

# ФИЗИКА РАДИАЦИОННЫХ И ИОННО-ПЛАЗМЕННЫХ ТЕХНОЛОГИЙ

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## COMPOSITION ADJUSTMENT OF VACUUM-ARC Ti-Al-N FILMS, DEPOSITED WITH USE OF TWO-CHANNEL FILTER

*D.S. Aksyonov, I.I. Aksenov, A.A. Luchaninov, E.N. Reshetnyak, V.E. Strel'nitskij*  
*National Science Center "Kharkov Institute of Physics and Technology",*  
*Kharkov, Ukraine*

*E-mail: strelnitskij@kipt.kharkov.ua*

Composite films were formed by vacuum-arc method using two plasma sources equipped with aluminum and titanium cathodes. The sources were coupled with a dual channel T-shaped magnetic filter. Ability of Ti-Al-N films composition adjustment by deposition of mixed plasma streams from the plasma sources was investigated. It has been found that film composition can be varied by changing arc currents in plasma sources. However adjustment range of film composition is rather limited, and can be expanded by changing magnetic field intensity in anode area of plasma sources. Obtained films have uniform composition and thickness on 180 mm diameter surface. Conditions for aluminum content adjustment range from 14 to 60 wt.% were found.

### INTRODUCTION

Multicomponent nitride-based films have higher (as compared to single-component ones) hardness, wear resistance, thermal stability and oxidation resistance. Such films are typically used as protective in high-speed cutting machinery in aggressive environments, where use of coolant and/or lubricant fluids are limited or inadmissible. Composite films usage lowers or eliminates need in said liquids and finally reduces production cost.

Multicomponent films can be produced by vacuum arc method using cathodes with predefined composition, corresponding to the desirable film composition. But such cathodes manufacturing is complicated, expensive and often is not possible. Composition of the obtained films can be changed only by changing the cathode itself. These circumstances make difficulties in experimental search of new film compositions.

Composite films can be obtained by simultaneous deposition of plasma streams from several plasma sources equipped with cathodes made of different (pure) materials. Single-channel [1, 2] or multichannel [3, 4] plasma filters can be used for this purpose. Plasma

source with a single-channel filter has one common anode and plasma duct and its cathodes are placed in vicinity to each other [1, 2]. Plasma streams from each cathode are being transported in common longitudinal magnetic field and so they are mixed weakly. This results in highly nonuniform composition of deposited films.

In multichannel systems plasma streams can be transported separately up to common output section. Here, each of them is transported by its "own" group of magnetic lines of force parallel to each other, so streams mixing degree is nearly none. As a result, such systems also can not provide a uniform distribution of film components. Thus, deposition of composition-uniform films is doubtful without using any additional mixing devices (homogenizers). Known mixers are not well studied; either they can not provide sufficient plasma mixing degree [3] or have material-demanding and difficult-to-made design [5, 6].

Previously [7] we have shown the ability to deposit Ti-Al-N films using separate plasma streams mixing. These films have uniform composition and thickness on 180 mm diameter surface. Aluminum content is about 40 wt.%. Two-channel T-shaped plasma filter was used for film deposition [8]. Magnetic system of the filter allows mixing of separate plasma streams without additional complications of the system design. Objective of this work is to investigate the system capabilities to deposit films with controllable composition and with uniform composition and thickness uniformity on 180 mm diameter surface.

### EXPERIMENTAL DETAILS

Investigated system is schematically shown in Fig. 1. It has two plasma sources 1 and 2, each of them respectively consists of cathode C1 and C2, anode A1 and A2, stabilizing coil S1 and S2 and focusing (anode) coils F11, F12 and F21, F22. The plasma sources are axially arranged and are attached to T-shaped plasma

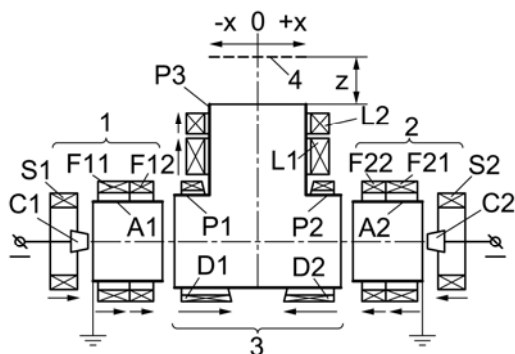


Fig. 1. Scheme of T-shaped magnetic filter equipped with two plasma sources

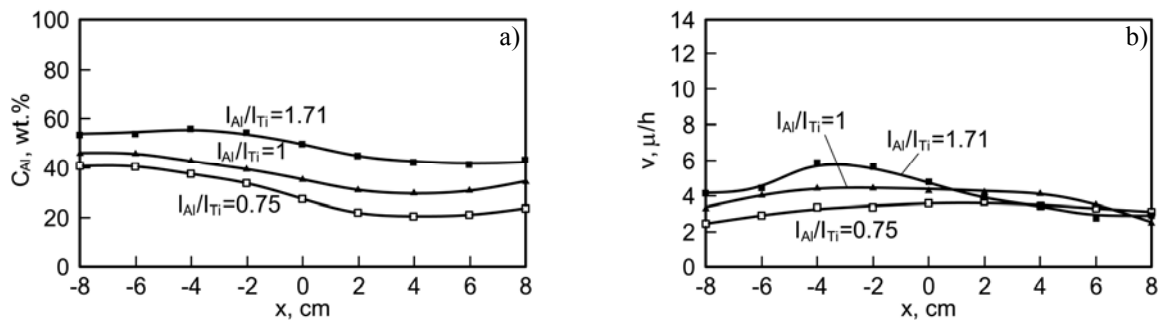


Fig. 2. Radial distribution of film aluminum content (a) and deposition rate (b) for different arc currents ratios.  $z = 25 \text{ mm}$ ,  $I_{F12} = I_{F22} = 0,5 \text{ A}$

duct 3 input sections P1 and P2. Filter output section P3 is located between input sections at the right angle to them. Input sections contain deflecting coils D1 and D2, output section contains two output coils L1 and L2. Plasma filter 3 has its output section attached to vacuum chamber of "Bulat-6" apparatus (not shown). Substrate holder 4 is placed away from magnetic filter output section on distance  $z$ . The system was described in more detailed way earlier [8].

Cathode C1 is made of aluminum and cathode C2 – of titanium. Films were deposited on polished  $20 \times 17 \times 1 \text{ mm}$  sized molybdenum samples.

Nine samples were used in each experiment. They were arranged in one row (along  $x$  in Fig. 1) with 20 mm offset.

Distance  $z$  between substrate holder 4 and system output section was 25 mm and 100 mm. The system was pumped out to  $2 \cdot 10^{-5}$  Torr residual pressure before each deposition cycle. Films deposited in nitrogen environment at 3 mTorr pressure level. Stabilizing, deflecting, output and focusing coils F11 and F21 had fixed current values, which respectively were  $I_{S1} = I_{S2} = 1.5 \text{ A}$ ,  $I_{F11} = I_{F21} = -0.4 \text{ A}$ ,  $I_{D1} = I_{D2} = 2 \text{ A}$ ,  $I_{L1} = 4 \text{ A}$ ,  $I_{L2} = -3 \text{ A}$ . Coil currents, which generate magnetic field directed accordingly to arrows in Fig. 1 will be mentioned hereafter as positive and vice versa. Films composition was controlled by means of discharge currents ratio  $I_{Al}/I_{Ti}$  variation in range from 0.75 to 2.14. Anode coils F12 and F22 current value and polarity were changed from  $-1$  to  $+0,5 \text{ A}$  to control plasma streams transport efficiency. Aluminum cathode arc current  $I_{Al}$  was changed in range 75...150 A, and titanium cathode arc current  $I_{Ti}$  – in range 70...100 A. Substrate was at floating potential during film deposition process.

Aluminum and titanium content (nitrogen content was not taken into account) were determined by X-ray fluorescent analysis using "SPRUT" spectrometer. Optical interferometer "MII-4" was used to determine film thickness. Eight measurements were made for each sample to determine film thickness: four at the top sample area and four – at the bottom one. Obtained thickness data were averaged.

## RESULTS AND DUSCUSSION

Earlier performed investigation [7] has shown that changing of such deposition process conditions as nitrogen pressure, negative substrate bias, magnetic field intensity at the system output and output-to-

substrate distance ( $z$ ) does not involve significant change of aluminum to titanium component ratio of the films. The conditions adjustment leads to components redistribution through the coating area. It is obvious that arc current variation will influence amount of plasma emitted by cathode spot. Thus, film elemental composition can be affected by controlling discharge currents ratio.

Such composition control technique during film deposition process in multiple-cathode systems was shown previously (see, for example, [3, 9]). Arc currents ratio influence on radial (along  $x$  in Fig. 1) distributions of film thickness and composition is shown in Fig. 2.a. It can be seen that current ratio change from 0.75 to 1.71 does not provide significant modification of Ti-Al-N film composition. Aluminum content value varies from 30 to 49 wt.%. Content range widening can be achieved by enhancement of available arc currents range, but it is not always possible as will be mentioned below.

Arc currents ratio unbalancing ( $I_{Al}/I_{Ti} \neq 1$ ) shifts plasma stream peak density towards plasma source with higher arc current. It can be observed in Fig. 2.b. Measurements were taken in the vicinity of the output section ( $z = 25 \text{ mm}$ ) where plasma streams are not completely mixed yet.

Regulation curves for the investigated system, adopted from work [9] and calculated from data presented in work [3] are shown in Fig. 3. A regulation curve reveals arc currents ratio change, which is needed to attain the desirable film components ratio. The higher the slope of regulation curve the more affect of arc currents ratio alteration on film composition. Shift of the curve indicates offset of available adjustment range.

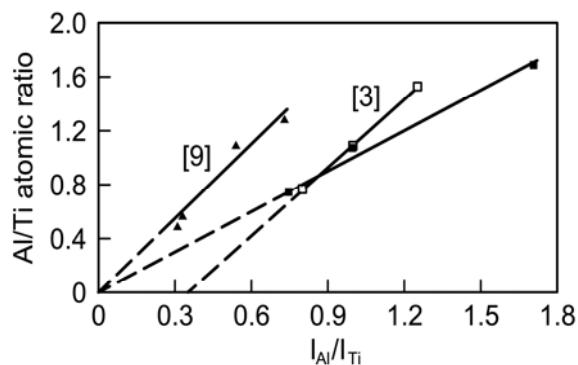


Fig. 3. Regulation curves of two-cathode systems

However, restrictions applied by power supplies and vacuum-arc maintenance or/and stability conditions make a wide range of component ratios unreachable.

That is, for Ti-Al-N film fabrication with 20 at.% of aluminum using the investigated system, titanium cathode arc current value must be 4 times higher than aluminum one. If arc currents values will not exceed available range for the system (see above), they will be 75 A for aluminum and 300 A for titanium or 25 A and 100 A respectively. For Y-shaped system [3] the difference is only 2 times, and so it can be implemented without any difficulties.

For the purpose of film composition adjustment range widening, it is necessary to additionally influence on plasma streams intensity or transporting efficiency, emitted by each of the cathodes or transported from them to deposition site. It can be supposed that magnetic field symmetry unbalancing (relatively to the system output axis) should significantly affect transporting capabilities of the system. That is, changing the intensity of magnetic field inside one of the anodes will change corresponding component transport conditions thereby changing intensity of plasma stream, which leaves plasma duct input section and reaches the system output. Dependencies demonstrated in Fig. 4,a prove that controllable reduction of one of the plasma streams intensity can be achieved by simultaneous unbalancing of arc currents and magnetic field symmetry. Thereby additional magnetic field symmetry unbalancing is extending available film compositions range. In our case adjustment of aluminum content becomes possible in range from 13 to 67 wt.% if arc currents ratio changed from 0.75 to 2.14 along with changing focusing coils currents. For minimum aluminum concentration in deposited film coil F12 current was changed from +0.5 to -1 A. For maximum aluminum concentration coil F22 current was changed from +0.5 to -1 A. Such approach reduces arc power supplies requirements: when magnetic field inside anode is weakened discharge voltage drop decreases, as a result - less powerful supply is needed to maintain arc discharge.

Moreover, decreased discharge power dissipation lowers heat load for corresponding anode and thereby reduces requirements for cooling system.

It should be noted that considered method of composition adjustment range expansion has unwanted side effect. Deposited films have significantly much less thickness uniformity (Fig. 4,b). Change of aluminum content permits thickness peak appearance and its growth. This peak is shifted towards plasma source with aluminum cathode for high aluminum content films and to the opposite side for low aluminum content. It is connected with plasma density maximum growth at the side of plasma source with increased arc current (see also Fig. 2,b) and magnetic field asymmetry, such shift is typical to knee-shaped filters having said asymmetry. The way to reduce this effect will be discussed further.

Nonuniformity of films thickness is greater for higher values of  $I_{Al}/I_{Ti}$  ratio. It can be explained by cathodes materials (aluminum and titanium) erosion rates differences [10]. This assumption may be supported by fact, that increase of total arc current ( $I_{Al} + I_{Ti}$ ) lowers integral deposition rate of the film. For curves shown in Fig. 2,b for 0.75, 1.0 and 1.71  $I_{Al}/I_{Ti}$  ratios, total arc currents are equal to 210, 200 and 190 A respectively.

If one assumes that specific erosion rates of cathode materials are equal, then alteration of total arc current should lead to proportional change of integral deposition rate. However, changing of total current on +5 and -5% changes integral deposition rate on -17 and +7.5% respectively (Fig. 2,b), and the current change by +10 and -12.5% changes integral deposition rate by +120 and -40% respectively (Fig. 4,b). In addition, increase of total arc current can either lower integral deposition rate (Fig. 2,b) or elevate it (Fig. 4,b). It means that integral deposition rate is dependent on both arc current ratio and their sum. Thus, aluminum and titanium have different erosion rates, which agree with existing data [10]. However, such strong dependence shown in experiments is unlikely the result of that relatively small difference between erosion

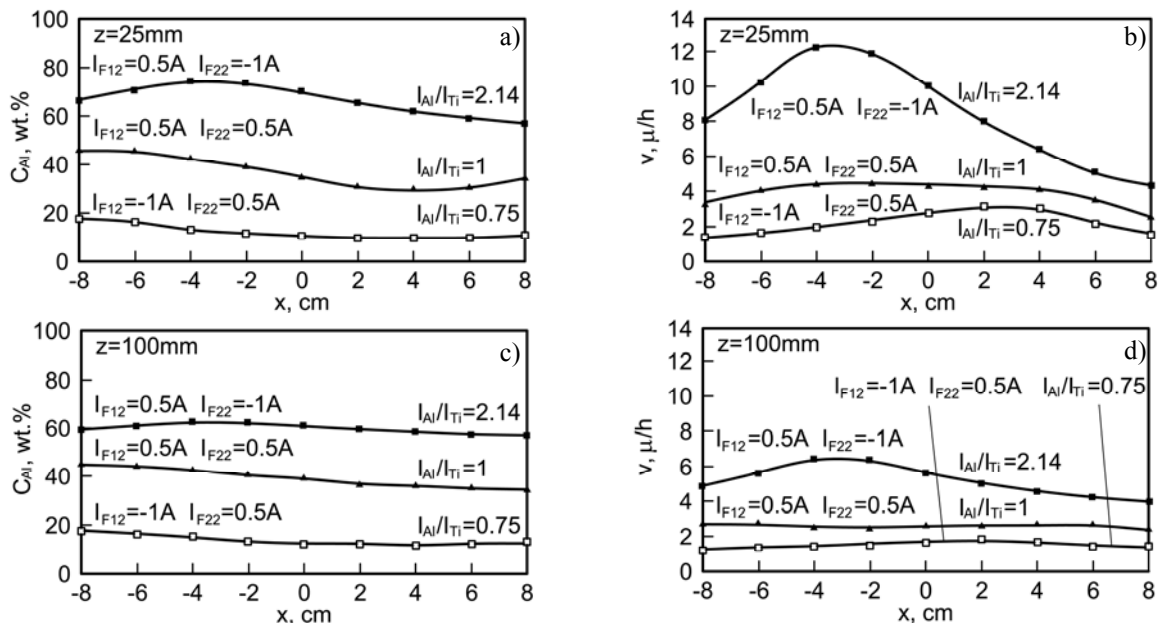


Fig. 4. Radial distribution of aluminum content (a, c) and deposition rate (b, d)

rates of aluminum and titanium, which are given in work [10].

To establish the mechanisms which are underlying discussed "contradictions" further study is required.

Increasing arc current in one of the plasma sources and total arc current, leads to increase of plasma stream density in corresponding channel and system output (from the side of this channel). Homogenization of plasma stream in this case requires extended (along system output axis) space with weakened magnetic field. It can be achieved by increasing the distance ( $z$ ) between output of plasma duct and substrate position.

From the practical application viewpoint of the investigated system, results obtained at distance  $z$  greater than 25 mm are more interesting. The measurement data shown in Fig. 4,c,d and earlier results [7] indicate that with increasing of distance  $z$ , deposited films become significantly more uniform in thickness and composition. It should be noted however, that deposition rate in this case notably decreases. Reasons of the phenomenon were discussed in previous work [7]. It can be clearly seen from Fig. 4,c that aluminum content adjustment range narrows with distance  $z$  increasing. At  $z = 100$  mm the range becomes 14...60 wt.% against 13...67 wt.% at  $z = 25$  mm.

Such adjustment range narrowing can be explained in the following way. Radial distribution of Al and Ti plasma stream components is not perfectly homogeneous: peripheral part of the stream close to plasma source with Al cathode is more aluminum-rich and vice versa. In the magnetic field with nearly zero intensity (inside vacuum chamber) plasma stream is dissipating and its peripheral part does not get to the substrate. If the stream is not centred on the system output axis then loses of its peripheral parts are equal (if the stream is symmetric about the axis). When plasma stream is shifted towards plasma source with Al cathode, the amount of Al-rich part losses become higher and Ti-rich – lower. As a result, amount of Al component reaching the substrate becomes lower too. Opposite plasma stream shift (towards plasma source with Ti cathode) for similar reasons produces opposite effect – aluminum concentration in deposited films increases.

## CONCLUSION

Possibility of Ti-Al-N films fabrication by simultaneous deposition of aluminum and titanium plasma streams from two vacuum-arc plasma sources using two-channel T-shaped magnetic filter was studied. It was shown, that Al and Ti film composition adjustment using traditional means – by changing plasma sources arc current ratio is possible in relatively small range: Al content can be changed from 30 to

49 wt.%. Adjustment range is limited by available arc currents values and vacuum-arc burning/stability conditions.

Range of film components content adjustment can be significantly widened by changing magnetic field intensity and geometry inside anodes of the plasma sources. Use of such method in discussed conditions has given an ability to deposit content-uniform films on 180 mm diameter substrate with adjustable aluminum concentration in range 14...60 wt.%.

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## **РЕГУЛИРОВКА СОСТАВА ВАКУУМНО-ДУГОВЫХ Ti-Al-N-ПОКРЫТИЙ, ОСАЖДАЕМЫХ С ИСПОЛЬЗОВАНИЕМ ДВУХКАНАЛЬНОГО ФИЛЬТРА**

*Д.С. Аксёнов, И.И. Аксёнов, А.А. Лучанинов, Е.Н. Решетняк, В.Е. Стрельницкий*

Исследована возможность регулировки состава Ti-Al-N-покрытий, получаемых вакуумно-дуговым методом путём смешения потоков плазмы от генераторов с титановым и алюминиевым катодом. Фильтрация потоков осуществлялась с помощью общего для обоих генераторов Т-образного двухканального фильтра. Установлено, что изменения содержания алюминия и титана в покрытии можно достичь путём регулировки соотношения разрядных токов в генераторах плазмы. Увеличение диапазона регулировки состава покрытия достигается путём подбора интенсивности магнитных полей в анодных секциях генераторов плазмы. Полученные покрытия однородны по составу на подложке диаметром 180 мм. Определены условия, позволяющие регулировать концентрацию алюминия в пределах от 14 до 60 вес. %.

## **РЕГУЛЮВАННЯ СКЛАДУ ВАКУУМНО-ДУГОВИХ Ti-Al-N-ПОКРИТТІВ, ОСАДЖУВАНИХ З ВИКОРИСТАННЯМ ДВОКАНАЛЬНОГО ФІЛЬТРА**

*Д.С. Аксёнов, І.І. Аксёнов, О.А. Лучанінов, О.М. Решетняк, В.Є. Стрельницький*

Досліджено можливість регулювання складу Ti-Al-N-покріттів, які отримано вакуумно-дуговым методом шляхом змішування потоків плазми від генераторів із титановим та алюмінієвим катодом. Фільтрація потоків здійснювалась за допомогою спільного для обох генераторів Т-подібного двоканального фільтра. Встановлено, що зміни вмісту алюмінію та титану в покритті можливо досягти шляхом регулювання співвідношення розрядних струмів генераторів плазми. Збільшення діапазону регулювання складу покриття досягається шляхом підбору інтенсивності магнітних полів в анодних секціях генераторів плазми. Отримані покриття однорідні за складом на підкладці діаметром 180 мм. Визначено умови, які дозволяють регулювати концентрацію алюмінію у межах від 14 до 60 ваг. %.