CHARACTERIZATION OF LASER-PEENED MATERIALS BY SYNCHROTRON RADIATION AND NEUTRON DIFFRACTION TECHNIQUES

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ABSTRACT
The effect of low energy laser peening (LP) without coating has been studied on metallic materials. A highly penetrable neutron beam was used to evaluate the overall distribution of residual stress. Compression in the top surface and its thermal stability were non-destructively confirmed through precise experiments with synchrotron radiation. The threedimensional (3D) image of fatigue cracks was clearly reconstructed by micro tomography (μCT) with phase contrast technique. It was shown that the growth of the cracks was retarded for the laser-peened materials.

KEY WORDS
Laser peening, Synchrotron radiation, Neutron, Diffraction, Residual stress, Micro tomography, Imaging, Fatigue crack

INTRODUCTION
Laser peening (LP) is a surface enhancement technology, which introduces compressive residual stress by irradiating intense laser pulses to metallic material in an aqueous environment [1-6]. The effect of LP is far superior to any other method such as shot peening, namely the depth of the compressive field imparted, which is essential to prevent the growth of cracks due to fatigue and stress corrosion [7]. LP has been utilized for U.S. jet fighters [8,9] to boost the tolerance against foreign object damage (FOD) of engine fan blades since 2002. However, the further application is limited due to the costly initial investment for custom-made high-power Nd:glass laser system.

A new process of LP was invented around a decade ago, which employs laser pulses with much lower energy from a compact and commercially available Nd:YAG laser [10]. The process does not require any surface coating (or so-called sacrificial overlay), which is formed on the surface prior to laser irradiation in order to protect the surface from melting in the conventional LP process [11-13]. Thanks to the lower pulse energy, the new process can utilize optical fiber to deliver laser pulses [14-16], which drastically improved the accessibility to three-dimensional objects [17]. Since 1999, the low energy LP process without coating has been used to prevent stress corrosion cracking (SCC) of aged nuclear power reactors in Japan [18,19].

The effects of LP without coating to enhance fatigue strength and to reduce SCC susceptibility have been demonstrated through various experiments [7,20-27], however, the mechanism to impart the compression and tactics to optimize the process are not fully elucidated yet. The authors have attempted to understand the fundamentals of LP through numerical simulation [28,29] and non-traditional experiments [30-33] with synchrotron radiation in SPring-8 and a neutron beam in JRR-3. In the present paper, we describe the recent advances in the characterization of laser-peened materials obtained in these state-of-the-art facilities, which could not be attained in conventional ones and provide indispensable information to understand the process and effect of LP.

LASER PEENING WITHOUT COATING
Basic Process
The process of the low energy LP without coating is illustrated in Fig.1. When an intense laser pulse with duration of several nanoseconds is focused on a metallic material through a water film, the top surface evaporates instantaneously through ablative interaction with the laser pulse. The water confines the evaporating material, and the resulting high density metal vapor is immediately ionized to form plasma by inverse bremsstrahlung. Subsequent laser energy absorption in the plasma generates a heat-sustained shock wave, which impinges on the material with an intensity of several gigapascals [34,35], far exceeding the yield strength of metals. The shock wave loses energy as it propagates to create a permanent strain [28,29]. After the propagation, the strained region is elastically constrained to form compressive residual stress on the surface.

In comparison with the conventional LP process, the newly developed process is characterized by lower pulse energy and smaller spot diameter in order to eliminate the coating required in the conventional process.

Experimental Procedure
The experimental setup of the low energy LP without coating is sketched in Fig.2. Near infrared light (λ = 1.06μm) of a Q-switched Nd:YAG laser is frequency-doubled to water-penetrable green light (λ = 532nm) by a
second harmonic generator (SHG). Laser pulses are delivered through optical fiber to a pencil-type irradiation head and focused by a concave mirror on the inside wall of a tube-shaped sample, as shown in Fig. 3. Pure water is continuously supplied to the surface of the wall, which sweeps the ablation products generated on the surface in order to minimize the scattering loss of the laser pulses. The sample is set on a turntable and the optical head is vertically driven by a linear stage so as to irradiate laser pulses on the inside wall spirally [36].

![Fig. 2 Experimental setup of low energy LP](image)

**Fig. 2 Experimental setup of low energy LP**

**RESIDUAL STRESS DEPTH PROFILE**

Non-destructive residual stress measurement was attempted for the tube samples with a highly penetrable neutron beam, since X-ray diffraction cannot be applied to the inside wall of small-diameter tubing without cutting out that may affect the distribution of residual stress.

**Sample Preparation**

The center region along the axis of an extruded carbon tool steel (JIS SK3) rod was drilled to make tube-shaped samples of 150mm long with 17mm outer and 9.5mm inner diameters. The wall thickness is 3.75mm. The middle part of 50mm long was laser-peened, as shown in Fig. 3, under the condition of 70mJ pulse energy, 0.7mm spot diameter, 100pulses/mm² irradiation density and 3800% coverage.

**Neutron Diffraction Experiment**

Lattice strain was measured with a neutron diffractometer in JRR-3 (Japan Research Reactor-3) of JAEA (Japan Atomic Energy Agency) as shown in Fig. 4. Strictly saying, strains in three directions are necessary for deducing the residual stress. In the present study, however, the strain measurement was made in circumferential (θ) direction throughout the pile wall and partially made for the radial (r) direction in order to confirm the trend of the residual stress, due to the limitation of available machine time.

![Fig. 4 Strain measurement by neutron diffraction in JRR-3](image)

**Fig. 4 Strain measurement by neutron diffraction in JRR-3**

The neutron beam was incident on the tube sample from the upper-right on Fig. 4. The neutrons diffracted by an α-Fe 211 plane were detected by a $^3$He counter. The incident and diffracted beams were collimated by cadmium (Cd) slits with a width of 0.3mm and a height of 15mm. The diffraction angle (2θ) was around 124.6deg. **Figure 5** shows the relation among the incident beam, diffracted beam and the gage volume for the strain measurement in the θ-direction ($\varepsilon_\theta$). The strain distribution over the tube thickness was obtained by alternately repeating the diffraction measurement and the positioning of the sample.

![Fig. 5 Gage volume in neutron diffraction](image)

**Fig. 5 Gage volume in neutron diffraction**

**Residual Strain and Stress**

**Figure 6** shows the distribution of the strain in the θ-direction ($\varepsilon_\theta$) over the tube thickness of the laser-peened and unpeened samples. LP introduced compressive strain to the depth of about 1mm from the inner surface. To the contrary, the strain in the outer side tended to become tensile so as to compensate the compressive strain. For the peened sample, the radial strain ($\varepsilon_r$) was also measured at the distances of 0.4mm and 1.9mm from the inner surface. By assuming the plane stress, it can be concluded that the residual stress near the inner surface is compressive due to
laser peening, whereas those in the center and outer regions are tensile [32].

Fig.6 Lattice strain of laser-peened and unpeened SK3 tubes with inner diameter of 9.5mm

RESIDUAL STRESS PROFILE IN TOP SURFACE
In the previous section, we have demonstrated that the low energy LP can introduce the compression up to 1mm from the surface. However, it still remains as an open question how the top surface could become compressive despite the possible thermal effect of the intense laser pulses directly irradiated to the surface without coating [11-13].

This section is devoted to give evidence showing that the surface after LP without coating is compressive and the residual stress imparted is stable under thermal loading. Flat samples were used in this experiment to determine the depth from surface accurately.

Sin²ψ Method
A preliminary experiment with usual sin²ψ method was conducted at BL19B2 of SPring-8 to compare the results with that of constant penetration depth (CPD) method. As shown in Figs.7 and 8, X-ray was collimated by a four-quadrant slit and irradiated to a high tensile strength steel (JIS SHY685) sample set on a four-circle goniometer. X-ray energy was adjusted to be 25keV with a silicon double crystal monochromator. Diffraction X-ray of an α-Fe 521 plane was detected by a scintillation counter via a soller slit. The scattering angle (2θ) was around 56.3deg. The maximum penetration depth of X-ray with 25keV energy was 22μm in this geometry.

Fig.7 Experimental scheme of CPD method

Figure 9 shows an example of so-called sin²ψ diagram obtained by a conventional ψ-goniometer method for a sample with a huge stress gradient. Because the residual stress is proportional to the slope of the diagram, the result is strongly affected by the range of ψ in the measurement.

Constant Penetration Depth Method
The constant penetration depth (CPD) method can provide relevant information on the surface residual stress, because the method controls the X-ray penetration depth constant during a series of exposure with various ψ angles [33]. Using this method with high energy and high brilliance X-ray of SPring-8, the residual stress depth profile in the top surface can be precisely evaluated non-destructively.

Sin²ψ diagrams obtained by the CPD method are shown in Fig.10 for the same sample as used in the conventional method in Fig.9. There is no ambiguity to deduce the residual stress since the data for each penetration depth make an almost straight line.

Fig.8 Experimental setup of CPD method in SPring-8

Fig.9 Conventional sin²ψ diagram for a sample with stress gradient on surface

Fig.10 Sin²ψ diagram by CPD method
Residual Stress Depth Profile
The CPD method was used to measure the residual stress depth profile of 20% cold-worked austenitic stainless steel (JIS SUS304). Low energy LP was applied to the sample without coating. The LP condition was 60mJ pulse energy, 0.7mm spot diameter, 70 pulses/mm² irradiation density and 2700% coverage.

The depth profile was measured non-destructively by detecting the diffracted X-ray from a γ-Fe 422 plane. The diffracted angle (2θ) was around 39.4deg. The sample was covered with a Kapton (polyimide) dome and heated up to 562K (289°C) and then 673K (400°C) with an electric heater. The sample was held at each temperature for one hour to attain the thermal equilibrium.

The profiles before, during and after the heat treatment are shown in Fig.11. The horizontal axis means the X-ray penetration depth from the surface of the material (SUS304). It is evident that the top surface is entirely compressive despite the direct irradiation of laser pulses to the sample without coating. The thermal loading up to 673K somewhat reduced the residual stress; however, the overall distribution was quite stable. This kind of in-situ measurement using the same sample could not be realized with the CPD method combined with high energy and brilliant X-ray of SPring-8.

![Residual stress depth profile of 20% cold-worked SUS304 laser-peened without coating](image)

Fig.11 Residual stress depth profile of 20% cold-worked SUS304 laser-peened without coating

3D VISUALIZATION OF FATIGUE CRACKS
The effect of the low energy LP without coating on impeding the propagation of fatigue cracks was evaluated by replication method and micro tomography (µCT) using synchrotron radiation of SPring-8 [37]. Taking advantage of highly parallelized brilliant X-ray with small divergence, a phase contrast technique was applied to obtain a clearer image of the cracks [38,39].

Sample Preparation
Test samples were prepared from an aluminum-silicon-magnesium cast alloy, JIS AC4CH. Impurities and defects in the bulk material were reduced by degassing in the casting process [22,40]. T6 heat treatment was applied to the cast blocks before machining to the samples. Figure 12 depicts the shape and dimensions of the fatigue samples. Microstructure is a typical dendrite with aluminum matrix and eutectoid silicon particles.

![AC4CH sample shape and dimensions](image)

Fig.12 AC4CH sample shape and dimensions

A small drill hole with a diameter of 0.3mm and a depth of 0.3–0.5mm was made at the center of the notched area of each sample, to initiate a fine crack from the drill hole. Then, a pre-crack with a length of about 2.5mm on the surface was introduced by rotating-bending fatigue with stress amplitude of 130MPa and frequency of 2760rpm in the ambient condition. LP without coating was applied to some samples with the conditions of 100mJ pulse energy, 0.6mm spot diameter, 273 pulses/mm² irradiation density and 770% coverage. Then, all samples were again subjected to rotating-bending fatigue loading of about 10^7 cycles.

Figure 13 shows the propagation behavior of main surface cracks under the stress amplitude of 130MPa [41]. The crack length was measured by replication technique. Each curve was shifted in a manner where the number of cycles at the crack length of 2.5mm became the origin of the horizontal axis. The cracks propagated acceleratingly in the unpeened samples. To the contrary, on the laser-peened samples, the cracks did not propagate during the additional loading of about 10^7 cycles [41].

![Fatigue crack growth behavior of AC4CH](image)

Fig.13 Fatigue crack growth behavior of AC4CH

Micro Tomography of Fatigue Cracks
The cracks in the fatigue samples were imaged by a µCT technique in SPring-8, as schematically shown in Fig.14. The X-ray energy was adjusted to be 28keV, considering the sample thickness and the contrast of the image.
The distance between the sample and the area detector (cooling X-ray CCD camera combined with scintillator and relay lens) was set for 800 mm to obtain phase contrast effect, which would enhance the edge of the cracks. Projection data of 1024 by 1024 was recorded every 0.5 degree from 0 to 180 degrees. The effective pixel size of the CCD camera including the magnification of the optics was about 6 μm. Slice images were reconstructed by an algorithm of filtered-back projection.

Fig. 14 Experimental setup for 3D crack imaging

Reconstructed three-dimensional (3D) images of the unpeened and laser-peened samples are shown in the left and right of Fig. 15, respectively. The shadows depicted in white might correspond to the opening of the cracks. The upper are the images viewed from the direction of the sample axis, while the lower are from the direction perpendicular to the axis. The initial drill holes can be identified at the center of the shadows.

The images well agree with the surface observation results by a microscope with replication technique (Fig. 13) and suggest that the crack growth is impeded for the laser-peened sample not only on the surface but also towards inside [32, 41], which could not be observed without highly-parallelized brilliant X-ray of SPring-8.

Fig. 15 3D image of fatigue crack in AC4CH samples after additional $10^5$ fatigue cycles

CONCLUSION

The effects of the low energy laser peening (LP) without coating upon the residual stress and crack growth in metallic materials were studied by means of neutron and synchrotron radiation. For the residual stress, the overall distribution in materials was measured with the highly penetrable neutron beam of JRR-3. The depth profile in the top surface was examined with high-energy brilliant X-ray of SPring-8 by strictly controlling the penetration depth to the materials. Fatigue crack growth behavior was also evaluated by micro tomographic (μCT) imaging at SPring-8, which emphasized the crack tip with phase contrast effect.

The results obtained are itemized as follows:

1. The distribution of residual stress through the wall of the tube samples was measured non-destructively with the neutron beam. The result showed that the compression was imparted by low energy LP without coating to about 1 mm depth from the inside wall of the SK3 tube sample with the inner diameter of 9.5 mm. Because the wall thickness is not sufficient, the change in the residual stress toward tension was observed on the opposite side so as to compensate the compression due to LP.

2. The compression of several hundred MPa was built in the top surface of the SUS304 sample after laser peening without coating, in spite of the direct irradiation of intense laser pulses to the surface. The absolute value of the residual stress imparted was somewhat decreased by the thermal loading, however, the distribution was still stable up to 673 K (400°C). These results were obtained for the first time using the constant penetration depth (CPD) method that strictly controlled the penetration depth of X-ray, in conjunction with the high energy and highly brilliant X-ray of SPring-8.

3. The effect of the low energy LP without coating on impeding the fatigue crack growth in cast aluminum alloy (AC4CH) was confirmed through the surface observation by microscopy with replication technique. Fatigue cracks on AC4CH were successfully visualized by micro tomography (μCT) with 28 keV X-ray of SPring-8. The retardation of the crack growth was confirmed for the laser-peened material not only in the surface direction but also towards inside.

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