Optik 164 (2018) 186–200

Contents lists available at ScienceDirect

Optik

journal homepage: www.elsevier.de/ijleo

Spectroscopic and morphological investigation of laser ablated silicon at various laser fluences

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ARTICLE INFO

Article history:
Received 25 July 2017
Received in revised form 10 February 2018
Accepted 6 March 2018

Keywords:
Silicon plasma
Laser-induced breakdown spectroscopy
Emission spectroscopy
Surface morphology
Laser ablation

ABSTRACT

The effect of laser fluence by using laser-induced breakdown spectroscopy on the ablation mechanisms of Si (100) has been investigated. For this purpose a Q-switched Nd-YAG laser with the wavelength of 1064 nm, pulse duration of 10 ns was employed. For the very first time by using linearly polarized Nd:YAG laser, the effect of fluence and optical emission spectroscopy has been carried out for various laser fluences ranging from 1.4×10^2 to 11.3×10^2 J/cm^2 under the ambient environment of He, Ne and Ar at a constant pressure of 200 torr. Boltzmann plot and Stark broadening method were used to determine the influence of laser fluences on plasma parameters such as electron temperature and number density. Our experimental results suggest that the laser fluence in different ambient environment play a significant role for plasma evolution, ambient gas causes a confinement effect that generation, recombination, and expansion of plasma and consequently affect the excitation temperature as well as electron density of plasma. The morphological analysis of laser irradiated Si was performed by Scanning Electron Microscope (SEM). Various kinds of structures e.g. Laser Induced Periodic Surface Structures (LIPSS) or ripples, droplets and craters have been generated and their density and size are found to be dependent upon the laser fluence and ambient environment. A quantitative analysis of crater depth measured from SEM images showed strong correlation between laser fluence and the growth of micro/nano structures on modified Si surface.

1. Introduction

LIBS analysis has been used for vast range of potential applications like environmental monitoring, material analysis, thin film deposition, micro-machining, nano structuring, biological identification and plasma characterization [1–9]. LIBS previously has been a qualitative technique but over recent years it has been developed into a pseudo-quantitative materials micro-analysis technique, capable of determining the elemental composition of solids, liquids and gases [1,2].

In this technique, when a powerful pulsed laser source is employed to a target surface. The high intensity of the laser vaporizes the target forming a transient plasma or plume which subsequently expands away from the sample surface [3]. The plasma consists of neutrals, ions and electrons, as well as excited species. The de-excitation of excited atoms generates the characteristic photons which can be measured with optical emission spectroscopy [4]. Because the temperature in the expanding plume is very high, plasma will be created by Multi-Photon Ionization (MPI) or Inverse Bremsstrahlung ionization (IBI) [2,3,5–8].Which leads to ionization and excitation of investigated material. Laser induced plasma parameters

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https://doi.org/10.1016/j.ijleo.2018.03.016

are dependent upon various factors e.g. laser fluence and the nature and pressure of background gases, physical, chemical properties of target material as well as the chemical composition of the target martial. The plasma parameters such as electron number density and electron temperature can be determined from the emission spectrum of the plume [2,9].

The laser fluences at a constant pressure with various ambient environments plays a significant role for the evolution and expansion of laser-induced plasma. It has been observed that shape, size and dynamics of the expanding plume are completely modified by introducing the ambient gas. The physical processes such as confinement effect, shielding effect, formation of shock waves and the interaction of the plume with an ambient gas makes it significantly important for various applications such as ion implantation using laser induced plasma as ion source, pulse laser deposition of thin films and micro/nano structuring of various kind of materials [9]. The electron temperature and number density of silicon plasma have been evaluated at various laser fluences. For both plasma parameters, an increasing trend up to a certain value of laser fluences is observed it is due to enhanced energy deposition. Afterword a decreasing trend is achieved which is attributed to the shielding effect. With further increase in fluence a saturation stage is achieved. The growth of laser induced surface structures is also strongly dependent upon the laser fluences [1]. The higher electron temperature and density leads more energy deposition to the lattice of the target and causes growth of various micro/nano structures on an irradiated surface. After laser irradiation nano- and micro textured silicon is extensively useful with several potential applications in the fields of microelectronics, photodetectors, solar cells, field emission devices and bio-medicine, etc. [10,11]. Experiment with some similarities such as effect of ambient environment and influence of its pressure variation on the mass ablation rate, the plasma temperature and the electron number density has been investigated by several groups specifically Shaikh et al. [3] have performed the laser induced breakdown spectroscopy of zinc. They have investigated the variation in electron temperature and number density as a function of laser irradiance and the distance of target surface. However the surface morphology have been rarely discussed in such studies. Farid et al. and Cowpe et al. [2,5] have investigated the effects of variation in ambient pressure in three different environments on LIBS parameters and the morphology of copper and silicon respectively. Hafeez et al. [12] have performed the laser induced breakdown spectroscopy of cadmium. They have investigated the variation in electron temperature and number density as a function of laser irradiance and the distance of target surface. However the surface morphology is missing in this study as well. Shaikh et al. [13] have presented the effects of 1st, 2nd and 3rd harmonics, laser irradiance in three different gases on Laser Induced Breakdown Spectroscopy of brass plasma. Again surface morphology has not been investigated.

In the present work, the variation of various laser fluences under a constant pressure of different ambient environments on emission spectroscopy of Si plasma has been investigated. Laser induced breakdown spectroscopy of silicon Si was carried out by exposing the target under ambient environment of inert gases of Ar, Ne and He for different laser fluences ranging from 1.4×10^2 to 11.3×10^2 J/cm^2 corresponding to laser pulsed energies of 25–200 mJ respectively. By employing LIBS technique. The plasma parameters such as electron temperature and electron number densities were determined by Boltzmann plot and Stark broadening method respectively. The surface morphological analysis of laser irradiated Si was performed by Scanning Electron Microscope (SEM). A correlation of excitation temperature and electron number density of plasma plume with the growth of surface structures of Si for various laser fluence under noble ambient environments have been evaluated for very first time and quantitatively justified by conducting a statistical analysis with the help of software.

2. Experimental details

Commercially polished single crystal Si (1 0 0) with dimensions of 10 mm × 6 mm × 1 mm, were selected as target materials. Prior to laser treatment, these substrates were cleaned by sonication in acetone (15 min) and in isopropanol (15 min) before being dried under an argon stream. A Q-switched Nd:YAG (1064 nm,10 ns) laser is employed for ablation and plasma production. A schematic of experimental setup is shown in Fig. 1.

The plasma plume is evolved after focusing the incident laser beam by 50 cm focal length lens. The incident beam propagation was perpendicular to the surface of the target. The silicon targets were mounted on target holder under vacuum chamber (10^{-6} mbar). For fresh surface exposures the target was rotated with the help of stepping motor to avoid non-uniform pitting and crater-formation. The radiations emitted from the Si plasma was collected by LIBS2500 (Broadband 200–980 nm, Resolution 0.1) plus spectrometer system (Ocean Optics USA). This spectrometer system consists of LIBS-Fiber bundle with seven silicon CCD array detectors. The data acquired to provide full spectral analysis by spectrometer was store in a PC through OOILIBS software.

The delay time between the laser trigger and data collection was 1.25 μs, which was kept constant for all measurements. In order to investigate the effect of three inert gases of Ar, Ne and He were filled in the chamber one by one at various fluences ranging from 1.4×10^2 to 11.3×10^2 J/cm^2 and a constant pressure 200 torr could be controlled with mbar precision. Laser fluence was evaluated by using the following relation

$$\text{Fluence} = \text{Energy}/\text{Area} \tag{1}$$

Where E is the pulsed energy of laser (ranging from 25 mJ to 200 mJ) and A is the area of focused laser spot which is acquired by the following equation [14].

$$\omega_f = (\lambda * f)/(\pi\omega_0) \tag{2}$$