

REACTIVE SPUTTERING OF TiN_x -FILMS BY MEANS OF CYLINDRICAL POST-MAGNETRON¹⁾

DUDAŠ, J.²⁾ Prague

The contribution presented describes a d.c. magnetron sputtering system with a spool-shaped Ti-cathode. Conditions of the reactive deposition of TiN_x -films and properties of these films are introduced. 100.

I. INTRODUCTION

TiN_x -films have a wide application field in industry as a wear resistant coating of machine parts and cutting tools [1], as a barrier layer in ohmic contacts of semiconductor elements [2], as anticorrosive coating [3], etc. For this reason a great attention is devoted to the deposition technology of TiN_x -films and their properties [4, 5].

II. EXPERIMENTAL

Some of the properties of d.c. magnetrons with a spool-shaped cathode were referred to by K i r o v [6] and T h o r n t o n [7]. The sputtering system we used (see Fig. 1) was placed inside a glass cylindrical vacuum chamber (160 mm i.d.). A magnetic field parallel to the cylindrical surface of the magnetron cathode was produced by coils placed outside the chamber. The water cooled cathode was the Poldi titan 45 (99.5% titanium) had the following dimensions: the post diameter 12 mm, the active length 13 mm, the diameter of the ending discs 17 mm. The Cu-sample holder was heated by a resistor wire and cooled by air. Before the deposition the chamber was evacuated by means of an oil diffusion pump ($2 \text{ m}^3/\text{s}$) to a pressure of about 10^{-4} Pa. The purity of both $Ar + N_2$ gases used in our experiments was 99.99%. The system enabled to clear the cathode by ionic bombardment before the deposition.

¹⁾ Contribution presented at the 6th Symposium on Elementary processes and Chemical Reactions in Low Temperature Plasma, JELŠAVA-HRÁDOK, June 9--13, 1986

²⁾ Institute of Plasma Physics, Czechoslovak Academy of Sciences, Pod Vodárenskou věží 4 182 11 PRAHA 8, Czechoslovakia

The degree of ionization was determined from the ionic part of the cylindrical probe characteristics after [8]. The heat flow to the substrate was determined from the temperature increase of a flat probe with small heat capacity after [9]. Microhardness of the films was measured by the Vickers indentor, electrical resistivity was determined by means of a 4-point probe and the intrinsic stress of the films was determined from the bending of flat coated substrates.

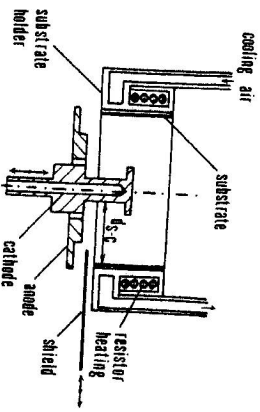


Fig. 1. Scheme of a magnetron sputtering system with a spool-shaped cathode.

III. RESULTS

The region of the stable magnetron discharge is shown in Fig. 2. If the magnetic field decreased below a certain value the magnetron discharge was interrupted abruptly or the discharge current began to oscillate.

The degree of ionization in the discharge plasma reached the values of up to $\alpha = 2 \times 10^{-3}$ at the distance of 7 mm from the cathode surface. It increased proportionally with the discharge power P_d and decreased rapidly with the increasing pressure p_{Ar} (Fig. 3).

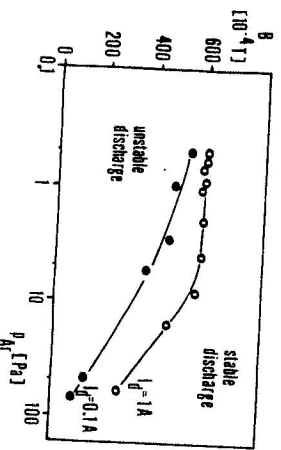


Fig. 2. Region of stable and unstable magnetron discharge for two different discharge currents I_d in the diagram of the magnetic fields B vs. argon pressure p_{Ar} .

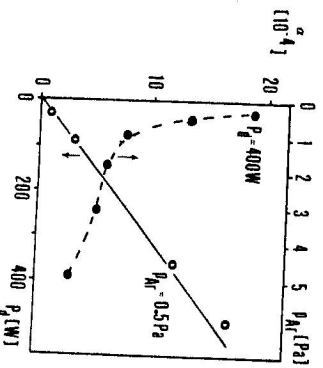


Fig. 3. Degree of ionization α of the magnetron discharge plasma vs. discharge power P_d and argon pressure p_{Ar} , 7 mm from the cathode surface.

The heat flow to the substrate is a parameter, which has great influence on the structure and other properties of the deposited films. It is caused by energetic particle bombardment and by discharge plasma radiation. Its value was 70 eV per condensing titanium atom in the nonreactive deposition of Ti-films ($p_{Ar} = 0.5$ Pa), it increased to 130 eV/at.Ti with the substrate bias $U_s = -50$ V (against the anode) and increased to the value of 300 eV/at.Ti in the reactive deposition of TiN_x-films in the atmosphere of pure nitrogen $p_{N_2} = 0.5$ Pa. The value of the heat flow did not depend on the discharge power and the cathode-to-substrate distance in agreement with [9].

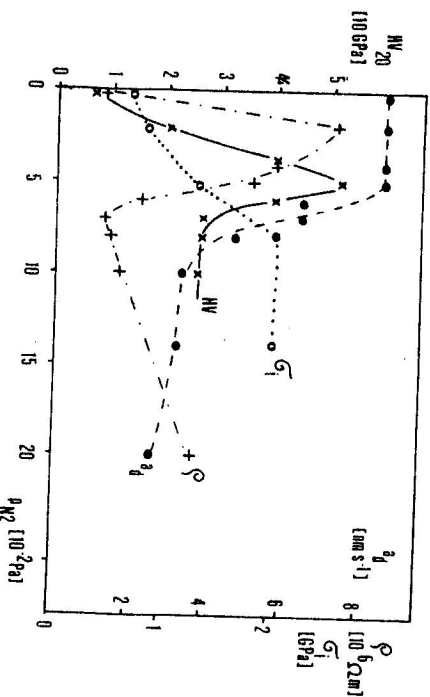


Fig. 4. The deposition rate a_d , microhardness HV_{20} , resistivity ρ and intrinsic stress σ_i vs. nitrogen partial pressure p_{N_2} for TiN_x-films on silicon. $I_d = 450$ W, $P_{in} = P_{Ar} + P_{N_2} = 0.5$ Pa, $d_{c-s} = 18$ mm, $B = 0.89$ T, substrate temperature $T_s = 473$ K.

The properties of reactively sputtered TiN_x-films strongly depend on the deposition conditions. The influence of individual parameters on the structure and other properties of these films was studied in previous works [10, 11].

The dependence of the most important parameters (deposition rate a_d , microhardness HV_{20} , resistivity ρ , intrinsic stress σ_i) on partial nitrogen pressure p_{N_2} for TiN_x-films on silicon substrates is given in Fig. 4. The shape of dependences $HV = f(p_{N_2})$ and $\rho = f(p_{N_2})$ is determined by the changes in the composition and the structure of the films caused by the changes of p_{N_2} . The high values of the compressive intrinsic stress are given by the atypical deposition conditions, where the substrate is close to the cathode surface ($d_{c-s} = 18$ mm) and is intensively bombarded by argon energetic neutrals reflected from the cathode surface.

IV. CONCLUSION

The described magnetron sputtering system is suitable for the coating of hollows. It is characterized by a high degree of ionization (10^{-3}) and high heat flows to the substrate (10^1 — 10^2 eV/at Ti). Reactively deposited TiN_x-films are characterized by great compressive intrinsic stress ($\approx 10^9$ Pa). The TiN_x-films deposited by this system gave good results as wear protective coatings on parts of textile machines.

ACKNOWLEDGEMENT

The author would like to thank Dr. J. Musil for many useful discussions.

REFERENCES

- [1] Nimmagada, R. R., Doerr, H. J., Bunschah, R. F.: *Thin Solid Films* 84 (1981), 303.
- [2] Noël, J. P. et al.: *J. Vac. Sci. Technol.* 42 (1984), 284.
- [3] Ramalingam, S., Winer, W. O.: *Thin Solid Films* 73 (1980), 267.
- [4] Schiller, S., Beister, G., Sieber, W.: *Thin Solid Films* 111 (1984), 259.
- [5] Sundgren, J. E., et al.: *Thin Solid Films* 105 (1983), 353.
- [6] Kirlov, K. I. et al.: *Vacuum* 26 (1976), 237.
- [7] Thornton, J. A.: *J. Vac. Sci. Technol.* 15 (1978), 171.
- [8] Kagan, Ju. M., Perel, V. I., Ripatt, P. O.: *Vest. len. univer., Ser. mat. fiz. chim.* 10 (1955), 129.
- [9] Thornton, J. A.: *Thin Solid Films* 54 (1978), 23.
- [10] Musil, J. et al.: *Proc. of 5th Intef. Conf. IPAT 85*. Munich 1985.
- [11] Dudaš, J., Musil, J.: *Czech. J. Phys.* (in press).

Received August 6th, 1986.

НАНЕСЕНИЕ ТОКНИХ ПЛЕНОК TiN_x МЕТОДОМ РЕАКТИВНОГО РАСПЫЛЕНИЯ В ЦИЛИНДРИЧЕСКОМ МАГНЕТРОНЕ СО ШТЫРЕМ

Данная работа описывает магнетрон постоянного тока для реактивного распыления с катушкообразным тигановым катодом. Сформулированы также условия для нанесения тонких пленок TiN_x методом реактивного распыления и описаны свойства этих пленок.