Mechanical Properties and Structure of TiAlSiN Coatings, Deposited in Conditions Magnetron Sputtering

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Abstract – The nanocomposite coatings by vacuum magnetron sputtering at simultaneous work of two magnetrons under the debalanced scheme were obtained. Influences of various sputtering parameters on a structural – phase condition, chemical compound, mechanical and tribomechanical properties of the coatings were investigated. On the basis X-ray analysis and mass-spectrometry of secondary ions laws of change of phases ratio in the coatings and their chemical compound from sputtering modes, the average size of subgrains of the basic phase, and also character of dependences phase and a chemical compound of TiAlSiN coating of discharge currents of magnetrons ratio were determined.

1. Introduction

Now for increase of wear resistance of cutting tool the titanium nitride coatings are widely used. However, at high rates of cutting because of low oxidizing resistance, TiN coating surface embrittles. Hence coating destroys. Because new biphase TiN coatings is developed.

The coatings, at the certain ratio in a crystal lattice of titanium and aluminium atoms, in some cases show a unique combination of properties: high hardness, wear resistance, oxidizing *y* resistance and, simultaneously, high elastic regenerating coefficient and low friction coefficient. But obtaining of alloy for similar coatings is connected to technical and technological difficulties. Therefore, in the given work, at the coating deposition, used targets from the titanium, aluminium and silicon alloys. Operation of structure and phase composition of the coatings is carried out by selection of sputtering modes, change of alloying element concentration, partial pressure of working gas mix and temperature of substrate, the appendix to a substrate of a negative displacement voltage.

The purpose of the present work was research TiAlSiN nanocomposite coatings obtained by vacuum magnetron sputtering at simultaneous work two magnetrons under the debalanced scheme and studying of influence of various parameters of sputtering on structural – phase state, chemical compound and mechanical properties of the coatings.

2. Materials and technique of experiment

Deposition of the coatings carried out on vacuum sputtering installation NNV-6.6-I1. In a basis of work of the deposition scheme lays simultaneous sputtering of the coatings with two magnetrons with target from the titanium and target from aluminium (91%) and silicon (9%) alloy in diameter 120 mm working from direct current sources, equipped with system of protection against microarches. On working surface of sample serially are sputtered layer behind layer titanium nitride and nitride of aluminium of silicon (AlSiN).

Power of the magnetron discharge was supported within the limits of 1–2 kW. The coatings deposited in the jet medium from argon and nitrogen mix gases the general 0.3 Pa pressure and partial pressure of nitrogen 0.03–0.06 Pa.

Heating of samples in the vacuum chamber before sputtering and maintenance of temperature in process sputtering of the coatings carried out with molybdenum heater. Measurement of temperature was carried out with the help chromel-alumel thermocouple with accuracy ± 5 °C. The coatings were deposited on substrates heated up to temperatures 300 °C to which additionally the constant displacement potential $U_s = -100$ V was put in.

As substrates samples as parallelepipeds with the sizes 6×6×15 mm from SHH-15 steel was used.

Working surfaces of the samples ground and polished up to $R_a = 0.08$ μm. Before a premise in the vacuum chamber, the samples were exposed to degreasing by organic solvents, washing by ethyl alcohol and drying. With the purpose of reduction of influence of experiment random errors on the obtained results at each sputtering mode 5–6 samples was investigated. X-ray analysis was carried out with the DRON-7 in an interval of corners – 20 … 140° in filtered Co–K_α radiation. Hardness of the coatings measured with the NanoHardnessTester firms CSM.

Concentration profiles of elements on coating thickness were investigated by mass-spectrometer of secondary ions MC-7201M in etching mode by beam of argon ions.

3. Results of experiment and discussion

For studying a structure and properties of the coatings changed chemical concentration Ti and AlSi with the change of discharge currents of magnetrons: first at the greatest possible value of a discharge current with Ti – target increased discharge current with AlSi – target from minimal possible up to greatest possible, then at the greatest possible discharge current a with AlSi – target reduced discharge current with Ti-target.

Research of a chemical compound of the coatings by MCVI method has shown, that change of discharge currents of magnetrons ratio (Table 1) can change it appreciably. Thus, concentration of nitrogen in the coating at level 55 ± 1 at. % is constantly. At passage of all range of variation of discharge currents of magnetrons concentration of the titanium in coating is decreases in ~ 3 times, and aluminium is increased in ~ 10 times.

Thus, it is possible to obtain nanocomposite coatings in an interval of structures from almost pure TiN, to be exact from Ti0, 42Al0, 03N0, 55, up to Ti0, 15Al0, 31N0, 54.

MCVI method has low sensitivity ions of silicon and as their relative concentration in ~ 10 times are lower than concentration of aluminium it appears behind a threshold of detection to the method.

At research of phase composition and coating structure by indexing of roentgenograms with ASTM card file in the TiAlSiN coatings three phases (Table 2) is revealed:

– firm solution TiAlN with a cubic lattice such as NaCl and parameter of a lattice in limits $a = 0.420$–0.423, dependent on contents Al in a solution;

– AlN with hexagonal lattice such as P6^3mc with parameters $a = 0.3114$ nm and $c = 0.4979$ nm;

– Si3N4 with hexagonal lattice such as P31C with parameters of lattice and = 0.7754 nm, and with = 0.5622 nm.

Medium-sized calculation of grains of the basic phase on difractograms has shown, that with other things being equal sputtering its value depends on Al concentration in coating and is in limits from 6 up to 20 nm. The least size of grains 6–8 nm is observed in coating 1HC31 at concentration $C_{\mathrm{Ti}} = 15$ at %, $C_{\mathrm{Al}} = 31$ at %, $C_{\mathrm{N}} = 54$ at % obtained at $I_{\mathrm{Ti}} = 2$A and I_{AiS}i = 4.5 A.

Change of superficial layer nanohardness at various values of discharge current difference of magnetrons ($I_{\mathrm{AlSi}} - I_{\mathrm{Ti}}$), is given in Fig. 1.

Under the diagram of nanohardness change in coordinates H_n – ($I_{\mathrm{AlSi}} - I_{\mathrm{Ti}}$), it is possible to notice, that with growth of current difference ($I_{\mathrm{AlSi}} - I_{\mathrm{Ti}}$), that is equivalent to increase of atoms concentration

Table 1. Chemical compound of TiAlSiN the coatings at different values of discharge currents of magnetrons targets from the titanium and AlSi alloy

Sample No.	I_{Ti}, A	I_{AlSi}, A	C_{Ti}, at %	C_{Al}, at %	C_{N}, at %
1HC23	4	2	42	3	55
1HC21	4	3	39	7	54
1HC19	4	4	35	10	55
1HC18	4	4.5	32	13	55
1HC28	3.5	4.5	28	16	56
1HC29	3	4.5	29	15	56
1HC30	2.5	4.5	20	25	55
1HC31	2	4.5	15	31	54

Table 2. Variation of TiAlSiN coating phase composition with discharge currents of magnetrons with Ti and AlSi targets at $p_{\mathrm{N}} = 0.06$, $p_0 = 0.3$ Pa

No.	I_{Ti}, A	I_{AlSi}, A	Phase composition		
			TiAlN, vol. %	AlN, vol. %	Si_3N_4, vol. %
HC23	4	2	100	–	–
HC22	4	2.5	100	–	–
HC21	4	3	93	7	–
HC20	4	3.5	94	6	–
HC19	4	4	79	21	–
HC18	4	4.5	88	12	–
HC17	4	4.8	88	12	–
HC28	3.5	4.5	69	31	–
HC29	3	4.5	51	44	5
HC30	2.5	4.5	49	44	7
HC31	2	4.5	22	61	17
HC35	1	4.5	10	74	16

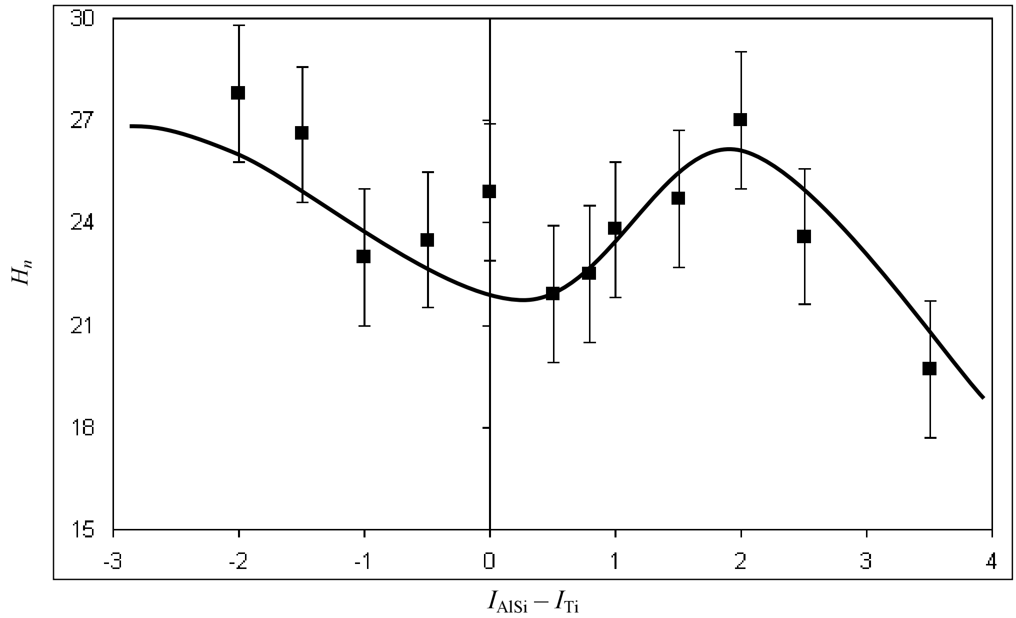

Fig. 1. Variation of nanohardness at change of magnetron discharge currents difference of ($I_{\text{AlSi}} - I_{\text{Ti}}$) with targets from AlSi-alloy and Ti

in coating, nanohardness in the beginning goes down, reaching the minimal value, and then again raises passing through a maximum.

The TiAlSiN coatings considered in the given work have much low mechanical and tribomechanical characteristics in comparison with TiAlN coatings, sputtering by magnetron with target from titanium (57 at. %) and aluminium (43 at. %) alloy. For comparison, we have taken samples with the best tribomechanical characteristics from TiAlSiN set. The comparative data are given in Table 3.

Table 3. Mechanical properties of the TiAlSiN and $Ti_{1-x}Al_xN$ coatings

	Nanohardness H_n, GPa	Microhardness H, GPa	Elasticity module E, GPa
1HC-30	27.1 ± 4.3	22.7 ± 0.3	291 ± 28
1HC-31	23.6 ± 1.5	22.5 ± 0.8	231± 9
$Ti_{1-x}Al_xN$	51 ± 4	43 ± 3	441 ± 25

At the Table 3 it is shown, the TiAlSiN coating nanohardness in ~ 2 times are lower than $Ti_{1-x}Al_xN$.

Apparently, at sputtering from two targets (Ti and AlSi) the coatings form nanolayers and replacement of Ti atoms by Al atoms in TiAlSiN crystal lattice a little.

4. Conclusions

1. Simultaneous deposition by two magnetrons with targets from different materials – from titanium and from of aluminium (91%) and silicon (9%) alloy at temperature 300 ºC nanocomposite coating are obtained consisting of three phases TiAlN, AlN, and Si_3N_4 with the size of grains from 6 up to 20 nm and homogeneous chemical compound on depth of coating.

2. The optimum ratio of sputtering parameters is determined: discharge currents of magnetrons to obtain homogeneous coating with minimal in the size of grain and, in same time, having the highest hardness.

3. Character of dependences of chemical and phase compositions, hardness of nanocomposite coatings of discharge currents of magnetrons ratio is found.

4. At simultaneous deposition of the coatings with use of two magnetrons with targets from titanium and of aluminium (91%) and silicon (9%) alloy replacement of titanium atoms by aluminium atoms in the crystal lattice of titanium nitride has not enough.

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