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Characterization of surface of TiN/CrN and TiN/NbN superlattices deposited on microelectronic materials using Atomic Force Microscopy

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In this paper we present the 3D images topography of TiN/NbN and TiN/CrN superlattices deposited by dc reactive magnetron sputtering on a <100> oriented monocrystalline Si surface and Si coated 200 nm thick Si_3N_4 film.

The Atomic Force Microscope (AFM) was used in these surface topography studies. The computer program Surface View was used to define the parameters of the studied surfaces. The 3D AFM images were constructed using 128x128 points matrix. The scan area was 950 x 950 nm up to 1650 x 1650 nm.

The TiN/CrN superlattices characterize with better flatness of the surface than TiN/NbN. The TiN/CrN/Si_3N_4/Si composite has distinctly larger grains than TiN/CrN/Si composite and exhibits better uniformity of the hardness distribution.

The TiN/CrN/Si_3N_4/Si superlattice has the highest kurtosis and the lowest skewness values. This composite demonstrates the best tribological behaviour.

Keywords: Superlattices, AFM, topography, MEMS.

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I. Introduction

One of the newest, introduced recently interesting ideas of coating technology are multilayer films, which are composed of two or more different materials deposited in layers of the thickness of single layer in the range 2-5 nm. Such multilayer structures called superlattices demonstrate entirely different behaviour than one layer solid films [1]. The studies of the tribological behaviour of such coatings demonstrate that they are superior in wear resistance, cracking and strength as compared with the one layer films [2, 3].

These novel physical properties of superlattices open new possibilities of their application in the construction of Micro Electro Mechanical Systems (MEMS) devices where very important is wear resistance of rubbing elements. The application of such resistant coatings seems also to be interesting in contact-mode operation of Atomic Force Microscope (AFM) to protect the tip of cantilevers against rapid wear. The mechanical properties, mainly hardness and wear resistance of typical microelectronic materials as Si, SiO_2, Si_3N_4 used in MEMS devices are not satisfying.

The deposition superlattice films about 200 nm thick on the mentioned materials effects in hardening the surface and increase of wear resistance.

The earlier studies [4, 5] of TiN/NbN, TiN/CrN superlattices deposited on Si or Si with Si_3N_4 underlayers has been demonstrated that the highest hardness (over 20 GPa) exhibit for: TiN/CrN superlattice /substrate Si and TiN/CrN superlattice/Si_3N_4 layer/Si substrate compositions. Moreover the TiN/NbN/ Si composition has the best uniformity of the film versus the depth. In this paper we present the 3D images topography of these compositions.

II. Experimental

TiN/NbN and TiN/CrN superlattices have been deposited on a n-type <100> oriented monocrystalline silicon surface. A pad dielectric layer was the 200 nm thick Si_3N_4 film deposited by Plasma Enhanced Chemical Vapour Deposition (PECVD). The superlattices were deposited by the dc reactive magnetron sputtering by the application of the targets Ti, Cr, Nb (99,95%), with the use of the LEYBOLD L400Sp system. The deposition was performed using continuous substrate rotation (350 rotation/h). TiN deposition rate was 0.1 nm/s, NbN - 0.2 nm/s and CrN –0,3 nm/s. A base pressure was kept below $1x10^{-6}$ mbar. During the sputtering process the power of magnetron was 0.4 kW. An Ar/N_2 mixture with partial pressures of 2.8 $x10^{-3}$ mbar and $3.8x10^{-4}$ mbar respectively, was used as a sputtering gas. The total

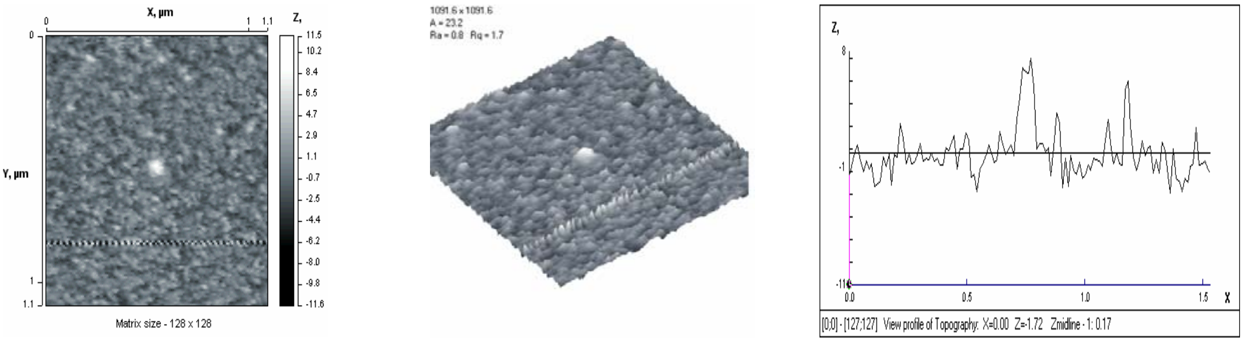

Fig. 1. Surface topography of superlattice TiN/CrN/substrate Si.

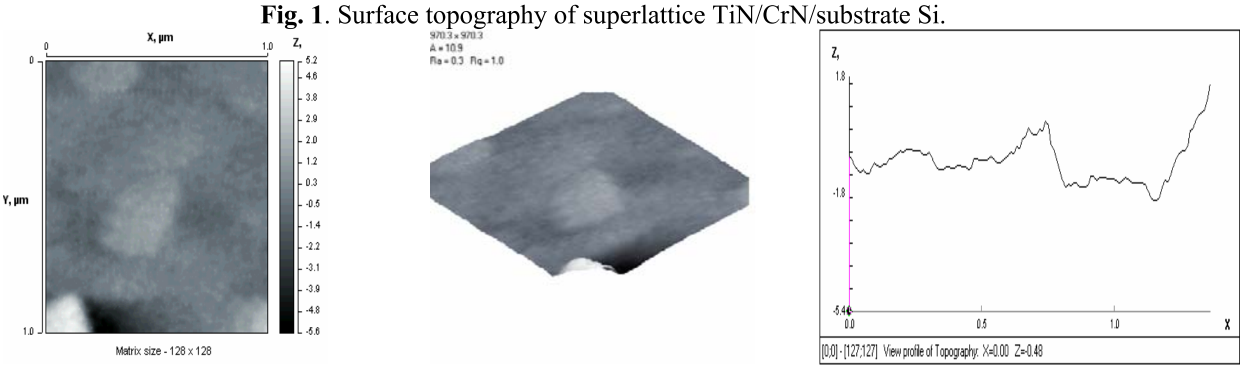

FIg. 2. Surface topography of superlattice TiN/CrN/layer of Si_3N_4/substrate Si.

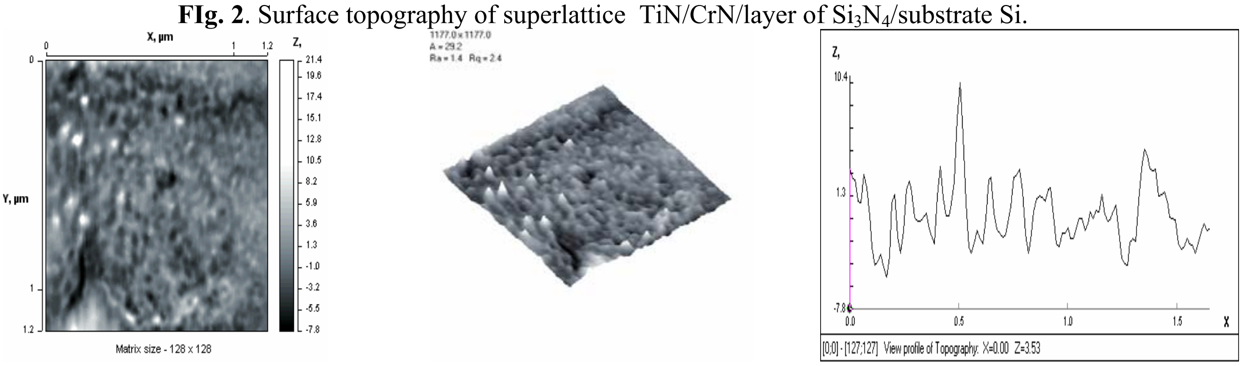

Fig. 3. Surface topography of superlattice TiN/NbN/substrate Si.

pressure in the chamber was kept $3.1x10^{-3}$ mbar.

The thickness of the single layers was measured using the VASE spectroscopy ellipsometer. The thickness of TiN single layer was 2.84 nm, CrN – 3.02 nm and NbN – 1.84 nm.

The total thickness of the superlattices applied as protective coatings in MEMS should be smaller as compared to the minimum sizes of MEMS components, which are in the range of micrometers. The investigations have been carried out on the TiN/NbN and TiN/CrN superlattices with 30 periods λ, where the total thicknesses of the films were 140.4 nm and 175.8 nm respectively.

The Atomic Force Microscope (AFM) constructed in the Metal-Polymer Research Institute in Gomel (Belarus) was used in these surface topography studies.

The computer program Surface View was used to define the parameters of the studied surfaces. The 3D AFM images were constructed using 128x128 points matrix. The scan area was 950x950 nm up to 1650x1650 nm.

III. Results and discussion

The 3D AFM images of the studied superlattices are presented in Fig. 1-3.

The 3D AFM images of the investigated superlattices demonstrate that the surface structure of the film depends on the material being deposited. The superlattices TiN/CrN characterize with better flatness of the surface than TiN/NbN. It was recognized also the effect of the substrate/underlayer on the structure of the superlattice. The same superlattice films (TiN/CrN) deposited on different substrates/underlayer have different topographies. This reflects on the hardness of the tested superlattices [5]. The superlattice TiN/CrN/Si_3N_4/Si with distinctly larger grains exhibits better uniformity of the hardness distribution than superlattice TiN/CrN/Si.

In this paper were defined the following parameters of the surface:

- Minimum H_{min} and maximum H_{max} peak-to-mean height.
- Average $H_{\alpha v}$ peak-to-mean height.
- Roughness parameters: R_a , R_{sk} , R_{ku} .
- Height distribution.
- Bearing Ratio.

H_{min} and H_{max} are the extreme values have been recorded for the studies surface.

$H_{\alpha v}$ is average height of roughness to line of mean profile. Value of H_{av} parameter was described by equation 1.

$$H_{av} = \frac{\sum_{i=1}^{n} H_i}{n}, \qquad (1)$$

where:
n – quantity of measurement points on test surface;
H_i – the height of successive measurement points to mean line.

R_a parameter (center-line average) is described by equation 2.

$$R_a = \frac{1}{N_x N_y} \sum_{j=0}^{N_y-1} \sum_{i=0}^{N_x-1} \left| Z_{i,j} - Z^{mid} \right|, \qquad (2)$$

where:
$N_x \times N_y$ – points of matrix (128x128),
Z_{ij} – an height of measurement point in a research space
Z^{mid} – an average height of roughness

$$Z^{mid} = \frac{1}{N_x N_y} \sum_{j=0}^{N_y-1} \sum_{i=0}^{N_x-1} Z_{i,j} \qquad (3)$$

The center-line average is average absolute value of profile deviation to mean line.

R_{sk} parameter (skewness) is described by equation (4)

$$R_{sk} = \frac{1}{R_q^{\,3}} \frac{1}{N_x N_y} \sum_{j=0}^{N_y-1} \sum_{i=0}^{N_x-1} \left(Z_{i,j} - Z^{mid} \right)^3. \qquad (4)$$

The skewness defines the variables with an asymmetric spread and represents the degree of symmetry of the probability distribution; symmetrical distribution function, have zero skewness.

R_{ku} parameter (kurtosis) is described by equation (5)

$$R_{ku} = \frac{1}{R_q^{\,4}} \frac{1}{N_x N_y} \sum_{j=0}^{N_y-1} \sum_{i=0}^{N_x-1} \left(Z_{i,j} - Z^{mid} \right)^4. \qquad (5)$$

The results of the AFM surface studies are presented in table 1.

In many tribological applications of the tested superlattices, height of the highest asperities above the mean line is an important parameter because damage of the interface may be done by the few high asperities present on one of the two surfaces. In the case of the possible additional lubrication, on the other hand, valleys may affect the lubrication retention and flow.

However, the height of R_a parameter is the most commonly specified in practice for rubbing components. The lower value relates to higher adhesive component of friction and higher values of R_a to higher mechanical component of friction force and higher, possibly abrasive wear. This parameter is seen to be primarily concerned with the relative departure of the profile in the vertical direction only.

The high kurtosis (and low skewness) value found for TiN/CrN/ Si_3N_4/ Si superlattice film is similar to the surfaces obtained by grinding, honing, milling and laser polishing processes [6]. The smaller values of kurtosis and higher together with higher values of skewness are more characteristic to the surfaces produced by turning, shaping and electrodischarge machining and therefore demonstrating worse tribological behaviour.

The Height distribution and Bearing area curve parameters are presented in Fig. 4

Bearing area curve obtained from a surface profile or a surface map, like in our case of 3D AFM images, can be used to describe approximately the real area of contact. It gives the ratio of air to material at any level, starting at the highest peak, called the bearing ratio or material ratio, as a function of level. The bearing area curve characterizes approximately the loading capacity of the surface, however, the relationship of bearing ratio to the fractional real area of contact is highly approximate as material is sliced off in the construction of the bearing area curve and the material deformation is not taken into consideration.

Table 1.

The results of the AFM surface studies

Roughness parameters of superlattices	Composition		
	TiN/CrN/Si	TiN/NbN/Si	TiN/CrN/ Si_3N_4/ Si
H_{min} [nm]	-11.64	-7.82	-5.44
H_{max} [nm]	11.54	21.36	4.96
$H_{\alpha v}$ [nm]	0.002	-0.541	-0.11
R_a	0.847	1.358	0.233
R_{sk}	0.396	0.856	0.100
R_{ku}	5.785	6.128	12.645

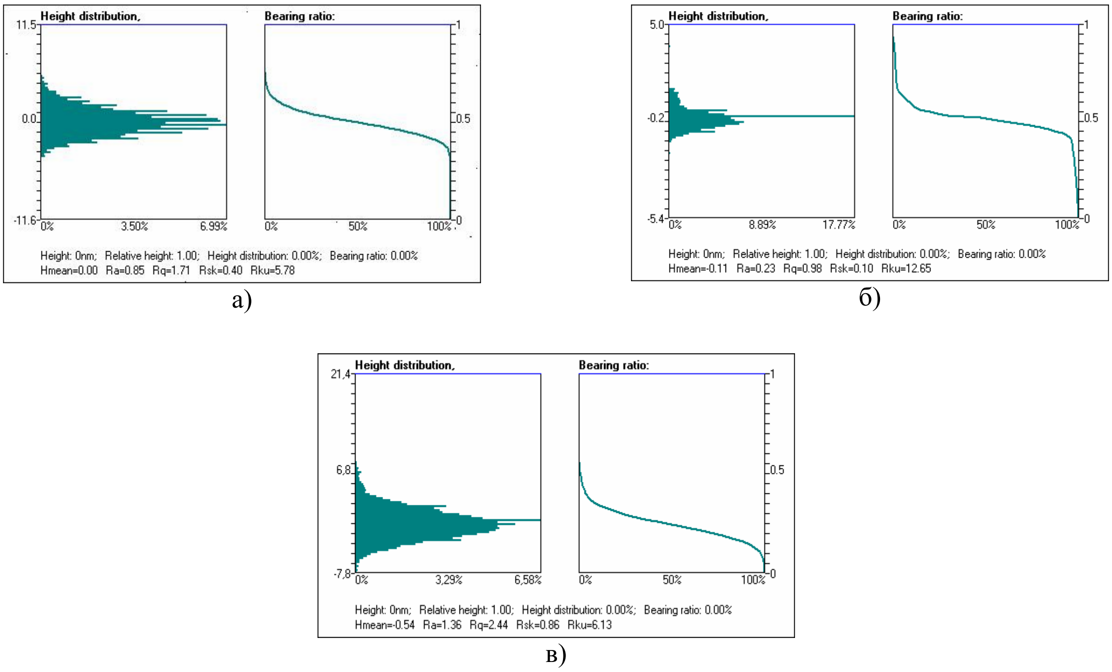

Fig. 4. Height distribution and Bearing Ratio parameters of superlattices:
a) TiN/CrN/Si, b) TiN/CrN/Si_3N_4, c) TiN/NbN/Si

IV. Conclusions

The 3D AFM images of the investigated superlattices demonstrate that the surface structure of the film depends on the material being deposited. The superlattices TiN/CrN characterize with better flatness of the surface than TiN/NbN. It was recognized also the effect of the substrate/underlayer on the structure of the superlattice. The same superlattice films (TiN/CrN) deposited on different substrates/underlayer have different topographies. This reflects on the hardness of the tested superlattices. The TiN/CrN/Si_3N_4/Si composite has the best uniformity of hardness. The statistical parameters are being described by Surface View computer program. The TiN/CrN/Si_3N_4/Si superlattice has the highest kurtosis and the lowest skewness values. This composite demonstrates the best tribological behaviour

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Дослідження поверхонь TiN/CrN і TiN/NbN надграток осаджених на мікроелектронних матеріалах, використовуючи атомний мікроскоп

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В даній статті ми представляємо 3-вимірну топографію зображень TiN/CrN і TiN/NbN надграток, які осаджені dc реактивним розпиленням на <100> орієнтовану монокристалічну поверхню Si, який покриває плівку Si_3N_4, товщиною 200 нм

В дослідженнях топографії поверхні використовувався атомний мікроскоп (АСМ). Комп'ютерна програма „Поверхневий вид" використовувалась, щоб визначити параметри поверхонь, які вивчались. 3-вимірні зображення АСМ були створені застосовуючи 128x128 точок матрицю. Область (площа) розгортки – 950x950 нм до 1650x1650 нм.

Надгратки TiN/CrN характеризуються кращою пологістю поверхні ніж TiN/NbN. Сполуки TiN/CrN/Si_3N_4/Si мають помітно більші зерна ніж сполуки TiN/CrN/Si, і показують кращу однорідність розповсюдження твердості.

Надгратка TiN/CrN/Si_3N_4/Si має найвищий ексцес і найбільш низькі значення асиметрії. Дана сполука демонструє найкращу трьохлогічну поведінку.