Damage threshold of monocrystalline silicon irradiated by a millisecond pulsed laser

MING GUO[a,b], YONG-XIANG ZHANG[c], LI HONG[a,b], NAN LI[a,b], JING ZHANGI[a,b], JING-YI LI[d], GUANG-YONG JIN[d,*]
[a]*Jilin Engineering Normal University, Institute for Interdisciplinary Quantum Information Technology, Changchun 130052, China*
[b]*Jilin Engineering Laboratory for Quantum Information Technology, Changchun 130052, China*
[c]*College of Optical and Electronical Information Changchun University of Science and Technology, Changchun 130022, China*
[d]*Changchun University of Science and Technology, Changchun 130022, China*

The laser damage threshold is an important index to describe the laser shock and protection. In order to determine the cleavage and the melting damage threshold of monocrystalline silicon irradiated by a millisecond pulsed laser, a temperature measurement system with a high-precision point thermometer was built. Based on the optical interference theory and on the Mach-Zehnder interference system, a real-time measurement method to determine the stress and the strain of millisecond pulsed lasers interacting with monocrystalline silicon was studied. The research results indicate that the covalent bond fracture damage of the monocrystalline silicon occurs initially under the action of the low-energy density laser; the stress damage dominates and the (100) crystal plane crack is of the "＋" type. The crack morphology depends on the surface crystal orientation of the monocrystalline silicon. For a large energy density, the thermal damage effect dominates. Under the action of a single pulse laser with a pulse width of 1.0–3.0 ms, the thermal damage threshold of monocrystalline silicon measures 33.0–57.2J/cm^2. The peak temperature of the center point of the laser increases as the number of pulses increases. The damage threshold of monocrystalline silicon decreases when the temperature and the stress exhibit a cumulative effect.

(Received June 18, 2019; accepted February 12, 2021)

Keywords: Millisecond laser, Monocrystalline Silicon, Threshold

1. Introduction

Silicon is a basic material in the electronic information technology and new energy industries [1-2]. Lasers can be used to process the surface of silicon and to manufacture submicron microdevices [3-6]. Semiconductor doping techniques can be carried out by using millisecond pulse lasers [7, 8]. The depth can reach to dozens of nanometers, the activation rate of the impurities in annealing process can be increased [9-11]. The silicon fine-scale electronic components in the laser trimming integrated circuit exhibit high-precision, high-speed, and low-cost integrated circuits. J Bonse [12] studied the threshold and the morphology of the femtosecond laser ablation on monocrystalline silicon. M.J. Buehler [13] experimentally investigated the dynamic crack of monocrystalline silicon controlled via the threshold crack speed. Zewen Li [14] simulated the field temperature distribution of a monocrystalline silicon irradiated by a millisecond pulsed laser. A two-dimensional transient solution model was established. The surface temperature change, melting, vaporization, as well as other phase transition processes, which take place after the irradiation of the monocrystalline silicon were analyzed. R. N. Oosterbeek [15] studied the effect of the type and concentration of silicon doping on the ablation threshold of the femtosecond laser-effect silicon. Currently, the study of laser-irradiated monocrystalline silicon is mainly carried out by using ultrashort-pulse lasers: The single laser pulse has high output energy, a high propagation efficiency in the air, and a high energy coupling efficiency during the irradiation. Moreover, short-pulse lasers with millisecond pulses have a higher degree of damage on the target when compared to short pulse lasers.

In this paper, the relation between the damage threshold and the laser parameters of a series of monocrystalline silicon targets is investigated. The experimental system was built and the thermal damage threshold and the stress damage threshold of monocrystalline silicon were measured. The mechanism behind the monocrystalline silicon damage caused by a millisecond pulsed laser was studied.

2. Characterization method of the damage threshold

When irreversible changes in the morphology of monocrystalline silicon, such as the cleavage and melting point, take place, an increase in the surface temperature occurs. Hence a high-precision point thermometer is used to monitor the temperature rise of the samples to test whether the monocrystalline silicon is damaged or not. A test method to reveal the surface morphology changes in monocrystalline silicon consisting in a 100-fold gold microscope was used. The measurement accuracy of the damage threshold was improved, and the measurement errors were reduced. The monocrystalline silicon damage threshold was characterized via the one-to-one method, which uses a constant laser energy density to act 10 points on the monocrystalline silicon surface. The laser energy density increases when its laser energy density exceeds the critical value. The surface morphology of the silicon can be clearly observed with a gold image microscope. Contrarily, when the laser energy density is lower than the critical value of the laser energy density, the surface morphology of the monocrystalline silicon cannot be fully observed with a 100-fold gold microscope. By averaging 10 energy density measurements acquired at the lowest energy density necessary to exceed the critical value, the damage threshold of the material is measured.

3. Experimental device

The samples manufacturer is Hefei Kejing Material Technology Company and the crystal plane orientation of the monocrystalline silicon was (100). The sample had a radius of 12.7 mm and a thickness of 4.0 mm and was polished on one side. It was ultrasonically cleaned in acetone and methanol for 15 minutes before testing. The experiment was conducted in air with an ambient temperature of 22°C, a relative humidity of 51％, and a pressure of 10^5 Pa.

The device for measuring the temperature of the monocrystalline silicon irradiated by the millisecond pulse is shown in Fig. 1. The laser source is a Meyer-100 1064 nm laser. In the experiment, the pulse width was set to 1.0 ms, 1.5 ms, 2.0 ms, 2.5 ms, and 3.0 ms, and the laser energy distribution focused onto the monocrystalline silicon surface was almost Gaussian. The laser provided a monopulse or pulse string output. In the setup, after the output laser power passes through the beam splitter, part of it is split via a dichroic prism and enters the VEGA FL500A's energy meter. This provides a measurement of the laser energy. The splitting ratio of the beam splitter is 1:49. The second beam passes through a focusing lens with a 500 mm focal length. The diameter of the spot on the monocrystalline silicon sample is approximately 2.0 mm. The monocrystalline silicon sample was clamped on a five-dimensional translation stage. A KBU1600-USB with a high-precision point thermometer was used to directly measure the temperature of the monocrystalline silicon surface during the experiment.

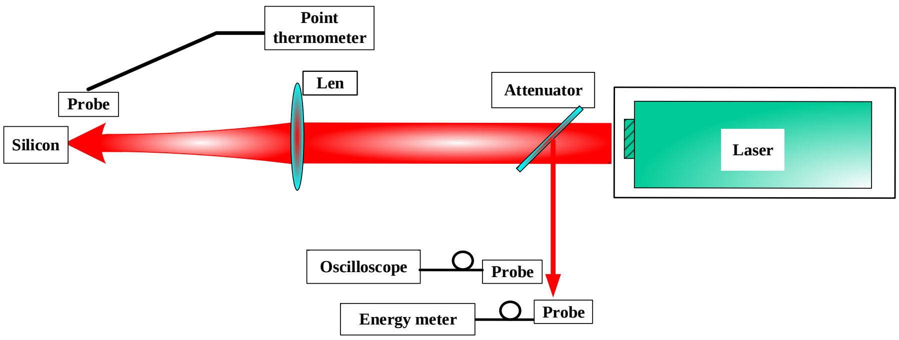

Fig. 1. Temperature measurement system for the monocrystalline silicon sample irradiated by a millisecond pulsed laser (color online)

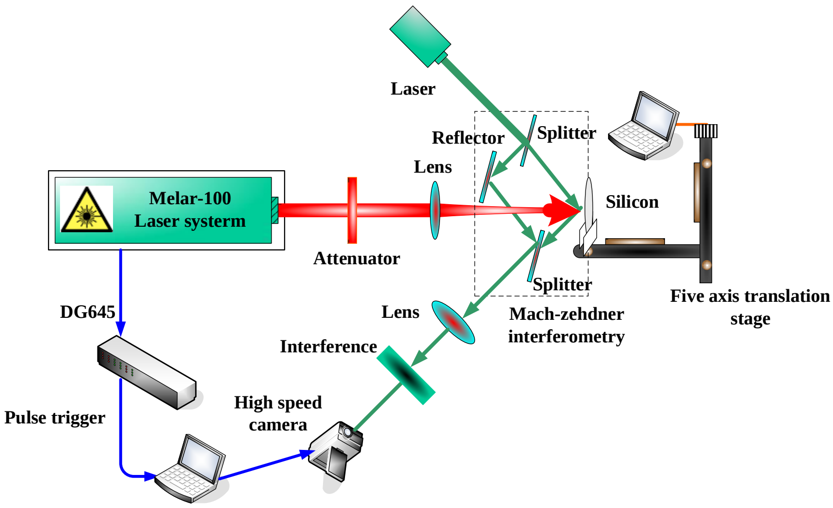

Fig. 2. Stress measurement system (color online)

Fig. 2 shows the on-line measurement system used to measure the stress generated by the pulsed laser on the monocrystalline silicon. The sample and laser parameters used in the experiment are identical to those used for the thermal damage threshold measurement. An attenuation system and a focusing lens are positioned in the optical path. The probe light is divided into two beams via a beam splitter and the monocrystalline silicon is irradiated. The spot size of the probe is larger than the spot size of the incident light. The second beam is irradiated onto a mirror. After the reflection, the two beams are combined and form a series of interference fringes after being focused by a lens. The semiconductor laser has a wavelength of 532 nm and a linewidth smaller than 0.7 nm. The high-speed camera has a frame frequency of 10,000 and is synchronously triggered with the laser. To prevent the camera from being damaged by the laser, an interference filter and a neutral filter were added before the camera to filter out the remaining reflected laser light and attenuate the backlight intensity.

A high-speed camera and a Mach-Zehnder optical interferometer system were used to convert the deformation of the monocrystalline silicon surface into a variation in the interference pattern; the fringes were then converted into strain values by processing their changes from frame to frame via the high-speed camera. In this way, the stress of the monocrystalline silicon target was calculated. The real-time damage morphology was obtained via a real-time measurement of the strain evolution of the monocrystalline silicon.

4. Results and analysis

A Leica DMI5000M metallographic microscope was employed to measure the two-dimensional morphology of a monocrystalline silicon sample irradiated by millisecond laser pulses. When the laser interacts with the monocrystalline silicon, multiple damage effects are often coupled together at the same time. The damage threshold is the result of a variety of coupling effects generated by these interactions. The morphological analysis of the laser damage threshold provides a basis to study the damage effect induced by a pulse laser on monocrystalline silicon.

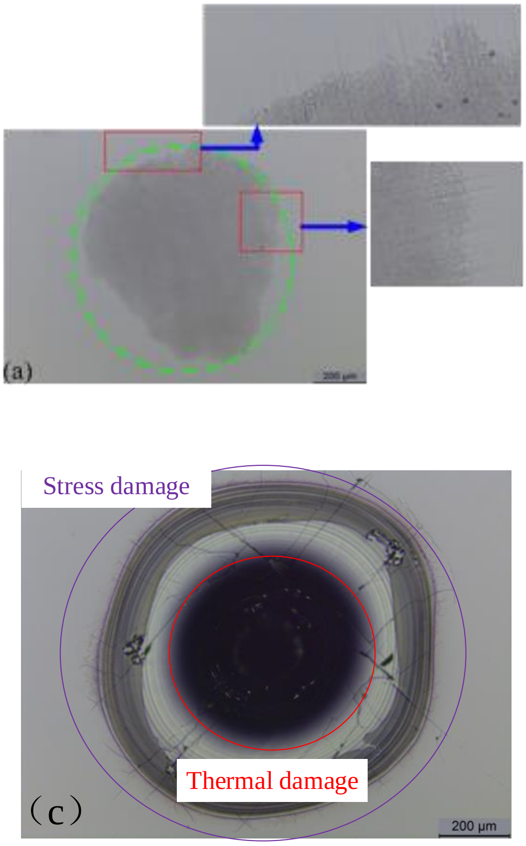

Fig. 3. Morphology of damaged monocrystalline silicon irradiated by a laser with different energy density values: (a) Ep = 26.2 J/cm^2, (b) Ep = 31.8 J/cm^2, (c) Ep = 287.5J/cm^2, and (d) Ep =325.4 J/cm^2 (color online)

The morphology of the damaged monocrystalline silicon sample is shown in Fig. 3. Inset 3 (a) presents the damage induced by a laser energy density of 26.2 J/cm^2. The surface of the monocrystalline silicon does not reach the melting temperature, and there are obvious microcracks at the edges of the laser-active area (green line). The results show that the microcracks are distributed vertically, indicating that the cleavage morphology is a "十

" type. Monocrystalline silicon is a brittle material with a narrow plastic zone. Its surface absorbs the laser energy, which diffuses toward the interior of the sample via heat conduction. The temperature field formed inside the material is inhomogeneous and generates a large temperature gradient on a millisecond scale. Due to the anisotropy of the sample, which has a typical diamond structure, the (111) crystal plane has the highest atomic surface density and the distance between (111) crystal planes is the largest. For this reason, the atomic bonds of the (111) crystal plane are the weakest and are easy to break. In the experiment, the surface of the monocrystalline silicon sample was oriented along the (100) crystal plane. The angle between the (111) crystal plane and the (100) crystal plane measures 90°. When the fracture occurs, vertical cracks are formed along the (100) crystal plane. When the thermal stress reaches the yield limit of monocrystalline silicon, brittle cracks become evident.

When the temperature of the laser energy absorbed by the monocrystalline silicon reaches the melting point of 1687 K, hot-melt damage occurs in the cleavable region defined by the center of the laser beam. Moreover, the stress cleavage damage occurs at the edges of the molten zone. As the laser energy density increases, the stress damaged area decreases, and the thermal damage area increases. Therefore, under the condition of a low energy density, the stress damage dominates when the monocrystalline silicon sample is irradiated by a millisecond pulsed laser. However, for a high energy density, the thermal damage effect dominates.

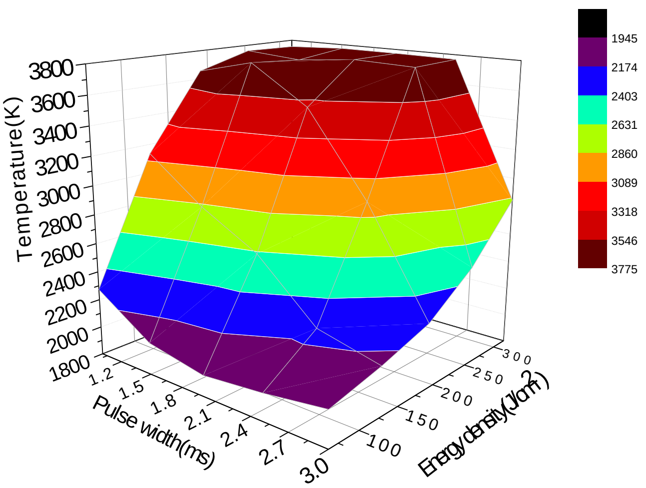

Fig. 4. Three-dimensional graph of the variation of the temperature as a function of the energy density and of the pulse width under a monopulse condition (color online)

Fig. 4 shows that (1) by setting a fixed pulse width, the temperature of the monocrystalline silicon sample increases upon the increase in the laser energy density, and the temperature rises faster; (2) For a fixed pulse laser energy density and upon the increase in the pulse width, the ramp is slow. The temperature at the center of the monocrystalline silicon surface is determined by the laser power density in that point. The longer the pulse width is the smaller becomes the power density of the laser, which enables the temperature in the central point of the monocrystalline silicon surface to drop.

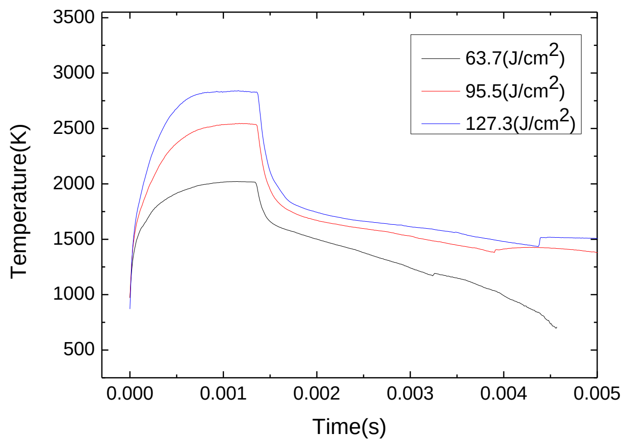

Fig. 5. Evolution of temperature at the center of the monocrystalline silicon (τ_p = 1.5 ms) (color online)

Fig. 5 shows that (1) within the laser pulse width time, the temperature of the central point of the sample gradually increases. Moreover, when the temperature reaches the melting point, the temperature rising curve begins to deflect. As the laser energy density increases, the solid-liquid phase increases. This variation generates a slow increase in the temperature trend, and a vaporization plateau is formed; (2) when the irradiation time is longer than the pulse width, the laser stops irradiating the monocrystalline silicon, and the temperature of the central point of the monocrystalline silicon surface drops. When the monocrystalline silicon undergoes the liquid-solid phase transition (1687 K), the slope of the curve is small and a plateau is visible when the temperature of the monocrystalline silicon changes rapidly after the liquid-solid phase transition occurs. The higher the laser energy density is, the longer is the plateau period.

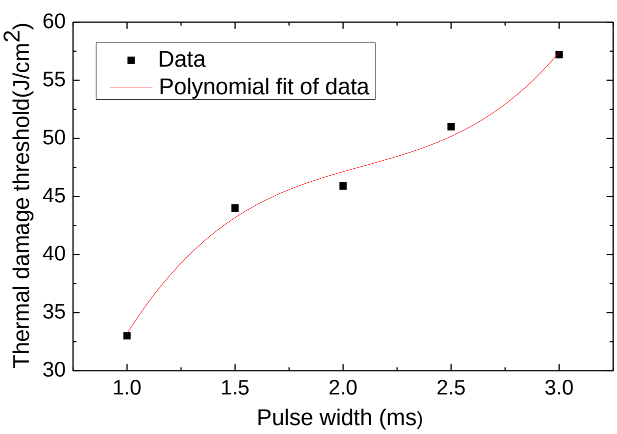

Fig. 6. Relation between the thermal damage threshold and the laser pulse width

Fig. 6 shows the variation of the thermal damage threshold of the monocrystalline silicon sample as a function of the pulse width under the single-pulse condition. The results reported in Fig. 6 show that the thermal damage threshold range of monocrystalline silicon with a laser pulse width of 1.0–3.0 ms is in the 33.0–57.2 J/cm^2 range. When the laser energy density is constant, as the pulse width increases, the laser power density is lower and the temperature of the irradiation central point on monocrystalline silicon surface decreases. Increasing the laser pulse width is equivalent to increase the duration of the action the time during which the heat conduction process takes place when the energy of the laser remains constant. In these circumstances, a higher amount of energy is lost due to thermal diffusion, leading to a slower temperature rise and a lower peak temperature. This generates an increase in the damage threshold as a function of the pulse width. The experimental data was processed by using a polynomial fit of the third order: the damage threshold increases rapidly.

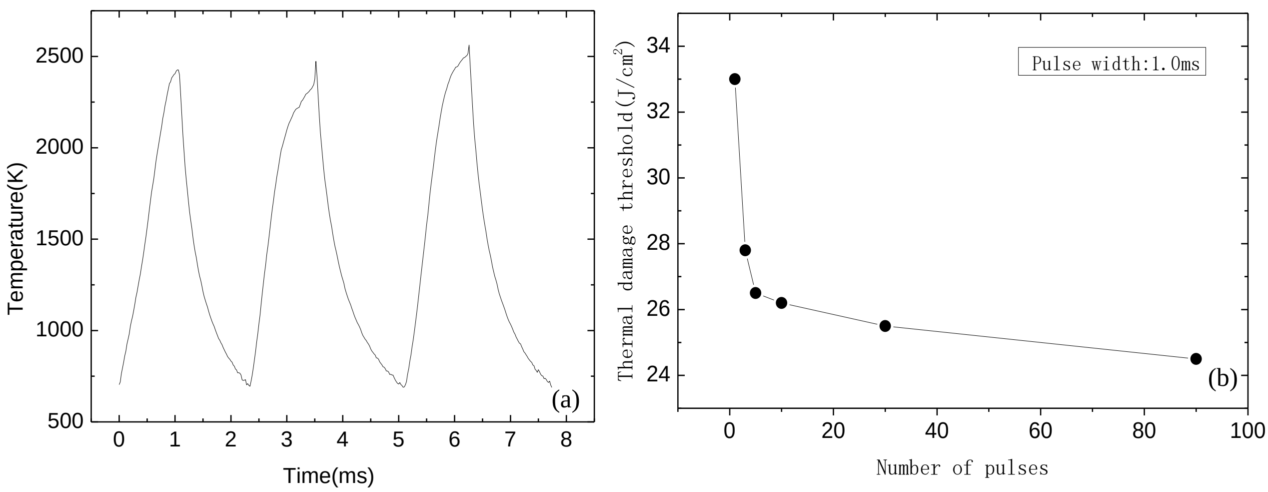

Fig. 7. Temperature evolution and thermal damage threshold under the action of a pulse string laser ((a) Temperature evolution, (b) Thermal damage threshold)

The temperature evolution of monocrystalline silicon sample is shown in Fig. 7(a). From the perspective of the temperature change process, monocrystalline silicon exhibits a pulse string effect after being irradiated with laser light. Under the action of the pulse string laser, the peak temperature of the sample continuously rises, indicating that the heath is continuously accumulated. This implies that the parameters of the damage region of the monocrystalline silicon target change and the absorption coefficient of the incident laser increases. When melting, the solidification time of the monocrystalline silicon target, and the time to reach the normal temperature starting from the melting point are longer, the laser energy deposited inside the monocrystalline silicon increases with the increase in the number of pulses.

Fig. 7(b) shows the relation between the thermal damage threshold of monocrystalline silicon and the number of pulses. From Fig. 7(b) it can be seen that upon the increase in the number of pulses, the laser damage threshold decreases, showing a change in the secondary e-index attenuation. When the number of laser pulses increases to 90, the damage threshold drops to 73.8% of the single-pulse damage threshold. The threshold of monocrystalline silicon damaged by the train of pulses is lower than the threshold measured by damaging with a single pulse laser, due to the presence of the heath accumulation effect induced by the train of pulses. When the monocrystalline silicon target is irradiated by the pulse train laser, the heat absorbed by the monocrystalline silicon cannot be completely consumed via heat conduction in the time interval between two pulses. After several cycles, the target experiences a cumulative effect. As the number of pulses increases, the laser energy continuously accumulates inside the monocrystalline silicon, causing the temperature of the monocrystalline silicon to increase continuously. For this reason, the monocrystalline silicon sample can be more likely damaged. When the pulse train laser is used, the absorption coefficient of monocrystalline silicon increases due to the thermal action of the laser pulses. This results in an increase in the pulse energy absorbed by the sample and the damage threshold in the case of a pulse string laser is lower than the one measured by using a single pulse device.

The evolution of the interference fringe pattern on the monocrystalline silicon surface is shown in Fig. 8. The laser energy density is 6.1 J/cm^2 and the pulse width is 3.0 ms. During the laser action, strain stress occurs on the surface of the monocrystalline silicon upon an increase in the lasing time. Moreover, the interference fringes on the surface of the monocrystalline silicon undergo a bending deformation. The first-order deformation occurs in the k+1 fringe, and the adjacent fringes are the k+1 and k+2 streaks, respectively.

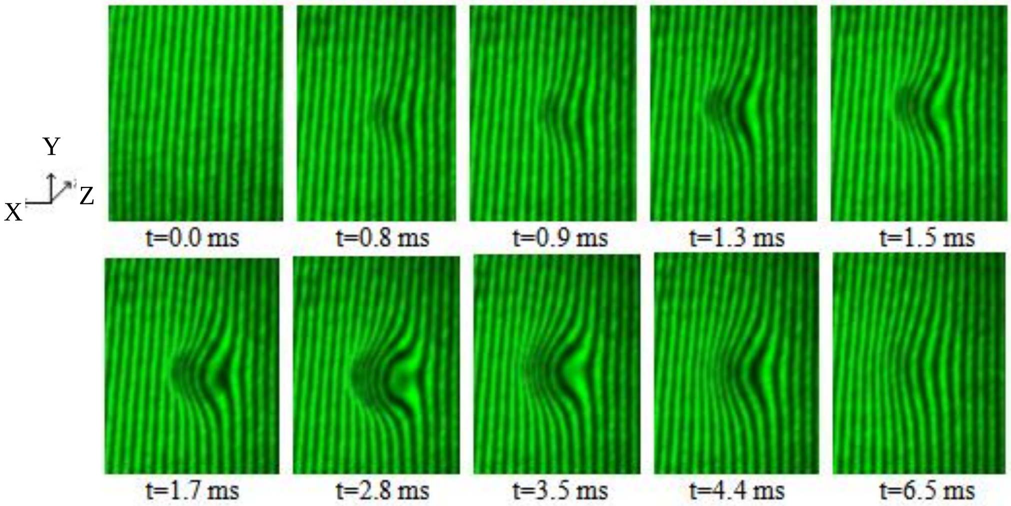

Fig. 8. Variations of the laser-centered Mac-Zehdner interference fringe in monocrystalline silicon at different times

The threshold measurement method was used to obtain the stress damage threshold of monocrystalline silicon for a laser pulse width of 1.0 ms, 1.5 ms, 2.0 ms, 2.5 ms, and 3.0 ms, as shown in Fig. 9.

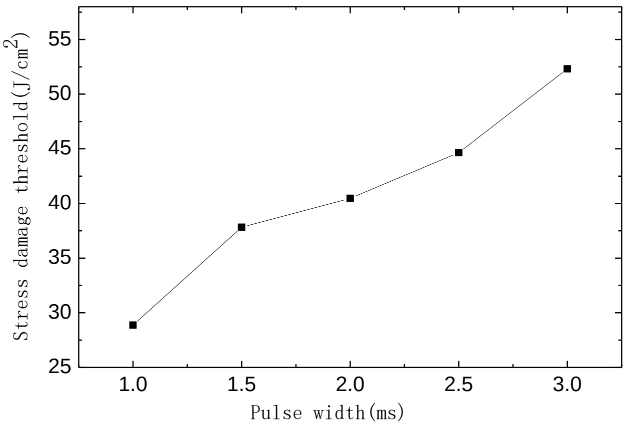

Fig. 9. Relationship between the stress damage threshold and the pulse width

Fig. 9 shows the stress damage threshold of monocrystalline silicon as a function of the change of the width of the laser pulse. The laser energy absorbed by the crystal lattice is quickly transformed into thermal energy, which causes the expansion and the contraction deformations that generate the thermal stress. When the thermal stress exceeds the yield stress of the silicon lattice, the crystal bonds inside the silicon crystal break and slip, generating damage in the lattice itself. Fig. 9 shows that the stress damage threshold of monocrystalline silicon increases with the pulse width when the laser pulse width measures 1.0–3.0 ms. The stress damage threshold induced by a pulse width of 1.0–3.0 ms is 28.9–52.3 J/cm^2. The silicon forbidden band width gradually narrows with the increasing temperature. The width of the band gap corresponds to the minimum energy required to generate an intrinsic excitation, and the temperature increase causes the excitation of several electrons. At room temperature, the intrinsic carrier concentration in silicon is very low, however it increases rapidly upon an increase in the temperature. When the temperature rises to a certain value, the carrier excitation becomes stronger. For a fixed laser energy density and with an increasing pulse width, the temperature becomes smaller and the damage threshold increases.

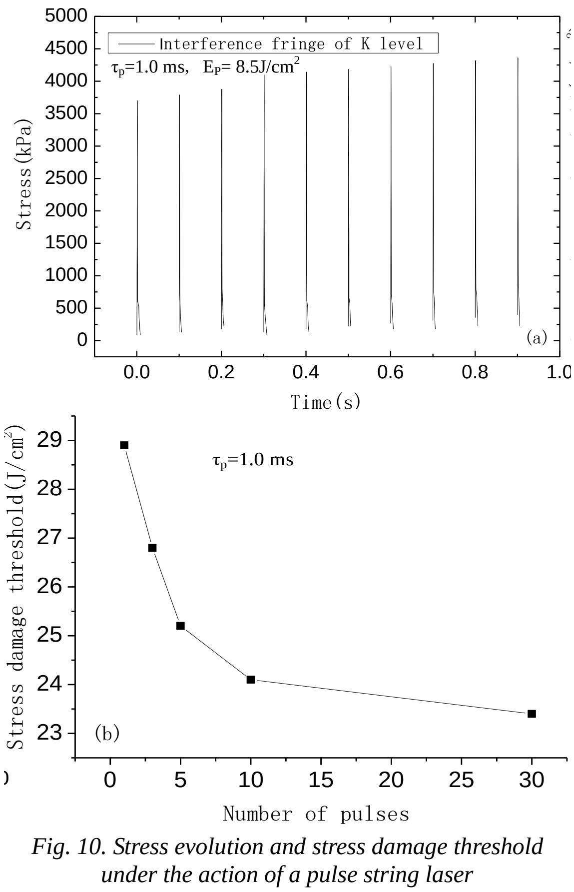

Fig. 10. Stress evolution and stress damage threshold under the action of a pulse string laser ((a) Stress evolution, (b) Stress damage threshold)

Fig. 10(a) shows that the change in the trend of each pulse is similar to that of a monopulse. Moreover, the maximum stress value increases from the 370 kPa of the first pulse to the 436 kPa of the 10th pulse, and this increment is 66 kPa. This indicates that the effect of the stress accumulation occurs under when a string laser mode is used.

Fig. 10(b) shows that when the pulse train acts on the monocrystalline silicon, the temperature rise area increases and it is no longer limited to the laser irradiation area, but it extends outwards. When the laser irradiation ends, the temperature gradient increases, providing a starting point for the thermal slip of monocrystalline silicon. This results in a larger thermal strain and thermal stress, which imply that the sample is more likely to be damaged. The irradiation generates tiny dislocations on the cleaved surface of the monocrystalline silicon sample due to the thermal action of the laser millisecond pulses of the string laser. Both the absorption coefficient and the thermal stress increase with the increase of the energy due to the pulse absorption. As the number of pulses increases and the temperature rises, the carrier excitation becomes stronger. For this reason, the pulse laser stress damage threshold of monocrystalline silicon is lower than the monopulse laser damage threshold.

5. Conclusion

An experimental setup to perform a series of temperature and stress on-line measurements on a monocrystalline silicon sample irradiated by a millisecond pulse laser was constructed. Monocrystalline silicon is brittle material, stress damage firstly occurs under the irradiation of pulsed laser. With the increase in the laser energy density, the monocrystalline silicon target melts, and the thermal damage effect dominates. The damage threshold increases as the pulse width increase. The threshold for a pulse width of 3 ms is 1.7 times higher than for a 1 ms pulse and measures 33.0–57.2 J/cm^2. Increasing the laser pulse width is equivalent to increase the duration of the laser action and of the heat conduction time, when the energy of the laser remains constant. Moreover, more energy is lost due to the thermal diffusion and the laser power density is reduced, leading to a slower temperature rise and a lower peak temperature. Under the action of a pulse train laser, the thermal damage threshold decreases with the increase in the number of pulses and exhibits a secondary exponential decay. By using a string laser, a significant temperature and stress accumulation effect can be detected on the monocrystalline silicon surface. The coefficient of the sample increases due to the thermal action of the laser pulses, resulting in an increase in the pulse energy absorbed from the material. For this reason, the damage threshold of the sample irradiated with a pulse string laser is lower than that measured with a single pulse one. As the pulse width increases from 1.0 ms to 3.0 ms, the stress damage threshold varies from 28.9 J/cm^2 to 52.3 J/cm^2. The results of this paper provide a theoretical and experimental reference to guide the processing and measure the laser damage threshold of monocrystalline silicon.

Acknowledgment

Project supported by the Youth Science of National Natural Science Fund Project(Grant No. 61905089), the 13th Five-Year Scientific Research Planning Project of the Jilin Provincial Department of Education(Grant No. JJKH20190765KJ), Specialized Fund for the Doctoral Research of Jilin Engineering Normal University (Grant No. BSKJ201824), School level project of Jilin Engineering Normal University (Grant No.XYB201819).

References

[1] G. Wang, E. Rosseel, R. Loo et al., Applied Physics Letters **96**(11), 637 (2010).
[2] Jostein Thorstensen, Energy Procedia **38**, 794 (2013).
[3] J. Liu, X. Jia, W. Wu et al., Optics Express **26**(5), 6302 (2018).
[4] C. T. M. Kwok, R. D. Braatz, S. Paul et al., Journal of Applied Physics **105**(6), 287 (2009).
[5] O. Garcia, J. J. Garcia-Ballesteros, David Munoz-Martin et al., Applied Surface **278**, 214 (2013).
[6] Y. Calahorra, O. Shtempluck, V. Kotchetkov et al., Nano Letters **15**(5), 2945 (2015).
[7] Li Fei Zhong, Journal of Hunan University of Science and Technology **32**(4), 31 (2011).
[8] Tie Chuan Zuo, Hong Chen, Journal of Mechanical Engineering **45**(10), 106 (2009).
[9] K. Mangersnes, S. E. Foss, A. Thogersen, Journal of Applied Physics **107**(4), 350 (2010).
[10] M. F. Ametowobla, Uni Stuttgart – Universitätsbibliothek, 2010.
[11] G. Hellings, E. Rosseel, E. Simoen et al., Electrochemical and Solid-State Letters **14**(1), H39 (2011).
[12] J. Bonse, S. Baudach, J. Krüger et al., Appl. Phys. A **74**(1), 19 (2002).
[13] M. J. Buehler, H. Tang, A. C. van Duin, Physical Review Letters **99**(16), 165502 (2007).
[14] Z. Li, H. Zhang, Z. Shen et al., Journal of Applied Physics **114**(3), H39-H41 (2013).
[15] R. N. Oosterbeek, C. Corazza, S. Ashforth et al., Applied Physics A **122**(4), 449 (2016).

*Corresponding author: laserqit@sina.com