CAAP Quarterly Report

Date of Report: July 10, 2016

Contract Number: DTPH56-14-S-N000006

Prepared for: Government Agency: DOT

Project Title: Laser peening for preventing pipe corrosion and failure

Prepared by: Board of Regents of the University of Nebraska for the University of Nebraska-Lincoln

Contact Information: Yongfeng Lu
Lott Distinguished Professor
Department of Electrical and Computer Engineering,
University of Nebraska-Lincoln,
209N Walter Scott Engineering Center,
Lincoln, NE 68588-0511
Phone: (402)472 8323
E-mail: ylu2@unl.edu

Other members: Leimin Deng (Postdoctoral researcher); Chenfei Zhang (Ph.D student); Shiding Sun (Ph.D student)

For quarterly period ending: June 30, 2016

Business and Activity Section

(a) Status Update of Past Quarter Activities

1. Major goal of the project

The ultimate goal of this project is to investigate and develop laser peening of stainless and carbon steels used for pipeline construction to improve their corrosion resistance. The corrosion resistance of pipelines will be enhanced via the compressive residual stress created by laser-induced shock waves during the laser peening. It is anticipated that using laser shock peening in

the construction of a pipeline will highly improve the reliability, safety, and lifespan of the nation's pipeline transportation system. As stated in our proposal, the major goals of this project will be achieved by organizing the project into four phases, each with specific objectives. Table 1 summarizes the current progress of each phase and its objectives during this reporting period.

Table 1. The completion rate for the objectives of each phase of the project

Phases	Major Goals and Milestones	Starting Date (mm/dd/yy)	Ending Date (mm/dd/yy)	Completion Rate (%)
Phase 1	1) Develop experimental setup for laser cleaning.	10/01/2014	12/31/2014	100
	2) Establish cleaning mechanism and parameter windows.	01/01/2015	09/30/2016	70
	3) Develop real-time monitoring of cleaning process.	01/01/2015	09/30/2016	0
Phase 2	1) Develop laser peening system.	10/01/2014	12/31/2014	100
	2) Establish parameter windows for laser peening.	01/01/2015	09/30/2016	100
Phase 3	1) Study surface morphology of the laser-peened surfaces.	01/01/2015	09/30/2016	100
	2) Determine residual stress of pipeline steels after laser cleaning and peening.	01/01/2015	09/30/2016	100
	3) Study corrosion resistance of laser-peened surface.	01/01/2015	09/30/2016	40
Phase 4	Design a laser peening prototype system.	06/30/2016	09/30/2016	50

2. Specific objectives during this reporting period

The results of our experiments provided in the previous reports have shown great improvement in mechanical property, residual stress, microstructure, and stress corrosion cracking (SCC) resistance of both stainless steel (SS) and carbon steel (CS) after laser shock peening (LSP) with a lab-based high energy Nd:YAG laser. These results could fulfill the project requirements and be used for the design of the proposed laser peening equipment. To further pave the way for widely applicable portable equipment which could be used for in situ laser peening in the future, a compact, low energy Nd:YAG laser was used for LSP experiments (during this reporting period of April 1, 2016, to June 30, 2016) instead of a delicate and ponderous high energy laser. Some key parameters were optimized for better peening effects, which were examined by surface morphology and residual stress measurements. In addition, metallographic imaging was also conducted for microstructure investigation. The objectives and specific content were as follows:

(1) Low energy laser peening system establishment with a compact Nd:YAG laser (Quantel DRL650).

(2) Parameter optimization of the low energy Nd:YAG laser peening: focused laser spot size, sample position, peening times, sacrificial coating, and spot-to-spot distance.

(3) Surface morphology and residual stress measurement of carbon steel.

(4) Metallographic investigation of carbon steel before and after laser peening.

3. Significant results

3.1. Experimental setup and procedure

3.1.1. Material preparation

During the reporting period, the samples tested were prepared from 1018 carbon steel (CS) and aluminum-magnesium (Al-Mg) alloy plates, which were cut into rectangular shapes with dimensions of 2 × 2 × 0.25 in. (length × width × thickness). Specifically, Al-Mg alloy was only used for the adjustment of the sample position, instead of CS, due to its softness, which could better reflect the LSP effect by morphology changes. Prior to the LSP treatment, mechanical polishing was carried out using sandpapers of different grades (400-1200 grift) to obtain mirror-like sample surfaces. If needed, the electrochemical polishing was conducted by immersing the CS into a specific polishing solution (an ethanol solution with 10 percent $HClO_4$) and positively biased at 25 V for a certain time. The polishing was followed by ultrasonic cleaning, first with acetone and then deionized water, to degrease the sample surface. All samples were prepared shortly before the LSP experiments.

3.1.2. Low energy Nd:YAG laser peening setup and procedures

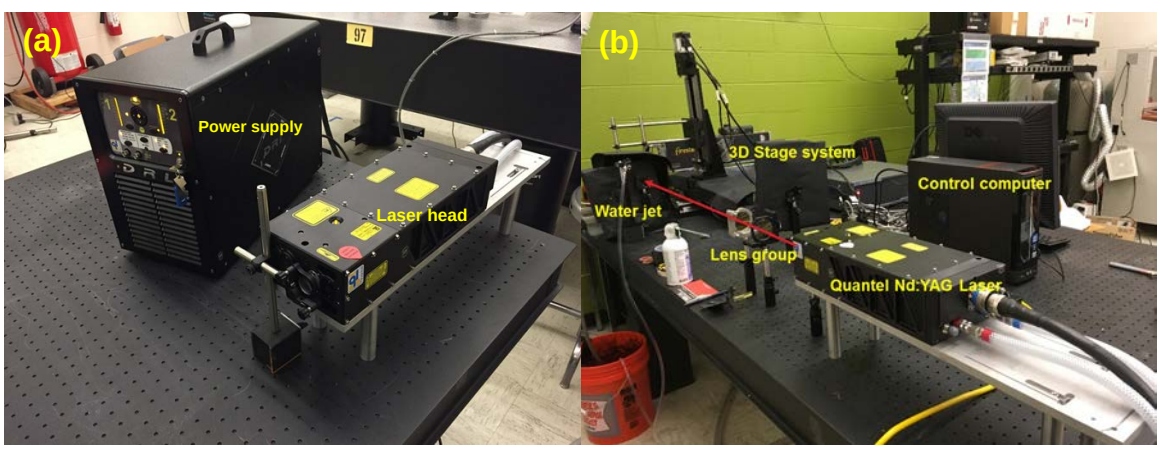

Fig. 1. Newly designed Nd:YAG laser peening system.

A new laser peening system (Fig. 1a) was established using a compact Quantel Nd:YAG laser (DRL650) and a programmable 3D stage, which is a test system for future portable laser peening

equipment. The laser is lightweight, with a 6.4 Kg laser head and an 18 Kg power supply. The size of the laser system is also very compact: the laser head measures only 94 × 432 × 160 mm, while the power supply is 406 × 236 × 500 mm. The laser head is completely sealed and filled with high-pressure gas, allowing it to operate in harsh environments such as underwater, high humidity, and serious vibration. The main parameters of the laser are: 1064 nm wavelength, 650 mJ maximum pulse energy, 15 ns pulse duration, 6.5 mm output beam diameter, and 1-30 Hz repetition rate. Although the laser repetition rate could be set from 1 to 30 Hz, we only used 5 Hz to avoid the incident laser from being seriously disturbed by the water spray induced by previous laser impacts. The entire laser peening system is shown in Fig. 1b. The sample was held by a three dimensional (3D) stage controlled by a computer to realize the scanning path and speed needed. The water jet was fixed to the stage to keep the water film stable on the sample surface while the stage was moving. A blower was used to prevent water sprays from reaching the optics. Two kinds of optical designs are shown in Fig. 2. For both of them, a plano-concave lens was used to expand the laser beam diameter followed by two plano-convex lenses to collimate and focus the laser beam. The laser spot diameter, after focusing, could be varied by changing the lens group setup; and the designs achieved two different focused spot diameters of 2.2 and 1.4 mm. Considering the influence of the sacrificial coating, LSP with and without 100 μm black tape was tested. The details of the laser parameters, confining layer, and sacrificial coating are shown in Table 2.

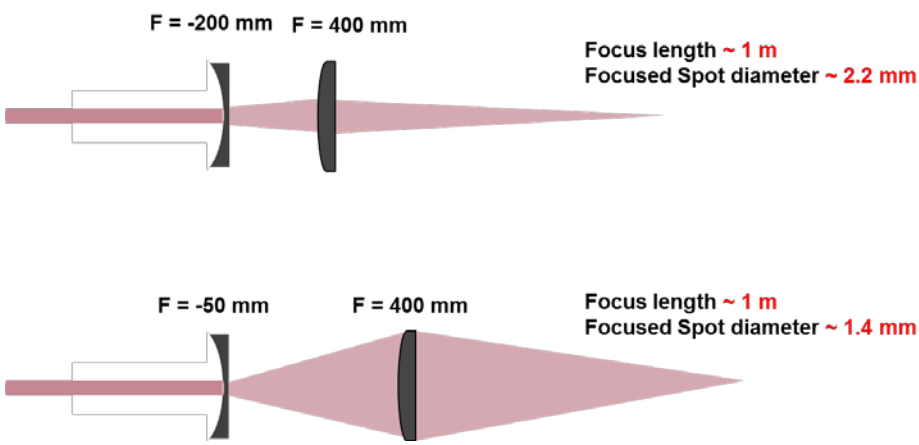

Fig. 2. Schematic of the two kinds of optical designs used during this reporting period.

Table 2. The processing parameters used in the LSP experiment.

Laser system	Quantel DRL650 laser; wavelength: 1064 nm; pulse energy: 650 mJ; pulse duration: 15 ns; frequency: 1-30 Hz; spot size after focusing: 2.2 or 1.4 mm
Confining layer	Water jet with a layer thickness of approximately 1 mm
Sacrificial coating	Black tape (~100 μm) or no sacrificial coating

3.1.3. Characterization methods

A 3D profile meter (New View 8000, Zygo Corporation) was used to characterize the surface morphologies after LSP to optimize the sample position. The surface residual stress measurements were conducted by X-ray diffraction (XRD) equipment (Smartlab, Rigaku) with copper (Cu) as the anode target and monochromator for the signal filter. The 2-theta angle was selected as 137°, while five incident angles were chosen as -45°, -37.76^o, -30°, -20.7^o, and 0°. The elastic modulus was selected as 200,000 MPa with a Poisson's rate of 0.29. In order to obtain the residual stress distribution along the depth, electrochemical polishing was used to remove thin layers from the sample surfaces with certain thicknesses. Nital, consisting of 4% HNO_3 and 96% ethanol, was used as the etchant for metallographic imaging of CS with a Keyence laser scanning microscope VK-X200K for the image capture.

3.2. Results and discussions

During this reporting period, the Quantel DRL650 Nd:YAG laser was employed instead of the previously used Continuum PR II 8010 laser due to its compact size, small weight, and adaptability to harsh environments (Sec. 3.1.2). The optical setups were as shown in Fig. 2 to obtain two different laser spot diameters at the focus point: 2.2 mm (large spot) and 1.4 mm (small spot). Laser peening without coating (LPwC) was carried out as another promising method in addition to LSP with sacrificial coating.The effects of the sample position, spot-to-spot distance, and laser peening times were also examined.

3.2.1. Large spot Nd:YAG laser shock peening experiments

With a large spot of 2.2 mm diameter at the focus point, both single spot laser peening for sample position optimization and large area laser peening with and without black tape were conducted.

(a) Optimization of the sample position

The sample position optimization was first conducted on Al alloy by single spot laser peening. One LSP impact with a pulse energy of 650 mJ was used with 100 µm black tape as the sacrificial coating. The LSP-generated dent depth and diameter at different sample positions are shown in Fig. 3a and 3b. The zero sample position was arbitrary. The dent depth increased from ~ 10 to ~ 20 µm with the sample position changing from -80 to 0 mm, topping at 0 to 30 mm, and decreasing slowly from 30 to 80 mm with strong variation. In Fig. 3a, the dashed blue square shows that in a large scale of about 100 mm length (-20 to 80 mm), the dent depth remains at a relatively large value (> 18 µm). In Fig. 3b, the dent diameter also shows stability at the same range with a value of approximately 1.8 mm. These results indicate similar shock wave intensity and profile among a large range of sample positions, which should be attributed to the laser characteristics. Different from our previously used Nd:YAG laser with a near Gaussian profile,

the Quantel laser has a flat top profile, which allows for uniform distribution of laser energy at the cross-section view and a longer depth of focus (DOF) along the propagation direction. These two laser characteristics result in a more uniform shock wave within a large range of sample positions, which means the low energy laser peening system is insensitive to laser-sample distance and is, therefore, beneficial for easy operation in practical environments. The sample position of 20 mm is used throughout the following parts of Sec. 3.2.1.

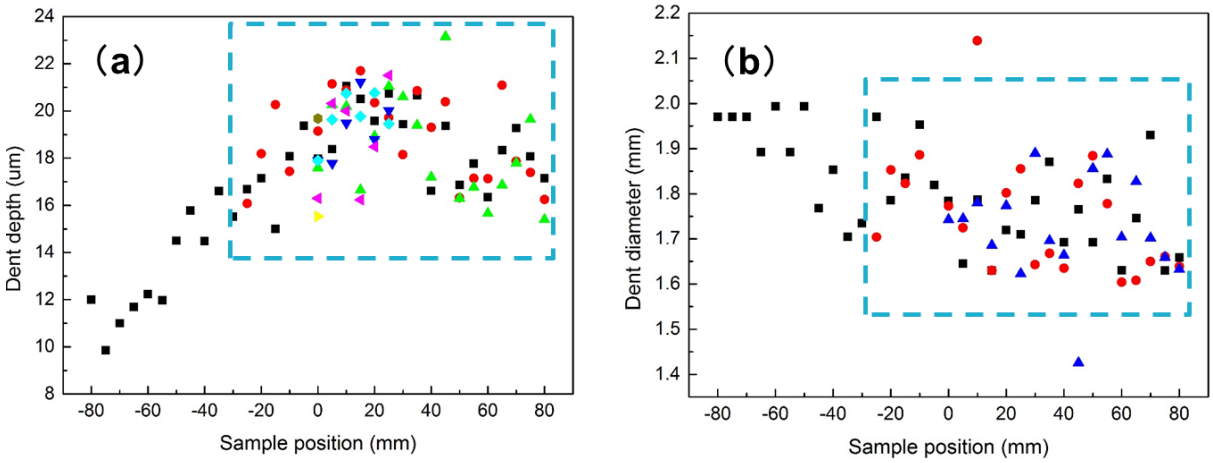

Fig. 3. Laser-peening-induced (a) dent depth and (b) dent diameter at different sample positions on an Al alloy surface.

(b) Large spot laser shock peening with black tape

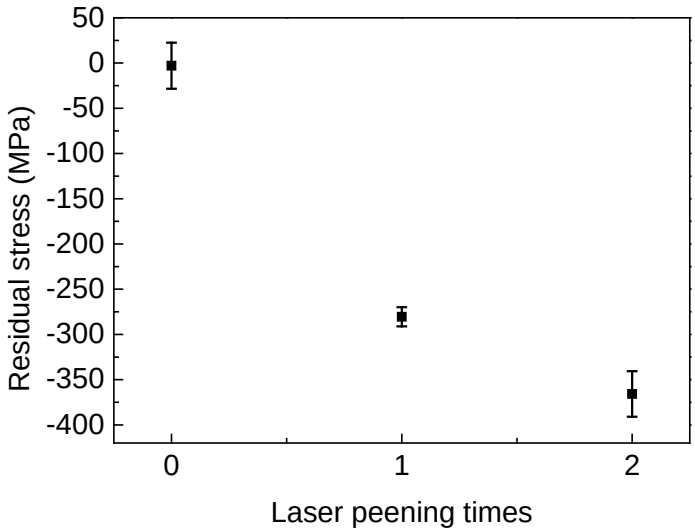

Fig. 4. Surface residual stress of the 1018 carbon steel after laser peening with black tape for different times (2.2 mm spot diameter, 650 mJ pulse energy, 0.5/0.75 spot-to-spot distance).

Large area LSP was carried out with a laser spot size of 2.2 mm diameter on the 1018 CS with black tape as the sacrificial coating. Considering the laser spot shape and diameter, the best spot-

to-spot distance was 0.5/0.8, which means a 0.5 mm spot-to-spot distance along the vertical direction and 0.8 mm along the horizontal direction. The CS was mechanically and electrochemically polished to obtain a stress-free surface and then laser peened one and two times. As shown in Fig. 4., the residual stress was - 3.0 ± 25.5 MPa without laser peening, which increased to - 280.5 ± 10.6 MPa after one laser peening impact and further increased to - 365.8 ± 25.2 after another impact. Thus, for LSP with black tape, the increase of the surface residual stress could be obtained by posting more peening impacts.

(c) Large spot laser shock peening without sacrificial coating

Laser peening without coating (LPwC) is a promising method since it avoids the use of sacrificial coatings, such as tapes, paints, and Al foils. It not only reduces the cost but also lessens contamination generated during the laser peening process. In order to suppress the laser ablation, LPwC could only be realized by low pulse energy, which is appropriate with the DRL650 Nd:YAG laser. Therefore, LPwC was carried out during this period in addition to the normal laser peening.

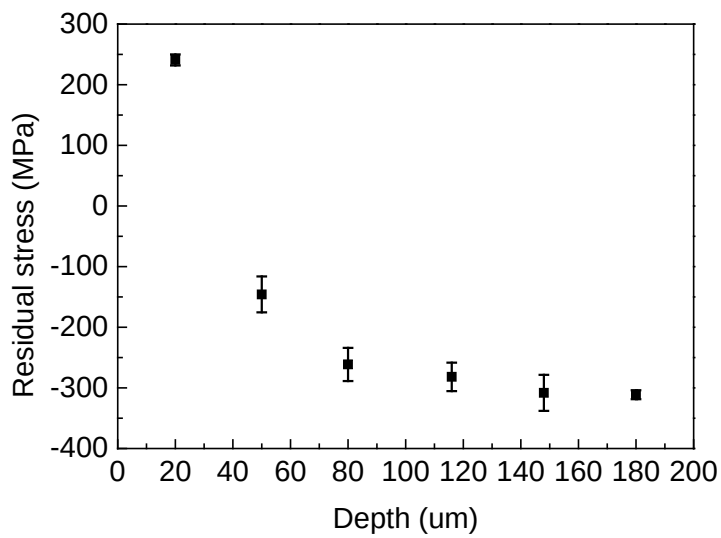

Fig. 5. In depth residual stress distribution of the 1018 CS after LPwC (2.2 mm spot diameter, 650 mJ pulse energy, 0.5/0.75 spot-to-spot distance, three times).

Fig. 5 shows the residual stress distribution of the 1018 CS along the depth after LPwC three times with a 2.2 mm spot diameter, a 650 mJ pulse energy, and a 0.5/0.75 spot-to-spot distance, which means a 0.5 mm spot-to-spot distance along the vertical direction and 0.75 mm along the horizontal direction. The near surface region had a tensile stress of 241 MPa due to the laser ablation, but the depth of the tensile region was less than 50 μm. At deeper regions, the residual stress became compressive, gradually increased to ~300 MPa, and remained stable within 180 μm. Although the tensile residual stress on the surface is a disadvantage for stress corrosion cracking resistance, the compressive residual stress after a 50 μm depth could prevent the

corrosion crack tips from growing into deeper regions. In this aspect, the LPwC was also a possible way to avoid the material and structure failure induced by the stress corrosion cracking in pipeline systems.

3.2.2. Small spot Nd:YAG laser shock peening experiments

A small laser spot of 1.4 mm diameter at the focus point was used both for single spot laser peening (sample position optimization) and large area laser peening with and without black tape.

(a) Optimization of the sample position

As shown in Fig. 6, the laser peening dent depth was measured on the 1018 CS surface after Nd:YAG laser peening at different sample positions (650 mJ pulse energy, 100 µm black tape, one LSP impact). Five repeated spots were measured and averaged for each sample position. The zero point was an arbitrary position. The single spot dent depth varied from 17 to 31 µm and peaked at the sample positions from -8 to 2 mm. With the smaller laser spot diameter of 1.4 mm at the focus point, the dent depth increased enormously to 31 µm compared to the 21 µm dent depth obtained with the 2.2 mm laser spot size. The increase of the dent depth reflects the increment of the laser-induced shock wave intensity due to the decrease of the spot size. On the other hand, the dashed blue square in Fig. 6 shows that in a much smaller scale of only 10 mm length (-8 to 2 mm), the dent depth remained at a large value (> 26 µm). This phenomenon may also be attributed to the small spot size, which makes the laser-induced shock wave easier to attenuate. The sample position of 0 mm is used throughout the following parts of Sec. 3.2.2.

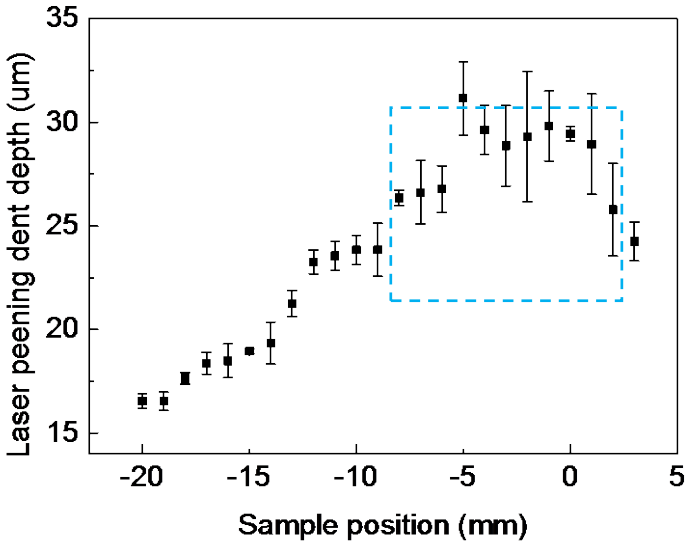

Fig. 6. The dent depth on Al alloy surfaces after laser peening with black tape at different sample positions.

(b) Small spot laser shock peening with black tape

Large area LSP was carried out with a laser spot size of 1.4 mm diameter on the 1018 CS with black tape as the sacrificial coating. Fig. 7 shows the effect of different spot-to-spot distances

(0.5/0.5, 0.5/0.6, 0.6/0.6, 0.6/0.7, and 0.7/0.7) and laser peening times (one and five times) on the surface residual stress. Take 0.5/0.6 for example, it means a 0.5 mm spot-to spot-distance along the vertical direction and 0.6 mm along the horizontal direction. After one laser peening, the surface residual stress became compressive except for the smallest spot-to-spot distance of 0.5/0.5, which was only 7.7 ± 11.3 MPa since the black tape was removed and the sample surface was partly ablated. With larger spot-to-spot distances of 0.5/0.6, 0.6/0.6, 0.6/0.7, and 0.7/0.7, the compressive residual stress increased slightly from -344.9 ± 16.6 to -398.5 ± 21.3 MPa as the spot-to-spot distance became smaller. After laser peening five times, the surface residual stress became compressive with a spot-to-spot distance of 0.5/0.5, even though the sample surface was ablated. On the other hand, the surface residual stress decreased slightly from -398.5 ± 21.3 to -351.7 ± -27.6 MPa with a spot-to-spot distance of 0.5/0.6 while the laser peening times increased from one to five. The in-depth residual stress increase should be the reason for the decrease of the surface residual stress after repeated LSP impacts.

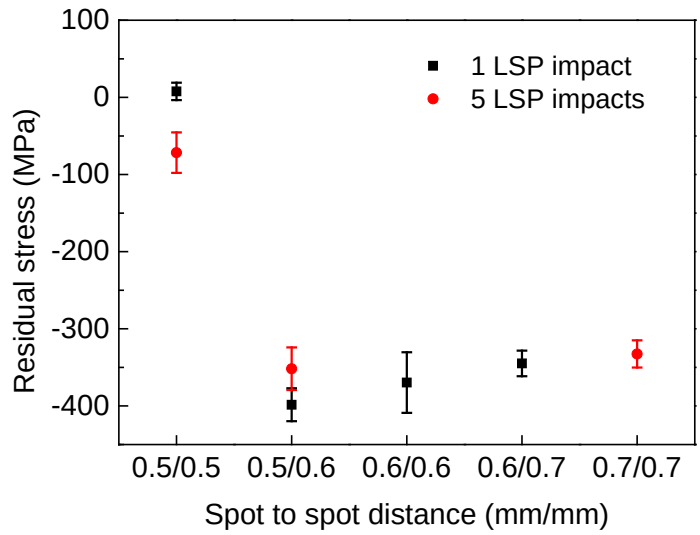

Fig. 7. Surface residual stress of the 1018 CS after laser peening with black tape with different spot-to-spot distances for different impact times (1.4 mm spot diameter, 650 mJ pulse energy, one or five times).

The two laser ablated samples (0.5/0.5, one time, and 0.5/0.5, five times) in Fig. 7 were further investigated for residual stress distribution along the depth. After laser peening one time, as shown in Fig. 8, the residual stress of the sample soon changed from tensile (at the surface) to compressive as the depth increased, peaking at 35 µm depth with -295.5 ± 29.9 MPa and then decreased to 181.5 ± 33.9 MPa at 69 µm and remained stable within 140 µm. On the other hand, after laser peening five times, the compressive residual stress increased rapidly from -71.6 ± 26.3 MPa at the surface to -352.6 ± 39.3 MPa at 20 µm depth and then remained nearly stable within 100 µm. Obviously, the increase of the laser peening times could improve the peak value of the in-depth compressive residual stress. It could also extend the heavily compressive region deeper

into the sample. According to the trend of the two curves, it is also reasonable to say that the laser peening affected depth increases with the increment of the laser peening times.

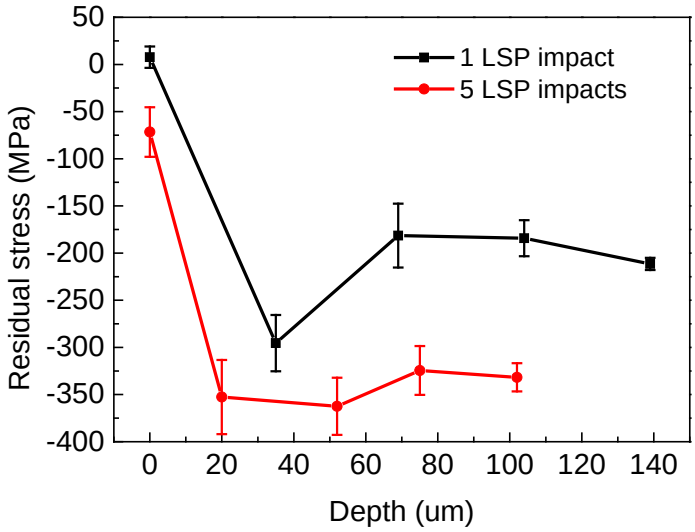

Fig. 8. In-depth residual stress distribution of the 1018 CS after laser peening with black tape for different times (1.4 mm spot diameter, 650 mJ pulse energy, 0.5/0.5 spot-to-spot distance, one or fivetimes).

(c) Small spot LSP without sacrificial coating

Laser peening without a coating was carried out with a laser spot size of 1.4 mm diameter on 1018 CS. As shown in Fig. 8, the residual stress distribution along the depth was investigated on two pieces of samples after LPwC for one time with different spot-to-spot distances of 0.5/0.5 and 0.7/0.7. Take 0.5/0.5, for example, it means a 0.5 mm spot-to-spot distance along the vertical direction and 0.5 mm along the horizontal direction. With the larger spot-to spot-distance of 0.7/0.7, the residual stress of the sample increased to -232.4 ± 9.6 MPa at 60 µm depth and then remained stable within 170 µm. On the other hand, with the smaller spot-to-spot distance of 0.5/0.5, the residual stress of the sample increased to -302.3 ± 24.6 MPa at 40 µm depth and then remained stable within 125 µm. It was observed that the decrease of the spot-to-spot distance resulted in the increment of the peak residual stress, while the laser peening affected depth also increased according to the trend of the curve.

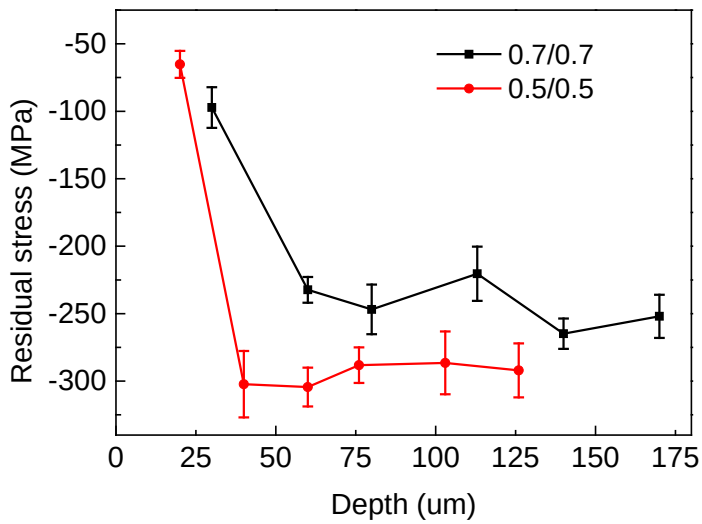

Fig. 9. In-depth residual stress distribution of the 1018 CS after LPwC with different spot-to-spot distances (1.4 mm spot diameter, 650 mJ pulse energy, 0.5/0.5 or 0.7/0.7 spot-to-spot distance, one time).

By comparing Figs. 8 and 9, the effect of the sacrificial coating (100 µm black tape) is revealed. With the same parameters (650 mJ, 0.5/0.5 spot distance, one LSP impact), the peak residual stress values along the depth were almost the same no matter whether the black tape was used or not, while the peak compressive residual stress remained stable in depth and could extend to deeper regions if the black tape was not employed.

3.2.3. Metallographic investigation on the 1018 CS surface

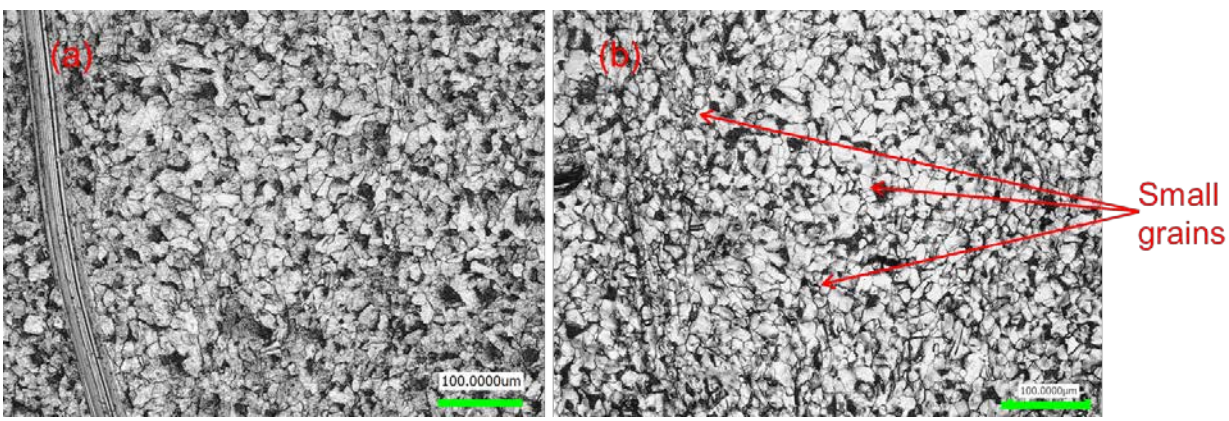

Fig. 10. Grain structure on the 1018 CS surface (a) before and (b) after laser peening with black tape (1.4 mm spot diameter, 650 mJ pulse energy, 0.5/0.6 spot-to-spot distance, ten times).

The microstructure variation on the 1018 CS surface due to the LSP with 100 µm black tape as the sacrificial coating (1.4 mm spot diameter, 650 mJ pulse energy, 0.5/0.6 spot-to-spot distance, ten times) was investigated by metallographic imaging (Sec. 3.1.3). Figs. 10a and 10b show the two photos captured in the same region of the sample surface before and after laser peening, respectively. Many small grains were observed after laser peening (Fig. 10b), while the grain

size before LSP was much larger (Fig. 10a). The grain refinement due to LSP could further increase the resistance of the CS to stress corrosion cracking besides the generation of the compressive residual stress, which was due to the much more complicated grain boundaries with smaller grains.

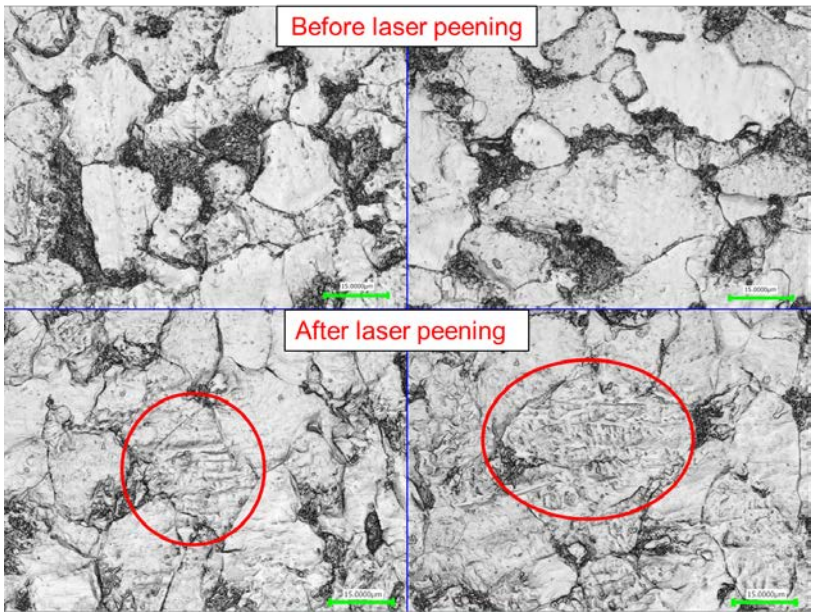

Fig. 11. Microstructure inside the grains on the 1018 carbon steel surface before and after laser peening with black tape (1.4 mm spot diameter, 650 mJ pulse energy, 0.5/0.6 spot-to-spot distance, ten times).

The structures inside the grains were also investigated by metallographic imaging. As shown in Fig. 11, serious slip bands were observed inside some of the grains after laser peening, which could barely be found before laser peening. The generation of extra slip bands reflects the existence of high compressive residual stress.

4. Key outcomes

During this reporting period, an laser peening system with a low energy DRL650 Nd:YAG laser was established. The optimization of the laser peening parameters was conducted, including the laser spot size after focusing, the sacrificial coating, the sample position, the laser peening times, and the spot-to-spot distance. The parameters were examined by the surface morphology, residual stress, and metallographic investigations. Some important conclusions have been summarized as follows:

(1) With a large laser spot size of 2.2 mm, the single spot laser-peening-generated dents are shallower than those obtained with a small laser spot size of 1.4 mm, while the suitable working scale is much larger (100 mm).

(2) The use of the sacrificial coating avoids the ablation damage on the sample and results in a

compressive residual stress on the surface. If the sacrificial coating is not used (LPwC) or is removed due to laser ablation, the compressive residual stress on the sample surface obviously decreases and might even change to a tensile stress. However, the residual stress turns to highly compressive as the depth increases, which is preferable for the increase of the SCC resistance. In addition, the LPwC-induced residual stress remains in a deeper region compared to that obtained after LSP with a sacrificial coating.

(3) The increase in the laser peening times and the decrease in the spot-to-spot distance both lead to the increase in the peak residual stress value and the laser peening affected depth.

(4) After LSP, grain refinement is observed at the same region, while slip bands can be observed in some of the grains on the sample surface.

Our previous results have shown great improvement of mechanical property, residual stress, microstructure, and SCC resistance of the stainless steel and CS after the LSP with a lab-based high energy Nd:YAG laser. In the next step, based on our results obtained during this period, comparable laser peening effects could be achieved with the compact, low energy Nd:YAG laser, which is promising for portable laser peening equipment in the future.

(b) Description of any problems/challenges

No problems were experienced during this reporting period.

(c) Planned Activities for the next quarter

The specifically planned objectives and activities to be accomplished during the next period (June 30, 2016, to September 30, 2016) are listed as follows.

Table 3. The planned objectives and activities in the next quarter.

Objectives	Activities
(a) Final verification of peening effects using lower energy Nd:YAG laser.	(1) Further verification of the corrosion resistance of laser-peened CS. (2) Further investigation of the corrosion resistance mechanism of peened CS using electron microscopy.
(b) Final optimization of laser cleaning process.	(1) Laser cleaning experiment using Nd:YAG laser. (2) Further optimization of laser cleaning process with microphone monitoring. (3) Design of laser cleaning head and integration.
(c) Configuration of the laser peening equipment	(1) Selection of suitable laser and relevant accessories. (2) Design of lab-based pipeline laser peening equipment.