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Profilometric, SEM and AFM Investigations of Titanium and Steel Surface Micro- and Nanoroughness Induced by Neutralized Krypton Ion Beam as a First Stage of Fractal Analysis

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This paper presents the influence of krypton ion irradiation of polycrystalline 99.5% titanium and 1H18N9T stainless steel (made in Poland) on resulting surface roughness and topography, examined by scanning electron and atomic force microscopes, as well as by means of profilograph, that enabled us to study surface morphologies in various scales, that may be important in fractal analysis of ion bombarded surface.

Keywords: surface roughness, surface topography, titanium, stainless steel, ion beam bombardment, GD ion gun.

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Introduction

Surface morphology (topography and especially roughness, which is considered as a set of protuberances and depressions existing on a target surface and defined by means of about 30 parameters and functions [1]) plays or could play an important role in various areas of science and technology, for example in:

a) microelectronics – as semiconductor dimensions shrink, resist line edge roughness (LER [2]) will be more important because roughness from the resist is transferred to the substrate with further processing steps,

b) surface analysis [3] where ion beam sputtering is widely used in depth-profile analytical techniques such as AES, RBS, SIMS, XPS – severe problem in analysing concentration profiles is broadening of the profiles by sputter induced roughening leading to a reduced depth resolution.

One of the most noticeable results of ion bombardment of solid surface is modification of its roughness, what can be observe even with the naked eye – ion beam modified part of a surface is more glittering (polishing process) or more matt (roughening process) than untreated one. To measure surface roughness parameters different methods are used, e.g. profilometrical [1], microscopic: STM, AFM [4] or optical [5]. Among various ion bombardment conditions, influencing on surface topography and roughness modification, angle of ion incidence [6], ion dose [7], ion energy [8], substrate temperature [9], kind of target material [10], and kind of bombarding ions [11] seem to be the most important. However, there are some problems with other factors which seem to be important as well, for example with the size of elementary segment l in profilometrical measurements or scanning area in microscopic observations. The method (profilometrical, microscopic, optical or other) used to measure surface roughness parameters is also important. Information about those problems is rather insufficient, especially in the case of such widely utilised materials like titanium and stainless steel modified by neutralized ion beam. We tried to bridge that gap and this paper is the result of our work on modification of titanium and stainless steel surface morphology (mainly roughness) induced by neutralized krypton ion beam in keV energy range, examined by means of profilograph, AFM and SEM (topography).

I. Experimental details

Two kinds of specimens in form of disks 15 mm × 1.5 mm (diameter × thickness) were examined:

a) polycrystalline 99.5% titanium, and

b) stainless steel of 1H18N9T (made in Poland).

Roughness (microroughness) parameters were measured: a) before ion irradiation, b) after each ion

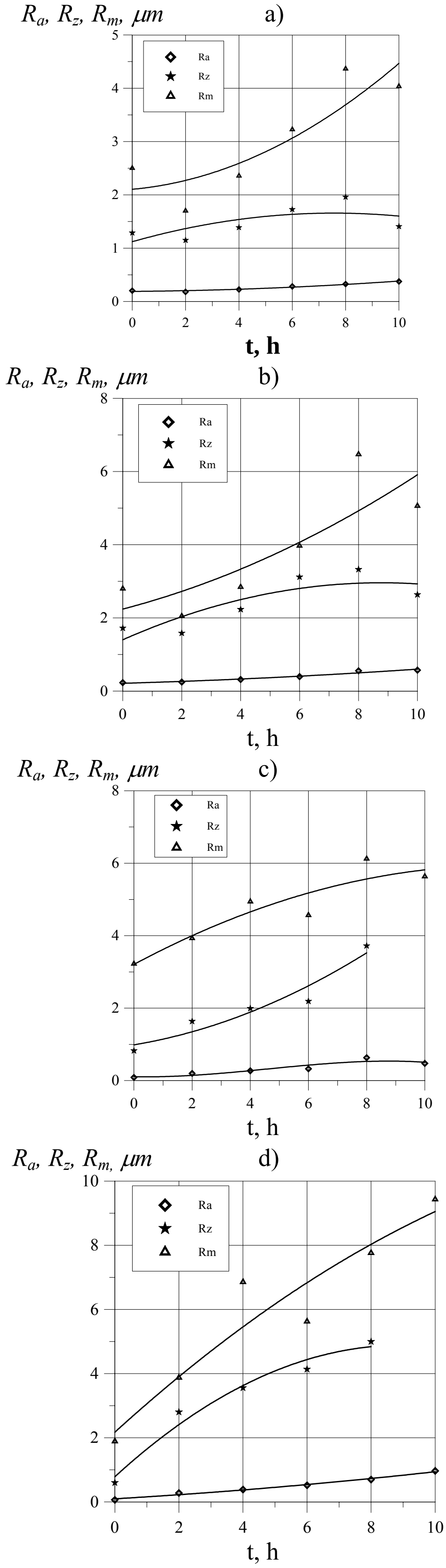


Fig. 1. Roughness parameters: R_a, R_z, R_m of 99.5% polycrystalline titanium and 1H18N9T stainless steel surface versus bombardment duration t for normal neutralized krypton ion beam incidence and various lengths of elementary segment l: (a) l = 0.25 mm, titanium, (b) l = 0.8 mm, titanium, (c) l = 0.25 mm, steel, (d) l = 0.8 mm, steel. The curves are the best polynomial fits.

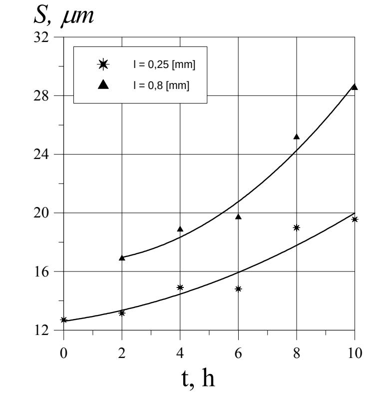


Fig. 2. Modification of horizontal parameter S induced by neutralized krypton ion beam bombardment (Θ = 0°) of mechanically polished surface of 1H18N9T stainless steel (made in Poland) versus time of irradiation.

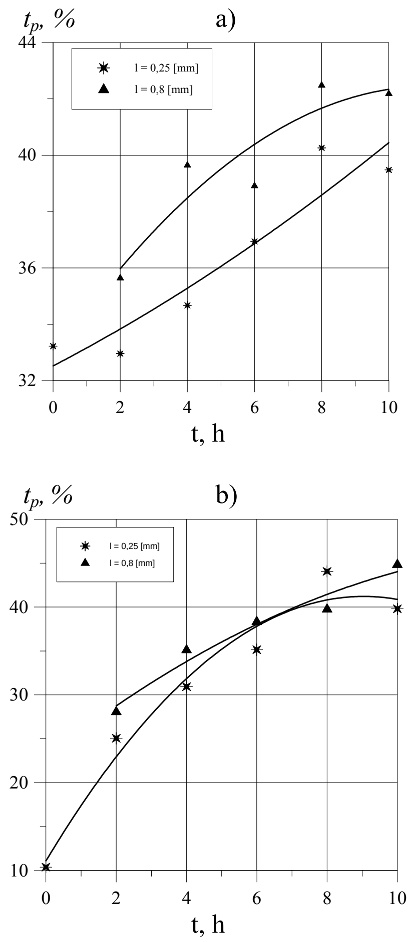


Fig. 3. Changes of parameter t_{p50} induced by perpendicular neutralized krypton ion beam irradiation on surfaces of (a) polycrystalline 99.5% titanium, and (b) 1H18N9T stainless steel.

bombardment process, using a high quality profilograph: Rank Taylor Hobson's *Talysurf* and calibrated atomic force microscope (made in our university). Ion irradiation processes were performed in an experimental apparatus with the glow discharge ion gun with hollow anode [12] (at applied voltage 6 kV and anode current up to 0.25 mA) used as a neutralized krypton ion beam source. Ion beam induced changes of five main roughness parameters were measured, i.e.:

a) arithmetical mean deviation of the surface profile R_a,

b) ten point height of irregularities R_z,

c) maximum height of the surface profile R_m,

d) mean spacing of local peaks of the profile S,

e) profile bearing length ratio t_p.

All parameters in question are well known and normalized [1]. Besides roughness changes, titanium and steel surface topographies were also examined (selected results are presented here) with Hitachi S-570 scanning electron microscope.

II. Results and discussion

3.1. Profilometrical microroughness measurements. Changes of the main roughness parameters examined by the profilograph and relating to vertical and horizontal features of the surface roughness profile shape of polycrystalline 99.5% titanium and stainless steel are shown in Fig. 1 (the smooth curves in presented figures are the best polynomial fits). As can be seen all three parameters: R_a, R_z, R_m concerning vertical features of roughness profile shape increase substantially with the increasing duration of ion bombardment. That proves the perpendicular neutralized krypton beam from glow discharge ion gun as a good surface roughening tool. Also the horizontal parameter S increases during krypton ion irradiation of steel surface (Fig.2), in spite of titanium (not presented here), where its dependence on bombardment duration is not so clear. That means widening of convex topographical forms through eliminating of small dimension features by more extensive elements, development of some topographical forms "at the cost" of the others. All roughness parameters were measured over a range of a conventionally determined elementary segment l (i.e. conventional value needed for determination of some roughness parameters, e.g. root-mean-square deviation, *RMS*). It is worth noting here that values of the parameters in question depend on the above-mentioned segment as shown in Figs. 1 and 2. If the length of element l (scale) was reduced (in our case from 0.8 mm to 0.25 mm), the surfaces appeared to be smoother. That "scaling effect" is very important and must be taken into account during roughness measurements. The same effect could also be observed in Fig. 3, that presents changes of profile bearing length ratio (parameter t_p) during ten hours of neutralized krypton ion beam bombardment of surfaces of both materials in question. The parameter in question t_{p50}, calculated for c = 50% (the middle of the distance between the mean line [1] and the maximum height of examined surface roughness profile shape) means probability of profile appearance for approach less than c value. That probability, calculated for l = 0.25 mm, increases substantially from about 10% for untreated steel surface to more than 40% for ion bombarded one (Fig. 3b). As mentioned, in the case of longer elementary segment (l = 0.8 mm), probability values are slightly higher. Ion bombardment induced changes of parameter t_{p50} for titanium surface (see Fig. 3a) are not so significant as observed for steel.

3.2. AFM nanoroughness measurements. Atomic force microscopy and scanning probe microscopy provide topographic information down to the Angstrom level. In atomic force microscopy the force interactions acting at the tiny microtip (Fig.4) are utilized for surface

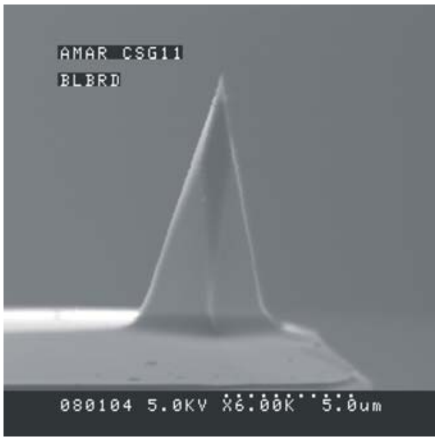


Fig. 4. Microtip of the silicon atomic force microscope cantilever.

characterization. In the experiments we applied homebuilt contact mode atomic force microscope for the investigation of surface roughness in a nanometre range. Deflection of the piezoactuator was measured by means of the fiber Fabry-Perot interferometer with the accuracy of 10nm. The measurement accuracy and resolution of

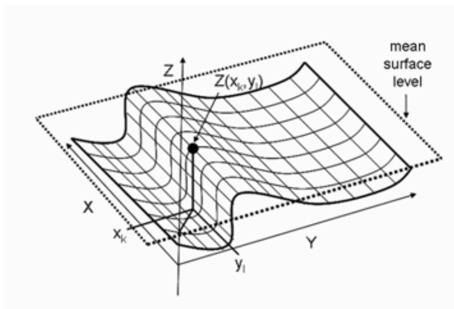


Fig. 5. Surface topography after slope correction – estimation of surface height $Z(x_k, y_l)$.

the piezoactuator deflections was estimated to be in the range of 10nm and 0.1nm, respectively. Surface topography was observed with the use of silicon cantilevers with force constant of 0.1N/m, scanresolution of 256x256 lines and scanning frequency of 0,5lines/s.

The calculation of surface roughness are routinely performed based on PN-EN ISO 42887 standard. However, in these calculations measurement data only

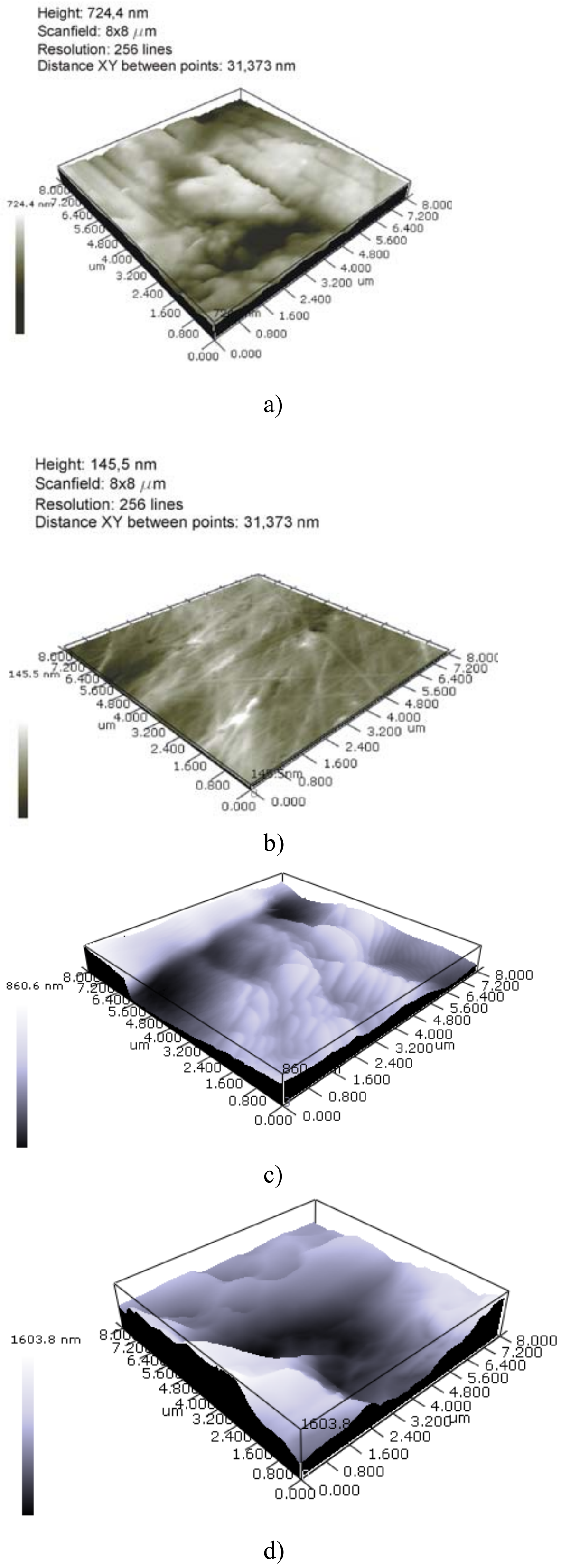


Fig. 6. AFM images (examples) of surface morphology of: (a) untreated 99.5% titanium, (b) untreated stainless steel, and krypton neutralized ion beam bombarded: (c) 99.5% titanium, t = 6h, (d) stainless steel, t = 6h.

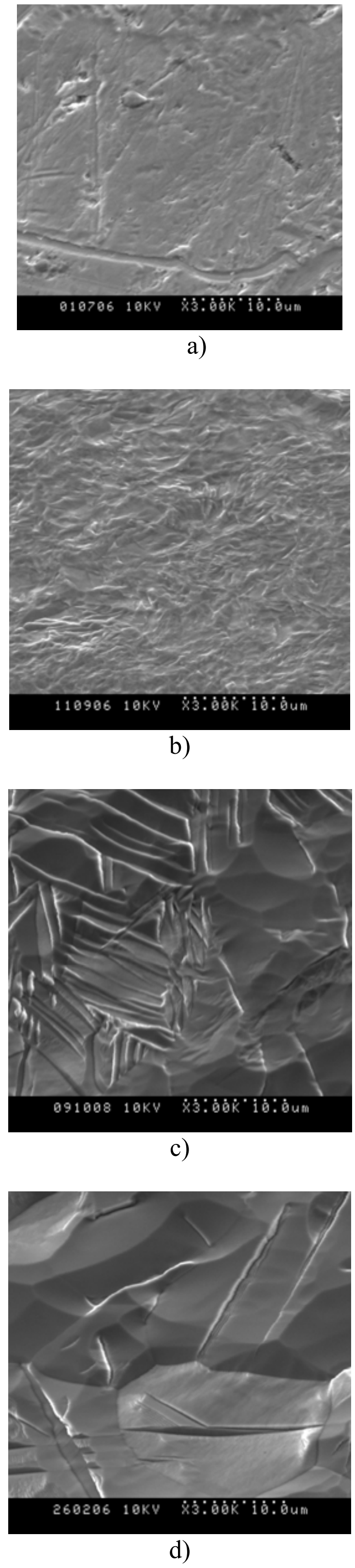
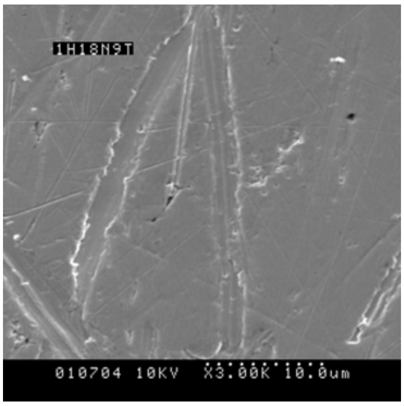
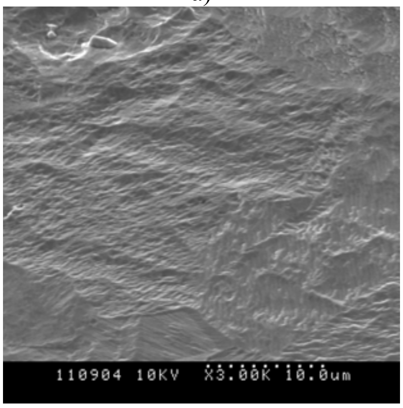


Fig. 7. Ex situ scanning electron microscopy images of polycrystalline 99.5% titanium surface topography after normal krypton neutralized ion beam bombardment: (a) 0 h, (b) 2 h, (c) 6 h, (d) 10 h.



a)



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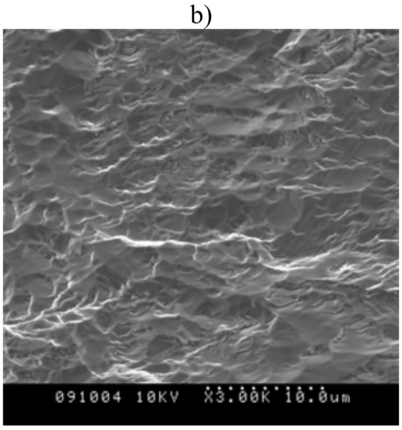
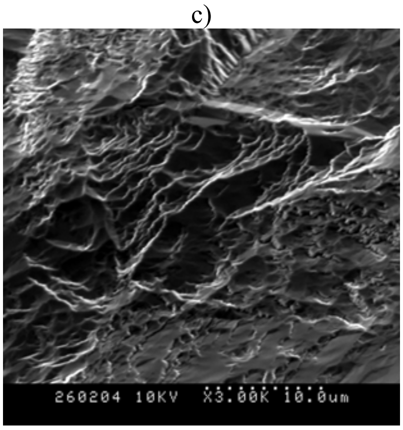


c)



d)

Fig. 8. Ex situ scanning electron microscopy images of stainless steel of 1H18N9T (made in Poland) surface topography after normal krypton neutralized ion beam bombardment: (a) 0 h, (b) 2 h, (c) 6 h, (d) 10 h.

from one surface profile line are considered. To estimate the surface roughness according to the image of 256x256 lines we developed the special procedure which includes data collected in the x and y plane of the surface (Fig. 5). In this procedure the two-dimensional (2D) surface roughness is defined as arithmetic average value of the surface height Z(x, y), which was calculated after slope correction:

$$\mathrm{Sa} = \frac{1}{\mathrm{XY}} \sum_{k=0}^{X-1} \sum_{l=0}^{Y-1} \left| Z(x_k, y_l) \right| \qquad (1)$$

where X, Y are numbers of lines measured in x and y direction, x_k, y_l are coordinates of each measurement point on the investigated surface.

Additionally we calculated the root mean square value of the surface roughness S_q, which describes the tallest and highest surface features:

$$\mathrm{Sq} = \sqrt{\frac{1}{\mathrm{XY}} \sum_{k=0}^{X-1} \sum_{l=0}^{Y-1} \left(Z\left(x_k, y_l\right)^2 \right)} \,. \qquad (2)$$

Results of AFM measurements and observations are presented in Table 1 and in Fig. 6 (selected examples). Generally, the changes of all roughness parameters examined by the microscope in question and relating to vertical features of titanium and stainless steel surface profiles are similar to that measured by profilograph but in much smaller range, nanometre range in this case. That is the result of scaling. Moreover, the experiments showed that GD ion source seems to be a good roughening tool not only in the micrometer range but also in nanoscale too.

3.3. SEM observations. There is a strong dependence of surface roughness upon surface topography (e.g. cone-textured surfaces are usually considered as extremely rough). For this reason, it is useful to verify the results of roughness measurements with topography SEM observations. To this end, a Hitachi S-570 microscope was used and the selected micrographs are presented in Figs. 7 and 8. Topographical changes induced by neutralized krypton ion beam from GD ion gun are spectacular, especially in the case of titanium, where some crystallographic effects are involved in modification phenomena (see Fig. 7c and Fig. 7d). Widening and deepening of topographical elements resulting from ion irradiation, as shown in the SEM micrographs, are in good agreement with profilometric measurements and calculations.

Conclusion

We have studied changes of surface morphology (main surface roughness parameters and topography) of polycrystalline 99.5% titanium and 1H18N9T stainless steel (made in Poland) induced by neutralized krypton ion beam from glow discharge (GD) ion gun. Results of quasi-tangential irradiation of titanium were already published [13]. Here, experimental results of normal bombardment of the materials in question were presented. GD ion gun with hollow anode was used as neutralized krypton ion beam source (accelerating voltage up to 6 kV) and scanning electron and atomic

Table 1

Changes of vertical roughness parameters of polycrystalline 99,5% titanium and stainless steel of 1H18N9T (made in Poland) surface induced by perpendicular neutralized krypton ion beam irradiation (Θ = 0°) and examined by means of atomic force microscope

Parameter [nm]	Time of titanium surface bombardment [h]					
	0	2	4	6	8	10
S_a	95,2	87-107	106,2-153,3	102,2-216,4	300,6	98,1-372,8
S_q	117,3	110-140	131,8-190,6	132,4-258,8	404,3	123,0-445,8
Parameter [nm]	Time of stainless steel surface bombardment [h]					
	0	2	4	6	8	10
S_a	11,1	88-128	165-166,1	129,3-250	365,5	77,4-377,7
S_q	14,3	110-162	198,3-200,3	157-313,2	435,3	95,4-489,5

force microscopes as well as high quality profilograph were applied to examine surface morphology modification. Such investigative procedure allows us to study the changes of roughness in various scales, that may be important in fractal analysis of ion bombarded surface. Results of profilometric measurements and calculations, obtained for various lengths of elementary segment *l*, as well as AFM investigations really showed “scaling effects”: generally similar changes (increase with bombardment duration) of roughness parameters as a consequence of ion irradiation could be observed but not in the same scale. Besides, the experiments proved the perpendicular neutralized krypton beam from glow discharge ion gun as a good surface roughening tool, not only in micrometer range but also in nanoscale.

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