Formation of Nanoparticles by Ion Beam Irradiation of Thin Films

Zs. Baji1, A. Szanyo1, Gy. Molnár1, A. L. Tóth1, G. Pető1, K. Frey2, E. Kötai3, and G. Kaptay4

1 Research Institute for Technical Physics and Materials Science, H 1525 Budapest. P.O. Box 49 Hungary
2 Institute of Isotopes, HAS, P.O. Box 77., H-1525 Budapest Hungary
3 Institute for Particle and Nuclear Physics, H 1525 Budapest P.O. Box 49 Hungary
4 BAY-NANO Research Institute for Nanotechnology + University of Miskolc, H 3515 Miskolc, Egyetemváros, E/7, 606

The possibility of fabricating nanoparticles by ion bombardment was investigated by the ion bombardment of indium films on oxide covered Si and Cr surfaces. The different masses of implanting specimen ensured the different energy transfer while the same Si substrate ensured the same thermal conductivity for the In and Cr layers. Chromium served as a reference for the effect of ion bombardment and as a substrate as well. The SRIM program was used to simulate the ion surface interaction process. The nanoparticles were detected by scanning electron microscopy (SEM). We found that the melting of the In layer results in the formation of nanoparticles of 50–300 nm diameter and 5–10 nm height. This method can be promising for nanoparticle formation of materials with low melting point.

Keywords: Nano-Particles, Ion-Bombardment, Indium, Chromium, Melting.

1. INTRODUCTION

Ion bombardment may change the composition and the structure of the surface of solids, including the mechanical, chemical, optical and even morphological properties of the surface.1-2 For example, it is widely known that the surface of germanium will be dramatically changed by heavy ion implantation.3-4

The preparation and examination of artificial low dimensional structures is one of the most challenging fields of solid-state research. Decreasing the quantity of a given material, as the geometrical size becomes comparable to the length parameter connected to a physical property, this quality becomes size dependent. The changes of the electron structure may be due to different phenomena. As the size of a nanostructure becomes comparable to the de Broglie wavelength of the electron, the electrons may get confined, and the electron structure can become quantized. As the size of the nanostructure is further reduced, the whole electron structure is rearranged. This is generally attributed to the fact that with the decreasing size, the surface/bulk proportion increases. As the surface atoms behave differently from the bulk particles, this logically leads to changes of the properties of the material, especially the electron structure.5 On the other hand, with size reduction, as the amount of surface energy increases compared to the volume energy, the melting points of materials dramatically decrease.

The effects of size reduction have the most striking results in the case of semiconductors, where some of the materials that have an indirect band gap in bulk state may even perform luminescence. In transition metals the d-states rearrange,6 which can also be followed by a metal-nonmetal transition.7 It is generally experienced, that the valence band of metals gets narrower with the decreasing cluster size.8 The catalytic properties of surfaces also change as an effect of ion bombardment, especially if nanosized islands are formed.9

Nanosized islands or nanodots have attracted attention because of their scientific curiosity and technical importance. Such clusters generally consist of 10–105 atoms; therefore they may show outstanding properties. As the electron structure changes many of the macroscopic properties change as well, among them the conductance, the magnetic and optical properties.

Although there are many special techniques to fabricate nanosized structures,10 generally these methods can be classified as top down and bottom up methods.
Ion implantation may be a simple but very versatile top down tool to decrease the size of a given system, or even to create nanodots.

Our earlier experiments9 show that after ion implantation the gold layer evaporated on silicon split up and formed nanosized islands. We supposed that the ion bombardment process with heavy ions can melt the layer, and depending on thermal and wetting properties islands, or even quantum dots may form.

The melting process may be explained and described by the thermal spike model.11,12 If dense cascades evolve, there may be a short period of time (∼10⁻¹² s) in which the majority of atoms in a very small volume moves with a kinetic energy more than a few electron volts—which means that the temperature in this volume exceeds the melting point—then the material rapidly cools down. This cooling process is extremely quick therefore unique, even metastable structures may form. Simulations proved the existence of this quasi-molten or perhaps even gas phase state.

The motivation of the recent study was therefore to examine which circumstances may generally lead to the formation of nanosized islands in different materials after ion bombardment. After our experiments on gold we supposed that thermal properties such as the melting point would be the crucial point of the phenomenon. In order to investigate this presumption we constructed films made of metals with different melting points. In order to see how the combination of the substrate and the layer changes the process, also three different types of surfaces were used. Then we irradiated these layers with a variety of ions. If our presumption was correct, then the more energy the ion implantation provides, the easier the layers could melt. We insured this difference in the energy transfers with different bombarding ions. This way we could investigate the effect of the different thermal parameters such as heat conductivity and melting point, the wetting and the mass of the bombarding ions on the resulting nanodots. The purpose of our research was to develop a well controllable method to fabricate nano-sized islands.

We classified the resulting morphologies with scanning electron microscopy.

2. EXPERIMENTAL DETAILS

The layers were evaporated in a VT 460 system in 10⁻⁵ Pa pressure by thermal evaporation. The deposition rate was 3 nm/min. During the deposition the substrate was at room temperature.

The substrate was a Si (100) wafer covered by native oxide. We examined 10 nm, 30 nm and 60 nm thick indium layers. In order to study the effect of wetting we evaporated some of the layers on a 5 nm chromium layer. As comparison we also prepared 60 nm thick chromium layers, and implanted them with the same ions, so we could see how a material with different thermal properties behaves under the same conditions.

The indium and chromium single- and multilayers were deposited in one vacuum cycle.

The samples were ion implanted by 40 keV argon, krypton and neon ions. We used a high dose ion implanter, and a magnetic mass separator. The ion current was 1 μA/cm² and the angle of the implantation was 0°. The ion beam was electrically swept. The dose was 10¹⁵ at/cm² in all cases. The ion implanter was evacuated by a turbo molecular pump and LN2 trap to 10⁻⁴ Pa during the Ar and Kr ion bombardment.

The morphologies of the as-deposited and ion implanted films were studied by a LEO 1540 XB type scanning electron microscope, and energy dispersive spectrometer (EDS).

We simulated the energy transfers and the sputter yields of the layers with the SRIM program.13,14 In ion bombardment the ions pass their energies on to the layer in two processes: elastic collisions with the electrons and inelastic collisions with the nuclei of the atoms. In our calculations we used the approximation that from the energy of the ions, the part that heats the layer is the part that the nuclei receive in the collisions.

3. EXPERIMENTAL RESULTS

In Figure 1 we can see the morphologies of a 10 nm thick In film deposited onto native oxide covered Si (100) before (a) and after Ar (b) and Kr (c) ion bombardment. The as deposited indium formed islands, therefore the layer is not continuous, as can be seen in the figure. The Ar and Kr ion bombardment did not induce large changes in the crystalline structure. The film consists of separate crystallites, probably single crystals of In. The average size of the crystallites is around 60 nm in the Ar- and 80 nm in the Kr ion bombarded samples. It is clear that a rather homogeneous crystalline system was formed by ion bombardment, with a very narrow size distribution. After argon irradiation the drops are rather rounded, but in the case of krypton irradiation the film turned into small, angular drops; consequently, in the course of the transition a recrystallization might have taken place.

The indium layers showed different morphologies depending on the layer thickness, even before the ion bombardment. While the 10 nm thick layer was continuous, the 60 nm thick layer was island-like with micron-sized structures, islands, as shown in Figure 2(a). On this thicker indium layer we can discover yet another phenomenon. After irradiated with krypton ions the smaller islands melted into the bigger shapes.

On Figures 3 and 4 we may see indium layers evaporated on a chromium base layer. The as deposited layer is continuous, as the EDS measurement showed. The effects of the ion bombardment with both ions and both sample...
Fig. 1. Morphology of the as deposited 10 nm thick In film onto native oxide covered Si (100) (a and b) after Ar (b) and Kr (c) ion bombardment.

Fig. 2. Morphology of the 60 nm thick In film deposited onto SiOx covered Si(100) substrate before ion bombardment (a) after Ar (b) and Kr (c) ion bombardment.

Fig. 3. Morphology of the 10 nm thick In film deposited onto Chromium substrate without any ion bombardment (a) and after Ar and Kr ion bombardment. (Figs. 3(b and c) respectively).

Fig. 4. Morphology of the 60 nm thick In film deposited onto Chromium substrate as deposited (Fig. 1(a)) Ar and Kr ion bombarded (Figs. 4(b and c) respectively) cases.
thicknesses are much smaller here. We attribute this to the fact that the wetting between chromium and indium is much greater than that between silicon oxide and indium.

As comparison we also examined chromium layers that have a higher melting point, these results are shown in Figure 5. We implanted these layers with the same types of ions, and got fundamentally different results. None of the different thickness chromium layers changed considerably, and this did not change with the different bombarding ions either.

**Table I.** Summary of experimental parameters and results.

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<th>Substrate Layer</th>
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<th>Melting D, nm</th>
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**Fig. 5.** Morphology of the 60 nm thick Cr film deposited onto SiO<sub>x</sub> covered Si (100) substrate before and after Ar ion bombardment (Figs. 5(a and b) respectively).

**Fig. 6.** The size distribution of the 10 nm thick In film on SiO<sub>x</sub> (a) and on Cr (b) after Kr ion irradiation, and of the 60 nm thick film on Cr, after Ar ion bombardment (c).

For the quantitative characterization of the resulting nanoparticles we fitted the size distributions from the SEM images with a lognormal distribution, which is shown in Figure 6. The resulting functions resample the distribution of the nanoparticles very well, their parameters are shown in Table I.
From the results on the different metals (and here, we may also refer to our previous experiments concerning gold layers), we can conclude, that the melting point of the target is a decisive parameter. The melting point of indium, which shows much greater changes, is 156 °C, while that of chromium is 1875 °C.

We also tried ion bombardment with neon ions, but then the layers showed no change at all. This also fits into the pattern, as neon has the smallest mass among the bombarding ions.

4. DISCUSSION

4.1. The Critical Size of the Breaking Droplet

Let us consider a liquid slab (right after it is melted) of thickness δ. The surface area of this slab can be divided into small squares, each of the surface area \( a ' \alpha \). Let us suppose that such a small slab transforms into the spherical cup of radius \( R \) in case of a partially wetting liquid:

\[
\delta \cdot a' = \frac{4}{3} \pi \cdot R^3 \cdot f(\Theta)
\]

with \( f(\Theta) \) is a function of the contact angle:

\[
f(\Theta) = \frac{2 - 3 \cdot \cos \Theta + \cos^3 \Theta}{4 \cdot \sin^3 \Theta} \quad \text{for} \quad 0' < \Theta < 90'
\]

\[
f(\Theta) = 2 - 3 \cdot \cos \Theta + \cos^3 \Theta \quad \text{for} \quad 90' \leq \Theta < 180'
\]

Using Eq. (1) and the coverage fraction (denoted by \( C \)) of the substrate surface by the droplet can be defined and calculated as:

\[
C = \frac{R^2 \cdot \pi}{a'^2} = \frac{3}{4 \cdot f(\Theta)}
\]

The thickness of the layer and the contact angle (and thus the value of \( f(\Theta) \)) are considered to be parameters of the system to be known in advance. Then, for each value of \( a', R \) can be calculated from Eq. (1). We are searching for the critical value of \( a' \), (and the corresponding critical value of \( R \)) at which the surface energy of the “spherical cup” configuration becomes more favorable. The energy change of the transition from the “slab” into the “spherical cup” configuration is defined as: \( \Delta G = G_{\text{cup}} - G_{\text{slab}} \). The definition of the critical \( R_{\text{cr}} \) is that at any \( R \geq R_{\text{cr}} \): \( \Delta G \leq 0 \). The total surface energy of the “slab” and “spherical cup” configurations, and also the difference of these energies are:

\[
G_{\text{slab}} = a'^2 \cdot (\sigma_{l} + \sigma_{v})
\]

\[
G_{\text{cup}} = A_{l} \cdot \sigma_{l} + A_{v} \cdot \sigma_{v} + (a'^2 - A_{l}) \cdot \sigma_{sv}
\]

\[
\Delta G = (A_{l} - a'^2) \cdot \sigma_{l} + (A_{v} - a'^2) \cdot \sigma_{v} + (a'^2 - A_{l}) \cdot \sigma_{sv}
\]

where: \( \sigma_{l}, \sigma_{v}, \sigma_{sv} \) are interfacial energies at the liquid/vapor, solid/liquid and solid/vapor interfaces, while \( A \) with the same indices are the interfacial areas in the “spherical cup” configuration for the same interfaces.

Substituting the Young equation into Eq. (4c), the following general equation is obtained for \( \Delta G \):

\[
\Delta G = \sigma_{lv} \cdot [(A_{l} - a'^2) + (a'^2 - A_{l}) \cdot \cos \Theta]
\]

As follows from Eq. (4d), the solution will be determined by the expressions to be derived for the liquid/vapor and solid/liquid interfacial areas of the spherical cup.

First, let us consider the wetting case, when \( 0' < \Theta < 90' \). From the geometry of the spherical cup for wetting liquids:

\[
A_{lv} = 2 \cdot \pi \cdot R^2 \cdot \frac{1 - \cos \Theta}{\sin^2 \Theta}
\]

\[
A_{v} = R^2 \cdot \pi
\]

Substituting Eqs. (1, 5a,b) into Eq. (4d), the energy change for the wetting liquids is obtained as:

\[
\Delta G = \pi \cdot R^2 \cdot \sigma_{lv} \cdot \left[ \frac{2 \cdot (1 - \cos \Theta)}{\sin^2 \Theta} - \cos \Theta - \frac{R}{\delta} \right]
\]

\[
\times \frac{2 - 3 \cdot \cos \Theta + \cos^3 \Theta}{3 \cdot \sin^3 \Theta} \cdot (1 - \cos \Theta)
\]

Making Eq. (5c) equal zero, the equation for the critical ratio of \( R/\delta \) is obtained as:

\[
\frac{R_{\text{cr}}}{\delta} = \frac{1}{(1 - \cos \Theta) \cdot 2 - 3 \cdot \cos \Theta + \cos^3 \Theta}
\]

\[
\times \left[ 2 \cdot \frac{1 - \cos \Theta}{\sin^2 \Theta} - \cos \Theta \right]
\]

One can see that the ratio of the critical radius of the droplet to the initial thickness of the slab depends only on the contact angle. The coverage of the surface by the droplets is calculated by substituting Eqs. (7a, 13d) into Eq. (11):

\[
C = \frac{1 - \cos \Theta}{(2 \cdot (1 - \cos \Theta)/\sin^2 \Theta - \cos \Theta)}
\]

In the non-wetting case, from analogous equations we obtain:

\[
\Delta G = \pi \cdot R^2 \cdot \sigma_{lv} \cdot \left[ \frac{2 \cdot (1 - \cos \Theta)}{\sin^2 \Theta} - \cos \Theta - \frac{R}{\delta} \right]
\]

\[
\times \frac{2 - 3 \cdot \cos \Theta + \cos^3 \Theta}{3 \cdot \sin^3 \Theta} \cdot (1 - \cos \Theta)
\]

\[
\frac{R_{\text{cr}}}{\delta} = \frac{1}{(1 - \cos \Theta) \cdot 2 - 3 \cdot \cos \Theta + \cos^3 \Theta}
\]

\[
\times \left[ 2 \cdot (1 - \cos \Theta)/\sin^2 \Theta - \cos \Theta \right]
\]

\[
C = \frac{1 - \cos \Theta}{(2 \cdot (1 - \cos \Theta)/\sin^2 \Theta - \cos \Theta)}
\]
The results of the calculations by Eqs. (5d–e, 6d–e) are shown in Figures 7(a–b). One can see that the critical size of the droplet becomes infinitely high when the contact angle of the liquid approaches zero (perfect wettability), and the liquid covers the substrate by a thin film. Increasing the contact angle, the critical size of the droplet gradually decreases, reaching 3 times the thickness of the layer at $\Theta = 90^\circ$ and 1.5 times the thickness of the layer at $\Theta = 90^\circ$. The coverage of the surface by the droplet as function of the contact angle gradually decreases from $2/3$ for low contact angles towards $1/2$ for $\Theta = 90^\circ$ and $\Theta = 180^\circ$, passing through a minimum point at $120^\circ$. Thus, from the point of view of separating the nanoparticles as much as possible, the contact angle of $120^\circ$ seems to be an optimum. As the contact angle in the In/SiO$_2$ system is $128^\circ$ on macroscopic objects, this material combination is ideal.

4.2. Kinetics of the Process

Ion beam irradiation of the deposited layer takes place at a constant rate. Under these circumstances the schematic time dependence of the temperature of the irradiated layer is shown in Figure 8. First the layer is heated up, then it melts (with a T-stop), then the liquid layer is further heated. When the dose is finished, the layer cools down. Depending on the total dose of irradiation the deposited layer might not melt ($D_1$ in Fig. 8), might just melt and then immediately cool down ($D_2$ in Fig. 8), or might be overheated considerably before cooling down ($D_3$ in Fig. 8).

![Fig. 8. Schematic time dependence of the deposited layer temperature during irradiation with a constant rate, using different total doses (ion/m$^2$). $D_1 < D_2 < D_3$.](image)

Depending on the total dose of irradiation, the size distribution of the droplets is expected to be different (see Fig. 9). Below a certain ion dose there is no droplet formation at all, as the thin film is not melted (see $D_1$ in Figs. 8, 9(a)). The ideal case is at $D = D_2$ (see Figs. 8, 9(a)) when droplets are formed, but they have no time to undergo Ostwald ripening. In this case all droplets have the same size, supposing the thickness of the initial thin film was ideally uniform. However, when the ion dosing is too high ($D_3$ in Figs. 8, 9(a)), the droplets have enough time to undergo Ostwald ripening, which leads to the wider and wider size distribution of the droplets. If the initial thickness of the thin film was not uniform, even at $D_2$ there is a relatively wide size distribution of droplets (see Figs. 8, 9(b)). The size distribution widens further with increased ion dosing (Fig. 9(b)).

![Fig. 7. The critical relative size of the droplet as function of contact angle of the droplet on the substrate (a), and The surface coverage as function of contact angle of the droplet on the substrate (b).](image)

![Fig. 9. Size distribution of the droplets after thin film irradiation as function of ion dosing for a uniform (a) and non-uniform (b) thickness of the thin film.](image)
5. CONCLUSIONS

The changes in the morphology depend on the energy the bombarding ions transmit. The differences of the sputter yields of Ar and Kr (as shown in Table I) do not justify such great changes in the morphology but the energy, the different ions transfer to the nuclei of the atoms of the layer is rather different. Thus we conclude that the morphological changes depend on the amount of energy transferred. Besides the mass of the ion, the melting point of the layer is just as important a factor. On the other hand wetting between the layer and the substrate is also crucial, that is, if the layer wets the substrate, then it is much harder to abrupt. From all these we can conclude that the formation of the nanoparticles is due to the melting and re-crystallisation of the layers.

As an effect of the ion bombardment the morphology of metal layers changed. Nanosized islands and nanodots were created by thermal processes. The above described process may be a general method for fabricating nanosized objects from materials with low melting points. In these cases the resulting structures may be controlled by the energy and the dosage of the implanting ions.

It has been theoretically shown that the radius of the droplets is proportional to the initial thickness of the layer, and that the coefficient of proportionality depends only on the contact angle, decreasing with increasing the contact angle. If the uniform film is ion beam irradiated with an optimum dosage, droplets with a relatively narrow size distribution can be produced.

If combined with formerly used methods where we changed the sizes of the nanodots with ion bombardment,9,15–17 this method may well become a versatile tool to construct nano-objects.

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References and Notes


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