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# The influence of coatings on subsurface mechanical properties of laser peened 2011-T3 aluminum

C. S. MONTROSS*, V. FLOREA
*Redstone Mining P/L Artarmon, New South Wales 2064, Australia*
M. V. SWAIN
*Department of Mechanical & Mechatronic Engineering, University of Sydney, Sydney, NSW 2006, Australia*

High power Q-switched laser systems are currently being developed for use in a process known as laser shock processing or "laser peening" which results in significantly improved fatigue properties in aluminum components. An ablative, sacrificial coating such as paint or metal foil is used to protect the aluminum component from surface melting by the laser pulse, which adversely affects fatigue life. This paper, using nano-indentation, analyzes the effect of the paint and foil coatings on the shock wave propagation into the aluminum specimen and the resulting change in mechanical properties versus depth. Near the surface, hardness was found to be increased by the laser peening, however this process decreased the measured elastic modulus. The laser pulse energy density and properties of the foil including its adhesion to the aluminum alloy were found to influence the change in surface mechanical properties. 

## 1. Introduction

High pressure shock waves in materials [1–4] with pressures from 1 GPa to over 1 TPa [5] have been generated with pulses from Q-switched lasers. When a laser pulse from a Q-switched laser approximately 50 ns in length hits a metal surface at energy densities greater than $\approx$0.5 GW/cm$^2$, the top micron surface layer is in effect instantly vaporized and forms a plasma. When this rapidly expanding vapor/plasma plume is confined to the metal's surface by a quartz or water layer, the confining layer will direct the pressure pulse into the metal as a shock wave. The physics of shock wave generation and relationships to laser power density and other variables are discussed elsewhere in detail [1–6]. The focus since the 1970's of applying the laser generated shock waves has been on metals for shock processing.

The effect of coatings and water overlays covering quartz pressure transducers on the peak pressures generated by a laser pulse have been reported [6–8], but not on any property changes beneath the laser target spot. When the [6] coatings were thin enough to avoid acoustic impedance mismatch but thick enough to avoid complete ablation, there was no difference in the shock wave pressures generated versus type of coating. Fabbro *et al*. [6] reported that the peak pressures generated can be varied and enhanced by the correct selection of a coating with a different acoustic impedance with respect to the target. In another investigation [7, 8], Al and Zn foils were used on quartz transducers and an aluminum vapor deposited coating and an aluminum paint coating were also used on quartz transducers. According to Fairand *et al*. [7] and Clauer *et al*. [8], changes in the peak pressure generated were reported to be affected by the absorption of the laser pulse by the coatings on the quartz transducers. They [7, 8] reported that the greater the absorption, the greater the peak pressure.

Direct interaction of the laser pulse with a bare aluminum surface results in melting and this detrimentally affects the fatigue properties even though a compressive layer had been created [9]. The use of black paint is commonly reported as both a thermally protective coating and for increasing the shock wave pressure [10–12], though only once was the use of a metal primer noted [13] or that the paint used was commercially available [14]. However, no detailed analysis of the property changes occurring below the surface due to the laser peening process was noted.

Self-adhesive foil has also been used to protect the surface during laser peening [15, 16]. It was reported [16] that when the reflectivity of the foil decreased, as when substituting stainless steel foil for aluminum foil, the depth of peening increased in aluminum components. However, no comparison of paint versus foil on subsurface properties has been noted in the literature.

* Author to whom all correspondence should be addressed. Corresponding address: Dept. of Mechanical and Mechatronic Engineering, University of Sydney, Sydney, NSW 2006, Australia.

Additional problems with coatings were noted in preliminary experiments in our laboratory. There was a tendency for the protective coatings surrounding the laser spot to debond and/or spall off due to the laser shock process. The result was when the specimen was indexed to the next area for laser peening, the spalled or debonded paint did not provide sufficient protection of the surface from the laser pulse. The need for adherent protective coatings for laser peening was clearly identified. It was noted elsewhere by Fabbro *et al.* [6] that the thermoprotective coatings used were recommended to have very good adhesive properties, in particular for cumulative or multiple laser peening processes.

The purpose of this paper is to report the effect of the ablative, sacrificial coatings on the laser pulse generation of shock waves and their propagation into the aluminum and the resulting change in properties below the surface versus depth. Property changes were measured using an Ultra Micro Indentation System (UMIS) with a diamond Berkovich nano-indentor on polished cross sectional surfaces going through the diameter of the laser peen spots. Three coatings were investigated: flat black paint with aluminum etch primer for aluminum metal, flat black paint with zinc chromate primer for aluminum metal, and self adhesive aluminum foil. All coating systems are commercially available and have been reported elsewhere [9–15]. The effect of the laser pulses on the spalling and debonding of these commercial coatings is also investigated and reported. Two laser pulse energy densities were investigated with the aluminum foil to determine if any decrease in properties due to the attenuation of the shock wave amplitude by the foil could be compensated by an increase in laser pulse energy density.

## 2. Experimental procedure

A commercially available as-received 2011-T3 alloy 25 mm diameter aluminum rod was used in the as-extruded state and machined into disks 10 mm thick. The T3 state consists of solution heat treated followed by cold working and natural aging to a stable condition. The elastic modulus for this alloy is given as 70 GPa [17].

The specimen's machined surfaces were sanded with 240 grit SiC paper then primers were used because of their increased adhesiveness as compared to paint. Two commercially available automotive aluminum metal primers, one which chemically etched the aluminum surface to increase adhesion (known as etch-primer), and a commercially available zinc chromate based primer, were investigated as undercoatings for a commercially available automotive non-reflective black paint. The average thickness of the combined primer and paint coatings near where the laser peening was conducted were measured at about 0.1 mm. The aluminum foil used was 3M #425 Al Foil Tape which has a foil thickness of 0.11 mm and an adhesive thickness of 0.076 mm.

The shock waves were induced by laser pulses from a Q-switched near-infrared laser of wavelength 1064 nm and a pulse length of approximately 40 ns and a rapid laser intensity rise time. A film of flowing water approximately 1 to 3 mm deep covered the coated surfaces and which only minimally absorbs the near-infrared laser radiation. This film of water constrains to the surface the rapid expansion of the high temperature and high pressure plasmas generated by the laser pulse and directs the shock wave energy into the metal specimen.

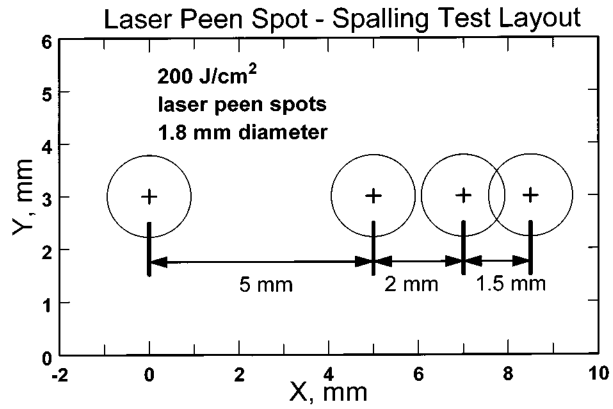


*Figure 1* Layout of the laser peen spots to test adhesion of protective coatings. Spots 1.8 mm in diameter for 200 J/cm$^2$ laser pulses are shown for example.

The energies used were approximately 6 J to 8 J delivered to the metal's surface with spot sizes varied between approximately 2 to 4 mm in diameter to vary the energy densities. At the metal's surface the laser energy densities of 100 and 200 J/cm$^2$ were used resulting in power densities of 2.5 and 5 GW/cm$^2$ respectively. From Fairand's *et al.* [3] fitted data shown in their Fig. 1 for a near infrared, Q-switched 1064 nm laser with an average pulse length of 40 nanoseconds, the resulting shock pressures are estimated to be 3.5 GPa and 6 GPa respectively.

Fairand's *et al.* [3] fitted data is preferred because the laser parameters that they used for their analysis are similar to the laser parameters used in this research. Additionally, the shock wave pressure was measured on the front surface of a cross-cut quartz pressure transducer where the shock wave was formed. Therefore, the shock wave pressures measured would not have been affected by any material properties during propagation.

Another research group, Berthe *et al.* [18], analyzed the peak pressure versus peak laser intensity by a different technique for different laser parameters than the ones used here. The velocity change of the back free surface of an approximately 0.5 mm (457 $\mu$m) thick aluminum foil accelerated by the laser generated shock wave was measured by a velocimetry interferometric technique. From this velocity data, the shock wave pressure at the back free surface of the aluminum foil was calculated. However, the effect of a solid's strength on the propagation and attenuation of shock waves becomes important for shock wave pressures at 10 GPa and below [19–22]. The laser generated shock wave pressures used by Berthe *et al.* [18] are considered to be below this pressure threshold where materials properties affect shock wave propagation behavior and velocity. The shock wave pressure at the back free surface of the aluminum foil is considered to be significantly different from the front surface where the shock wave was generated by the laser pulse.

To investigate the effect of spacing between indexed laser peen spots on the adhesion of the coatings, a series of 100 J/cm$^2$ laser pulses were delivered to the coated surfaces. The distance between the centers of the successive spots was decreased from 5 mm to 2 mm to 1.5 mm which resulted in an eventual overlap of the spots. For the aluminum foil coated specimens, an additional series of 200 J/cm$^2$ were used to investigate whether increasing the energy density compensated for any shock wave attenuation by the foil. An example of the spatial arrangement is shown in Fig. 1 where the final two laser spots overlapped for a series of 1.8 mm diameter, 200 J/cm$^2$, laser pulse spots on the aluminum foil. After each shock wave generation by the laser pulse, the laser peened spots and the areas surrounding them were visually inspected for spalling and/or debonding of the coatings used.

Cross sectional surfaces, through the diameter of the spots, were cut and polished to metallographic quality. Using a diamond Berkovich nano-indentor, both the hardness and elastic modulus were measured [23–25] of the polished cross section. In the method used by the UMIS nano-indentor, load-displacement data is measured during one complete cycle of loading and unloading. Using a model of an elastic half space relating the deformation caused by an elastic punch to its contact area at peak load, the elastic modulus can be calculated from the unloading data. Fig. 2 shows one complete loading-unloading cycle of a typical load-displacement behavior for the aluminum specimens. From the slope of the initial unloading data, S, the initial unloading stiffness can be determined. From this data and the elastic modulus and Poisson's ratio for the indentor, the elastic modulus can be determined for the specimen at that location.

A line of nano-indentation measurements were made in a non-laser peened area from the as-received, prepared surface to determine if the sample preparation developed any residual property changes. With careful preparation and experimental technique [26], elastic moduli of the specimens can be determined within ±5%.

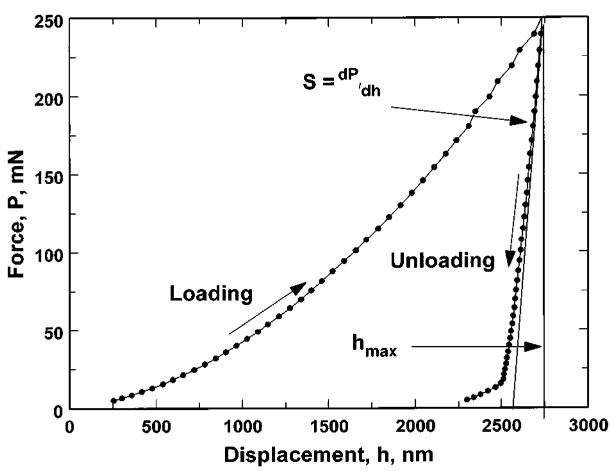


*Figure 2* Example Force – Displacement diagram for the nano-indentation of a polished surface of a 2011-T3 aluminum alloy specimen.

Lines of nano-indentation measurements were made on the polished cross sectional surface from the center of each laser spot into the aluminum to identify the depth of the metal affected by the shock waves. The average value of the hardness and elastic modulus in each aluminum sample was calculated based on measurements of the polished, cross sectional surface away from the laser peen, shock wave process area.

## 3. Results

### 3.1. Adhesion of coatings

In Table I, the relationship between the adhesive behaviour of the coating and the laser energy density is shown. For both of the primer/paint combinations, there was spallation of the paint coating from the surface around the laser spot. For the zinc chromate primer/black paint combination, the spallation didn't extend as far from the edge of the spot as it did for the etch primer/paint combination. Additionally, there was some surface melting of the aluminum specimen through the coating with the etch primer/paint combination. As the distance between the spots decreased, the degree of spallation increased, thereby affecting successive laser peen spots.

With the use of an aluminum foil coating, the foil retained adhesion after laser peening with a laser energy density of 100 J/cm$^2$, even when the distance between the laser peen spots decreased. When the laser energy density was increased to 200 J/cm$^2$, the foil coating debonded directly under the laser spot and in a small region around the spot. When successive laser spots started to overlap previous laser peen spots, the degree of debonding increased affecting the local area.

### 3.2. Change in hardness and elastic modulus

The baseline hardness and elastic modulus of the aluminum specimen away from any laser peening spots was measured versus depth. Analysis of the data of the as-received, prepared surface without laser peening indicated that there were no surface residual stresses or property changes due to sample preparation techniques prior to laser peening. With a careful measurement technique, the average hardness was measured at 1.61 GPa with the average elastic modulus measured at 71.7 GPa, which is within the ±5% accuracy possible noted by Oliver and Pharr [21].

TABLE I Relationships between adhesion of coatings and laser pulse energy

| Laser Energy Density, J/cm$^2$ | Coating | Behaviour of Coatings |
|---|---|---|
| 100 | Etchant primer, black paint | Spallation |
| 100 | Zinc chromate primer, black paint | Spallation |
| 100 | Self-adhesive Aluminum foil | Adhered |
| 200 | Self-adhesive Aluminum foil | Loss of adhesion |

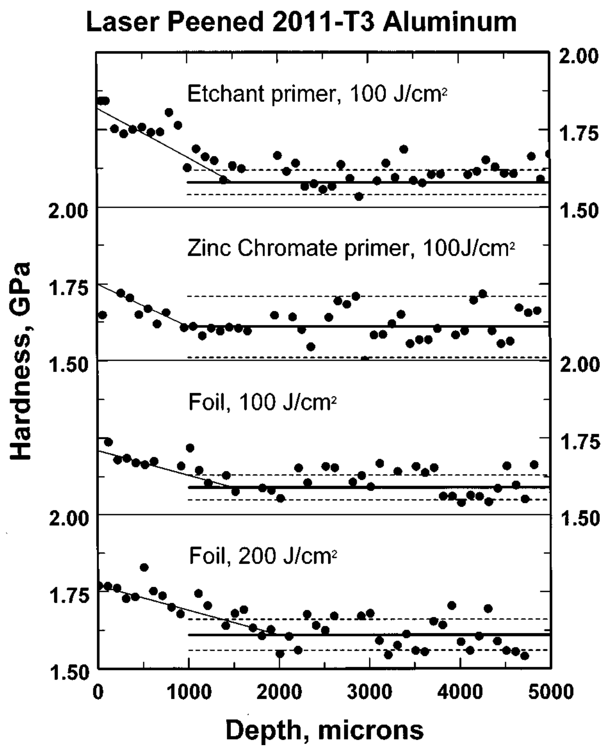


*Figure 3* Comparison of Hardness versus Depth of laser peened black paint/etchant primer coated 2011-T3 aluminum, black paint/zinc chromate primer coated 2011-T3 aluminum, self-adhesive aluminum foil coated 2011-T3 aluminum (all three laser peened with a 100 J/cm$^2$ laser pulse), compared with self-adhesive aluminum foil coated 2011-T3 aluminum laser peened with a 200 J/cm$^2$ laser pulse.

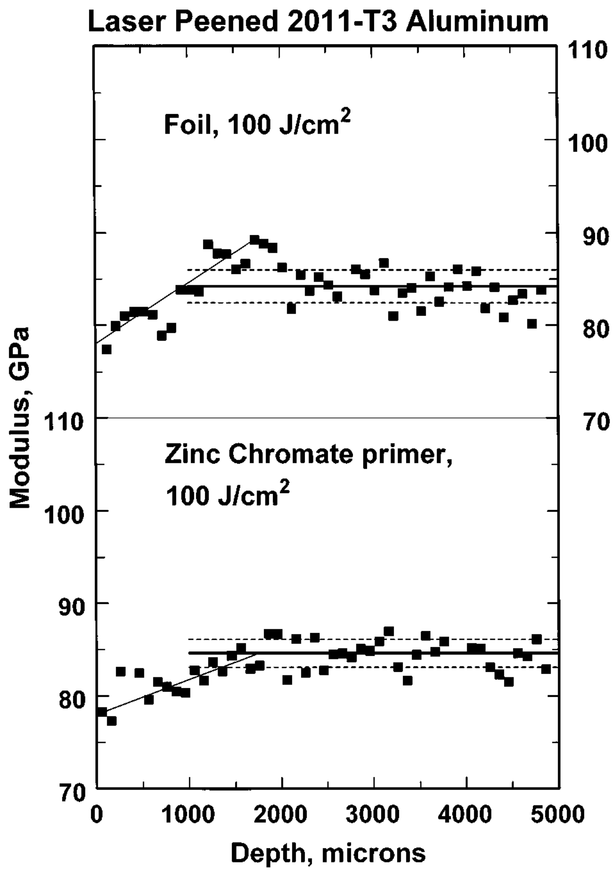


*Figure 4* Elastic modulus versus depth of black paint/zinc chromate primer coated 2011-T3 aluminum compared with the elastic modulus of self adhesive aluminum foil coated 2011-T3 aluminum, both laser peened with a 100 J/cm$^2$ laser pulse.

In Fig. 3, the hardness and elastic modulus are plotted versus depth for the etchant primer/paint combination, zinc chromate primer/paint combination, foil with a 100 J/cm$^2$ laser energy density peen spot, and foil with a 200 J/cm$^2$ laser energy density peen spot, respectively. From these figures, the depth of hardness change and the depth of elastic modulus change below the impacted surface are listed in Table II.

The average hardness, the increase in hardness, the average measured elastic modulus, and the surface elastic modulus are listed in Table III with respect to the coating and laser energy density.

The aluminum surface for all coatings increased in hardness by 7.5% to 15%, from a bulk average of 1.6 GPa to approximately 1.7 to 1.8 GPa. The depth of penetration of the shock wave into the aluminum varied with the coating and with the energy density applied to the metal foil coating. There was more scatter in the measurements as compared to the baseline measurements of the hardness and elastic modulus of the aluminum specimen away from any laser peening spots.

TABLE II Relationships between coating, laser pulse energy, and depth of property changes

| Laser Energy Density, J/cm$^2$ | Coating | Depth of Hardness Changes, mm | Depth of Modulus Changes, mm |
|---|---|---|---|
| 100 | Etchant primer, Black paint | 1.5 | 2.5 |
| 100 | Zinc chromate primer,black paint | 1.0 | 2.0 |
| 100 | Aluminum foil | 1.5 | 2.0 |
| 200 | Aluminum foil | 2.0 | 1.0 |

The elastic modulus was reduced at the surface for all specimens from −5% to −12% due to the laser peening. In Fig. 4, the change in elastic modulus versus depth for the black paint/zinc primer coated specimen is compared with the foil coated specimen. The reduction of elastic modulus for the primer/paint based coatings increases linearly from the surface into the bulk but the depth of reduction did not correspond to the depth of the hardness change. For the foil coated specimens, the elastic modulus at the surface was less than the average but linearly increases to a peak slightly above the average before decreasing and reaching the bulk average. The depth of the elastic modulus change did not correspond with the depth of the hardness change.

## 4. Discussion

### 4.1. Thermoprotective coatings

The effect of different thermoprotective coatings for laser peening ranging from paint to foil has been discussed in the literature [6, 10–16]. Other than the need for the thermoprotective coatings to have very good adhesion to the target [6], little comparison or discussion has been noted. In this research on laser peening with coatings of similar thickness, if change in surface hardness was the chief criteria, the etch primer/paint coating

TABLE III Relationships between coating, laser pulse energy, and property changes

| Laser Energy Density, J/cm$^2$ | Coating | Average Hardness, GPa | Surface Hardness, GPa | Average Elastic Modulus, GPa | Surface Elastic Modulus, GPa (% Change) |
|---|---|---|---|---|---|
| 100 | Etchant primer, black paint | $1.58 \pm 0.04$ | 1.82 (+15%) | $78.3 \pm 1.3$ | 69.0(−12%) |
| 100 | Zinc chromate primer, black paint | $1.61 \pm 0.1$ | 1.75 (+8.7%) | $84.6 \pm 1.5$ | 78.3(−7%) |
| 100 | Aluminum foil | $1.59 \pm 0.04$ | 1.71 (+7.5%) | $84.2 \pm 1.8$ | 78.0(−7%) |
| 200 | Aluminum foil | $1.61 \pm 0.05$ | 1.77 (+10%) | $84.9 \pm 1.6$ | 80.7(−5%) |

would produce the greatest change. However, the etch primer/paint coating spalls off during laser peening. If retention of the coating during multiple laser peening or overlapping of the laser peening spots, the aluminum foil would be the best when the laser energy density is 100 J/cm$^2$.

### 4.2. Elastic modulus measurement

The difference in the measured elastic modulus for the laser peened specimens and the book value is due to a slight difference in measurement technique. All indentation measurements were run overnight to minimize disturbances from vibrations and thermal fluctuations. Due to the volume of data points that needed to be measured, 21 points for the baseline measurements versus 50+ for the peened specimen, and the time constraint for the overnight run, the measurements of the peened specimen were conducted with shorter dwell times.

The elastic modulus is calculated based on the slope of first eight unloading points. Variability in these data points near the maximum force and displacement will contribute to changes in the elastic modulus. This variability in the data points is due to the plastic deformation plus a small amount of creep behaviour of the material during indentation and unloading which influences the slope of the unloading curve and thereby overestimating the modulus. More accurate measurement of the elastic modulus may be obtained if the indentor to be loaded and unloaded a few times before the load – displacement behaviour becomes perfectly reversible [26]. This was not practical in this research. Another way is to slow the loading and unloading steps to allow better equilibration of the stresses. This resulted in good data for the baseline measurements but was not practical for the others.

### 4.3. Hardness changes

The depths of hardening by laser peening of aluminum alloys, typically 1 to 2 mm, and the increases in surface microhardness, from approximately 1.6 to 1.7 or 1.8 GPa, reported in the literature [27–31] correspond closely to the results measured here. It is interesting to note that in Fig. 3 for the etchant/black paint coated aluminum, the reduction in shock wave intensity after traveling approximately 500 microns resulted in a reduction in hardness from 1.81 GPa to approximately 1.75 GPa. It is expected that the shock wave pressure intensity in the shock wave pressure versus laser intensity measurements by Berthe *et al.* [18] would similarly be reduced after traveling 457 microns through the aluminum foil.

The type of coating used affected the changes in the hardness and elastic modulus. Increasing density of the coating, from the etch primer/paint coating, zinc chromate primer/paint coating, to the foil coating, resulted in decreasing change in surface hardness and elastic modulus. Increasing the energy density from 100 to 200 J/cm$^3$ for laser peen spots on the aluminum foil only slightly increased the change in hardness and may not be considered worthwhile doing due to the debonding of the foil from the specimen. The foil coating is considered the better coating since it doesn't spall off during laser peening and costs are ignored.

The depth of hardness and elastic modulus change decreased with increasing density of the coating, going from the etch primer/paint coating to the zinc chromate primer/paint coating, but the aluminum foil had an equivalent depth of change as the etch primer/paint coating. A possible explanation is that other than providing thermal protection, the etch primer/paint coat effectively doesn't exist during shock wave propagation. Therefore the attenuation of the shock wave will be similar to the attenuation through the foil into the specimen.

### 4.4. Elastic modulus changes

The reduction in surface elastic modulus due to laser peening has been reported only once before regarding a black paint coated, laser peened aluminum weldment [32]. The results here are similar to the changes in elastic modulus that were reported.

In the analysis of metals shocked by explosives or from dynamic fracture tests, the focus has been on phase changes, microstructural changes, and physical changes caused by the shock wave such as shear band, void, and/or spall formation [33]. When the peak shock wave pressure was greater than the dynamic yield strength, extensive plastic deformation and increased dislocation density resulted [34]. A uniform distribution of dislocation tangles has been observed in 2024-T3 aluminum alloy laser shocked at 6.5 GPa and the dislocation tangles are reported to be similar to that produced by other shock wave generation methods [35]. It was noted by Jones [36] that the density of dislocations caused by shock wave propagation is several orders of magnitude greater than an equivalent amount of cold working though actual quantitative densities were not given.

The reduction in elastic modulus by explosively driven shock waves in annealed 1018 steel [37] and 2024 aluminum alloy [38] was reported to be due to the dislocation loops generated by the shock waves. For the 2024 aluminum alloy shocked at 0.15 GPa and 1.1 GPa, increasing the shock pressure increased the reduction in elastic modulus. Upon annealing, the elastic modulus recovered to within 95% of the elastic modulus prior to being explosively shocked.

The cause of the complex behavior of the change in elastic modulus with depth for the foil protected aluminum specimens is not well understood. The reduction in the surface elastic modulus in the laser peened aluminum weldment was almost linear with depth [27] as seen in the primer/paint specimens in this work. It is hypothesized that the laser pulse accelerated the 0.11 mm thick (110 $\mu$m) protective aluminum foil into the bulk aluminum specimen similar to the flyer plate – target technique used in the generation of shock waves by explosives. The adhesive layer between the foil and the specimen was of such a low impedence compared with the aluminum foil and specimen, it effectively didn't exist with respect to the shock wave. Therefore the foil and bulk specimen behaved similar to the flyer plate – target technique. In Murri *et al*. [39], a schematic of a flyer plate soon after impact with a target is shown in their Fig. 1; from the interface between the flyer plate and target, shock waves propagate out into both the flyer plate and the target. The shock wave reflects off the air-flyer plate interface becoming a rarefaction wave which propagates into the flyer plate and target.

Intense laser driven shock waves generated on the surface of aluminum foils 435 $\mu$m and 475 $\mu$m thick have resulted in spallation of the back surface of the foils where the compressive shock waves are converted into reflected rarefaction waves at the foil-air interface [40, 41]. In this situation, it is hypothesized that following the initial laser generated shock wave, rarefaction waves and additional compressive shock waves of lesser magnitude interacted with the specimen. This resulted in the unusual behavior of the elastic modulus in the foil protected specimens.

## 5. Conclusions

From the research results presented, the following conclusions can be made.

a. Laser pulses from a Q-switched near-infrared laser of wavelength 1064 nm and a pulse length of 40 ns and energy densities of 100 and 200 J/cm$^2$ generated shock waves which increased the surface hardness of the 2011-T3 aluminum samples. The increase in surface hardness matched that reported in the literature. The depth of the hardness change was similar to the depth of induced compressive stresses reported in the literature measured using the strain gauge rosette technique.

b. A change in elastic modulus due to the laser generated shock waves was found in this research. The decrease in elastic modulus at the surface ranged from −5% to −12% and occurred to a depth greater than the measured change in hardness.

c. The decrease in the surface elastic modulus from both laser generated and explosively generated shock waves has been reported before and is due to the generation of dislocation loops and other defects by the shock wave.

d. Self adhesive foil is considered to be the better protective coating when laser peened with a 100 J/cm$^2$ laser pulse because it does not spall off unlike the primer/paint combinations. However, the change in surface hardness during laser peening is not as significant as that attained with the etch primer/paint coating.

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