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Surface morphology development during ion sputtering: roughening or smoothing?

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Abstract

We report on STM studies of ion-sputtered surfaces, applying sputtering conditions which were shown to produce a relatively smooth surface. The height correlation function was calculated for the nickel layer in both the as-received and sputtered conditions. The large-scale roughness of the as-received specimen was reduced by ion sputtering according to expectations derived from Auger depth profiling. On the other hand, the small-scale roughness was increased due to sputtering. Self-affine scaling regions are identified, and the exponents are compared to theoretical and numerical results.

Keywords: Ion bombardment; Ion–solid interaction; Surface structure, morphology, roughness, and topography

There are several analytical techniques (e.g. SIMS, Auger and XPS depth profiling) that apply low-energy (less than 5 keV) ions. Similarly, there are important technological processes like plasma etching, ion-assisted deposition, specimen preparation by ion thinning, etc., where the surface of the specimen is subject to low-energy ion bombardment. These treatments, despite the low ion energy, also generate considerable damage, like vacancies, interstitials or such extended damage as surface roughening and ion mixing on the irradiated surface. For optimal application of the above techniques, the understanding of ion-sputtering induced damage processes is of great importance. Though the damage processes occur simultaneously and are strongly interrelated, we will focus our attention in this letter to surface roughening alone.

When a target is bombarded with ions, depending on the ion energy, angle of incidence, type of ion and type of target, various topological features may develop. The study of these features by means of transmission electron microscopy has a long history [1]. Recently, scanning tunnelling microscopy (STM) has frequently been used for this purpose [2]. The advantage of this method is that besides the image, it provides directly the height function along the surface, which can in principle be used for a more quantitative characterization of the changes of the surface morphology due to ion bombardment. STM has also made it possible to check the validity of theoretical predictions of surface roughening due to sputtering. For example, Eklund et al. [3] studied the roughening of graph-

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ite under 5 keV Ar ion bombardment by STM and calculated the height correlation function. They found that correlated structures developed due to sputtering characterized by a length that diverged with increasing ion flux. Agreement with linear response theory was poor. Krim et al. [4] studied sputtered epitaxial Fe film with 5 keV Ar and observed well-defined self-affine behavior. Chason et al. [5] used 1 keV Xe with to sputter Ge(001), and observed temperature-dependent roughening.

The sputtering conditions chosen in the works cited are not considered favourable, however, if one wants to obtain a smooth surface. There is now a consensus (based on for example, TEM and Auger depth profiling studies) that surface roughening can be considerably reduced by introducing specimen rotation, a grazing angle of incidence and low sputtering energy (some hundreds of eV) [6–8]. Specimen rotation reduces shadowing effects and anisotropic sputtering, while with a grazing angle of incidence, the probability of channeling (which strongly contributes to surface roughening) is negligible.

In this letter we report on surface morphology development using specimen rotation and a grazing angle of incidence. The surface before and after sputtering was characterized by STM, and the height correlation function was calculated.

Nickel, chromium, copper and silver were deposited on a well-polished silicon substrate. The evaporated layers were ion sputtered in our dedicated AES depth profiling device [9] to remove a layer 50 nm thick. Ion sputtering was carried out on a rotating sample (at a speed of 30 rpm) by a special Teletwin-type ion gun [10]. The angle of incidence of Ar ions was 86° relative to the surface normal, and the incidence energy was 1 keV. The diameter of the ion beam was about 0.3 mm, with an ion current of some, μA. The slope of the crater produced by the sputtering was calculated (by measuring the distances of the subsequent layers on the wall of the crater) to be less than 10^{-5} rad, which means that the removal of material took place in an almost layer-by-layer manner. The electron beam diameter was less than 50 μm. Using these conditions the depth resolution in Auger depth profiling does not depend on the thickness of the removed layer, and thus surface roughening does not depend on the fluence [8–10].

STM micrographs of various horizontal resolutions were taken from as-received and sputtered surfaces using an RHK-100 microscope and a UHV-635 head. The size of the scanned regions varied from 5 to 750 nm. In all cases the resolution of the STM micrographs was 256 × 256 pixels. The average surface roughness was calculated by the software of the instrument and it was found that for all cases, it was lower after sputtering. For a better understanding of the change of the morphology, we calculated the height correlation function. Results concerning the nickel layer will be presented here.

Figs. 1 and 2 show typical STM images of the as-received and sputtered surfaces of the nickel layer, respectively.

If a surface is essentially two dimensional (i.e. the overhangs are irrelevant), then it can be characterized by a single valued height function, $h(\mathbf{r})$ ($\mathbf{r}=(x,y)$). A standard method for investigating surface morphology is to study the height–height correlation function, which is defined as

$$c^2(r)=\langle (h(\mathbf{r}+\mathbf{r}')-h(\mathbf{r}))^2\rangle_{\mathbf{r}'}. \quad (1)$$

If the surface is isotropic in the x and y directions (as in our case), then the correlation function depends only on the magnitude of $\mathbf{r}(|\mathbf{r}|=r)$. The value of the correlation function at a given distance r is related to the roughness of the interface on length scale r. We make use of this interpretation of the correlation function later.

Let us now very briefly review the self-affine characterization of surfaces. A self-affine isotropic surface is statistically invariant under an affine transformation. In terms of the height function, this means

$$h(x,y)\approx b^{-H}h(bx,by), \quad (2)$$

where H is the self-affine (or Hurst) exponent. Such interfaces are referred to as self-affine fractal surfaces [11]. It can be shown that for a self-affine fractal surface, its correlation function scales as

$$c(r)\sim r^{H}. \quad (3)$$

This relation is often used in self-affine analysis of surfaces. Due to physical limitations, the above

Fig. 1. Three-dimensional shaded view of a 50 nm × 50 nm STM image of nickel evaporated on silicon substrate (as-received specimen).

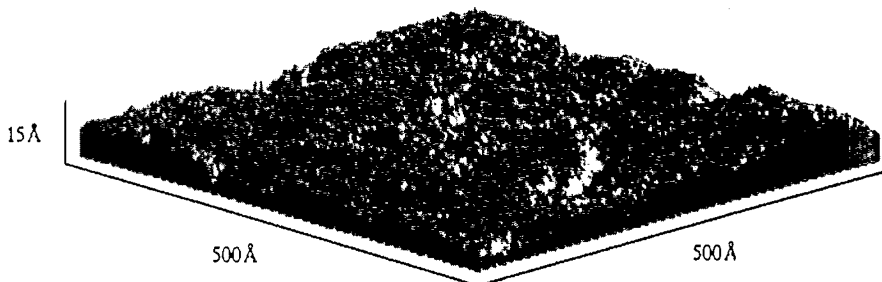

Fig. 2. As Fig. 1, but after sputtering.

expression holds only in a restricted range of lengths. Breakdown of the scaling relation (Eq. (3)) is expected both on small and large length-scales. The scaling region in experiments is usually 1–2 decades, although a larger range can be obtained in computer simulations.

In our case, the surface function $h(r)$ was obtained directly from the STM micrographs. We have evaluated the correlation function (Eq. (1)) by taking every possible pair of positions, calculating the square of the height difference, and averaging for equal distances. The correlation function was discarded for lengths beyond half the size of the sample and for very small lengths (smaller than three pixels). Processing scans with various spatial resolutions and resealing the correlation functions, we could extend the range covered by our analysis. It should be noted that the collapse of the correlation functions is not perfect, slight deviations are seen. They are caused by automatic surface levelling occurring during scanning. In Fig. 3 we present the correlation function for a sample before sputtering. The sample does not show long-range self-affine behavior. The scaling region consists of two sections of different slopes (0.77 ± 0.04 and 0.42 ± 0.03). The smaller exponent is quite close to

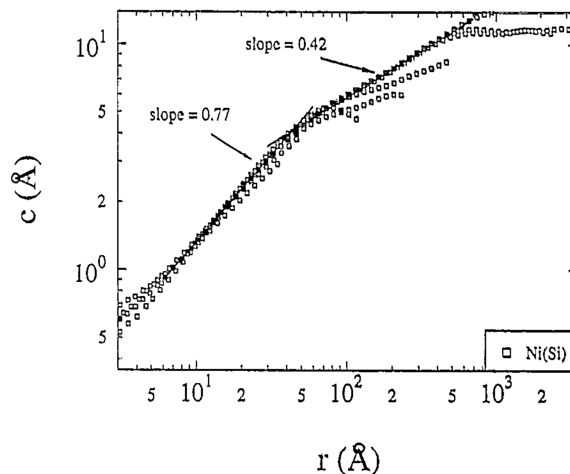

Fig. 3. Correlation function for the surface shown in Fig. 1.

the slope obtained from numerical solutions [12] of the Kardar–Parisi–Zhang equation [13]. The greater exponent is most probably indicative of surface diffusion [5,14,15], which results in a roughness exponent close to 1. The same plot for the sputtered sample is shown in Fig. 4. The scaling exponent (0.49 ± 0.05) is closer to $\alpha = 1/2$, which is characteristic for a Brownian surface rather than the KPZ value.

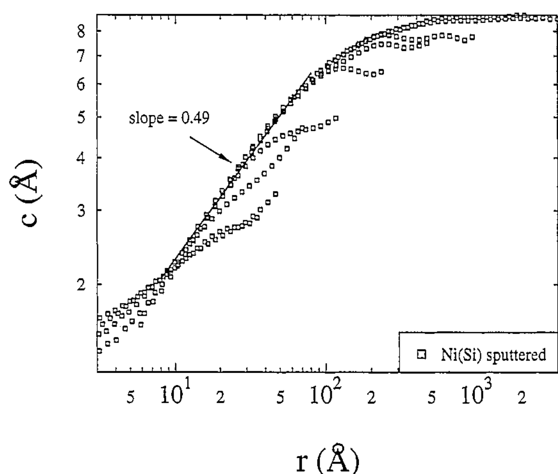

Fig. 4. Correlation function for the surface shown in Fig. 2.

In order to show the changes in surface morphology we plotted in the correlation function for a sample before and after sputtering (Fig. 5). The common feature of the two plots is the existence of lower and upper cut-offs. These cut-offs are further apart for the non-sputtered sample, i.e. the scaling range is wider.

After the sputtering process, the surface morphology changes. The scaling region becomes narrower, which means that the surface becomes more similar to a random surface. It loses its fine (small scale, $r < 10$ Å) structure, and the saturation value of the correlation function (which characterizes the roughness) on small scales grows by 2 Å, corresponding to strong roughening. On the other hand, on large scales ($r > 300$ Å) one can observe the decrease of surface roughness, from 12 to 8 Å.

The smoothing observed at large scales can be explained by the phenomenological model of Barna [6]. This model uses the well-established fact that the sputtering yield depends on crystallographic orientation and that the ion current density changes with the change of the surface normal. In the case of the sputtering conditions applied here, the model predicts smoothing. The more refined model of Chason et al. [5] also offers an explanation of the smoothing by considering surface diffusion and viscous flow. The large-scale smoothing in our case most probably cannot be explained by this model, because of the large distances involved. Roughening was observed, however, at the finer scale. Thus, either the supposed surface diffusion is not fast enough to recover the damage, or the model of Chason cannot be applied to this case. Roughening at the finer scale can on the other hand, be explained by the random arrival of the sputtering ions. This roughening, in the case of improper sputtering conditions (e.g. non-rotated specimen, not preferable ion energy or angle of incidence) might develop to a larger-scale roughness which can show self-affine [4] or self-similar morphologies [3]. However, properly chosen sputtering conditions omit such a large-scale development of surface roughness.

The sputtering conditions used here result in a good-quality sputter surface for a wide variety of cases, for several elements, and for crystalline and amorphous materials. Thus the above idea is valid for great variety of cases. The depth resolution of Auger depth profiling is fortunately not affected by this roughness, since it is smaller (by some Å) compared to other effects (such as ion mixing, and intrinsic interface roughness) which cause damage in the range of more than 10–15 Å.

This work draws attention to the fact that more parameters should be introduced into theoretically derived general equations of surface morphology evolution during ion sputtering [16] in order to account for the various sputtering conditions or/and material parameters which result in various surface morphologies.

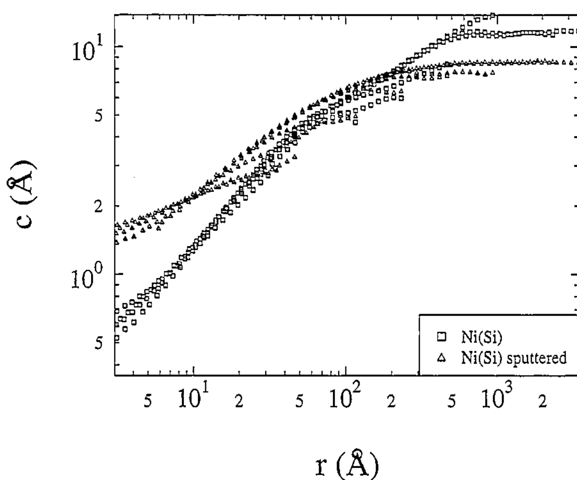

Fig. 5. Comparison of the correlation functions for the as-received and sputtered surfaces.

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In summary, the ion sputtering introduces surface roughening most probably because of the random arrival of the bombarding atoms, but the roughness created cannot develop. This results in a rough surface in a microscopic range, if properly applied sputtering conditions are used.

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