear is strongly accelerated). The wear data accumulated in this test indicate that the deposition of “duplex” coatings, i.e.  $\sim 3 \mu\text{m}$  arc TiN layer topped with  $1 \mu\text{m}$  magnetron composite TiN/Si<sub>3</sub>N<sub>4</sub> layer on WC plates may extend the lifetime by  $\sim 60\%$  (Table 1, Fig. 5).

Table 1. Lifetime to „arbitrary blunting” wear of all tested cutting plates

Number of cutting edge	I series (non-coated)	II series magnetron TiN/ Si <sub>3</sub> N <sub>4</sub> coated	III series arc TiN coated	IV series duplex TiN/ Si <sub>3</sub> N <sub>4</sub> /TiN coated
	[min]	[min]	[min]	[min]
1	22.0	25.7	40.4	41.8
2	22.0	28.4	38.8	45.9
3	22.0	39.4	44.2	48.4
4	40.3	34.8	37.7	60.5
5	40.3	36.0	37.8	62.3
6	41.8	53.2	33.2	63.5
7	33.0	24.8	31.3	42.6
8	33.1	26.3	37.5	43.7
9	30.3	31.7	40.2	45.8
average value	31.6	33.4	37.9	50.5
measurement error	6.3	6.9	3.0	6.9

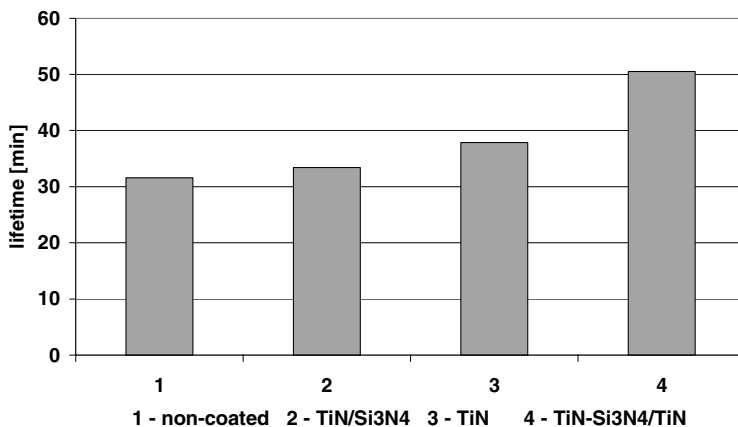


Fig. 5. Average lifetime to “arbitrary blunting” wear of all tested cutting plates

The statistical measurement error was estimated at 0.95 confidence level for the respective series of cutting plates. The analysis of the results with t-student test and taking the data from the I-st series (i.e. non coated plates) as a reference gave values of 0.677, 0.055 and 0.00026 for II, III and IV series, respectively. The obtained values describe probability that the differences in average values are accidental, i.e. the result of t-student confirmed the statistical correctness of the performed test.

The average lifetime of the analysed series of cutting plates show strong correlation (at a level of ~96%) with their hardness (Fig. 6). The presence of such correlation indicates that adhesion is good enough not to influence the coated plates' lifetime in any significant way. It is also an indication that the expensive and time consuming work tests might be, to some extent, substituted with simple and fast micro-hardness measurements.

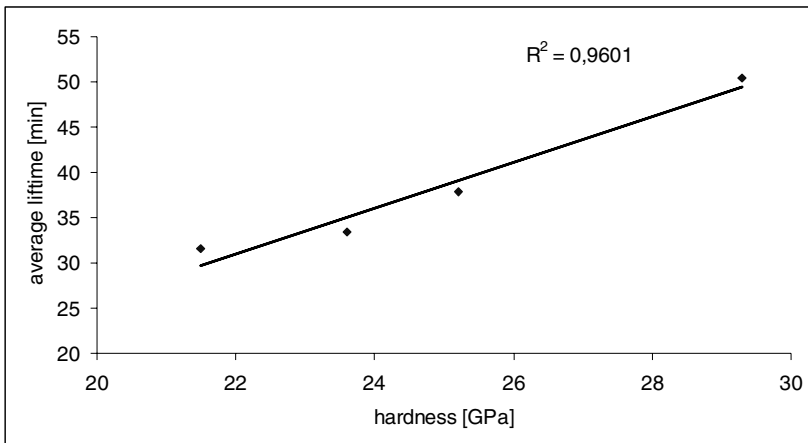


Fig. 6. Tested cutting plates hardness against their average lifetime

## Conclusions

The microstructure observations showed that the arc-TiN layer is filling all surface holes, pittings and cracks, producing a much smoother surface for the next magnetron deposited ultra-hard composite crystalline-amorphous TiN/Si<sub>3</sub>N<sub>4</sub> layer. The ~60% lifetime extension of this “duplex” coatings observed in the work test supports the idea for their industrial application.

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## **Powłoki typu “duplex” z układu Ti–Si–N zastosowane na płytkach frezarskich z WC**

### **Słowa kluczowe**

Powłoki duplex, warstwy TiN/Si<sub>3</sub>N<sub>4</sub>, płytki skrawające WC.

## Streszczenie

W pracy przedstawiono wyniki badań eksploatacyjnych płytek frezarskich TPKN 1603PPR SM 25T/P25 wykonanych z węglików spiekanych pokrytych powłoką typu „duplex” złożoną z twardej warstwy buforowej TiN oraz ultratwardej krystaliczno-amorficznej TiN/Si<sub>3</sub>N<sub>4</sub>. Warstwa buforowa TiN nanoszona była metodą łukową na stanowisku PUSK-83, a warstwę wierzchnią TiN/Si<sub>3</sub>N<sub>4</sub> naniesiono z użyciem magnetronu planarnego WM-50 ze specjalnym targetem wykonanym ze spieku TiSi4% at. Badania mikrostruktury powłok z użyciem mikroskopu transmisyjnego wykazały, że warstwa buforowa ma znacznie bardziej grubokrystaliczną budowę w stosunku do kompozytowej warstwy wierzchniej. Równocześnie zastosowanie warstwy buforowej TiN prowadzi do wypełnienia pęknięć i zagłębień na powierzchni płytek oraz wygładzenia powierzchni pod warstwę TiN/Si<sub>3</sub>N<sub>4</sub>. Płytki z powłoką „duplex” porównywano z takimi samymi pokrytymi jedynie tzw. warstwą buforową TiN, jak też z płytkami bez pokrycia. Potwierdzono istnienie korelacji pomiędzy trwałością ostrzy badanych płytek a twardością naniesionej warstwy oraz możliwość wydłużenia czasu pracy płytek z powłokami typu „duplex” o 60%.