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# INFLUENCE OF THE PUMPING SPEED ON V-A CHARACTERISTICS OF THE DC MAGNETRON DISCHARGE IN AN Ar—$N_2$ MIXTURE[1])

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In the DC magnetron sputtering of titanium in an Ar + $N_2$ mixture the V-A characteristics of the discharge were measured and the influence of the pumping speed on their shape was studied. There is a hysteresis loop in the characteristics at low pumping speeds. At high pumping speeds the hysteresis disappears.

## I. Introduction

DC planar magnetrons have been used for thin film deposition of different materials. Compared to the diode sputtering their advantage is in the possibility of sputtering the cathode with a high current density while the discharge voltage is several undreds volts. This fact is closely related to the typical shape of V-A characteristics of the magnetron discharge [1].

In the reacative magnetron sputtering the reactive gas (e.g. nitrogen) forms a compound with the cathode material (e.g. titanium) not only on the substrates but also on the cathode itslef. This effect causes a decrease of the sputtering yield of the cathode material. Changes of the flow rate of nitrogen gettered by the sputtered titanium cause changes of the partial pressure of nitrogen and it reacts back on the covering of the target by the nitride. That is why the hysteresis effect can be observed in the reactive sputtering with two stable regimes (termed metallic and nitride modes) differing by the coverage of the target by the nitride. The history of the process determine which mode is present [2]. In the metallic mode the coverage of the target by the nitride is low, the sputtering yield is high and the paratial pressure of nitrogen is low. In the nitride mode the target is covered by the reaction product, the sputtering yield is lower and the partial pressure of nitrogen is high. Jumpings between the two modes occur at the boundaries of the hysteresis loop. The hysteresis effect can be undersirable because it can prevent forming films with proper stoichiometry.

In the reactive sputtering the shape of the V-A characteristics is influenced by the presence of the reactive gas. The characteristics are strongly modified compared to those observed in pure inert gas especially when a hysteresis loop occurs in the characteristics [2, 3].

This paper presents V-A characteristics of the DC planar magnetron in the reactive sputtering of titanium in an Ar-$N_2$ atmosphere and compares the characteristics with the behaviour of the partial pressure of nitrogen for different pumping speeds.

## II. EXPERIMENTAL ARRANGEMENT

The experiments were made in a device schematically shown in Fig. 1. The deposition chamber is evacuated by means of an oil diffusion pump (2000 $ls^{-1}$).

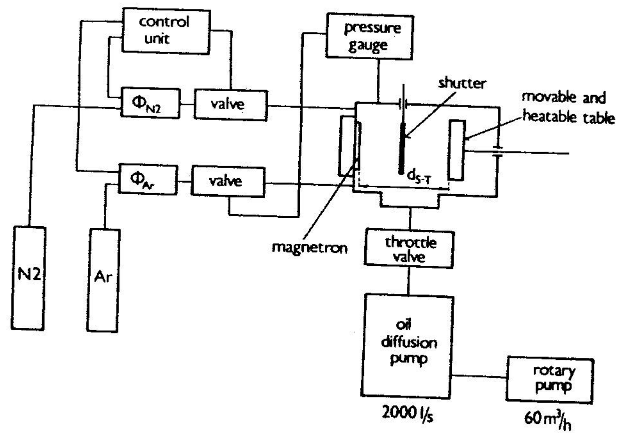


Fig. 1. Schematic illustration of the experimental device.

The pumping speed was varied by a throttle valve placed between the diffusion pump and the deposition chamber. The deposition process was carried out at a constant total pressure $p_T$ measured by means of a high pressure triode ionization gauge:

$$p_T = k_{N2}p_{N2} + k_{Ar}p_{Ar} \qquad (1)$$

where $k_{N2}$, $k_{Ar}$ are the sensitivies of the gauge for both gases, $p_{N2}$, $p_{Ar}$ are their partial pressures. The amount of $N_2$ in the gas mixture was controlled by setting

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up the ratio $\varphi = \Phi_{N2}/\Phi_{Ar}$, i.e. the ratio of flow rates of nitrogen $\Phi_{N2}$ and argon $\Phi_{Ar}$. For this control the automatic MKS pressure/flow controller, equipped with mass flow meters and electronic valves, was used.

The diameter of the planar circular magnetron used was 120 nm. The target was made from Poldi Titan 45 (99.5%). Experiments were made in the arrangement shown in Fig. 2. The electromagnet current $I_m$, the total pressure $p_T$ and the ratio of flow rates $\varphi$ in the experiments were kept constant.

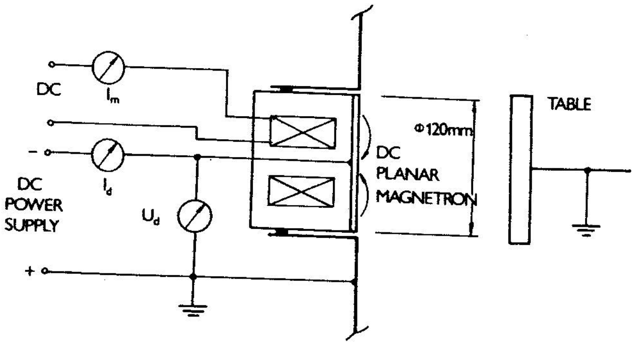


Fig. 2. Experimental arrangement used for measuring the V-A characteristic.

## III. RESULTS AND DISCUSSION

The V-A characteristics of the magnetron discharge, i.e. the function of the discharge voltage $U_d$ vs. the discharage current $I_d$, are shown in Fig. 3. Constant parameters are $p_T = 0{,}9$ Pa, $I_m = 2$ A, the pumping speed for nitrogen $S_{N2} = 91\ \mathrm{ls}^{-1}$. When $\varphi = 0$, the V-A characteristic has a shape typical for a magnetron discharge in an inert gas atmosphere. If nitrogen is mixed with argon ($\varphi > 0$), then the V-A characteristics fall into three parts. In the metallic part of the characteristic corresponding to comparatively high currents $I_d$ the curve is near to that observed when $\varphi = 0$. In the nitride part of the characteristic, with low currents $I_d$, the discharge voltage is higher than without nitrogen. A transition region lies between these two parts of the characteristic. The transition region is shifted towards higher currents when the ratio $\varphi$ is increased. The shift indicates that the shape of V-A characteristics is influenced by covering the target by the reaction product and by changes of the nitrogen partial pressure.

To confirm this statement we have suggested an experiment in which the partial pressure $p_{N2}$ with the discharge was measured simultaneously with the V-A characteristics. The determination of $p_{N2}$ is based on measuring the argon



flow rate without nitrogen $\Phi_{Ar}(0, I_d)$ and with nitrogen $\Phi_{Ar}(\varphi, I_d)$. Equilibrium state in the sputtering chamber is described by the following formulae:

$$\Phi_{Ar} = p_{Ar}S_{Ar} + {}^{s}\Phi_{Ar} \tag{2}$$

$$\Phi_{N2} = p_{N2}S_{N2} + {}^{s}\Phi_{N2} \tag{3}$$

where $S_{N2}$ and $S_{Ar}$ are the pumping speeds of the vacuum system including the throttle valve and ${}^{s}\Phi_{Ar}$, ${}^{s}\Phi_{N2}$ are the flow rates of gases gettered by the sputtered titanium.

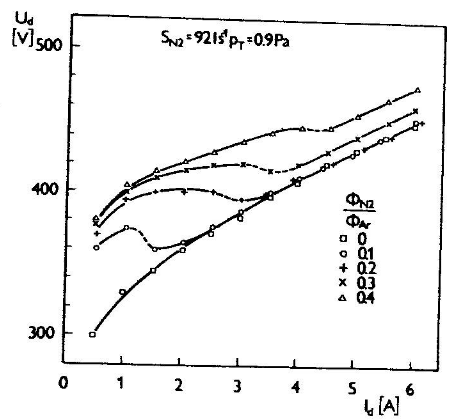


Fig. 3. A set of V-A characteristics of the magnetron where the ratio of flow rates $\varphi = \Phi_{N2}/\Phi_{Ar}$ is a parameter.

Using the same method as in ref. [5] the partial pressure of nitrogen $p_{N2}$ can be determined with the help of the following formula

$$p_{N2}(I_d) = \frac{1}{hS_{N2}}(\Phi_{Ar}(0, I_d) - \Phi_{Ar}(\varphi, I_d)) \tag{4}$$

where

$$h = \frac{k_{N2}}{k_{Ar}}\frac{S_{Ar}}{S_{N2}} = 0.55.$$

The comparison of $U_d$, $\Phi_{Ar}$ and $p_{N2}$ as functions of the discharge current $I_d$ is shown in Fig. 4 with constant parameters $S_{N2} = 41\ \mathrm{ls}^{-1}$, $p_T = 2$ Pa and $I_m = 2$ A. This detailed measurement shows a hysteresis loop in the V-A characteristics when $\varphi = 0.1$. Similar loops can be seen in the functions $\Phi_{Ar}$ and $p_{N2}$ vs. $I_d$, too. Moreover, the jumps between both parts of the curves $U_d(I_d)$, $\Phi_{Ar}(I_d)$ and $p_{N2}(I_d)$ occur at the same discharge currents $I_d$. This fact confirms the hypothesis that a close relation exists between the gettering effects and the shape of the V-A characteristics.

In the metallic part of the functions ($\varphi = 0.1$ in Fig. 4) the partial pressure $p_{N2}$ is low and the target coverage by the reaction product is low, too. Therefore the discharge voltage $U_d$ is nearly the same as without nitrogen ($\varphi = 0$). On the other hand, in the nitride mode the partial pressure $p_{N2}$ is comparatively high and the taraget is strongly covered by the nitride. In this mode the voltage $U_d$ is higher than in pure argon.

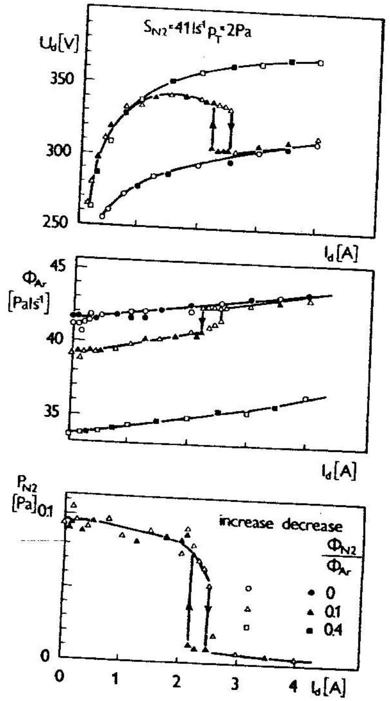


Fig. 4. Discharge voltage $U_d$, argon flow rate $\Phi_{Ar}$ and nitrogen partial pressure $p_{N2}$ as functions of the discharge current $I_d$ at a low pumping speed of the vacuum system.

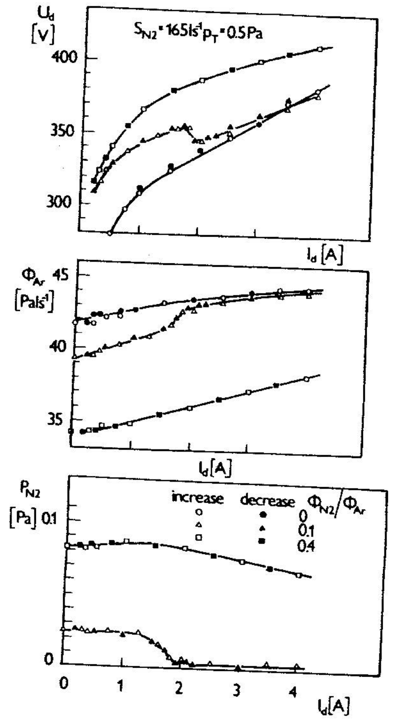


Fig. 5. The same functions as in Fig. 4 at a high pumping speed of the vacuum system.

The increase of $U_d$ in the reactive mode is typical for TiN. Some other materials behave in an opposite manner, e.g. AlN and $SiO_2$ [3, 4]. The covering of the taraget by the reaction product causes a change in the secondary emission yield of the target and this fact is probably the basic reason for the difference in the discharge voltage between metallic and nitride modes. Effects occurring in the gas phase have to be considered, too, but the influence of these effects on $U_d$ is probably weaker. Our measurements confirm this statement. When $I_d < 1$ A, the values of $U_d$ obtained for $\varphi = 0.1$ and $\varphi = 0.4$ are nearly equal, although the partial pressures $p_{N2}$ differ by a factor of 4 (see Fig. 4).



The hysteresis effect in the reactive sputtering is closely related to the pumping speed of the vacuum system. If the pumping speed is increased above a particular critical value, then the hysteresis loop disappears [5]. In Fig. 5 it is shown that the same effect is observable also in the functions of the partial pressure $p_{N2}$, the flow rate $\Phi_{Ar}$ and the voltage $U_d$ vs. the discharge current $I_d$. Here the pumping speed $S_{N2}$ is increased to 165 ls$^{-1}$; $p_T = 0.5$ Pa, $I_m = 2$ A. In Fig. 5 it can be seen that if the pumping speed is sufficiently high, then all the three functions are unambiguous and without jumps. However, also in this case the transition between the nitride and metallic parts of the V-A characteristic corresponds to a region of a fast change in $p_{N2}$, which is in an indication of a fast change in the target coverage by the nitride.

## IV. CONCLUSION

The V-A characteristics of the DC magnetron discharge in the reactive sputtering of titanium in the Ar-$N_2$ gas mixture indicate the state of coverage of the target by the reaction product. The hysteresis loop observed in the characteristics can bring valuable information about the state of the system, especially when the measuring of the partial pressure of the reactive gas is not available. Moreover, the shape of the V-A characteristics shows what the pumping speed is, if higher or lower than the critical one.


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# ВЛИЯНИЕ СКОРОСТИ НАКАЧКИ НА ВОЛЬТ-АМПЕРНЫЕ ХАРАКТЕРИСТИКИ РАЗРЯДА В СМЕСИ $Ar + N_2$, ПРОИСХОДЯЩЕГО В МАГНЕТРОНЕ ПОСТОЯННОГО ТОКА

В работе приводятся результаты измерений вольт-амперных характеристик разряда и изучено влияние скорости накачки на их форму при распылении титана в смеси $Ar + N_2$, которое происходило в магнетроне постоянного тока. Обнаружено, что при низких скоростях накачки в вольт-амперных характеристиках существует петля гистерезиса. Эта петля гистерезиса при высоких скоростях накачки исчезает.