Deformation behavior of single crystal silicon induced by laser shock peening

Yuanxun Liu, Xianqian Wu, Xi Wang, Yanpeng Wei, Chenguang Huang*

Key Laboratory for Mechanics in Fluid Solid Coupling Systems, Institute of Mechanics, Chinese Academy of Sciences, Beijing 100190, People's Republic of China

ABSTRACT

Laser shock peening can significantly improve the fatigue life of metals by introducing plastic deformation and compressive residual stresses near the surface. It has been widely applied on metals for surface strengthening. The plastic deformation behavior of brittle materials such as single crystal silicon under LSP is rarely studied. In the present research, the surface integrity and residual compressive stress of P-type single crystal silicon in <100> orientation shocked by LSP at imposed high temperature were measured to investigate the plastic deformation mechanism at high temperature and high compressive stress. The surface morphology of shocked silicon, observed using optical microscopy, showed that the cracks on the shocked silicon surface became less and the fragments were smaller while the temperature or the laser power density increased, which indicates that the plasticity of single crystal silicon is improved at high stress and temperature. However, the excessive laser power density would lead to local damage of the shocked silicon. The residual stress, measured using Raman scattering method, showed that the compressive residual stresses with magnitude of a few hundreds of MPa were introduced in the surface layer of silicon after LSP at imposed high temperature, and it increased with respect to the temperature and the laser power density. The experimental result indicates the material has experienced the plastic deformation and provides a potential processing method to improve the mechanical behavior of brittle material like single crystal silicon.

Keywords: Laser shock peening; single crystal silicon; plastic deformation; surface morphology; residual stress.

1. INTRODUCTION

Single crystal silicon is an important semiconductor material in the electrics industry. The presence of crystalline defects, including dislocations, is detrimental to the processing and service life device. As a result, the dislocation properties and plasticity of single crystal silicon as a model material are widely studied. The dislocation mobility of single crystal silicon is very low at low temperature even at high pressure ^[1]. However, the dislocation mobility increases exponentially at elevated temperature ^[2]. Therefore, it is able to introduce compressive plastic strain and residual stress in single crystal silicon by elevated temperature and high pressure.

Laser shock peeening (LSP) can significantly improve the fatigue life of metals by introducing plastic deformation and compressive residual stresses near the surface ^[3-8]. It has been widely applied on metals for surface strengthening. Through the interaction of a pulsed high-intensity laser beam and energy absorption layer on the metallic target surface, a

*E-mail address: huangcg@imech.ac.cn.

2nd International Symposium on Laser Interaction with Matter (LIMIS 2012), edited by Jingru Liu, Jianlin Cao, Stefan Kaierle, Proc. of SPIE Vol. 8796, 87962M · © 2013 SPIE · CCC code: 0277-786X/13/\$18 · doi: 10.1117/12.2011314

shock wave with high amplitude can be generated and propagates into the target. If the amplitude of shock wave exceeds the Hugoniot elastic limit (HEL) of the target material, plastic deformation occurs and residual compressive stresses are induced near the surface, which results in the enhancement of fatigue life.

The plasticity of single crystal silicon shocked by laser shock peening (LSP) is rarely studied except by Cheng, et al ^[9-11]. In the LSP experiments, they found single crystal silicon showes ductile behavior when the temperature reaches 850K and the applied laser power density is 6GW/cm². Besides, they studied the effect of process conditions on the dislocation behavior of silicon in LSP via dynamic dislocation simulation. However, the macroscopic dynamic behavior of single crystal silicon has not been studied or clearly described, such as stress waves propagation and the surface integrity after LSP, which is significantly important for understanding the deformation mechanism. The present work focuses on the deformation behavior of single crystal silicon under high temperature LSP. The mechanism of deformation behavior is studied by experimentally observation of surface morphology and residual stress of shocked silicon. The effect of experimental parameters such as temperature and laser power density on the deformation behavior is also investigated.

2. EXPERIMENTAL METHOD

2.1 Laser shock peening

2.1.1 Laser

The LSP experiment was performed with a Q-switched high power Nd: YAG pulse laser having a wavelength of 1064 nm. A typical temporal profile of the laser pulse, which is in the near Gaussian shape, is recorded as Fig. 1. The pulse duration (full width at half maximum, FWHM) is approximately 10.0 ns, and the output energy is about 2.4 J. Its spatial profile is modulated to a nearly Gaussian shape. A 600 mm focal lens was used to adjust the beam size such that various laser intensities can be achieved at the specimen surface.

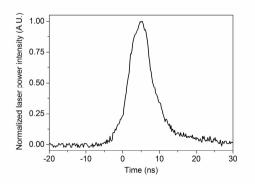


Fig. 1. Temporal profile of laser power density.

2.1.2 Target

The target material is 500-µm-thick P-type single crystal silicon wafers in <100> orientation. The samples has been polished before LSP. Two samples were stacked together in each experiment to investigate not only the decay of the shock stress but also the difference of deformation behavior of the samples at different stress amplitude. A 0.06-mm-thick copper foil, chosen as an absorption layer for its high threshold of vaporization, was coated with the

target surface tightly. The samples were sandwiched between two pieces of 2-mm-thick fuse silica glass. The upper glass placed against the copper foil is used as confined layer, and the lower one placed closely against the lower silicon sample is taken as a cushion to avoid the catastrophic breakdown induced by large deformation in LSP. The fuse silica glass is taken because of its transparency to the laser and high melting point. The copper foil and two pieces of fuse silica glass were fully-clamped with the target by a specially designed hold as shown in Fig. 2. In experiment, a high temperature resistance heater was used to heat the single crystal silicon evenly to different temperature varied from room temperature to 1000K. The heating rate was controlled around 5 K/min. After LSP, the target was air cooled down to room temperature. As the heating and cooling rates are very small, the residual stress introduced by the temperature gradient load is negligible.

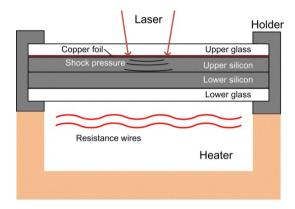


Fig. 2. Schematic of experimental.

2.2 Surface morphology

The surface morphology of shocked silicon wafers is investigated in this study to characterize the deformation behavior for different pressure and temperature. The surface morphology was observed using optical microscopy. The samples used for surface morphology observation were prepared from the recovered shock target. After shocked the silicon samples were etched using nitric acid to wash away the copper foil sticking on them, and then the surface was cleaned with acetone. As the sample had been polished before LSP, the deformation character of the shocked sample was obviously.

2.3 Residual stress

After shock processing, the surface residual stress across the shocked region was measured using Raman scattering. It was performed on a Raman scattering machine in Via-Reflex operating with a wavelength 532 nm. The incident light, that emitted by an argon-ion laser, is focused on the surface of the measured region using a 50 times objective lens. The diameter of the focused light is about 3 μ m. The scattered light is collected with the same objective lens. In Raman scattering, the scattered spectrum is a function of the Raman shift and contains the material's physical and chemical characteristics. For a stress-free single crystal silicon, the Raman spectrum has a single peak with a Raman shift ω_0 of ~520cm⁻¹. When the silicon is loaded with the external stress, the change of the Raman shifts is obtained ^[12],

$$\Delta \omega = \frac{\lambda_3}{2\omega_0} = \frac{q(s_{11} + s_{12}) + ps_{12}}{2\omega_0} \sigma_{xx}$$
(1)

where s_{ij} are elements of compliance tensor of single crystal silicon, σ_{xx} is the uniaxial stress applied on the sample.

Proc. of SPIE Vol. 8796 87962M-3

According to Chandrasekhar et al, the relation between σ_{xx} and $\Delta \omega$ is

$$\sigma_{xx} \approx -434\Delta\omega \tag{2}$$

where σ_{xx} is in unit of MPa and $\Delta \omega$ in cm⁻¹. A Lorentz function is fitted to the silicon Raman peak at each position in order to determine the peak frequency as accurately as possible.

3. RESULTS AND DISCUSSION

The plastic deformation of single crystal silicon is carried out mainly by dislocation motion. The motion of dislocations can be assisted by the imposed temperature and the applied external stress. The elementary mechanism of dislocation motion is associated with energy barriers. It has to overcome high Peierls potentials of the first and second kinds. At the beginning, kink pairs are generated while crossing the energy barrier in the direction of dislocation motion. Then, each kink propagates sideways along the dislocation line. The dislocation velocity v in single crystal silicon is a function of temperature T and applied stress τ as the following phenomenological law ^[13],

$$v = v_0 \left(\frac{\tau}{\tau_0}\right)^m \exp\left(-\frac{U}{k_B T}\right)$$
(3)

where τ and T are applied stress and temperature, v_0 and τ_0 are material related constants, k_B is the Boltzmann's constant, the exponent m is usually between 1 and 2, and U is the total activation energy. The dislocation velocity increases with respect to the temperature and stress. Hence, the stress and temperature have great impact on the plastic deformation behavior for single crystal silicon. From a macroscopic point of view, the yield point of the stress-strain curve of single crystal silicon depends on the imposed temperature and external stress according to Alexander and Haasen^[14-15],

$$\tau_{yield} \propto \left(\dot{\gamma} \exp\left(\frac{U}{k_B T}\right)\right)^{1/(m+2)}$$
(4)

where $\dot{\gamma}$ is the resolved strain rate. The plastic deformation behavior of single crystal silicon is related to the strain rate, the applied stress and temperature. For instance, plastic deformation could be obtained in single crystal silicon under a hydrostatic pressure of 5 GPa at temperature as low as room temperature and 150°C with a imposed strain rate of 5×10^{-5} s⁻¹, while it occurs under a pressure of 1.5 GPa at 450°C with a imposed strain rate of 2×10^{-6} s^{-1 [13]}. However, the applied stress and strain rate can reach up to several GPa and 10^6 s⁻¹ respectively in LSP. The deformation behavior will be much more complex and undiscovered under high temperate LSP loading. As the strain rate is almost the same for each experiment, the influences of temperature and applied stress on the deformation behavior of single crystal silicon are given, which depicts the deformation behavior of single crystal silicon under ultra-high strain rate, high imposed temperature, and high applied stress. Moreover, the residual stress is also given to aid to the understanding of dynamic behavior of single crystal silicon.

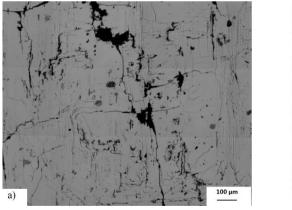
3.1 Surface morphology

Surface morphology is significantly important for understanding the deformation mechanism. The observation of surface morphology can be directly used to investigate the plastic deformation behavior as well as the influences of temperature and applied stress. The surface morphologies of the upper samples after shocked with laser power density of 7.8 GW/cm² at 25 °C and 630 °C, respectively, are depicted in Fig. 3, where the shock pressure is 6 GPa and the strain

rate is up to 10^6 s⁻¹. The shock pressure for given laser power density is calculated by the following formula according to Fabrro's model ^[16],

$$P(GPa) = 0.01 \left(\frac{\alpha}{2\alpha + 3}\right)^{1/2} Z^{1/2} \left(g \bullet cm^{-2} \bullet s^{-1}\right) I^{1/2} \left(GW \bullet cm^{-2}\right)$$
(5)

where P is the shock pressure, α is a fraction of the internal energy representing the thermal energy (α =0.1 here), Z is the combined shock-wave impedance of the taget and the confining medium (Z= 1.47×10^6 g • cm⁻² • s⁻¹ for the silicon/glass interface here), and I is the laser power density. At 25 °C, the sample was fragmentized after shocked. A lot of major cracks are generated in the shocked region, which indicates the sample is brittle even at compressive stress of about 6 GPa. The stress cannot induce plastic deformation under the ultra-high strain rate loading but is higher than 5 GPa, which can do it at quasi-static loading at the same temperature. This is in accord with equation (4): the yield stress increases with rising strain rate. As the shock pressure is applied on the <100> axes, the major cracks are in horizontal and vertical directions. The cracks at the marginal impacted region are ascribed to the large impulse and shear stress induced by the shock pressure. Compared with the room temperature condition, the surface morphology shocked at 630 °C changed greatly. Although the cracks are also generated at this condition, the sizes of fragments are significantly changed. The major cracks are vanished instead of numerous short cracks with elevated imposed temperature, which indicates the ductile behavior of the single crystal silicon is initiated at this condition. The dislocation mobility is elevated with respect to the temperature. Hence, the plasticity of the sample is promoted at high temperature. Unlike the room temperature shocked condition, the sample shocked at high temperature deformed plastically firstly at the impact region. Then it generates the numerous small size cracks to absorb the remaining shock energy as fracture energy. It implies that the plastic deformation is exhibited at the condition of 6 GPa compressive stress, 630 °C temperature and strain rate up to 106 s⁻¹.



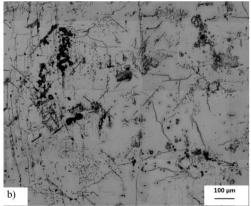


Fig. 3. Surface morphologies of the upper samples after shocked with laser power density of 7.8GW/cm2 at (a) imposed temperature 25 °C (b) imposed temperature 630 °C.

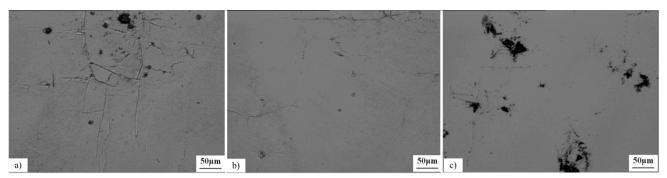


Fig. 4. Surface morphologies of the lower samples shocked at imposed temperature 730 with laser power densities of (a) 4.5 GW/cm² (b) 6.4 GW/cm² (c) 12.6 GW/cm².

Fig. 4 gives the surface morphologies of the lower samples shocked with laser power densities of 4.5 GW/cm^2 (compressive stress 4.5 GPa), 6.4 GW/cm² (compressive stress 5.4 GPa) and 12.6 GW/cm² (compressive stress 7.6 GPa) at imposed temperature 730 °C, respectively. To improve the capability of the plastic deformation, the higher imposed temperature was imposed. Unlike the upper sample, the surface morphologies of the lower samples are much more integrated. There are two reasons: the decay of the compressive stress and imposed impulse. In other words, the upper samples suffered much higher stress and impulse leading to more cracks to absorb the remaining shock energy as fracture energy, while the lower ones suffered appropriate stress and impulse producing more integrated surface. Although more or less cracks still generate on the surfaces of lower samples at this condition, the cracks are markedly different from the cracks on the surface of sample shocked at 25 $^{\circ}C(Fig. 3(a))$. And the shear stress needed for plastic deformation in silicon under guasi-static loading at 700 $^{\circ}$ C is in the magnitude of 100 MPa [13, 15]. Thus, it is inferred that the imposed stress and impulse on the lower samples is high enough to cause plastic deformation and even fractures. As a result, more or less cracks still generate in lower samples. For the laser power density of 4.5 GW/cm², the major cracks are visible in and around the impact region. When the laser power density increases to 6.4 GW/cm², the major cracks disappear, which indicates the capability of plastic deformation of the single crystal silicon is able to dissipate the impact energy applied on the material. However, this tendency is not monotone while the laser power density increases to 12.6 GW/cm², where several destructive morphology is localized on the shocked surface.

3.2 Residual stress

The residual stress of the shocked surface is measured to investigate the plastic deformation behavior of single crystal silicon under LSP loading at imposed temperature. At the beginning, the residual stress of the upper sample along the diameter cross the impact region is measured with Raman scattering method as depicted as Fig. 5, where the laser power density is 7.8 GW/cm² and the imposed temperature is 500 °C. The maximum residual compressive stress of the impact region is about 600 MPa, and it decreases with the increasing distance from the center because of the Gaussian spatial distribution of the laser power density. This confirms that single crystal silicon experiences plastic deformation in LSP at high temperature and considerable residual stress can be induced, meaning that LSP is potential for silicon enhancement. In addition, the maximum residual stress is not located at the center of the impact region, which could be ascribed to the initiation of the major cracks. After the major cracks initiated, the residual stress distribution on the impact region would be changed because of the release of residual stress nearby the cracks.

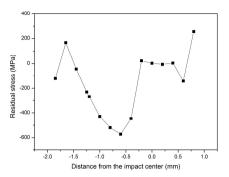


Fig. 5. Residual stress of the upper sample along the diameter cross the impact region shocked with laser power density of 7.8 GW/cm^2 at imposed temperature 500 \therefore

The residual stress on the top surface of the lower sample shocked with laser power densities of 4.5 GW/cm², 6.4 GW/cm², 12.6 GW/cm² at imposed temperature of 730 °C are shown in Fig. 6. The maximum residual stresses measured on the top surface of the lower samples are less than 200 MPa, which significantly decrease compared with the upper sample. While the shock wave propagates in the samples, the energy is dissipated quickly as plastic strain energy and fracture energy, which leads to a much smaller compressive stress applied on the lower one. Additionally, the maximum residual stresses shocked with the laser power densities of 4.5 GW/cm² and 12.6 GW/cm² are not at the center of the impact region, which is attributed to the major cracks as mentioned above. As the surface after shocked with laser power density of 6.4 GW/cm² is more integrated, the residual stress distribution on the shocked region is more reasonable. The residual compressive stress decreases with the increasing distance from the impact centre; the residual tensile stress with an amplitude of about 50 MPa appears at the marginal region because of transverse extrusion effect. Besides, for the sample has experienced larger plastic deformation under higher stress, the maximum residual stress increased with respect to the laser power density. However, the maximum residual stress decreased at excessively high laser power density because of the release of residual stress around the initiating fractures.

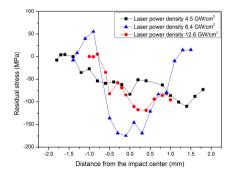


Fig. 6. Residual stress on the top surface of the lower sample shocked at imposed temperature of 730 with laser power densities of 4.5 GW/cm², 6.4 GW/cm² and 12.6 GW/cm².

4. CONCLUSION

The plastic deformation behavior of single crystal silicon under LSP condition at imposed high temperature was studied. The surface morphology and residual stress distribution of the shocked samples were investigated. The main conclusions are as follows,

- a) The capability of plastic deformation of single crystal silicon increases with respect to the imposed temperature because of enhancement of dislocation mobility at high temperature. It is also elevated while raising the laser power density. However, it decreases if the laser power density is so high that fractures initiate severely.
- b) Residual compressive stress with amplitude of several hundreds of MPa can be introduced in single crystal silicon by LSP method with imposed high temperature, which might be a possible processing method for semiconductors to improve the mechanical properties especially for fatigue life.
- c) At LSP loading condition with imposed high temperature, the compressive stress is excessively high for the single crystal silicon, which will lead to the initiation of fractures.

Considering the strain rate and the stress under high temperature LSP is much higher than it in ordinary processing technologies of silicon crystal and the related research is limited. More study of the deformation behavior under high temperature LSP is needed.

ACKNOWLEDGEMENTS

The authors thank the National Natural Science Foundation of China (Grant No. 10972228, 11002150 and 91016025) and Basic Research Equipment Project of Chinese Academy of Sciences (Grant No. YZ200930) for financial support.

REFERENCES

- [1] Loveridge-Smith, A. Allen, J. Belak, et al., "Anomalous elastic response of silicon to uniaxial shock compression on nanosecond time scales," Phys. Rev. Lett. 86(11), 2349–2352 (2001).
- [2] Cai, W., Bulatov, V. V., Justo, J. F., Argon, A. S. and Yip, S., "Intrinsic mobility of a dissociated dislocation in silicon," Phys. Rev. Lett. 84(15), 3346–3349 (2000).
- [3] Montross, C. S., Wei T., Ye L., Clark G. and Mai Y. W., "Laser shock processing and its effects on microstructure and properties of metal alloys: a review," Int J Fatigue 24(10), 1021-1036 (2002).
- [4] Clauer, A. H. and Lahrman, D. F., "Laser shock processing as a surface enhancement process," Key Eng. Mater. 197, 121-144 (2001).
- [5] Nikitin, I., Scholtes, B., Maier, H. J. and Altenberger, I., "High temperature fatigue behavior and residual stress stability of laser-shock peened and deep rolled austenitic steel AISI 304," Scr Mater 50, 1345–1350 (2004).
- [6] Liu, Q., Yang, C., Ding, K., Barter, S. and Lin, Y., "The effect of laser power density on the fatigue life of laser-shock-peened 7050 aluminium alloy," Fatigue Fract Eng Mater Struct 30(11), 1110-1124 (2007).
- [7] Zhang, Y. K., Chen J. F. and Xu R. J., "Experimental research of laser shock strengthening AM50 magnesium alloy," Zhongguo Jiguang 35(7), 1068-1072 (2008).

- [8] Peyre, P., Berthe, L., Scherpereel, X. and Fabbro, R., "Laser-shock processing of aluminium-coated 55C1 steel in water-confinement regime, characterization and application to high-cycle fatigue behaviour," J Mater Sci 33(6), 1421-1429 (1998).
- [9] Cheng, G. J., Cai, M., Pirzada, D., Guinel, M. J. f., and Norton, M. G., "Plastic deformation in silicon crystal induced by heat-assisted laser shock peening," J Manuf Sci Eng Trans ASME 130, 011008 (2008).
- [10] Cheng, G. J. and Shehadeh, M.A., "Dislocation behavior in silicon crystal induced by laser shock peening: a multiscale simulation approach," Scr Mater 53, 1013–1018 (2005).
- [11] Cheng, G. J. and Shehadeh, M.A., "Multiscale dislocation dynamics analyses of laser shock peening in silicon single crystals," Int J Plast 22, 2171–2194 (2006).
- [12] Wolf, I. D., "Stress measurements in Si microelectronics devices using Raman spectroscopy," J Raman Spectrosc 30, 877–883 (1999).
- [13] Hirth, J.P. and Kubin, L., [Dislocations in Solids], Elsevier, North-Holland, 47-108 (2010).
- [14] Alexander, H. and Hassen, P., "Dislocations and plastic flow in the diamond structure," Solid St. Phys. 22, 27-158 (1968).
- [15] Moulin, A., Condat, M. and Kubin, L.P., "Mesoscale modeling of the yield point properties of silicon crystals," Acta mater 47(10), 2879-2888 (1999).
- [16] Fabbro, R., Fournier, J., Ballard, P. and Virmont, J., "Physical study of laser-produced plasma in confined geometry," J Appl Phys 68(2), 775-784(1990).