JOURNAL OF MATERIALS SCIENCE 25 (1990) 3875–3879

Ion-bombardment modification of the surface morphology of solids

Part 1 *Changes of surface roughness*

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One of the aspects of ion-bombardment modification of the surface morphology of solids (IBMSM) is surface roughness alteration. The influence of ion-beam sputtering on changes in the surface morphology is presented and discussed. Theoretical concepts (a simple theory), together with experimental verification including narrow- and broad-beam sputtering-induced modification of surface roughness of various materials, such as metals (aluminium, titanium), alloys (stainless steel 1H18N9T and SS316LC) and alumina ceramic (99.5% Al_2O_3), are the main area of interest here. These rather unexplored problems are very important from theoretical and practical points of view because there are many technologies and experimental techniques in which they are, or may be, used.

1. Introduction

In spite of a large number of published articles, the problem of ion-bombardment modification of the surface morphology of solids is not fully understood or comprehensively described. Moreover, the term "surface morphology" is generally identified with surface topography, surface profile or surface shape, and this is misleading. Recently, it has been suggested [1, 2] that the modification in question is a very complicated phenomenon which must be considered from various points of view, and that different aspects of this phenomenon must be taken into account. One of the more important aspects is surface roughness alteration. Unfortunately, information about the influence of ion sputtering on roughness modification is rather sparse [3–6]: only a few articles have been published in this research area. Taking into account the importance of the question, some theoretical concepts and experimental results are presented here.

Every surface contour of a target material (which can be measured, for example, by means of a profilograph, see Fig. 1) consists of three main profiles:

(a) surface shape profile,
(b) surface waviness profile, and
(c) surface roughness profile.

The criterion of this classification is a quantity (value) of coefficient f, where

$$f = \frac{D}{H} \tag{1}$$

where D is the mean distance between two consecutive surface protuberances (surface contour maxima) and H is the height of the largest (extreme) protuberance (see Fig. 2).

If $f < 40$, the studied profile is a surface roughness profile. If $f \geqslant 1000$, the analysed profile is a surface shape profile. Between these two values we assume that the real profile is a waviness profile.

2. Theoretical considerations

The main subject of this article is the surface roughness considered as a set of protuberances and depressions existing on a target surface. There are several parameters which give quantitative information about surface roughness (see Fig. 3). The first is the maximum roughness height, $R_{\max}$, (or maximum roughness, see Fig. 3a), i.e. the distance between two lines which are parallel to the mean line and pass across the top of the highest protuberance (the upper line) and the lowest point of the deepest depression (the lower line). This parameter is not as important as the mean arithmetical deviation of a profile from the mean line, R, and is considered to be the main roughness factor (often called the mean roughness). It is defined as the mean value of the distances ($y_1, y_2, \ldots, y_n$) of points of a profile from the mean line measured over a range of the elementary segment l (see Fig. 3b), i.e.

$$R = \frac{1}{l}\int_0^l |y|\, dx \tag{2}$$

The parameter in question is usually measured using a profilograph but can also be calculated directly from the profile shape recorded by this apparatus (see Figs 3a and b). To this end, the mean distance, h, of the base line from the surface profile (see Fig. 3a)

$$h = \frac{1}{l}\int_0^l y_i\, dx \tag{3}$$

which gives the possibility of determination of the mean line, must be calculated. Having the mean line, it is easy to obtain the factor R (see Fig. 3b)

$$R \approx \frac{1}{n}\sum_{i=1}^{i=n} |y_i| \tag{4}$$

The parameter in question, very useful in the case of untreated (non-ion-irradiated) surfaces is not as

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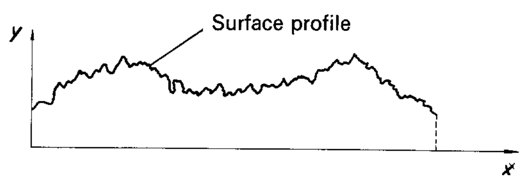

Figure 1 The real profile of a target surface which consists of: the surface shape profile, the surface waviness profile, and the surface roughness profile.

precise for ion-sputtered materials, because values of the mean arithmetical deviation, R_u, of various sample surfaces of the same material before ion bombardment are not identical and therefore it is very difficult to interpret the results of mean roughness, R, measurements obtained after ion sputtering. A good solution, which enables one to avoid this difficulty, is to introduce a new factor, K (relative roughness), which takes into account the influence of the initial deviation, R_u, on the final one, R, (i.e. after sputtering)

$$K = \frac{R}{R_u} \tag{5}$$

Taking into consideration Equation 4 and the dependence

$$R_u \approx \frac{1}{m} \sum_{i=1}^{i=m} |(y_0)_i| \tag{6}$$

where (y_0) is the distance of a profile point from the mean line of the untreated surface, we can write

$$K \approx \frac{\frac{1}{n} \sum_{i=1}^{i=n} |y_i|}{\frac{1}{m} \sum_{i=1}^{i=m} |(y_0)_i|} \tag{7}$$

In these cases, when elementary segments l_u and l of untreated and sputtered surface profiles, respectively, are equal, and the numbers of measuring points in both segments are identical, i.e. $m = n$ we obtain

$$K \approx \frac{\sum_{i=1}^{i=n} |y_i|}{\sum_{i=1}^{i=n} |(y_0)_i|} \tag{8}$$

It can be shown that changes in the heights of certain surface profiles induced by ion sputtering depend on the ion-beam incidence angle, θ. Fig. 4 shows the erosion of unit area, A, of the target surface after unit time, $\mathrm{d}t$, of ion-beam bombardment. From

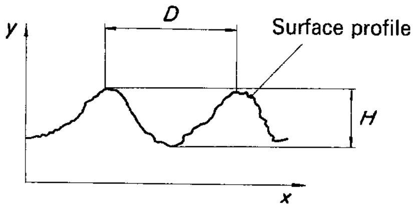

Figure 2 The real surface profile, usually measured using a profilograph, where the mean distance, D, between two consecutive surface protuberances and the height, H, of the largest protuberance, are indicated.

the definition of sputtering yield

$$Y = \frac{N_a}{N_j} \tag{9}$$

with

$$N_a = NA\,\mathrm{d}L \tag{10}$$

and

$$N_j = \phi\,\mathrm{d}t \tag{11}$$

where N_a and N_j are average numbers of ejected atoms and incoming ions, respectively, N is the number of atoms per unit volume of target material, $\mathrm{d}L$ and $\mathrm{d}H$ are heights of the sputtered unit volume (measured in the ion-beam direction) and sputtered part of the surface profile (measured perpendicular to the surface), respectively, and ϕ is the ion flux.

From Equations 9 to 11 it follows that

$$Y = \frac{NA\,\mathrm{d}L}{\phi\,\mathrm{d}t} = \frac{N\,\mathrm{d}L}{\Phi\,\mathrm{d}t} \tag{12}$$

where Φ is the number of ions per second striking the unit area, A.

According to Equation 12 the height

$$\mathrm{d}L = \frac{Y\Phi}{N}\,\mathrm{d}t \tag{13}$$

but, as can be seen in Fig. 4, it can also be expressed by

$$\mathrm{d}L = \frac{\mathrm{d}H}{\cos\theta} \tag{14}$$

Combining Equations 13 and 14 one readily obtains

$$\mathrm{d}H = \frac{\Phi}{N}\,Y(\theta)\cos\theta\,\mathrm{d}t \tag{15}$$

The height, H, of the ion-irradiated surface profile (see Fig. 4) which can be measured (perpendicular to the

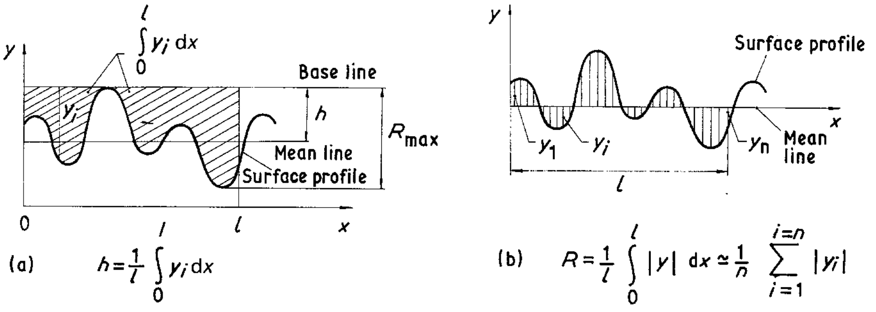

Figure 3 The method of surface roughness calculation showing determination of (a) the mean distance, h, of the base line from the surface profile, and (b) the mean arithmetical deviation, R, of the surface profile from the mean line.

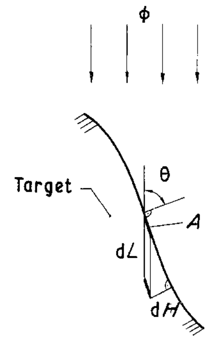

Figure 4 The erosion of unit area, A, of the target surface after unit time, $\mathrm{d}t$, of ion-beam bombardment. Ion flux is denoted here as ϕ; $\mathrm{d}L$ and $\mathrm{d}H$ are heights of the sputtered volume (measured in the ion-beam direction) and the sputtered part of the surface profile, respectively, and θ is the angle of ion-beam incidence.

surface) after t sec sputtering can be obtained from Equation 15 after integration over time

$$H = \int_0^t \frac{\Phi}{N} Y(\theta) \cos\theta \, \mathrm{d}t = \frac{\Phi t}{N} Y(\theta) \cos\theta \quad (16)$$

For given values of t and Φ, and N being constant, we can expect changes of H proportional to $Y(\theta) \cos\theta$, i.e. for $\theta = 0$ rad

$$H = CY(0) \quad (17)$$

where

$$C = \frac{\Phi t}{N} \quad (18)$$

and for $\theta = \pi/2$ rad

$$H = 0 \quad (19)$$

The information about the changes of height, H, very important from theoretical and practical points of view, cannot be directly used in studies of ion-beam-induced modification of surface roughness, R, where knowledge of heights y_i (see, for example, Fig. 3b) and especially heights Δy of sputtered parts of the surface profile (parallel to the y-axis direction) are essential.

The relation between the heights $\mathrm{d}H$ (normal to the surface) and $\mathrm{d}y$ (parallel to the y-axis and/or perpendicular to the mean line, see Fig. 4) can be calculated based on Fig. 5. It seems that two main kinds (types) of profiles could be distinguished, the first is the so-called "increasing" profile (Fig. 5a) and the second the "decreasing" one (Fig. 5b). According to Fig. 5a and Equation 15 the height $\mathrm{d}y^+$ of part of the "increasing" surface profile eroded after unit time, $\mathrm{d}t$, of ion sputtering can be expressed by

$$\mathrm{d}y^+ = \frac{\Phi}{N} Y(\theta) \cos\theta \cos(\theta - \psi) \, \mathrm{d}t \quad (20)$$

where ψ is an angle between the ion-beam direction and the y-axis. Integrating Equation 20 over time and taking into account Equation 18, one readily obtains

$$\Delta y^+ = CY(\theta) \cos\theta \cos(\theta - \psi) \quad (21)$$

In the same way one can calculate the height Δy^- for the "decreasing" surface profile (see Fig. 5b)

$$\Delta y^- = CY(\theta) \cos\theta \cos(\psi - \theta) \quad (22)$$

It is well known [7–9] that the equilibrium state is reached for planes (lines) which are perpendicular ($\theta = 0$ rad) or parallel ($\theta = \pi/2$ rad) to the ion flux, as well as inclined at an angle θ_m. Therefore, after time, t, of ion sputtering those planes (lines) are predominant at the surface. It seems that the angles in question, together with $\theta = \theta_g$ (the grazing incidence), are also interesting in this consideration. For $\theta = 0$ rad

$$\Delta y^+ = CY(0) \cos(-\psi) \quad (23)$$

and

$$\Delta y^- = CY(0) \cos\psi \quad (24)$$

For $\theta = 0$ rad and $\psi = 0$ rad

$$\begin{aligned} \Delta y^+ &= \Delta y^- \\ &= CY(0) \end{aligned} \quad (25)$$

whereas for $\theta = 0$ rad and $\psi = \pi/2$ rad

$$\begin{aligned} \Delta y^+ &= \Delta y^- \\ &= 0 \end{aligned} \quad (26)$$

The same result is also observed for $\theta = \pi/2$ rad. Ion sputtering of a solid surface at grazing incidence, i.e. at $\theta = \theta_g \approx 0$ rad, gives changes in heights, Δy, close to zero

$$\begin{aligned} \Delta y^+ &= \Delta y^- \\ &\approx 0 \end{aligned} \quad (27)$$

According to Equations 23 to 25, perpendicular bombardment ($\theta = 0$ rad) leads to changes in heights, Δy, which are greater than zero

$$\begin{cases} \Delta y^+ > 0 \\ \Delta y^- > 0 \end{cases} \quad (28)$$

which should mean an increase of mean surface roughness

$$R(\theta) = R(0) > R_u \quad (29)$$

or

$$K(\theta) = K(0) > 1 \quad (30)$$

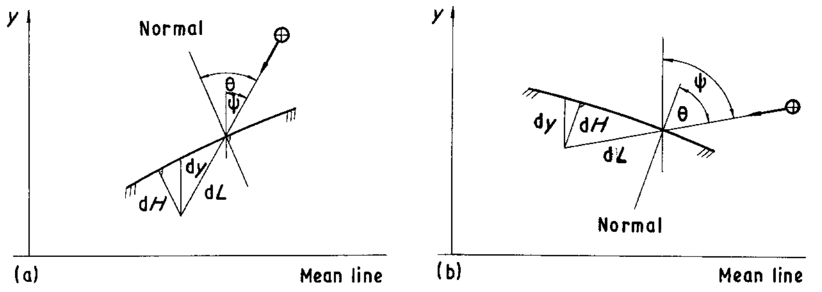

Figure 5 Changes of the surface profile heights, $\mathrm{d}y$ (parallel to the y-axis and/or perpendicular to the mean line) induced by ion-beam sputtering: (a) an example of so-called "increasing" profile, (b) the "decreasing" surface profile.

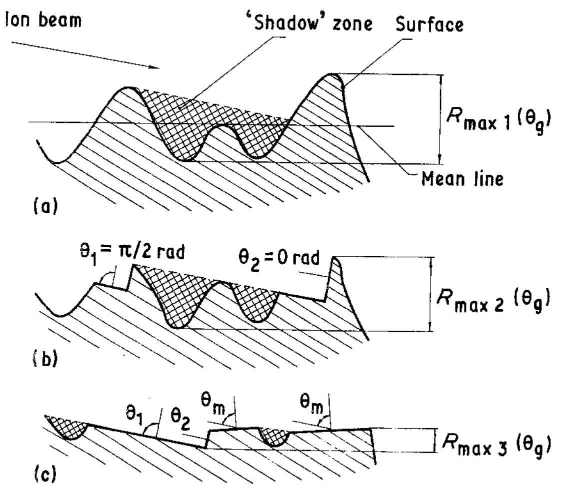

Figure 6 Changes of surface roughness (maximum roughness height, R_{max}, in this case) induced by sputtering using a very oblique ion beam, (a) $R_{max\,1}(\theta_g)$ after time t_1 of ion irradiation, (b) $R_{max\,2}(\theta_g)$ after t_2 sec of sputtering, (c) $R_{max\,3}(\theta_g)$ after time t_3 of ion bombardment. Maximum roughness: $R_{max\,1}(\theta_g) > R_{max\,2}(\theta_g) > R_{max\,3}(\theta_g)$, and time of sputtering $t_1 < t_2 < t_3$.

For parallel bombardment ($\theta = \pm\pi/2$ rad) there are no changes in Δy (see Equations 21, 22 and 26) and therefore

$$R(\theta) = R(\pm\pi/2) = R_u \tag{31}$$

and accordingly

$$K(\theta) = K(\pm\pi/2) = 1 \tag{32}$$

More interesting, especially from the practical point of view, is not $\theta = 0$ rad, but the grazing incidence of the ion beam, θ_g, where changes $\Delta y^+ = \Delta y^- \approx 0$ should be expected. This means

$$R(\theta_g) \approx R_u \tag{33}$$

or

$$K(\theta_g) \approx 1 \tag{34}$$

In real systems, where apart from the $Y(\theta)$ relation, secondary effects also have been considered (see, for example, [10]), much more pronounced changes of roughness, as can be expected from Equations 29, 30 and 33, 34, could be observed. For perpendicular bombardment ($\theta = 0$ rad) of a smooth target surface (see for instance Fig. 4 in [10]), the mean roughness $R(0)$ of the ion-sputtered surface is much greater than the unsputtered one, R_u, i.e.

$$R(0) \gg R_u \tag{35}$$

or

$$K(0) \gg 1 \tag{36}$$

On the other hand, ion sputtering of a surface with a very oblique ion beam ($\theta = \theta_g$) may lead to a decrease of roughness (see Fig. 6)

$$R(\theta_g) \lesssim R_u \tag{37}$$

or

$$K(\theta_g) \lesssim 1 \tag{38}$$

From Equations 29 to 38 it follows that

$$K(0) > K(\theta_g) \tag{39}$$

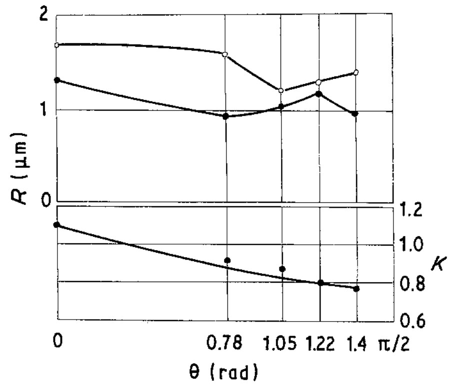

Figure 7 Angle, θ, of narrow ion-beam incidence variations of mean, R, and relative, K, roughnesses for two selected materials [5]. (○) 99.9% Ti, (●) 99.5% Al_2O_3.

When the values of mean arithmetical deviation, R_u, of untreated surfaces of samples are comparable, one can also write

$$R(0) > R(\theta_g) \tag{40}$$

The last equations show the great influence of ion-beam incidence on solid surface roughness, which can be used in controlled modification of the property in question, over a wide range of $K(\theta)$, from $K(\theta) \gg 1$ for $\theta = 0$ rad to $K(\theta_g) < K(0)$ for grazing ion-beam incidence.

3. Experimental verification

It must be stated at the outset that "experimental material" is rather poor. There are only a few articles with suitable results concerning the problem in question. Moreover, the results of the greater number of experiments refer to single (individual) samples. Generally, there are no systematic studies (excluding [3] and [4]) taking into account results of experiments done on a large number of specimens.

The experimental results presented here refer to targets of different materials, such as metals (aluminium, titanium), metal alloys (stainless steel of SS316 LC and 1H18N9T) and alumina ceramic (99.5% Al_2O_3), sputtered with the use of narrow and/or broad ion beams. Generally, two types of ion source have been used: narrow-beam glow discharge with a hollow

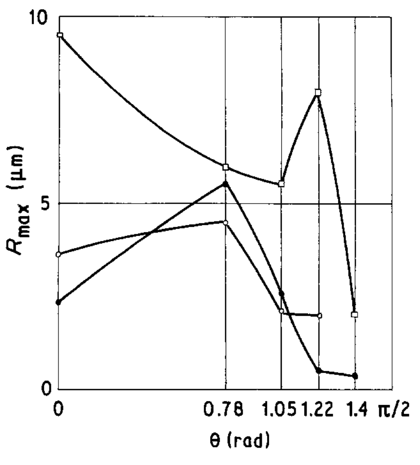

Figure 8 Angle, θ, of narrow ion-beam incidence variations of maximum roughness, R_{max}, for three selected materials [5]. (□) 99.9% Ti, (●) 99% Ta, (○) SS 316 LC.

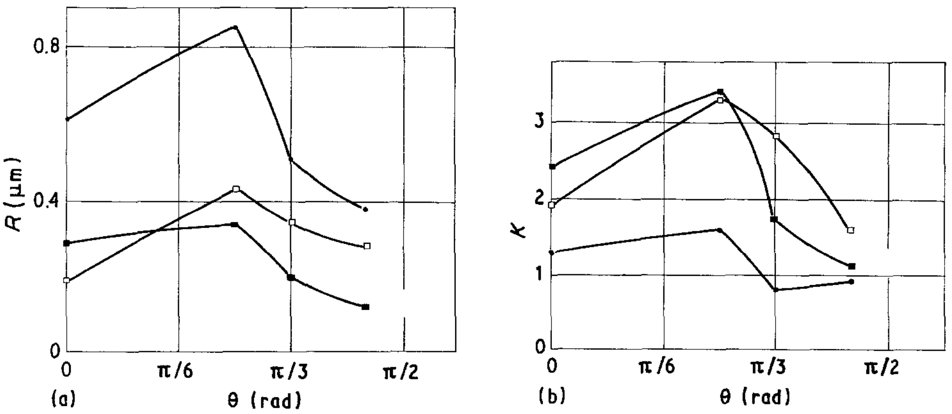

Figure 9 Angular dependence of (a) mean roughness, R, and (b) relative roughness, K, for broad argon-ion irradiation of (●) 99.9% Al, (□) 99.9% Ti and (■) stainless steel type 1H18N9T; see [6].

anode gun, and a broad-beam Kaufman-type source. Fig. 7 shows the angle, θ, of ion-beam incidence variations with the mean arithmetical deviation, R, of a surface profile for two selected materials (Fig. 7a), together with variations of factor K obtained for 99.5% Al_2O_3 (Fig. 7b). All the materials have been sputtered with a narrow argon-ion beam (up to 0.1 mA and 7 kV) for 2 to 4 h [11]. The results presented here are in a good agreement with the simple theory of roughness changes induced by ion sputtering, discussed in Section 2. It is easy to verify (see Fig. 7b) that $K_{Al_2O_3}(0) > 1$, $K_{Al_2O_3}(1.4\,\mathrm{rad}) \lesssim 0$, and $K_{Al_2O_3}(0) > K_{Al_2O_3}(1.4\,\mathrm{rad})$ confirm Equations 30, 38 and 39, and also the relations (see Fig. 7a) $R_{Al_2O_3}(0) > R_{Al_2O_3}(1.4\,\mathrm{rad})$ or $R_{Ti}(0) > R_{Ti}(1.4\,\mathrm{rad})$ are in good agreement with Equation 40.

It must be stated that good agreement with the theory can also be observed in the case of maximum roughness height, R_{max}, with the angle, θ, of ion-incidence measurements (at least for the material surfaces studied and presented here, Fig. 8). Theoretical concepts discussed in the Section 2 have also been proved by broad argon-ion beam bombardment of various materials, as can be seen in Fig. 9. The only exception is titanium, where extreme values of R_u influence the resulting mean roughness $R(\theta)$ and therefore Equation 40 is not fulfilled. In the experiments, a 12 cm Kaufman source ($0.5\,\mathrm{mA\,cm^{-2}}$, 0.8 kV and 3 h bombardment) and a special method of examination have been used [6].

4. Conclusion

A simple theory of surface roughness changes induced by ion sputtering of solids is presented. The main parameters of surface roughness have been defined, i.e. mean (R) and relative (K) roughnesses as well as the less important and rarely used maximum roughness (R_{max}). It has been shown that changes in the real surface profile, Δy, caused by ion erosion and measured perpendicular to the mean line, depend on the angle of ion-beam incidence, θ, and the angle, ψ, between the beam direction and the y-axis. A general formula for the variation of angles θ and ψ with the main roughnesses in question (i.e. R and K) has been proposed and experimentally verified. Investigation of ion-bombardment-induced surface roughness is not only a theoretical question. It seems that the practical aspect is also very important and interesting, because there are many branches of research work and technology where it is, or may be, used (for example, mechanics, materials science, optics, medicine, etc.). Therefore, further studies in this field are necessary, especially much more systematic research based on a large number of sputtered samples of the same material, as well as investigation of the influence of the angle θ and sample rotation on surface roughness.

Acknowledgement

The author thanks Mr Z. Pochrań for his help in preparation of this and other papers published in this journal.

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Received 4 May
and accepted 29 September 1989