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ABSTRACT

The density of glow discharge deposited hydrogenated amorphous silicon obtained at 250°C on high purity Al foil was measured by the density gradient method. The density of the films is 2.18 g/cm^3, i.e. there is a ~6 % density deficit compared with c-Si which in our case is not due to its porous structure. We found the density gradient method a quick, convenient and very sensitive method for checking the quality of a-Si:H films.

АННОТАЦИЯ

Нами изучалась плотность пленок a-Si:H, полученных с помощью разложения силана (SiH_4) в высокочастотном газовом разряде методом градиента плотности. Плотность пленок составляет 2,18 г/см^3, т.е. по сравнению с плотностью c-Si имеется дефицит ~6%, который в нашем случае не связан с пористой структурой пленок. Метод градиента плотности оказался быстрым, удобным и очень чувствительным для контроля качества пленок a-Si:H.

KIVONAT

A ködfénykisülés módszerével nagytisztaságu Al fóliára 250 C°-on leválasztott hidrogénezett amorf szilicium sürüséget a sürüséggradiens módszerével vizsgáltuk. A filmek sürüsége 2,18 g/cm^3. A kristályos sziliciuménál mintegy 6 %-kal kisebb sürüséget esetünkben nem a filmek porózus szerkezete okozza. A sürüséggradiens módszert egyszerünek, gyorsnak és igen érzékenynek találtuk az a-Si:H filmek minõségének ellenõrzésére.

INTRODUCTION

Hydrogenated amorphous silicon (a-Si:H) thin films prepared by the glow discharge (GD) deposition of silane are considered as promising active material for solar cells, high current diodes, light flux meters, etc. [1]. Because these films can also be recrystallized by laser light of appropriate wavelength and intensity [2] new fields of application are opened up.

It is well known that the properties of the films depend first of all on the preparation conditions but they can be further modified by heat treatment and irradiation. The parameters to be controlled during the plasma deposition process are quite a few, making the task of obtaining samples with reproducible properties rather difficult.

Structural investigations have shown that films prepared under different conditions differ remarkably: they might contain pores or be compact with densities 60-90 % of that of the crystalline silicon [3,4].

Aiming at a quick and convenient method to check the quality of GD deposited films we arrived at measuring their density. The density gradient method has been found simple, highly reliable and sensitive for this purpose. This communication is an account of our first density measurements carried out by this method on GD deposited a-Si:H films prepared on different substrates and annealed at different temperatures.

SAMPLE PREPARATION

We used an inductively coupled plasma-decomposition system similar to those used by others working in this field. The diameter of the pyrex glass reaction tube was 120 mm with a gas inlet tube at the top. The distance between the horizontal sample holder and the lower edge of the RF coil was 50 mm. Silane was diluted with Ar at a ratio of 1:50. The operating conditions (gas pressure ~0.1 torr, RF power ~5 W) were chosen in such a way as to eliminate the formation of yellow dust particles. The substrate temperature was held at 250^{o}C.

We tried but failed to use NaCl monocrystals and pressed KBr discs as substrates. Good quality a-Si:H films remaining intact after the substrate had been dissolved we could get only on high purity aluminium foils. To free the film the aluminium substrate was dissolved in $HgCl_2$ solution, e.g. in 5.5 % water solution, 15-30 minutes was needed to separate the a-Si:H films. They were subsequently washed several times in distilled water and dried in exsiccators at room temperature.

For annealing the free a-Si:H films they were placed in between two silica plates and heated for one hour at either 500 or 700^{o}C in a quartz tube flushed continuously with pure nitrogen gas. The nitrogen flow was on before the commencement of the heat treatment and was stopped after the sample had cooled down to room temperature.

DENSITY MEASUREMENT

The density gradient method we used is described in detail elsewhere [5]. The vessel used by us consisted of two bulbs of ~150 cm^3 connected together with a 300 mm tube with an internal diameter of 18 mm (see inset in Fig. 1). An ideally linear density gradient formed in this connection tube in two days if the two liquids of different density were introduced carefully and then

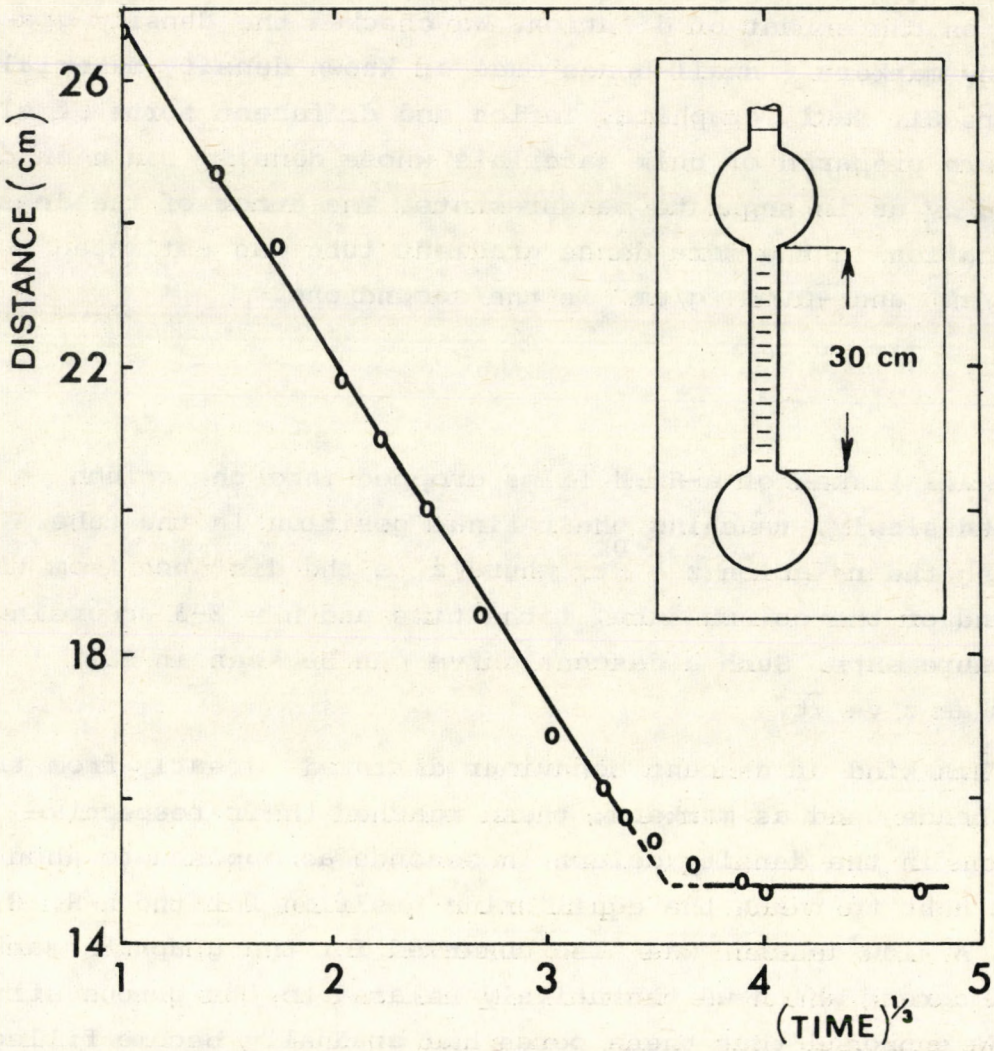

Fig. 1. Descent of GD deposited a-Si:H film flakes in the linear density column. For details see the text. Inset: sketch of density column vessel.

mixed as described in [5]. The density gradient remained linear for several days if the vessel was thermostated with an accuracy of $\pm 0.2^{\circ}C$.

We used two columns: one filled with methylenebromide and ethylenebromide with densities 2.7 and 2.2 g/cm^3 respectively, and another with ethylenebromide by itself and diluted with

ethanol covering the density range 2.2-2.1 or 2.2-2.0 g/cm^3 depending on the extent of dilution. We checked the density gradients by markers - small beads made of known density materials, e.g. pure Si, NaCl, graphite, Teflon and different sorts of glass. These were prepared of bulk materials whose density had been determined by us in separate measurements. The error of the density determination in the more dense gradient tube was estimated to be $\pm$0.01 g/cm^3 and $\pm$0.001 g/cm^3 in the second one.

RESULTS

Small flakes of a-Si:H films dropped into the column descended slowly, reaching their final position in the tube following the relation $z \propto \sqrt[n]{t}$, where z is the distance from the upper end of the column tube, t the time and n = 2-3 according to our measurements. Such a descent curve can be seen in Fig. 1 plotted as z vs $\sqrt[3]{t}$.

This kind of descent behaviour differed greatly from that of the beads used as markers; these reached their respective positions in the density column in seconds as opposed to about half an hour to reach the equilibrium position for the a-Si:H flakes. A slow descent was also observed for the graphite markers (pencil cores) which we tentatively related to its porous structure. We supposed that these pores had gradually become filled with the column liquid resulting in its slow descent. Influenced by the analogy of the descent curves of a-Si:H films and porous graphite pieces we checked the structure of the former by SEM. As shown in Fig. 2 no pores were found in our a-Si:H films: they were compact and showed no columnar structure at a $2.2 \cdot 10^4$ magnification.

We could not detect any difference in the densities of the as-prepared and the annealed samples: in all cases the density was 2.18 g/cm^3. The density of the samples deposited on glass and NaCl at room temperature varied between 2.0 and 2.1 g/cm^3.

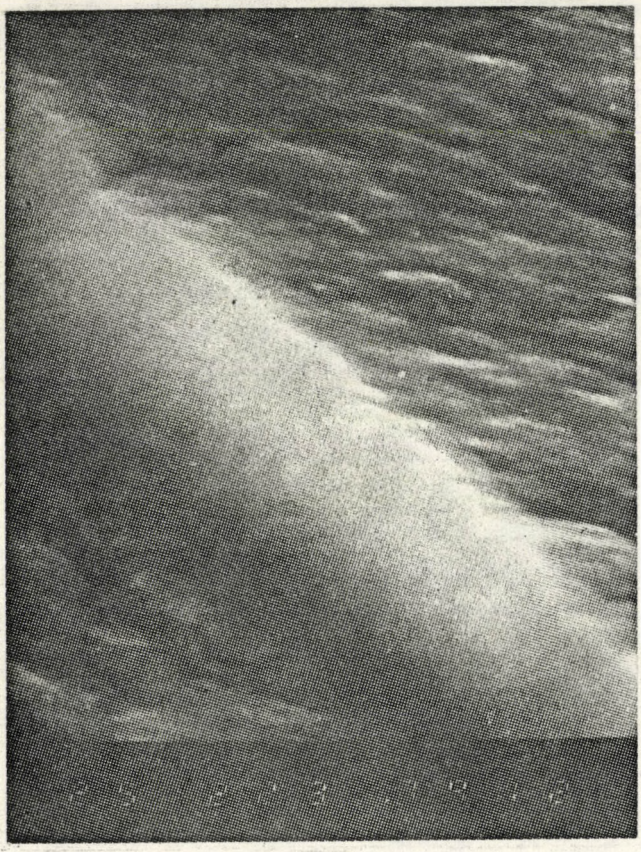

Fig. 2. SEM picture of fructured a-Si:H film. Magnification 22000.

No systematic measurements were made with the latter samples.

CONCLUSIONS

The density of good quality GD deposited a-Si:H films on high purity aluminium foil substrate at 250°C is 0.935 relative to the density of crystalline Si ($d = 2.33\ g/cm^3$). The density deficit in our sample was not due to pores found by others in films deposited in different conditions. The small ~6 % density deficit can probably be accounted for by the density fluctuations accompanying the polygonal domain networks first shown in a-Ge films by Barna et al. [6] with the help of TEM and substantiated theoretically by J.C.Phillips (see e.g. [7]).

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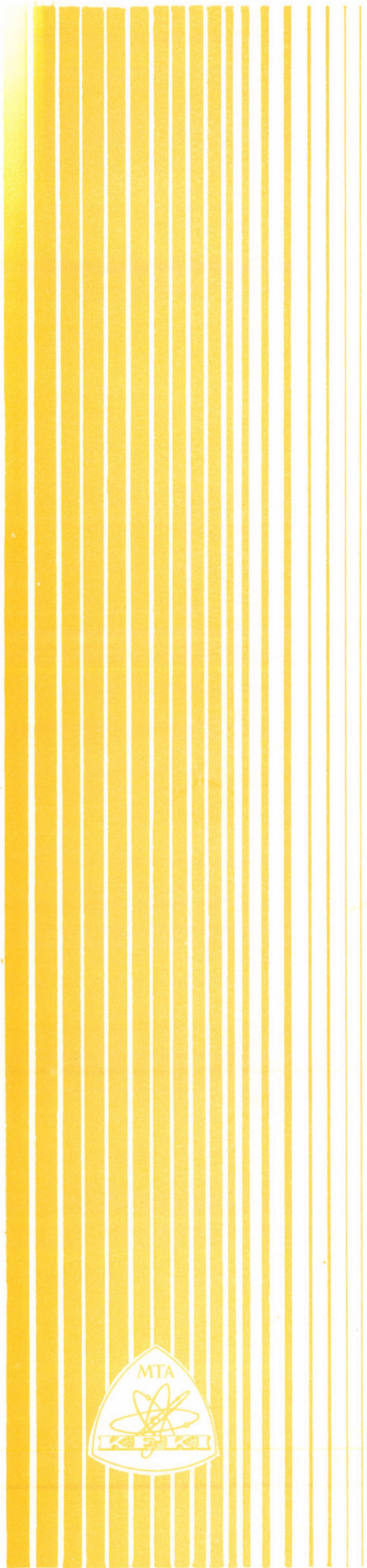

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