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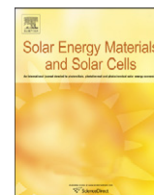

Structural study of device quality silicon germanium thin films deposited by pulsed RF plasma CVD

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ABSTRACT

Microstructures in silicon germanium thin films deposited by pulsed rf plasma CVD have been studied with the help of small-angle X-ray scattering (SAXS) and high-resolution transmission electron microscopy (HRTEM). With lowering of the pulse duty cycle the size of the particles incorporated in the films from the plasma decreases. However, the particles become more symmetric in shape and crystalline in nature. At 75% duty cycle the films have the highest photosensitivity. The increase in SAXS scattering at 75% has been explained by the formation of uniform-size nanocrystallites of SiGe. Urbach energy variation with the duty cycle also suggests the formation of nanocrystallites.

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1. Introduction

We have recently shown that the transport properties like diffusion length (L_d) and mobility-lifetime ($\mu\tau$) product in a-$Si_{1-x}Ge_x$:H thin films can be significantly improved by controlling the duty cycle (DC) in square wave pulse-modulated rf (13.56 MHz) plasma-enhanced chemical vapour deposition (PECVD) using a pulse frequency of 1356 Hz [1]. The particles incorporated from the plasma have very strong influence on the microstructure of the a-Si-related films [2,3]. Hence, proper attention is needed to obtain a clear picture of the dependence of the microstructure of these films on the duty cycle. Furthering this work, we report in this article, studies of the microstructure in the films by small-angle X-ray scattering (SAXS) and high-resolution transmission electron microscopy (HRTEM). The photosensitivity and the sub-band gap absorption of these films were measured and correlated with the microstructural data.

2. Experimental

The a-$Si_{1-x}Ge_x$:H samples were deposited from a mixture of silane, germane and hydrogen at the flow rates of 2.8, 0.7 and 96.5 sccm, respectively, by square-wave pulse-modulation (SWPM) of rf (13.56 MHz) PECVD with the rf power fixed at 25 W. The pulse frequency was 1356 Hz. The values for pulse duty cycle defined by $DC = T_{on}/T_{on}+T_{off} \times 100\%$, where T_{on} and T_{off} are the plasma "on" and "off" times, respectively, were 100% (continuous mode), 85%, 75% and 50%. The plasma "on" time under the pulsed conditions varied between 368 and 627 μs. For SAXS study thick (2 μm) samples were deposited on 99.999% pure Al foils. SAXS study was performed by using Cu-K_α radiation ($\lambda = 0.154$ nm) with the scattering angle 2θ ranging between 0.13° and 8.8°, corresponding to momentum transfer q ($=(4\pi/\lambda)\sin\theta$) value between 0.1 and 6.2 nm^{-1}. For HRTEM study thin (500 Å) samples were deposited on C-coated Cu grids. For optical absorption measurements and conductivity measurements, ~5000-Å-thick samples were deposited on Corning 7059 glass substrates. The sub-band gap absorption characteristics of the samples were studied by dual-beam photoconductivity (DBP) method.

3. Results

3.1. SAXS

SAXS can detect inhomogeneity in a-$Si_{1-x}Ge_x$:H films primarily due to nanostructural electron density fluctuations on a scale from 1 to 30 nm but can also indicate the presence of larger size (>30 nm) features. From SAXS intensity $I(q)$ vs. q plots for the four samples deposited at DC = 100% (continuous mode), 85%, 75% and 50%, the micro/nanostructural heterogeneities defined by $Q_N = \int I(q)q^2\,dq$ have been calculated and plotted in Fig. 1. The larger size features quantified by the Porod constant A obtained from the relation $I_L = Aq^{-3}$, where I_L is the scattering due to features of dimension >30 nm [4], are also shown in the same figure. We observe that as the plasma is changed from continuous to the SWPM mode, Q_N increases but the magnitude of A decreases. Decrease of A at low duty cycles implies decrease of

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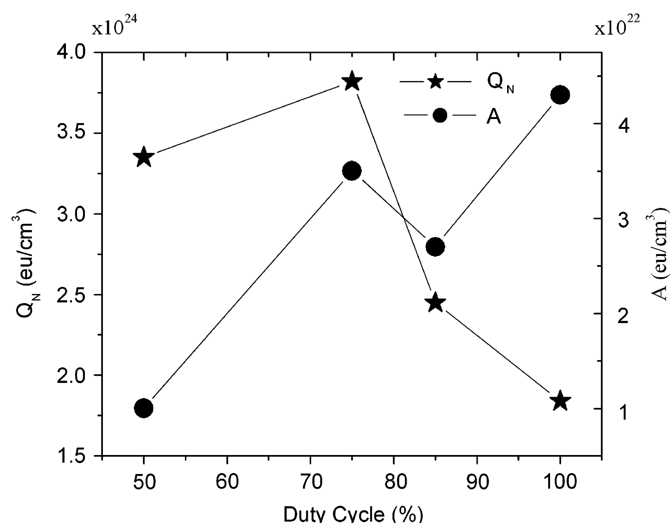

Fig. 1. Variation of SAXS microstructure factor Q_N and the Porod constant A in a-$Si_{1-x}Ge_x$:H films deposited at different pulse duty cycles (lines are guides to the eye).

Table 1
Properties of a-$Si_{1-x}Ge_x$:H films.

Duty cycle (%)	Urbach energy (E_u) (meV)	DOS (cm^{-3})	$Q_N(0^\circ)/Q_N(45^\circ)$
100	65	1.35e16	3.1
85	88	2.0e16	
75	88	3.0e16	1.3
50	88	3.5e16	

large size (>30 nm) objects within the films. The increase of Q_N can be due to several possible reasons such as nanovoids, Ge-density fluctuations and nanocrystallites formation [5]. Also, if the microstructure becomes more anisotropic with elongated features along the growth direction, the SAXS can be significantly enhanced. This effect is revealed by taking the ratio of Q_N with the sample in non-tilted (0°) mode and in the 45° tilted mode relative to the X-ray beam. SAXS tilting results show that the ratio $Q_N(0^\circ)/Q_N(45^\circ)$ decreases sharply from 3.1 at DC = 100% to 1.3 at DC = 75% (Table 1). This observation implies that the film deposited at DC = 75% contains objects more isotropic (spherical) than those within the films deposited in continuous mode. The values of Q_N for all the films under this study are, however, low compared to those reported for silicon germanium films of the same band gap deposited by conventional PECVD technique [5].

3.2. HRTEM

The HRTEM micrographs of the films deposited at different duty cycles show embedded nanoparticles in the films (Figs. 2 and 3). We observed a gradual decrease in the number and average size of these particles with the lowering of the duty cycle. Morphologically the films appear to be quite different. A comparison of the particle size distribution within the films deposited at DC = 100% and 75% is given in Fig. 4. The DC = 100% film has a large variation in the size of the nanoparticles with an average size of about 13 nm. Most of these nanoparticles are amorphous and only a few isolated clusters of nanocrystallites are observed. The fringe pattern of one such cluster is shown in the inset of Fig. 2. On the other hand, the DC = 75% film has large number of smaller size nanocrystallites distributed uniformly within the film. These nanocrystallites have better uniformity in size than the DC = 100% films and their average size is about 8 nm. The fringe patterns of the nanocrystallites correspond to crystalline SiGe with compositional variation between $Si_{0.4}Ge_{0.6}$ and $Si_{0.6}Ge_{0.4}$ (inset Fig. 3).

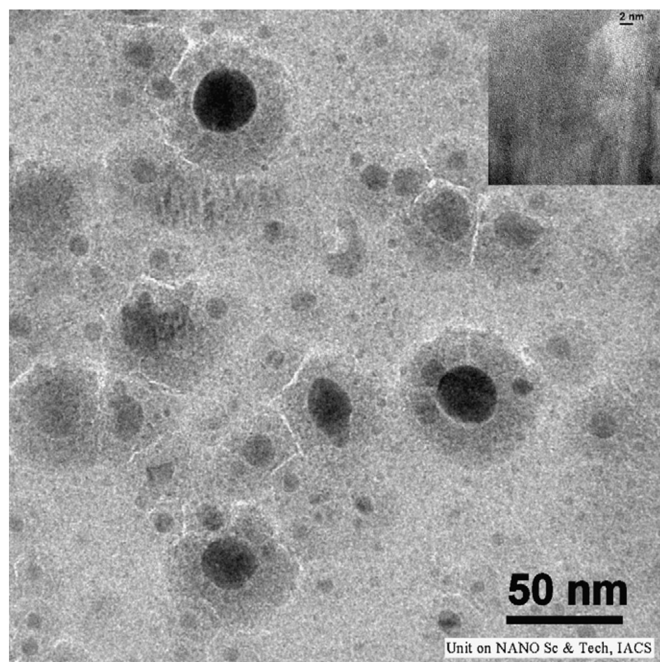

Fig. 2. HRTEM micrograph of 100% duty cycle (continuous mode) a-$Si_{1-x}Ge_x$:H sample. The inset shows the fringe patterns due to formation of SiGe nanocrystallites.

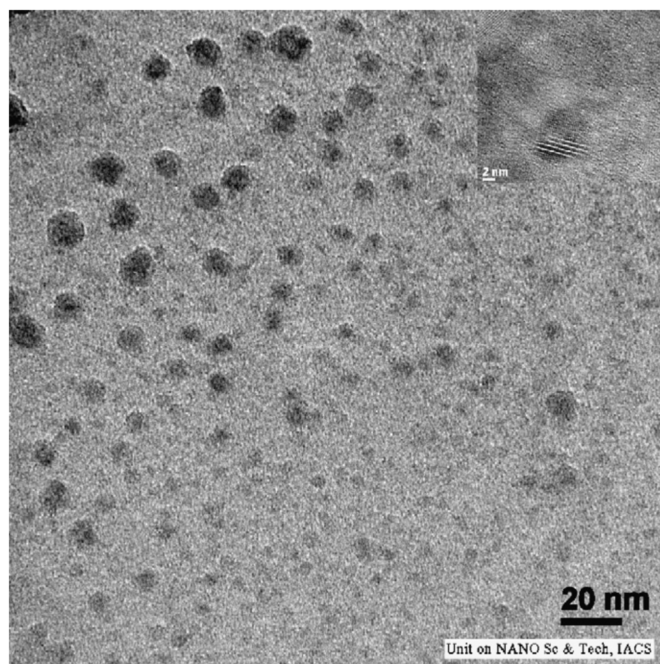

Fig. 3. HRTEM micrograph of 75% duty cycle a-$Si_{1-x}Ge_x$:H sample. The inset shows the fringe patterns due to formation of SiGe nanocrystallites.

3.3. Photosensitivity

Photosensitivity (σ_{Ph}/σ_D) of the films deposited at different duty cycles are shown in Fig. 4. Photosensitivity sharply increases as we go from the continuous mode to the SWPM mode. The film deposited at

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