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Structural and photo-luminescence properties of nanocrystalline silicon films deposited at low temperature by plasma-enhanced chemical vapor deposition

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Abstract

Nanocrystalline silicon (nc-Si) films were prepared by a plasma-enhanced chemical vapor deposition method at a deposition temperature below 220 °C with different dynamic pressures (P_g), hydrogen flow rates ([H_2]), and RF powers, using $SiH_4/H_2/SiF_4$ mixtures. We examined the photo-luminescence (PL) spectra and the structural properties. We observed two stronger and weaker PL spectra with a peak energies around $E_{PL} = 1.8$ and 2.2–2.3 eV, respectively, suggesting that the first band was related to nanostructure in the films, and another band was associated with SiO-related bonds. The nc-Si films with rather large PL intensity was obtained for high [H_2] and/or low pressure values, However, effects of [H_2] are likely to be different from those of P_g. The average grain size (δ) and the crystalline volume fraction (ρ) at first rapidly increase, and then slowly increase, with increasing P_g. Other parameters exhibited opposite behaviors from those of δ or ρ. These results were discussed in connection with the changes in the PL properties with varying the deposition conditions.
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1. Introduction

The recent trend in the development of semiconductors usable as optical devises is to prepare quasi-direct-gap semiconductors, as nanorystalline silicon (nc-Si) films, which exhibit rather strong visible photo-luminescence (PL) spectra at room temperature [1], from indirect-gap semiconductors. So, many research workers investigated semiconductors exhibiting such strong PL spectra [1–3], in which, the mainstream was based on the quantum confinement effects in the semiconductors, such as nc-Si [1], Si-based luminous alloys prepared using siloxene [2], and polysilane [3]. Furthermore, formation of nc-Si structures was attempted to utilize different techniques; as the formation of porous Si [1,4], and plasma-enhanced chemical vapor deposition (PECVD) [5–8], sputtering [9,10], evaporation [11,12], and ion beam synthesis [13].

However, mechanism of the PL process has not yet been understood well, which was the issue for the device application. In this work, we mainly changed the dynamic pressure, P_g, along with the changes in the hydrogen flow rate, [H_2], in the supplied RF power below 30 W, and in the deposition temperature, T_d, below 220 °C. For these nc-Si films, we studied the optical properties, such as PL, Fourier-transform infra-red (FT-IR) absorption and optical absorption, and the structural properties, such as X-ray diffraction (XRD), Raman scattering and electron spin resonance (ESR). Furthermore, changes in the PL properties were discussed in connection with the structural properties.

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2. Experiments

PECVD nc-Si films were deposited at T_d = 100 or 220 °C with different P_g, ranging from 20 to 80 Pa by RF glow-discharge (13.56 MHz) of $SiH_4/H_2/SiF_4$ mixtures, in a hot wall-type inductively coupled fused quartz-reactor. In this work, we used the H_2 flow rate of $[H_2]$ = 5, 15 or 30 sccm under a fixed condition of $[SiH_4]$ = 0.5 sccm. The SiF_4 gases were diluted with He (SiF_4; 5%, He; 95%) and the net SiF_4 flow rate was $[SiF_4]$ = 0.1 sccm. In a previous paper [14], we found that the average grain size at the <1 1 1> direction, which were estimated using the width of the corresponding XRD signal, exhibited the minimum value at around $[SiF_4]$ = 0.1 sccm. So, we selected the deposition conditions of this $[SiF_4]$ value in this work.

The values of the average grain sizes, $<\delta$ (1 1 1)$>$ and $<\delta$ (1 1 0)$>$ at the <1 1 1> and the <1 1 0> directions, respectively, were estimated utilizing Scherrer's formula [15] using the full-width at half maximum (FWHM) values of the corresponding XRD signal measured by an XRD instrument (SHIMAZU XD-D1). The Raman spectra were measured by a Raman spectrometer excited with 35-mW Ar–ion-laser light at 488 nm, having a double monochrometer (Joibin Yvon RAMANOR HG 2S) coupled with a cooled phto-multiplier tube (Hamamatsu R649S). The difference in the sensitivity factor for the XRD spectra with different textures was corrected using the intensity of the corresponding signal observed for c-Si powder. The Raman spectra were divided into two elements as follows: the 520 cm^{-1} component due to crystalline phase (c-phase), and the 480 cm^{-1} component due to amorphous phase (a-phase). The FWHM values for the 520-cm^{-1} component were assessed. A change in the FWHM values should reflect the degree of the strain around the crystallites. Furthermore, in order to investigate the effects of He addition in the SiF_4 gas, we prepared a new sample with [He] = 9.5 sccm only. The deposition conditions are the same as those for other samples. Furthermore, we used the supplied RF power conditions of 10 or 30 W. we cleaned the substrates by exposing them for 20 min, in 90-W nitrogen plasma followed by the cleaning process in 90-W hydrogen plasma, immediately before the film deposition.

The crystalline volume fraction (crystallinity), ρ, was determined from the intensity ratio of the 520-cm^{-1} component to the 480-cm^{-1} component, using the procedure by Tsu et al. [16]. However, practically, the Raman spectra should include the third component with frequencies between 480 and 520 cm^{-1}, which would be caused by the component composed of smaller crystallites that cannot be detected as the 520 cm^{-1} components, and its intensity was smaller than those of other two components, Therefore, we ignored the contribution of the third component to crystallization of the films. The PL spectra were also observed utilizing the above Raman spectrometer. These films were simultaneously deposited on Corning 7059 glass substrates for XRD and Raman scattering measurements, on n-type-(100) single crystalline Si (c-Si) substrates having a resistivity higher than 10^3 Ωcm for FT-IR absorptions, and on fused quartz substrates for PL, optical absorption and ESR. The film thickness was fixed to be approximately 0.35 (±0.05) μm. The difference in the film thickness was also corrected using the X-ray absorption coefficient for Si.

Optical absorption was measured using a spectrophotometer (JASCO V-570). The optical band gap, E_g^{opt}, was obtained through the Tauc's plots. Structural properties were assessed by a FT-IR absorption spectrometer (JASCO FT/IR-610). We also observed ESR spectra under room temperature using an x-band spectrometer (JEOL JESRE 1X) with the magnetic-field modulation frequency of 100 kHz. Although the ESR profiles were complex and two or more kinds of defects may be included in a film, we evaluated the ESR spin density, N_s, using the integrated intensity for simplicity. Then, in the ESR measurements, details in determining these ESR parameters are described elsewhere [17]. For a part of the ESR measurements, we also measured for new samples grown on a c-Si substrates with 0.1-μm-thick thermally grown SiO_2 layer to clarify the effects of different substrates on the ESR spectra. As a result, we found no significant difference in the defect properties between both films. This is because the use of crystalline Si-based substrates with SiO_2 layers would be hard to perform other experiments, such as the PL, the XRD and the Raman measurements, in addition to the use of the quartz or the glass substrates. Unfortunately, we did not examine these effects.

3. Results and discussion

3.1. Results

As well as P_g, $[H_2]$, RF power and T_d were varied to examine the effects on PL spectra. Fig. 1 shows the PL spectra for nc-Si

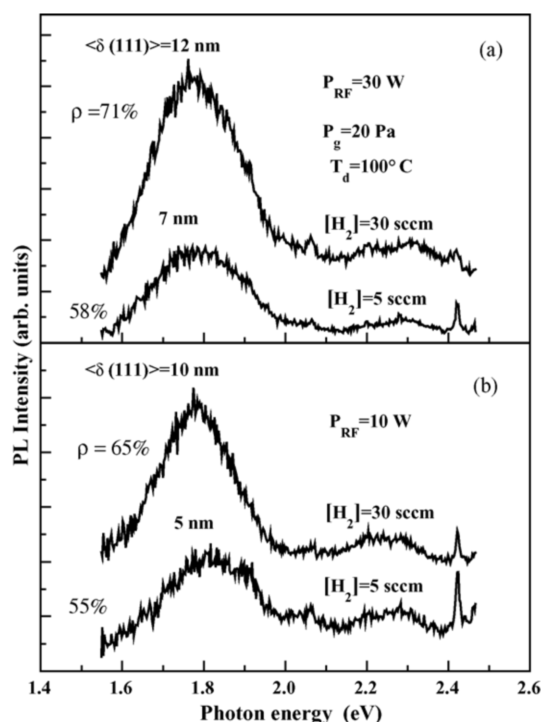

Fig. 1. The PL spectra as a function of photon energy for the nc-Si films with T_d = 100 °C: (a) an RF power of 30 W and a fixed P_g value of 20 Pa, and (b) the RF power of 10 W under the same deposition conditions as those in (a). Furthermore, the values of $<\delta$ (1 1 1)$>$ and ρ were shown for comparison.

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