CAAP Quarterly Report

Date of Report: January 10, 2015

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

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For quarterly period ending: December 30, 2015

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 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	20
	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	80
Phase 3	1) Study surface morphology of the laser-peened surfaces.	01/01/2015	09/30/2016	70
	2) Determine residual stress of pipeline steels after laser cleaning and peening.	01/01/2015	09/30/2016	60
	3) Study corrosion resistance of laser-peened surface.	01/01/2015	09/30/2016	20
Phase 4	Design a laser peening prototype system.	06/30/2016	09/30/2016	20

2. Specific Objectives During This Reporting Period

During this reporting period of the project (October 1, 2015, to December 31, 2015), preliminary laser cleaning experiments was carried out with high power nanosecond (ns) fiber laser. In addition, the experimental setup for Nd:YAG laser peening was modified for effectiveness and convenience. Single spot and large area laser peening experiments were performed to show the improvement. The metallographic investigation and improved residual stress measurement of carbon steel were also implemented. The objectives and specific content are as follows:

(1) Preliminary laser cleaning experiments with a high-power ns fiber laser

(2) Setup improvement and parameter optimization for Nd:YAG laser peening experiments.

(3) Metallographic investigation and improved residual stress measurement of the carbon steel.

3. *Significant Results*

3.1 *Experimental setup and procedure*

3.1.1 Laser cleaning setup with high power ns fiber laser

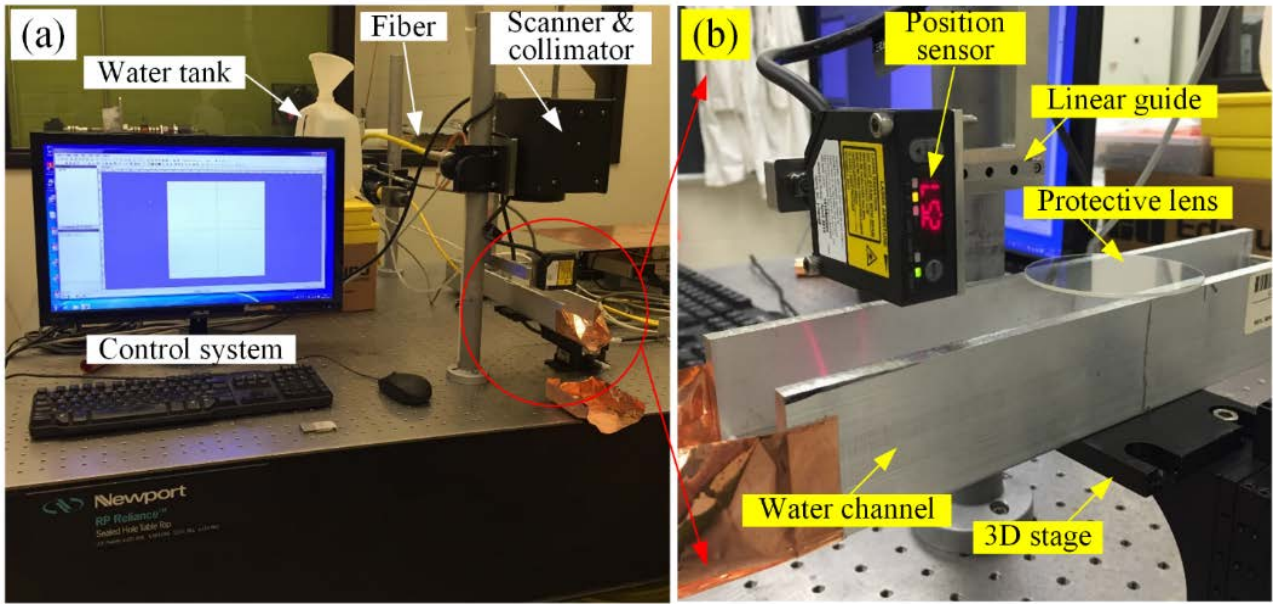

Fig. 1. The laser cleaning system established using a high power ns fiber laser.

A laser cleaning system was set up using the high power fiber laser, as shown in Fig. 1. The high power fiber laser used in this reporting period is a prototype multi-modal laser with a maximum pulse energy of 50 mJ (loan from IPG, main parameters shown in Table 2).

This system includes a computer to control the fiber laser and a Galvano scanner to carry out experiments (Fig. 1a). A 3D stage with an accuracy of 25 μm was used to adjust the sample position; A highly precise laser position sensor with an accuracy of 1.5 μm was used to measure the relative displacement of the samples; The flowing water in the water channel was introduced to wash away the contamination particles; A protective glass was used to isolate the smoke to prevent the contamination from reaching the optical systems (Fig. 1b).

The sample selected for the fiber laser cleaning experiments was an aluminum-magnesium (Al-Mg) alloy plate but not stainless steel (SS) because it is easy to be affected and damaged by laser cleaning, so a better material protection of stainless steel can be achieved. The Al-Mg alloy plate was cut into rectangular shapes with dimensions of approximately 50×25 × 6 mm (length×width×thickness). Prior to the laser cleaning treatment, the samples were polished mechanically with sandpaper of different grades of roughness to achieve a mirror-like surface. 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 laser cleaning

experiments. The mixture of black paint, rubber coating, and high temperature resistance coating were used as contaminative layer on surfaces for the laser cleaning experiments.

Table 2. The processing parameters used in the fiber laser cealning experiment.

Laser system	IPG YLP-HP-50-100-10-500 ns fiber laser; wavelength: 1064 nm; pulse energy: 15-50 mJ; pulse duration: 25, 40, 60, 100, and 200 ns; frequency: 2-50 kHz;
Confining medium	Flowing distilled water with a layer thickness of approximately 1-2 mm.
Contaminative layer	Black paint

3.1.2 Improved Nd:YAG laser peening experimental setup

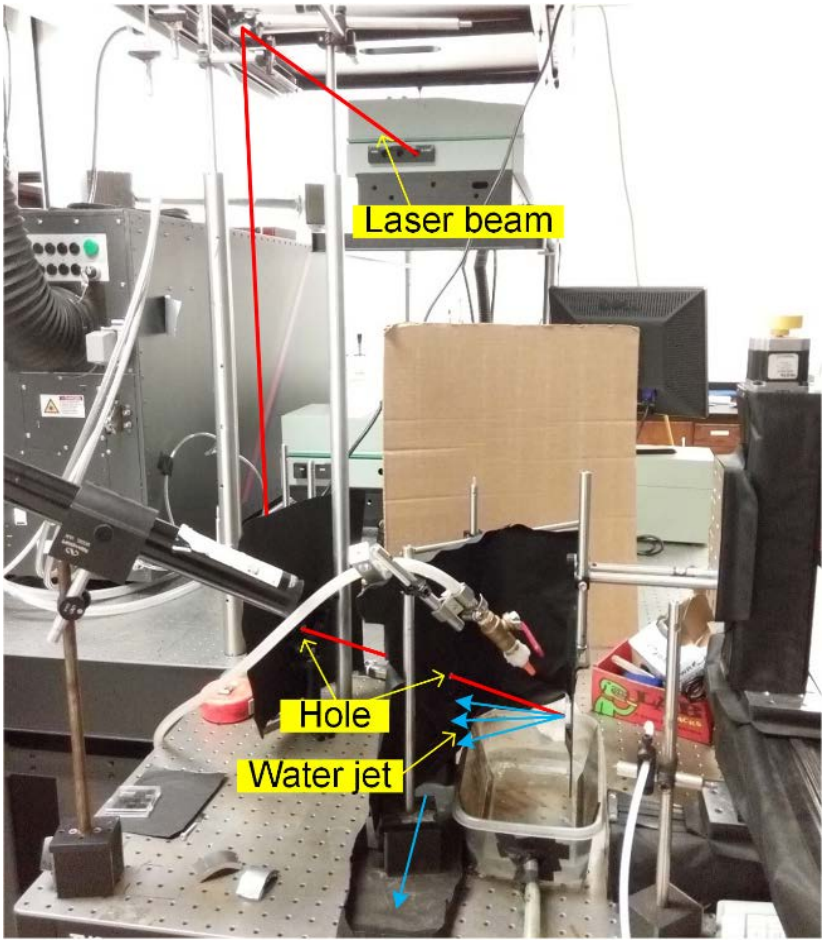

Fig. 2. Improved Nd:YAG laser peening system

With our previous Nd:YAG laser peening setup, serious water jet and particles generated in the laser peening process tend to contaminate the optical system and shield subsequent laser pulse energy. Therefore, the optical setup was modified for higher effectiveness and convenience in

this period (Fig. 2). By introducing a small tilt angle to irradiate the laser beam and adding two fenders with small holes, contamination particles and water jet were prevented from reaching the reflective mirrors, which were both generated during the laser peening process. In such a way, the protective glass and compressed air were no longer needed, reducing the loss of laser energy and increasing/stabilizing the laser peening effect, which is also beneficial for compact laser peening system design. In addition, the laser repetition rate was reduced from 10 to 5 Hz to avoid the incident laser from being seriously disturbed by the water spray induced by previous laser impact. Moreover, the sacrificial layer for Nd:YAG laser peening process was changed from black tape with 177 μm thickness to black tape (100 μm) or black paint (50 μm) with smaller thickness, which reduces the shock wave attenuation in the sacrificial layer and enables better peening effect. For these experiments, Al-Mg alloy (50×25×6 mm) was also selected for the parameter optimization because it could show the peening effect obviously. The laser source is a continuum Q-switched Nd:YAG PR II 8010 laser. Laser pulse with wavelength and energy of 1064 nm and 850 mJ were used. The focus position was carefully optimized by comparing morphology changes on the Al-Mg alloy sample surface after laser peening. On the other hand, carbon steel (12×12×3 mm) was laser-peened with improved Nd:YAG laser peening setup. The residual stress was measured along depth as well as the metallographic imaging.

3.1.3 Characterization

3D profile meter (New View 8000, Zygo Corporation) was used to characterize the sample morphologies after laser peening for optimizing the single spot and large-area laser peening parameters. The micromechanical properties of the samples were carried out using Tukon 2500 Vickers micro-hardness tester with a square-based diamond pyramid indenter. Surface residual stress measurements were conducted by X-ray diffraction (XRD) equipment (Smartlab, Rigaku) with Cu as the anode target and monochromator for signal acquirement. In order to obtain the in-depth residual stress distribution, electro-chemical polishing was used to remove thin layers from the sample surfaces with certain thickness. Nital consisting of 4% HNO_3 and 96% ethanol was used as the etchant for metallographic imaging of carbon steel with optical microscopy for image capture.

3.2 Results and discussion

3.2.1 Laser cleaning experiment using the high power ns fiber laser

The high power fiber laser with Galvano scanner was considered efficient for laser cleaning. Therefore, several laser cleaning experiments were carried out.

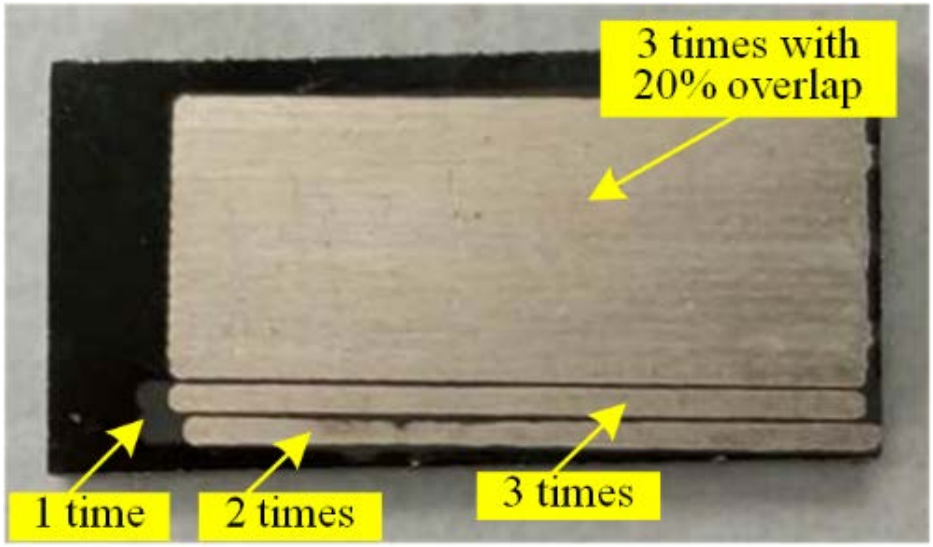

Fig. 3. Laser cleaning experiments using high power fiber laser with different impact times (in air)

After laser scanning at the focal position with a 500 W output power and a 500 mm/s scanning speed for one time, about half depth of the contamination layer was removed, as shown in Fig. 3. During the second scanning, almost all of the contamination layer was removed except some black residual dust inside the gaps or pits on the sample surface. After the third scanning, all contamination layer was removed completely without damaging the Al alloy surface.

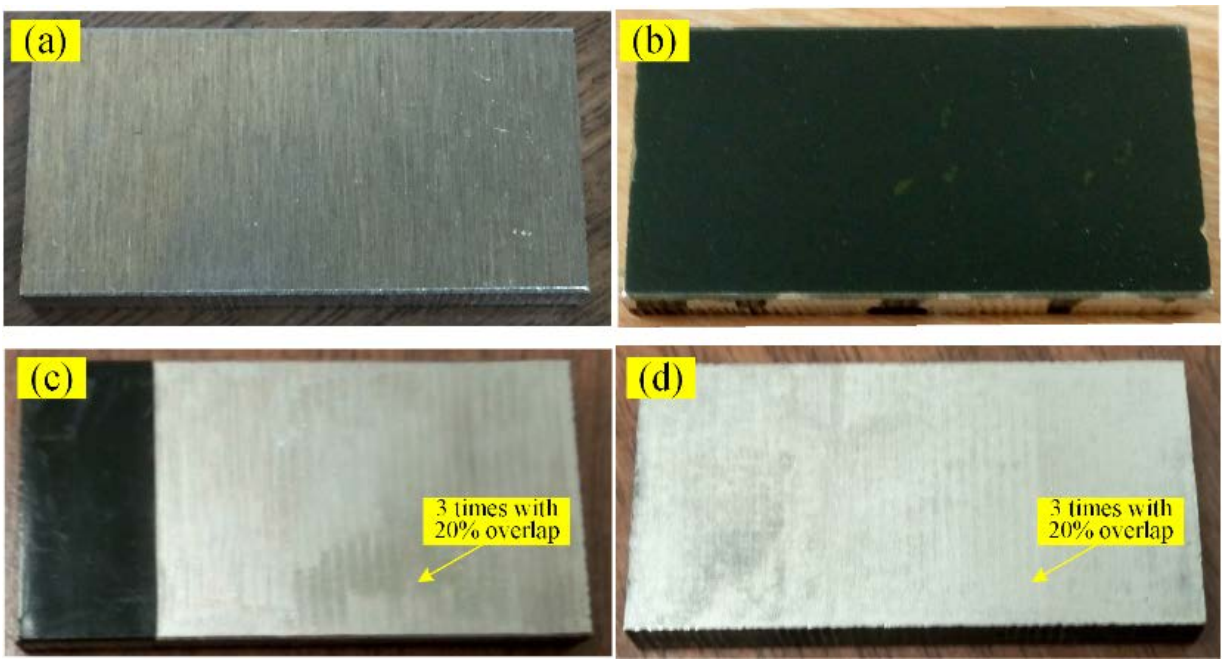

Fig. 4. Laser cleaning of Al alloy (in air) using the high power fiber laser. (a) original surface; (b) surface coated with contamination layer; (c) contamination layer removed partly; (d) contamination layer removed totally.

Fig. 4. shows the large-area cleaning procedure using the high power fiber laser. After 3 times of laser scan, the contaminants on the sample surface were totally removed. Meanwhile, even some oil contamination residuals produced in the fabrication procedure of the Al alloy were also removed. The comparison of the sample surface topography and profile before and after indicates that the Al alloy surface was almost unchanged after laser cleaning except a decrease in the surface roughness, which might be induced by cleaning of the original oil residuals, as shown in Fig. 5. Since Al alloy is easier to be damaged by laser, the laser power should also work for stainless steel and carbon steel without surface damage.

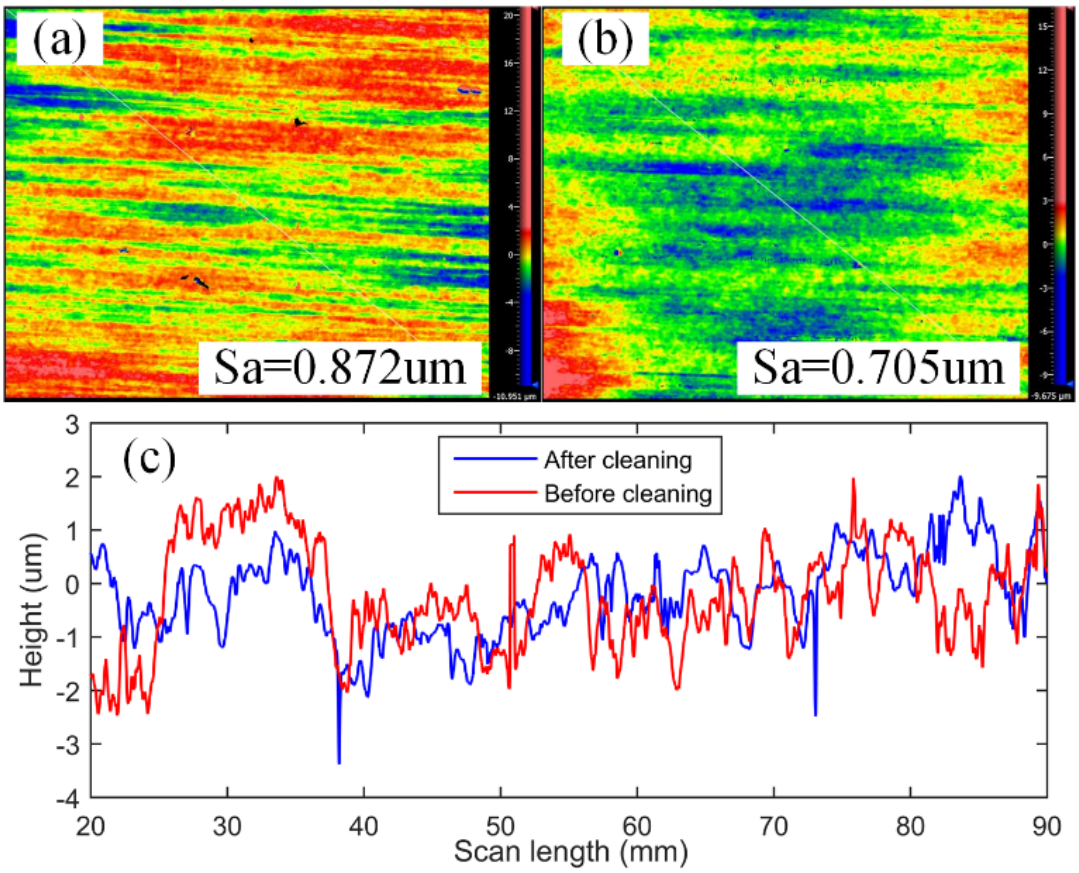

Fig. 5. Al alloy surface before and after laser cleaning: surface topography (a) before laser cleaning; (b) after laser cleaning; (c) comparison of the surface profiles;

3.2.2 Nd:YAG laser peening experiments with improved setup

In order to improve the peening effects, Nd:YAG laser peening experiments were further implemented with modification. Black paint with a thickness of 50 μm was used as the sacrificial layer for single spot laser peening due to its small thickness and strong peening effects, which makes it easy for us to discover the best focus length. Black tape with a thickness of 100 μm was also used as the sacrificial layer for large-area laser peening experiments since black paint would be easily removed in this case. In the single spot experiment, the effect of focal positions on

sample morphology was investigated, while in large-area experiments, surface residual stress was investigated to demonstrate the effect of laser peening impact times.

(A) Single spot Nd:YAG laser peening experiments at different focal positions

The effects of focal position on the Al alloy sample surface were examined in single spot Nd:YAG laser peening experiments with black paint as a sacrificial layer. The surface morphology of the laser peening induced deformation dents were investigated.

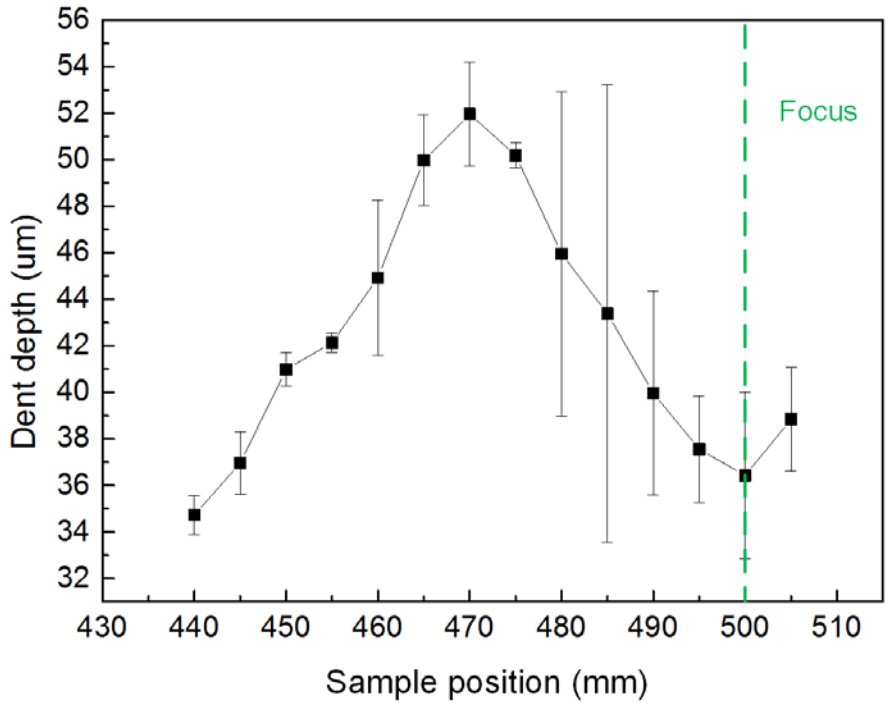

Fig. 6. The dent depth of laser peening on Al alloy surfaces at different sample positions with black paint as the sacrificial layer.

As shown in Figure 6, the laser peening dent depth increased from 36 to 52 μm and then decreased to 35 μm, when the sample position with respect to the lens (focal position) decreased from the focal length (500 mm) to 440 mm. The peak is at the focal position of 470 mm, which is 30 mm shorter compared to the focal length. This phenomenon is attributed to the planar-like shock wave at 470 mm and sphere-like shock wave at 500 mm. Although the shock wave is stronger at the focal point due to the higher laser peak power density, the sphere-like shock wave is easier to be attenuated in the sacrificial layer, while the planer-like wave generated at 470 mm could keep the shock wave intensity at a relatively higher level when arriving at the Al alloy surface. In addition, the planer shock wave itself could deform the sample easily. It should be noted that due to the use of the black paint with a thickness of 50 μm as the sacrificial layer, the laser peening dent depth increases significantly from ~26 to ~52 μm compared to that obtained with the black tape (100 μm thickness), while dent depth was less than 20 μm in our previous

experiments with unimproved setup and procedures. Therefore, the smaller thickness is considered the most important factor for sacrificial layer selection.

The laser peening dent diameter was also measured at different sample positions as shown in Figure 7. The dent diameter keeps increasing as the sample-to-lens distance (focal position) decreases, which is induced by the increase in the laser spot size. According to Figures 6 and 7, the most preferred sample position for our setup is 470 mm, where both of the dent depth and diameter are relatively high.

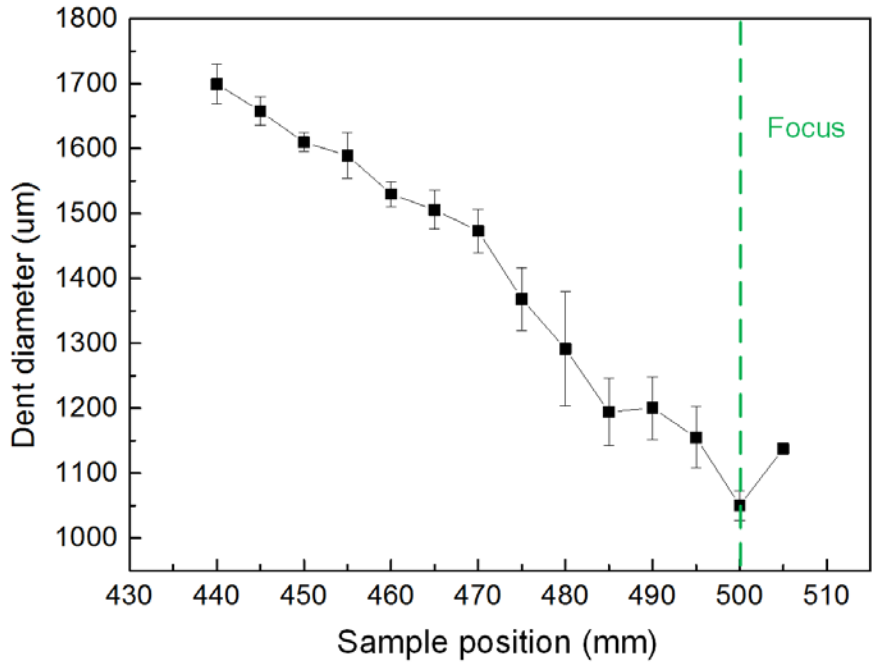

Fig. 7. The laser peening dent diameter on Al alloy surface at different sample positions with the black paint as the sacrificial layer.

(B) Large area Nd:YAG laser peening experiments

Large area Nd:YAG laser peening experiments have been further performed and improved to achieve larger and uniform residual stress. The experimental setup has been adjusted as shown in 3.1.2. with black tape (100 µm) as sacrificial layer. These modifications improved the simplification and effectiveness of the laser peening system, which could be demonstrated using the following results.

With the black tape (100 µm) as the sacrificial layer, several experiments have been performed. The results show that 50% overlapping rate causes the fully removal of the sacrificial layer and damage on the Al alloy surface. Therefore, a new method was employed to obtain high overlapping rate of 50%, in which the samples were scanned twice with a lower overlapping rate (25% along one direction, 50% along the perpendicular direction, durable for the black tape). By replacing the peened black tape and changing the starting point slightly of the second scan, 50%

overlapping rate could be obtained. As shown in Figure 8, 4 samples were processed by laser peening with the procedure described above to obtain the 50% overlapping rate and laser peened for 1, 2, 3, and 4 times, respectively. As we can see, the high overlapping rate results in a uniform surface morphology. The surface residual stress was measured by XRD ($2\theta = 135.7^{o}$) and shown in Figure 8b. The compressive residual stress increases from 246.8 to 270.6 MPa with the increment of the impact times. The residual stress is much higher than the one achieved with the 177 μm black tape, indicating significant improvement of the laser peening effects. The surface hardness was also measured and summarized in Figure 8c, which also increases with the increment of the laser peening impact times but saturates after 3 impacts.

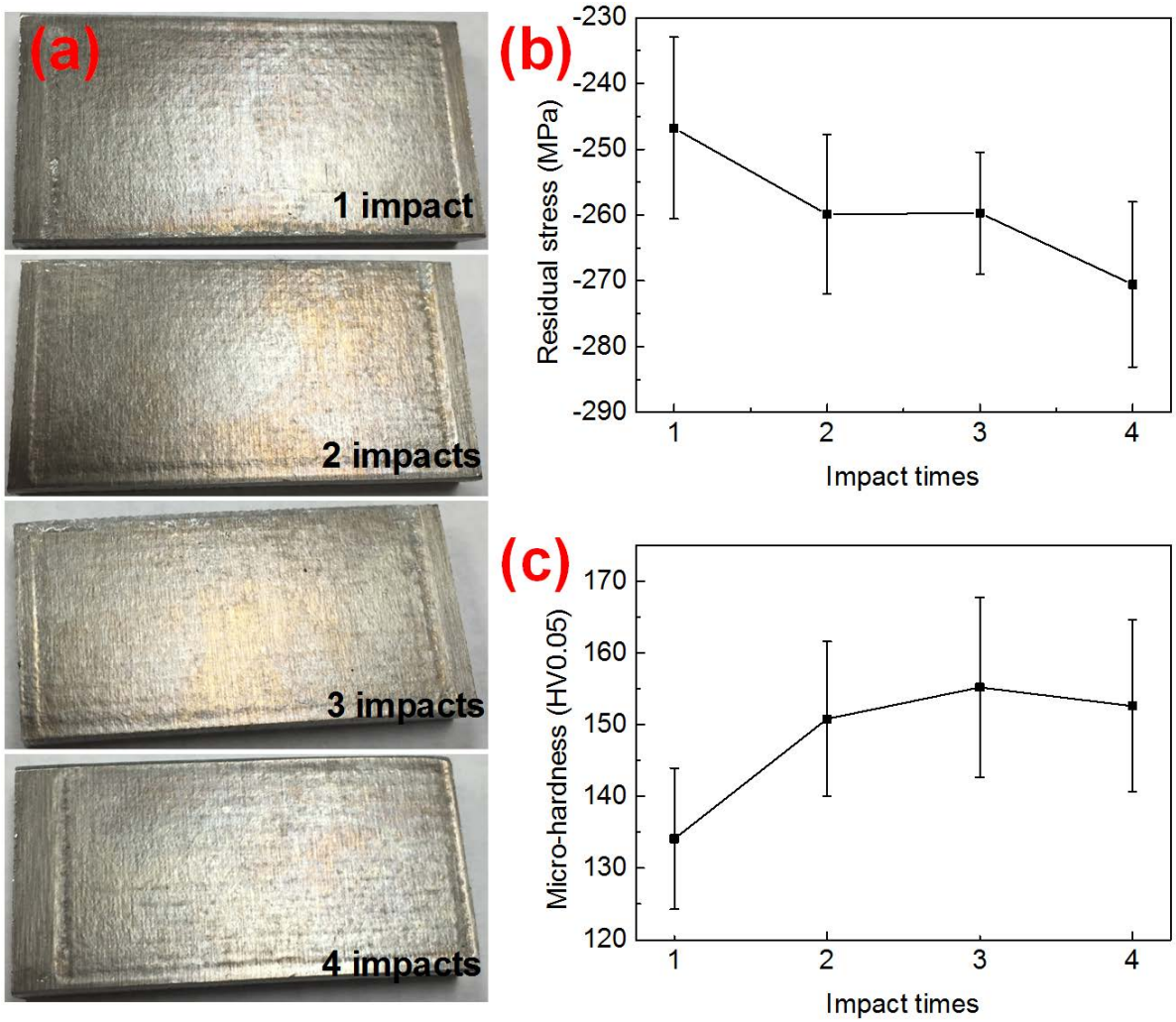

Fig. 8. (a) Al alloy samples after laser peening with 50% overlapping rate, 1-4 impact times, and the black tape (100 μm thickness) as the sacrificial layer; (b) Surface residual stress and (c) micro-hardness as a function of impact times

Both the single spot and the large area laser peening results show better peening effects after the modification of Nd:YAG laser peening setup and procedures since the dent depth, hardness and residual stress of Al alloy samples were improved obviously.

3.2.3 Residual stress measurement and metallographic investigation of the carbon steel

The improved Nd:YAG laser peening setup and procedure were also used for carbon steel processing. The residual stress and metallographic image were measured/captured on the surface and at different depth to show the depth effect of laser peening. Electrochemical polishing was used to remove thin layers from the sample surface without introducing extra residual stress. The metallographic imaging was obtained after etching and captured by optical microscopy. The residual stress was measured by X-ray diffraction ($2\theta = 116.2^{o}$). By using the monochromator, more reliable Smartlab with Cu as the anode could be used to do the residual stress measurement for carbon steel.

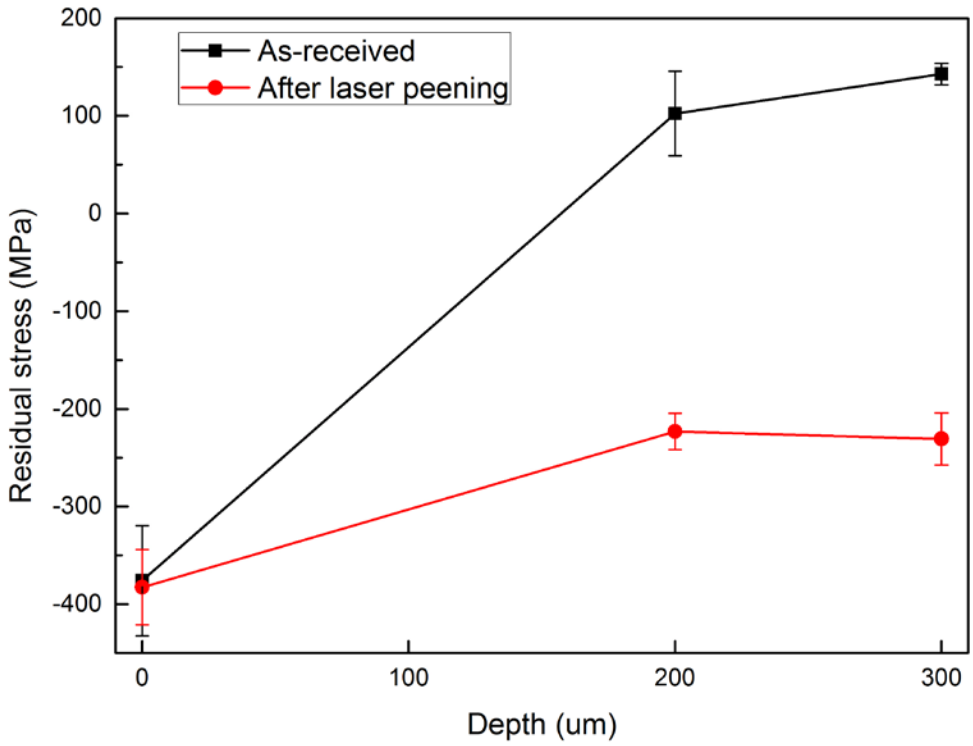

Fig. 9. Residual stress of the as-received carbon steel sample (red) and sample after laser peening (black, 850 mJ, 50% overlap, 100 µm black tape, improved setup) as a function of depth.

Figure 9 shows the residual stress distribution of low carbon steel samples along depth. The red line indicates the sample after laser peening with improved setup/procedure while the black line shows the as-received sample. On the surface of the low carbon steel, similar high compressive residual stress was observed on both of the samples (-376 and -383 MPa), indicating that the samples were surface processed by the manufacturer and the existing surface residual stress is

too high to be further enhanced by laser peening under current conditions. However, after removing a surface layer of 200 µm by electrochemical polishing, the residual stress of the as-received sample changes from compressive (-376 MPa) to tensile (102 MPa), while the sample after laser peening only decreases a little and still remains compressive (from -383 MPa to -223 MPa). It reveals that the process by the manufacturer did not affect the sample under 200 µm depth, while laser peening is still effective at that depth. After removing another 100 µm layer of the carbon steel samples, the residual stress remains almost unchanged. Although the surface with high residual stress is hard to be further improved by laser peening, in depth residual stress improvement could be achieved apparently.

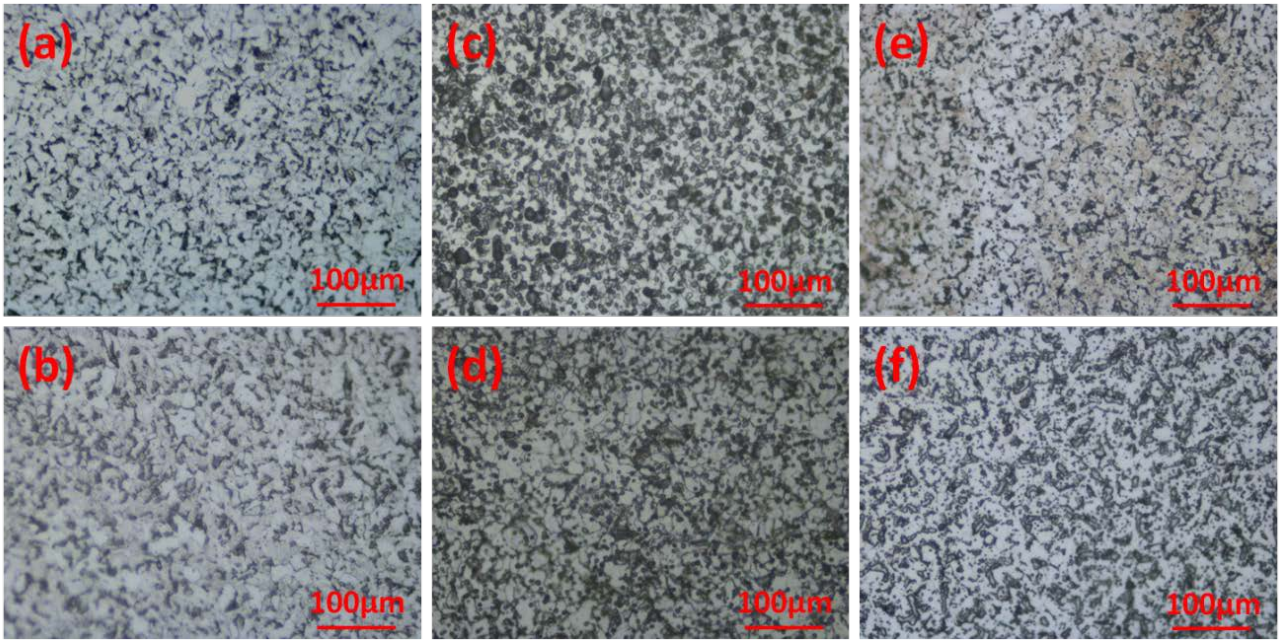

Fig. 10. Metallographic image of low carbon steel by optical microscope. (a) surface, (c) 200 µm depth, (e) 300 µm depth of the as-received sample; (b) surface, (d) 200 µm depth, (f) 300 µm depth of the sample after laser peening.

The corresponding metallographic image of the carbon steel after etching (Nital consisting of 4% HNO_3 and 96% ethanol) is shown in Figure 10. Two phases were observed for all the images with some corrosion pits. The grain size change is tiny and difficult to recognize. Thus, the residual stress difference is most likely to be induced by defects generated inside independent grains, which need to be further observed by TEM.

4. Key Outcomes

In this reporting period, preliminary laser cleaning experiments was performed by high power fiber laser. The Nd:YAG laser peening setup and procedure were modified with single spot/large area experiment performed to show the improved effectiveness. With the improved setup, carbon

steel was laser-peened with residual stress and metallographic image measured and captured. Some important conclusions have been summarized as follows:

(1) The high power fiber laser performed perfectly in the preliminary laser cleaning experiments, fully removed the mixed contamination layer consisting of black paint, rubber coating, high temperature resistance coating, and some oil residual on Al alloy surfaces with 1.7 mm spot diameter, 500 W output power, 500 mm/s scanning speed, 20% overlapping rate, and 3 scanning times. The optical image and morphology after laser cleaning show that no surface damage was generated.

(2) The setup and procedure of Nd:YAG laser peening were improved for convenience and effectiveness. In the single-spot laser peening experiments with black paint as sacrificial layer, the optimized focal position was determined as 470 mm. Due to the use of 50 μm black paint as the sacrificial layer, the laser peening dent depth increases significantly to ~52 μm. In large-area laser peening experiments, with black tape (100 μm) as the sacrificial layer and 50% overlapping rate achieved by new scanning method, the residual stress on Al alloy surfaces reaches 246.8 MPa with 1 impact and further increases with more impact times.

(3) Carbon steel samples were processed with improved Nd:YAG laser peening setup and procedure. The surface residual stress does not increase due to the existing high residual stress, while the in-depth residual stress increases obviously. The grain size at different depth before and after laser peening does not change obviously, indicating the existing of the in-grain defects.

Description of any problems/challenges

No problem was experienced during this reporting period.

Planned Activities for the next quarter

The specifically planned objectives and activities to be accomplished during the next period (Jan 1, 2016, to March 31, 2016) are listed as follows.

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

Objectives	Activities
(a) The corrosion resistance test of laser-peened samples.	(1) Optimize corrosion solution and testing conditions (2) Optimization of Nd:YAG/fiber laser peening processing parameters of stainless steel and carbon steel samples for the corrosion test;
(b) Surface evaluation of laser-peened samples.	(1) Residual stress measurement of stainless and carbon steel on the surf and along depth; (2) To further investigate the microstructure mechanism of LSP impacts stainless steel and carbon steel by means of cross-sectional opti microscopy, electron backscatter diffraction (EBSD), and transmiss electron microscopy observation.

(c) Laser cleaning parameter window and mechanism analysis	(1) Nd:YAG and fiber laser will be used to clean oil and sand on stainl steel and carbon steel surface using different processing parameters; used study laser cleaning process by measuring the spectroscopy during plas development and expansion. (2) Laser-induced breakdown spectroscopy (LIBS) technique will be used.