Materials Science-Poland, Vol. 26, No. 2, 2008

Surface nanomodification induced by a neutralized ion beam

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The paper presents results of surface nanomodification induced by neutralized ion beam from the glow discharge ion gun with a hollow anode. Processes like surface polishing, surface roughening, generation of various surface/subsurface structure elements (e.g., waves, ripples) resulting from ion bombardment were investigated. All those events are crucial in such areas of science and technology as preparation (ion beam thinning and polishing) of samples for transmission electron microscopes; surface analysis where ion beam sputtering generating unwanted surface structures like e.g., waves is widely used in depth-profile analytical techniques such as secondary ion mass spectroscopy, Auger electron spectroscopy, X-ray photoelectron spectroscopy, Rutherford backscattering spectrometry; formation of patterns in sub-100 nm regime for micro/nanoelectronics where reduction of line etch roughness (LER) must be taken into account (as semiconductor dimensions shrink, LER will be more important because roughness from the resist is transferred to the substrate with further processing steps).

Key words: *surface nanomodification; neutralized ion beam; ion polishing; surface roughening*

1. Introduction

Material modification processes like sample thinning, surface polishing, surface roughening, revealing and/or generation of surface/subsurface structure elements can be achieved by the use of ion beam irradiation. The processes in question are very important and must be considered in various areas of science and technology. For example, in surface analysis [1] ion beam sputtering is widely used in depth-profile analytical techniques such as Auger electron spectroscopy (AES), Rutherford backscattering spectrometry (RBS), secondary ion mass spectroscopy (SIMS), X-ray photoelectron spectroscopy (XPS) where a severe problem in analysing concentration profiles is broadening of the profiles by sputter induced roughening (i.e., formation of topographical/structural elements like, for example, waves or ripples presented in

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Fig. 1) leading to a reduced depth resolution. Next, in microelectronics shrinking semiconductor dimensions cause that line resist etch roughness (LER) [2] will be more important because roughness from the resist is transferred to the substrate with further processing steps. Finally, in transmission electron microscopy, ion beam thinning and polishing are widely used processes of sample preparation thanks to well known advantages in comparison to conventional methods.

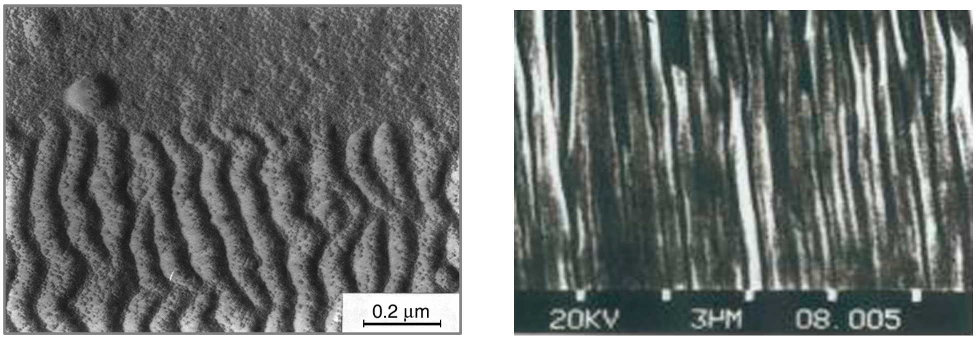

Fig. 1. Unwanted morphological elements induced by neutralized argon ion beam: a) waves on Corning 7059 glass surface [3], b) ripples on 1H18N9T stainless steel surface [4]

The paper presents results of surface nanomodification, i.e. surface polishing, surface roughening, surface etching leading to sample thinning as well as generation of various surface and/or subsurface structure elements reminiscent of waves or ripples in sub-micrometer (nanometer) regime induced by neutralized ion beam from a glow discharge ion gun with a hollow anode. Taking into consideration imposed limitations on the length of the paper, only some selected experimental results have been presented.

2. Experimental

GD ion source. The ion bombardment induced modifications of solid surfaces were performed in apparatuses equipped with a well known ion beam source – a glow discharge (GD) ion gun with a hollow anode. Simplicity of the gun and d.c. high voltage power supply design, ease of continuous operation, convenience in use and maintenance, and facility of sputtering of non-conductive materials without additional neutralizing systems (the beam is a "mixture" of ions, electrons and neutrals) are only the principal virtues of this source. The gun with earthed cathode and anode at a positive potential (up to 7 kV), yielding ion current of up to 0.1 mA and current density of up to 5 $\mu A/mm^2$ has been used to modify surfaces of various materials in micrometer and submicrometer (nanometer) range. The diameter of the neutralized ion beam depends on the cathode orifice diameter Φ_C and we used this electrode with optimal value of $\Phi_C = 1$ mm.

Surface modification measurements and observations. To investigate ion beam induced surface polishing or roughening it is convenient to measure changes of roughness parameters. We measured all the main parameters relating to horizontal and vertical features of the surface profile. Here, only results of measurements of the roughness parameter R_a (arithmetical mean deviation of the surface profile) are presented. The parameter was measured: a) before ion irradiation, b) after each ion bombardment process, using a high quality profilograph (Rank Taylor Hobson's Talysurf) and a calibrated atomic force microscope made at this University [5]. Changes of surface roughness are directly connected with alteration of surface topography. Therefore, the surface topographies were also studied (only selected results are presented here) with scanning electron microscopes (SEMs, e.g. Hitachi S-570, Jeol JSM-5800LV) and a transmission electron microscope (TEM, binary carbon replicas method) in the micrometer range as well as with AFM in the nanometer range. These microscopes were also utilised to observe ion beam revealed and/or generated surface and subsurface defects or structures.

3. Results and discussion

3.1. Ion polishing

The neutralized ion beam from GD gun seems to be a good polishing tool not only in the micrometer but also in submicrometer (nanometer) range as is shown in Fig. 2.

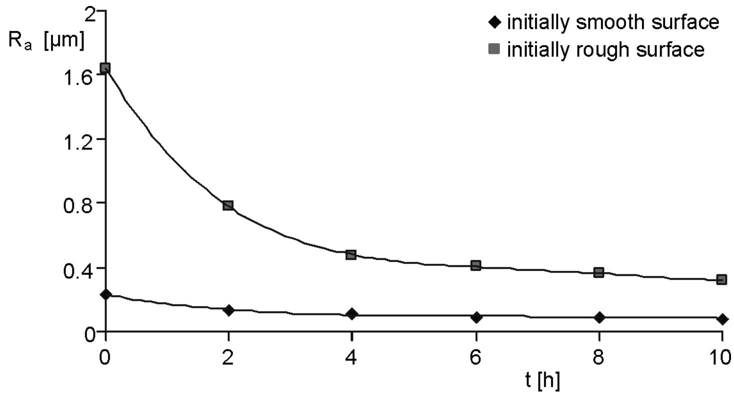

Fig. 2. Micrometer and nanometer changes of the roughness parameter R_a generated by a very oblique ($\Theta = 87°$) neutralized argon ion beam irradiation of initially rough and initially smooth 99.5% polycrystalline titanium surfaces; the curves are the best polynomial fits

Two kinds of titanium surfaces: initially rough and initially smooth were polished with a very oblique neutralized argon ion beam. After 2–4 h of polishing, the intensity of the process decreases and the parameter R_a diminishes at a rate of several to several dozen nanometers per hour. The capability of the neutralized ion beam to nanopolishing is especially visible in Fig. 3 where a submicrometer modification of the parameter in question is generated on stainless steel and titanium surfaces by means of a very inclined ($\Theta = 87°$) argon ion beam. In the case of steel, the parameter R_a decreases (on average) less than 4 nm per hour.

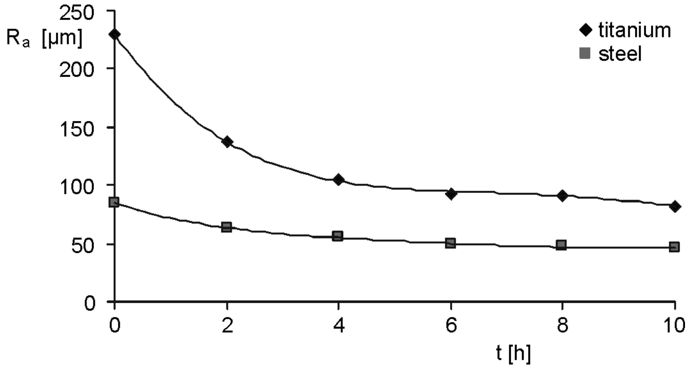

Fig. 3. The influence of neutralized argon ion beam bombardment duration at $\Theta = 87°$ on vertical roughness parameter R_a of 1H18N9T stainless steel (made in Poland) and 99.5% polycrystalline titanium; the curves are the best polynomial fits

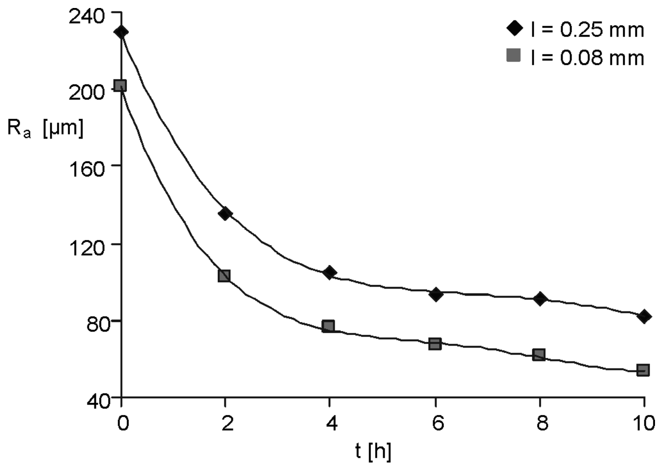

Fig. 4. "Scaling effect" observed on $R_a(t)$ changes induced on mechanically polished surface of 99.5% polycrystalline titanium by neutralized argon ion beam bombardment at the incidence angle $\Theta = 87°$; the curves are the best polynomial fits

In profilometric measurements, all roughness parameters are measured over a range of a conventionally determined elementary segment l. It is worth noting here that the values of the parameters in question depend on the above-mentioned segment as is shown in Fig. 4. When the length of element l (scale) was reduced (in our case from 0.25 mm to 0.08 mm), the surface appeared to be smoother. That "scaling effect" is very important and must be taken into account during roughness measurements.

3.2. Ion thinning

It is well known that ion thinning is used in specimen preparation for TEM and that efficiency of the process, i.e. ion thinning rate depends on many factors. Two measuring methods were used to study the parameter in question: profilometric and microscopic (ion sputter-induced step was examined by means of a profilometer and SEM, respectively).

In the case of the GD source with the cathode and anode holes diameters $\Phi_C = 1$ mm and $\Phi_A = 4$ mm, respectively, the thinning rate of stainless steel irradiated perpendicularly with a neutralized argon ion beam is about 0.063 μm/min for the microscopic method and 0.058 μm/min for profilometric one. After a few dozen hours of gun's work, the cathode orifice diameter Φ_C changes (increases to $\Phi_C = 1.5$ mm) due to ion sputtering process but the rate is the same (as determined by microscopic method) or almost the same (profilometric method) as can be seen in Table 1.

Table 1. Argon ion beam thinning rate of 1H18N9T stainless steel for two selected cathode orifice diameters [6, 7] ($U_A = 5$ kV, $I_A = 0.35$ mA, $t = 2$ h)

Diameter of cathode hole Φ_C [mm]	Thinning rate [μm/min]	
	Microscopic method	Profilometric method
1.0	0.063	0.058
1.5	0.063	0.062

It seems that the neutralized ion beam from the GD gun is a good and precise thinning tool giving thinning rates in a submicrometer/min range.

3.3 Ion roughening

Ion roughening is a process leading to an increase of solid surface roughness due to ion bombardment inducing modification of its surface morphology. The neutralized ion beam from the GD gun applied to roughen surfaces has shown its ability to do that not only in micrometer but also in submicrometer range. That can be seen in Fig. 5 presenting the dependence of $R_a(t)$ for stainless steel and titanium surfaces irradiated perpendicularly ($\Theta = 0°$) with a krypton neutralized ion beam. The mean value of R_a increases due to nanoroughening process about 20 nanometers per hour for titanium and about 40 nanometers per hour for steel. Taking into account the results of *ex situ* SEM examinations (not presented here), one can conclude that on increasing of surface roughness, a widening of topographical forms can be observed due to eliminating small dimension features by more extended elements.

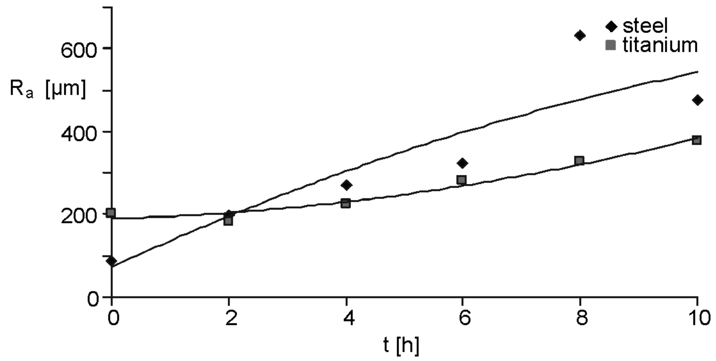

Fig. 5. Time dependences of the roughness parameter R_a of stainless steel (1H18N9T) and 99.5% polycrystalline titanium surfaces irradiated with a normal neutralized krypton ion beam; the curves are the polynomial fits

Table 2. Vertical roughness parameters R_a (maximum values) [nm] induced by perpendicular neutralized krypton ion beam irradiation ($\Theta = 0°$) and examined by means of atomic force microscope (see [5])

Material	Bombardment duration [h]					
	0	2	4	6	8	10
Polycrystalline 99.5% titanium	95.2	107	153.3	216.4	300.6	372.8
1H18N9T steel	11.1	128	166.1	250	365.5	377.7

Ion roughening of metal targets with the use of a neutralized ion beam in question was also studied by means of a homebuilt contact mode atomic force microscope (see e.g., [5]). All AFM results presented in Table 2 are smaller than R_a parameters obtained with the use of a profilometer (compare Fig. 5) even though they were measured for the same sample surfaces. The reason of those discrepancies are "scaling effects" mentioned in Section 3.1 – the AFM elementary segment (8 μm) is much smaller than that of the profilometer (250 μm) used in our experiments.

3.4. Revealing/generating of surface/subsurface structure elements

Neutralized ion beam from the GD gun allows to reveal surface and/or subsurface structures (e.g. grains and grain boundaries) as well as various structure elements like voids, pores, macrodefects, etc. (for further information, see e.g. [6]). On the other hand, there are some unexpected and unwanted results of the beam bombardment, e.g. generation of waves or ripples on irradiated surfaces as was shown on TEM and SEM images presented in Fig. 1. The mean period of waves and ripples is about 75 nm and

700 nm, respectively. Those nanostructures on the surface mean increased roughness – a serious obstacle in surface analysis (see Section 1).

4. Conclusion

Neutralized ion beam from GD gun to surface nanomodification has been used in, e.g., surface polishing, surface roughening, surface etching (leading to sample thinning) as well as generation of various surface and/or subsurface structure elements reminiscent of waves or ripples in sub-micrometer regime. Taking into account results of our experiments (published here and unpublished) it can be stated that the neutralized ion beam in question could be a good modification tool, not only in micrometer but also in nanometer range.

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Received 28 April 2007
Revised 16 February 2008