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PHASE COMPOSITION OF AMORPHOUS Se ISLAND LAYERS [*]

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Results of investigations of island amorphous selenium layers evaporated upon a carbon film are presented. The layers were heated at various temperatures. In all cases, selenium formed islands of almost perfectly hemispherical shape with various diameters. Such well-defined shape of the islands made it possible to calculate total mass of the islands. Experimental data indicates that heating of the layer causes an increase of the mass of islands up to 7 times in comparison with their mass prior to thermal processing. This is possible only if the islands grow by accepting atoms of the same kind adsorbed on the substrate. It follows that a part of the layer mass is accumulated in the gas adsorbed on the substrate. This part depends on the layer temperature, substrate material, evaporation rate, etc. Investigation of distribution of island diameters immediately after layer evaporation and during thermal processing of the layer allowed determination of conditions that are needed in order to be able to estimate defect density in the substrate from the measured density of the islands.

Keywords: thin film, selenium, island, defect, decoration

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1. Introduction

Recent years have been marked by rapid development of nanotechnology, extensive application of very thin layers, miniaturization of equipment, use of non-continuous layers. Some properties of the latter layers can vary with time. This can be caused by adsorbed molecules which have not yet merged into stable islands. Some works [2–5] provided a theoretical description of formation of stable nuclei and their growth from gaseous phase. However, it is difficult to identify such gaseous phase molecules visually, because their interaction with the electron beam in the electron microscope is very weak due to their low density. Besides, the adsorbed gas molecules can have rather high mobility, therefore they would be difficult to observe by other means (e. g., using atomic force microscopes), too. Therefore, in order to detect molecules of the evaporated material adsorbed on the substrate, indirect methods must be used. The purpose of this work is to demonstrate a method of measuring the quantity of gas adsorbed on the substrate during various stages of formation of the island layer, as well as a method of measuring defect density on the substrate, using an island layer. An amorphous island selenium layer has been used for the investigations.

2. Investigation technique

Island layers of amorphous selenium were evaporated onto carbon films secured on copper grids that are used as object holders in transmission electron microscopes. The samples were prepared in vacuum at a pressure of 10^{-4} torr by means of selenium evaporation from a directly heated tungsten trough. The process of evaporation from the trough was observed visually. When the material in the evaporator heated up and evaporation started, the damper between the evaporator and the substrate was opened. At the end of evaporation, this damper was closed in order to avoid further heating of the evaporated layer by the evaporator. The mass of evaporated material placed into the tungsten trough prior to evaporation was known. If this mass is sufficiently large, continuous layers are obtained. Composition of those layers can be determined by an interference microscope and, if density of the evaporated material is known, the unit area mass of the layer can be found. Thus, an empirical dependence of the layer unit area mass on the total mass evaporated from the tungsten trough was found. The substrate temperature was stabilized using a water

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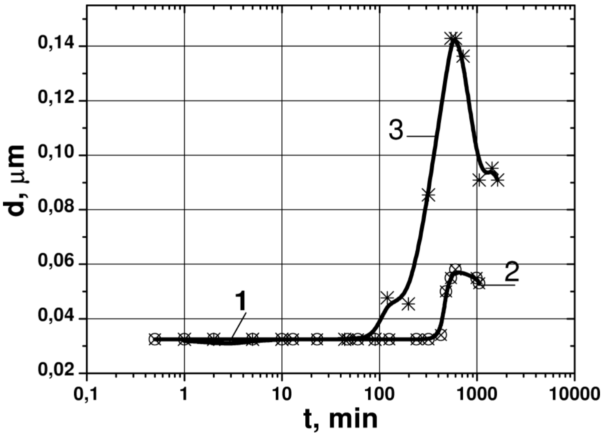

Fig. 1. Dependence of the minimum island diameter on heating duration. Layers are heated at temperatures 87 °C (*1*), 71 °C (*2*), and 57 °C (*3*).

thermostat. The layers were evaporated simultaneously onto 20 to 30 grids with carbon films. In this way, many samples evaporated under identical conditions were obtained. The samples were prepared at substrate temperature 20–25 °C, because at higher substrate temperatures larger amorphous selenium islands are obtained, whereas the mass of gas adsorbed on the substrate is smaller. The obtained layers were subsequently heated at temperatures 57, 71, and 87 °C. All islands formed in the samples were round in shape and not in contact with each other (the islands had not started coalescing yet). Consequently, the only possible mechanism of mass exchange between islands was via atoms adsorbed on the substrate. In the obtained island layers, largest and smallest island diameters as well as diameter distribution and the total perimeter of the islands were measured and the mass of the islands was calculated. The mass of an island can be calculated only when its shape is known. As it was mentioned above, electron microscope investigations of the islands have shown that they are round in shape. The island profile can be measured in the places where the carbon film is damaged and bent at an angle of 90°. In those places, the island profile is clearly visible. It was found that both the smallest and the largest islands are hemispheric. When the island shape is known, its volume can be calculated easily, and the product of the volume and material density gives the mass of the island.

3. Results

During heating of a layer at various temperatures, the minimum diameter of an island remains constant for a prolonged period of time (Fig. 1).

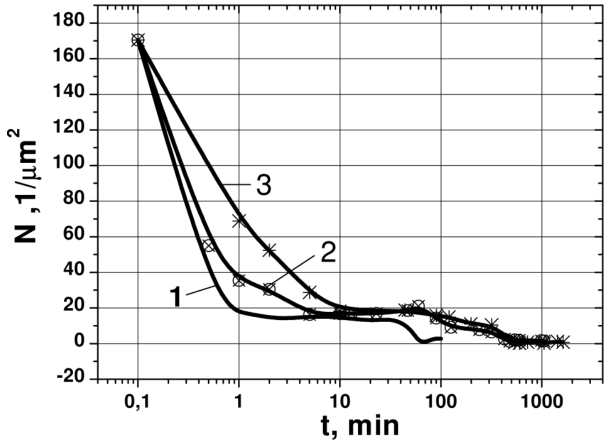

Fig. 2. Dependence of island density on heating duration. Layers are heated at temperatures 87 °C (*1*), 71 °C (*2*), and 57 °C (*3*).

Histograms if island diameters indicate that density of islands having the smallest diameter remains constant for about 100 min since the start of heating. The smallest islands do not disappear in the electron microscope during heating of the sample. If the islands were formed by homogeneous mechanism, then the minimum island diameter would decrease during heating. It follows that islands are formed by heterogeneous mechanism, i. e., on substrate defects [6, 7]. Thus, island density provides information about defect density on the substrate. However, island density does not always coincide with substrate defect density [8]. For example, at substrate temperature of 57 °C, the smallest islands disappear in 100 min, and at a temperature of 71 °C they disappear in 130 min (Fig. 1). Thus, in the case of prolonged heating of the layer, not all substrate defects will be marked by islands of the growing layer. In the case of short durations of thermal processing of the layer (e. g., immediately after the layer evaporation), island density is not equal to substrate defect density, too, because some islands can form by the homogeneous mechanism, i. e., in places where defects are absent. When such freshly evaporated layer is heated, mass exchange between islands begins. After evaporation, all islands are small and their diameters are similar. Therefore, only the islands that have been formed on substrate defects will remain after heating, whereas the other islands will disappear. Thus, during initial stage of heating of a freshly evaporated layer (Fig. 2), island density decreases (the homogeneously formed islands disappear). Later on, island density remains constant for some time: islands formed on substrate defects survive the heating, so that island density (about 18 μm^{-2}) during that time coincides with substrate defect density and each island corresponds to a unique defect of the substrate. With further heating of

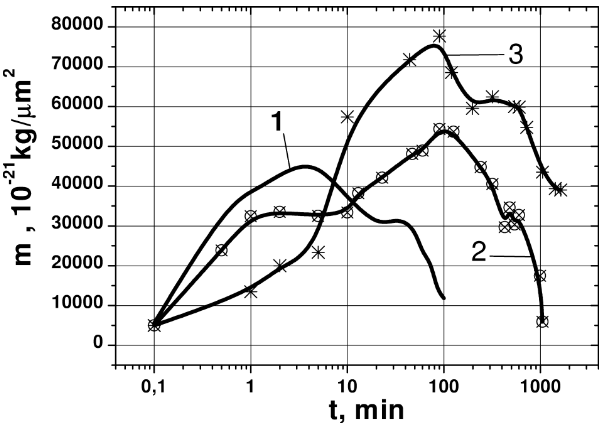

Fig. 3. Dependence of island mass on heating duration. The layers are heated at temperatures 87 °C (*1*), 71 °C (*2*), and 57 °C (*3*).

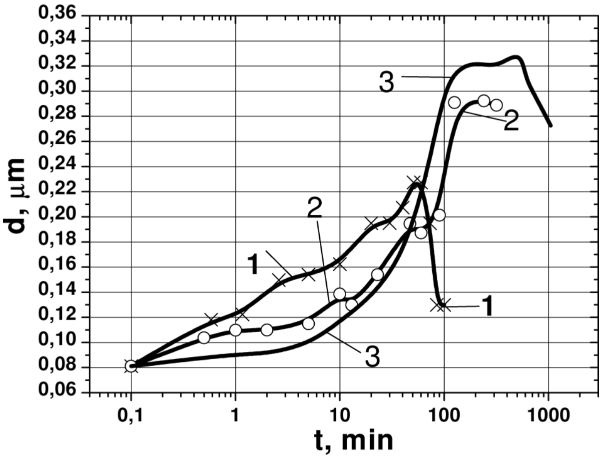

Fig. 4. Dependence of island maximum diameter on heating duration. The layers are heated at a temperature of 87 °C (*1*), 71 °C (*2*), and 57 °C (*3*).

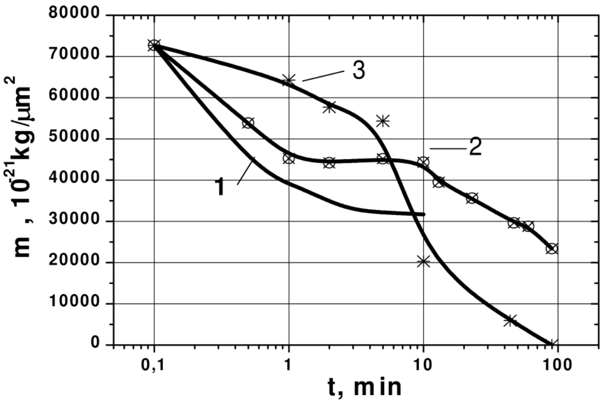

Fig. 5. Dependence of the mass of gaseous phase part of the layer on heating duration. The layers are heated at 87 °C (*1*), 71 °C (*2*), and 57 °C (*3*).

the layer, island density begins to decrease again (this corresponds to disappearance of islands formed on substrate defects – during this interval of time island density will be smaller than substrate defect density).

During heating of the layer, the part of its mass that is accumulated in the islands at first increases and then begins to decrease (Fig. 3). Since there is no evaporation from the evaporator, the only possible reason of the increase of island mass is inclusion of molecules of evaporated material absorbed on the substrate into the growing islands. The island mass reaches a maximum and then starts to decrease with further heating of the layer. This is a result of layer's gradual vaporization. The decrease of island maximum diameter with further heating of the layer (Fig. 4) confirms this conclusion. The total mass of the layer consists of the total mass of the islands and the mass of adsorbed gas. During the measurements, the total mass of the layer does not change until layer vaporization starts. The total mass of the layer can be found knowing the amount of material evaporated from the evaporator or assuming that the maximum mass accumulated in the islands is equal to the total mass of the layer. We determined the total mass of the layer using the second method. When this mass is known, the mass of adsorbed gas at various moments of time can be found easily (Fig. 5). As we see in Fig. 5, the mass of adsorbed gas of evaporated material monotonously decreases after preparation of the layer. The rate of this decrease increases with substrate temperature. In a freshly prepared layer, the larger part of the layer mass can be accumulated in the gas adsorbed on the layer surface. Those adsorbed molecules are much more mobile than molecules in the islands, because they do not form strong bonds with each other. Therefore, the adsorbed gas can react more easily with molecules of another material that reach the substrate (e. g., during evaporation of another layer), and it can form crystals more easily than on the layer surface or in its bulk (crystals often grow between amorphous islands). Thus, it is important to know the amount of adsorbed gas on the layer surface.

4. Summary

We have shown in this work that defect density on a substrate can be determined by deposition of an island layer. This is done during the period of layer heating after island density stops decreasing but before the smallest islands start disappearing: then island density is equal to substrate defect density. The mass contained in the gaseous phase part of the island layer can be determined if the layer has not yet started vaporizing (the maximum island diameter and thus the mass of the layer contained in islands have not yet started decreasing). Then, by knowing the total mass of the layer and

the mass contained in islands (which can be calculated, if the shape of the islands is known), we can determine the mass of adsorbed gas of the evaporated material on the substrate.

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AMORFINIO Se SALELINIŲ SLUOKSNIŲ FAZINĖ SUDĖTIS

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Santrauka

Tyrinėjami amorfiniai saleliniai seleno sluoksniai, užgarinti ant anglinių padėklų. Sluoksniai buvo kaitinami įvairioje temperatūroje. Visais atvejais seleno salelės turėjo pusės sferos formą. Tai leido apskaičiuoti sluoksnio masę, sukauptą salelėse. Sluoksnį kaitinant bendra salelių masė didėja iki 7 kartų. Tai įmanoma tik tada, kai prie salelių prisijungia įgerti ant padėklo užgarintos medžiagos dujinės fazės atomai. Todėl galima teigti, kad po užgarinimo dalis sluoksnio masės yra dujinės fazės. Dujinės fazės kiekis priklauso nuo sluoksnio garinimo spartos, nuo padėklo temperatūros, padėklo medžiagos.

Ištyrus salelinio sluoksnio salelių tankio, didžiausio ir mažiausio salelių skersmens kinetiką, esant įvairiai padėklo temperatūrai, galima įvertinti padėklo defektų tankį ir net konkrečiai nurodyti defektą ant padėklo.