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Diffraction Grating and Periodic Surface on Porous Silicon

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Abstract

Fabrication of micrometer size laser-induced periodic surface structures (ripples) on single crystalline silicon by laser beam irradiation of wavelength (810 nm) in HF electrolyte has been reported. Nonlinear changes of the refractive index are observed due to the interaction of laser light with silicon nanoparticles. Spatial self-phase modulated optical fringes were used to study the nonlinear optical response of nanocrystalline silicon produced by laser-induced etching process.

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الخلاصة

تم تحضير سطح سليكوني مسامي ذو تركيب دوري متكرر باستخدام طريقة الليزر المحتث وذلك بواسطة التشعيع بطول موجي 810nm في حامض الهيدروفلوريك المركز. لوحظت التغيرات اللاخطية لمعامل الانكسار من خلال تفاعل الليزر مع السليكمون النانوي. استخدمت اهداب التاخل البصري المتظمنة للطور الذاتي الفضائي لدراسة الاستجابة البصرية اللاخطية للسليكون النانوي المنتج بعملية التنميش المحتث بالليزر.

1. Introduction

Laser-induced periodic surface structures have been reported for many kinds of materials [1-4]. The period Λ is usually close to the laser wavelength λ. These experiments were sufficiently explained by the theory of interference between the incident laser and the surface scattered wave [5,6]. The observation of a strong reflection from the modified regions occurring only along the direction of the polarization of the writing laser has given the evidence of periodic behavior. The subwavelength grating like distribution in the irradiated spot inside the silicon could also explain the birefringence phenomenon [7]. Surface ripples with a period equal to wavelength of incident laser radiation and that are likewise aligned in a direction perpendicular to the electric field of light wave have been observed in many experiments involving laser deposition [8] and laser ablation [9]. Such gratings are generated as a result of interference between the light field and the surface plasmon-check wave launched because of initial random surface inhomogeneties [10].

The phenomenon is interpreted in terms of interference between the incident light field and the electric field of bulk electron plasma wave, resulting in periodic modulation of electron plasma concentration and permanent structural changes in porous silicon.

In semiconductors, a large number of grating investigations have been performed for the following reasons; First, forced light scattering at laser – induced gratings allows the study of changes in optical material properties due to a variety of nonlinear optical mechanisms. The absorption and

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refractive index changes are important for applications in real – time holography, phase conjugation, optical bistability, optical grating and laser mode – locking. These applications are facilitated because semiconductors are fairly well understood materials available in excellent quality and their physical properties can be controlled by chemical or radiation treatment. Second, lasers – induced gratings in semiconductors are of interest in studying the mobility of electron and holes, especially their transport and decay parameters. These material properties are important for the fabrication and understanding of electronic and optoelectronic devices like transistors, photo detectors and laser. There are three possibilities for the origin of the scattering in porous silicon thin film: air / P-Si interface or the inner surface of the material and the P-Si / bulk Si interfaces.

The photosensitivity of silicon was given in 1992 by Noguchi [11], who synthesized porous silicon simply by illuminating a Si substrate in a hydrofluoric acid solution. This effect is expected to be amplified owing to the extraordinarily large specific surface of the material. On other hand, by changing the porosity of porous silicon, it is possible to cover a broad range of refractive indices, which has also made possible the production of waveguide structures, [12] and optical superstructures like Bragg reflectors and Fabry-Perot filters [13]. In another case, PS structures are generated in the layer plane, the principle based on the photosensitivity of the material and on an interference technique. Holographic techniques and surface lithography are already used in industry to obtain diffraction gratings. Diffraction gratings on porous silicon were obtained rather uniform energy distribution between diffraction orders [14].

Laser-induced periodic surface structures (LIPSS) have been extensively investigated on different materials for over two decades using a wide variety of laser sources from low power CW laser [8] to nanosecond [15] and femtosecond [16,17] in the wavelength range from 0.240 to 10.6 μm [18,19].

The formation of periodic structures or ripples on the surface of semiconductors is useful for the fabrication of gratings. Periodic or ripple structures have a variety of potential applications, where increased surface area is desired. Reactivity of the submicron and nanometer size ripple structures on the semiconductor surface would be enhanced drastically due to the large surface to volume ratio and the probability of dangling bonds on the surface [20].

When an intense laser beam is incident on a medium containing nanoprticles of Si, the refractive index is altered due to the intensity of the laser beam. Changes in the refractive index lead to a velocity distribution of the laser beam in the transverse plane. Thus the spatial phase variation occurs in the plane perpendicular to the beam which leads to a visible optical fringe pattern. This phenomenon is known as self-phase modulation (SPM) [21,22], where the change in refractive index $|\Delta n(r)|$ for a nonlinear medium is proposed as a function of laser power density *P(r)*

$$|\Delta n(r)| = g_L P(r) \qquad \text{.......(1)}$$

The nonlinear coefficient g_L includes the size dependent refractive index and is written as

$$g_L = \int_{L_1}^{L_2} f \frac{N(L)}{L} dL \quad \text{........(2)}$$

Where f is the coupling constant of light with the medium containing nanoparticles of Si. $N(L)$ is a Gaussian distribution function for the sizes of nanoparticles. L_1 and L_2 are the minimum and maximum size of the Si nanoparticles used in the Gaussian distribution.

The far-field diffraction intensity distribution of the optical generated fringe pattern is given by the relation [23].

$$I(x) = u_0 \left(\frac{2p}{lZ} \right)^2 \left| \int_0^w rdrJ_0\left(\frac{krx}{2Z} \right) \exp\left(-2\frac{r^2}{w^2} - if(r) \right) \right|^2 \quad \text{..........(3)}$$

Where $I(x)$ is the far field diffraction of the Gaussian beam, $J_o(x)$ = zero order Bessel function, k is the wave number in free space, Z the distance from the sample to the sample observation point, w is the beam waste and $f(r)$ is the phase factor.

In the present work, we are investigating the optical fringe patterns in the laser light reflected by nano-Si during the LIE process (in situ). This study could provide important details on the nano-particle sizes and their distribution in addition to some optical properties.

2. Experimental work

A commercially available crystalline Si phosphoric doped n-type (111) oriented with resistivity (1-15 Ω.cm), thickness 550 μm has been used. Samples were immersed in HF acid of concentration 40% and 30% in a plastic container. Each wafer was put on two Teflon plates in such a way that it allowed the passage of current from the bottom surface to the laser irradiated top portion through the electrolyte [24]. Laser etching was accomplished by utilizing IR diode laser of 1.53 eV photon energy. The beam was focused to a circular spot of 1mm diameter, thereby, obtaining laser power density of 15 W/cm^2 for time duration 20 minutes. After etching, the samples were rinsed with ethanol and dried in air. Then, samples were placed in a vacuum system, which was evacuated to a pressure of 10^{-3} Torr. A white screen was placed at a distance of one meter from the surface of the crystal to observe the optical fringe patterns formed by the reflected beam in nearly back reflection geometry. Finally, the optical fringes formed for different etching time were recorded by charge-coupled device (CCD) camera and stored in the form of a video picture. The fringes reported here are the stand-still photographs of the kinetic picture taken by a computer to analyze the optical fringe patterns by image processing program. The surface morphology of the prepared samples was also investigated by a high resolution scanning electron microscopy (SEM) (ZEISS, EVO 50) and high resolution optical microscopy (Kruss.) supported with digital camera.

3. Results and Discussion

A. Optical fringes from silicon nanocrystallites

Light reflected from the surface of the sample manifests itself into a concentric optical fringe pattern on the observation screen. However, it is observed that the evolution of the optical fringe pattern depends upon the fabrication parameters such as the irradiation time, probing laser power densities, wavelength of the laser probe, resistivity of crystalline silicon and the hydrofluoric acid concentration. Figure (1) shows the evolution of optical fringe patterns with irradiation time. The experimentally observed fringe patterns as shown in figure (1) were fitted with a theoretical model obtained by Eq.(3). Initially, when the silicon surface illuminated with laser beam, no fringes were observed and only an intense reflected spot could be observed on the screen. As the illumination process proceeds, low number of weak intensity fringe patterns were emerged after few minutes. We have recognized that those patterns have become brighter and larger in number with time. As the etching progress, a fringe pattern is observed moving around the reflected spot on the screen and then disappears. The pattern again reappears and then disappears. This process gets repeated after every few seconds. It seems that the appearance and disappearance of fringes are related with the size of the nanocrystallites. Fringes disappear when cluster of Si atoms (i.e., nanoparticles) collapses. This is in agreement with the results of Iqbal et al. [25] and Shukla et al [26], where they have shown that the instability of nanocrystallites of size less than 3 nm is due to the increase of free energy and lattice expansion. After few seconds, appearance of fringes was observed again as a new layer of Si containing the nanocrystallites which was established with etching. Nanocrystallites of larger size are formed in another layer and the size gradually decreases with etching time.

The resolution of the center for the fringe pattern is not good enough in this case and is related to the high intensity of the reflected laser light which causes saturation in the camera's detection. One could get better resolution if a neutral filter (ND) is used. The transverse beam showed the doughnut-shape profile as described in Fig.(1). Due to the excessive etching in the porous layer, this will lead to remove the silicon nanocrystallites sizes from the photosenthesized porous silicon layer. In other words, there are two etching stages according to the model of Koker and Kolasinski [27], the two stages are: (1) formation of new porous layer due to the absorption within the porous layer and (2) removing of the first formed porous silicon with its silicon nanocrystallites layer. Table (1) summarizes the estimated sizes of nano-Si and the fitting parameter used for the optical fringe pattern shown in Fig.(1).

Figure (2) illustrates the nanocrystallites sizes in the PS layer as a function of irradiation time for laser-induced etching.

B. Surface Morphology

The irradiation of the silicon surface with a high penetration depth wavelength (810 nm) lead to form a ripple-like structure. The evolution of ripple structure on Si surface in various aqueous HF acid concentration 30% and 40% is shown in Fig. (3). For 40% HF acid concentration, ripple structures with micrometer periodicity and a maximum valley depth of (20 µm) are observed as shown in Fig.(3a). The absorption depth of 810 nm laser light in Si is approximately 12 μm and the observed ripple structure has a large valley height. However, for diluted HF electrolyte, the valley depth increases significantly during the laser irradiation. This enhancement is caused by large photo-induced charge carrier density. Photoelectrochemical etching induced by the excited carriers provides a feedback mechanism that reinforces the laser-induced etching formation because the rate of etching is related to the density of excited carriers.

Moreover, two different regions are clearly observed with a regular step of (35μm) and line width of (20 μm) corresponding to the image of the interference pattern as shown in Fig.(3a). The surface plot and plot profile of the grating reveals ripple and microchain structure as shown in Fig.(3a) and (3b), respectively.

Moreover, on decreasing HF acid concentration to the 30% wt., the valley depth of the ripple structure further decreases to 15 μm with regular step 30 μm and the line width is (35 μm). The ripple structure is attributed to the optical interference of the incident laser with a surface scattered wave, as seen in figure (4 a, b and c). Where ordered structure is distinguished.

4- Conclusions

Optical fringe patterns from silicon nano-particles are utilized to calculate the nonlinear changes of the refractive index by the SPM model. Moreover, we have synthesized PS structure using a laser-induced etching technique combined with photosensitivity of the material. The interference pattern can be reproduced very precisely in the PS layer even at submicron scale. We found that it is possible to modulate the refractive index in directions parallel to the layer plane which, in addition to the diffraction properties of such structures, will probably give rise to applications in integrated optics and photonics.

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Table (1) The estimated nanocrystallite sizes for different irradiation time

Time(min.)	No.of fringes	L_1(nm)	L_2(nm)	L_o(nm)	Dn_{max}(r=0)	Dn_{min}(r)	DR(r=0)
3	3	3	8	5	0.232	0.031	0.388
7	4	3	8	4.5	0.238	0.032	0.379
12	5	1.5	7.5	3.9	0.385	0.056	0.198
15	4	3	8	5	0.24	0.033	0.375

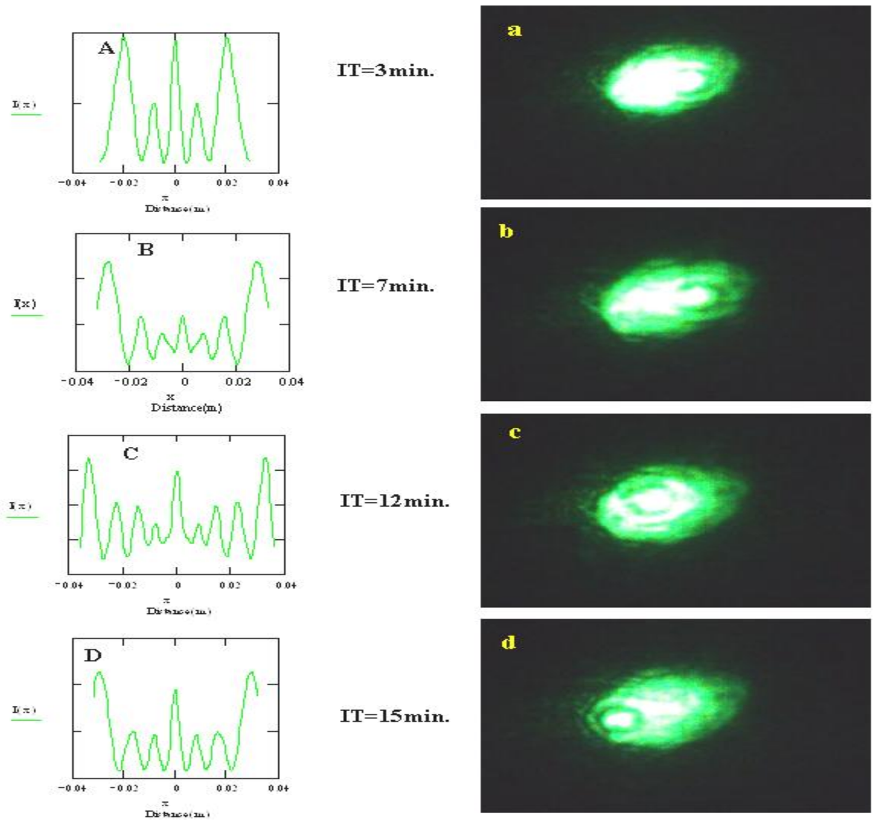


Figure (1) the left column represents the radial intensity distributions of the irradiation pattern while the right column is the temporal evolution of the reflected beam as a PS etched at laser power density (25 W/cm^2) (810 nm wavelength)

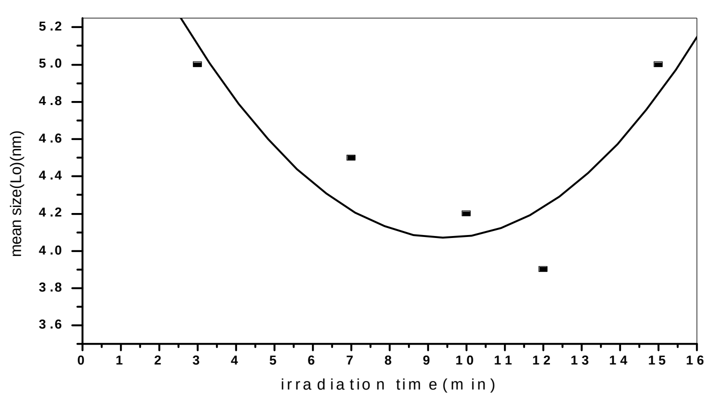
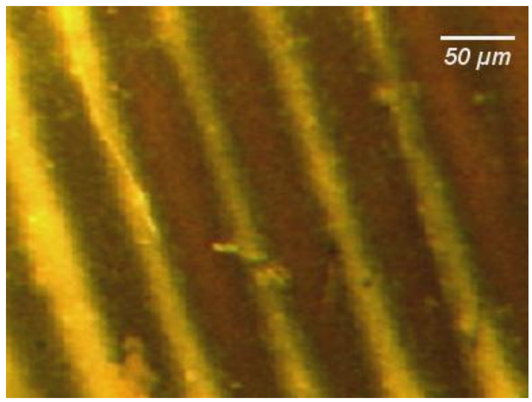
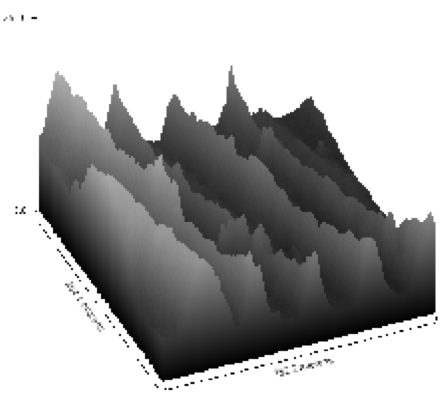


Figure (2) Dependence of average Nan crystallite size on the irradiation time

a


b


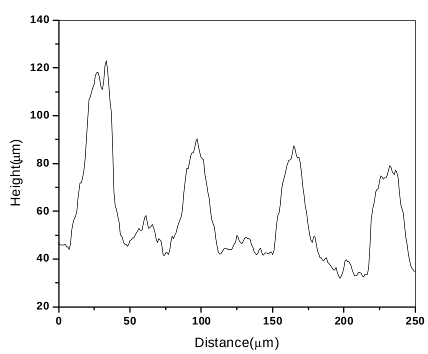
c


Figure (3):(a) micrograph of n-type PS lateral superlattice produced by LIE process with 40% wt HF concentration (b) the surface plot of grating (c) plot profile of the micrograting

a

b

c

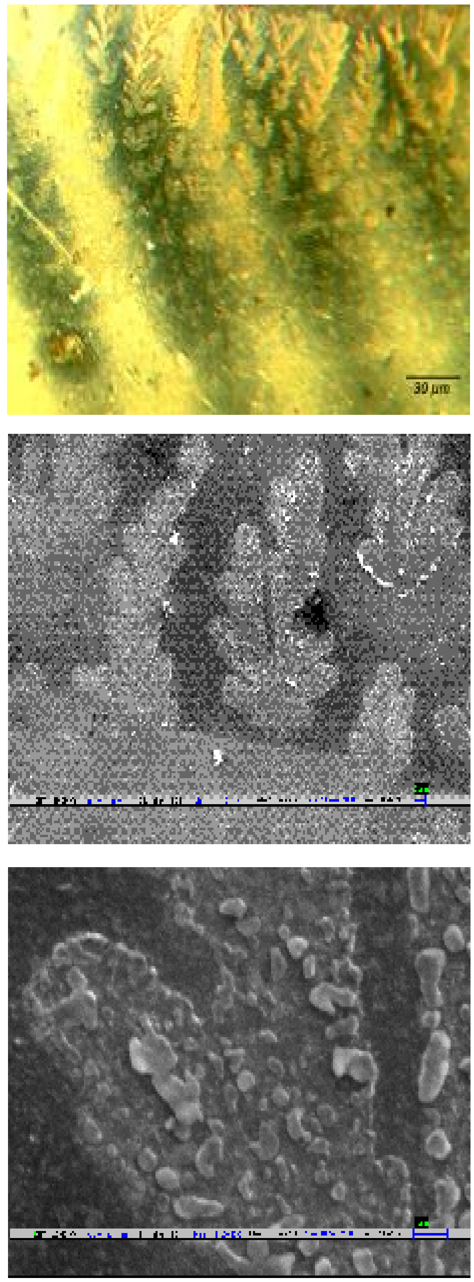


Figure (4): (a) the top view of the high resolution optical microscope of n-type PS structure produced by LIE process (b) and (c) A magnified SEM of PS structure