



Studies on ultra-short pulsed laser shock peening of stainless-steel in different confinement media

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ABSTRACT

We investigate the role of liquid confinement media on ultra-short pulsed Laser Shock Peening (LSP). The LSP of stainless-steel 316 and 316 L was studied using Ti: Sapphire laser pulses of about 2 ps duration, maximum energy of about 1 mJ and pulse repetition rate of 5 kHz in different liquid confinement media of Ethanol, Deionized water and separate aqueous solutions of NaCl and Glycerol. It is found that the laser fluence and/or energy attenuating mechanisms like self-focusing, filamentation, plasma breakdown in the confinement media are less significant with ps laser pulses than those with sub-ps or fs pulse durations. It is shown that the resulting surface hardness of the peened steel as a function of laser fluence depends significantly on the confinement media and the relative increase in the hardness increases monotonically with the acoustic impedance of the liquid of the confinement medium used during LSP.

1. Introduction

Laser shock peening (LSP) in the ultra-short pulse durations regime of pico-second (ps) to femto-second (fs) is a potentially important area of research for surface enhancement of engineering components of different metals and alloys even though this area is still in its infancy. This is because in this time regime the pressure of the shock waves generated is correspondingly orders of magnitude higher than that generated for example in nano-second or micro-second time regimes and the depth of the residual compressive stress is much finer [1–3]. Also, the process of ultra-short pulsed shock peening is fundamentally non-thermal in nature because the laser pulse duration in this case is comparable or shorter than the electron-phonon coupling time. The heating and cooling rates of the surface in this case can be as large as $\sim 10^{12}$ Kelvin per second [4] resulting in ultra-fast ablation, which is not possible with longer laser pulses resulting in slower thermal processes like melting and vaporization. The end result of these initial processes is the generation of sharp shock-wave [5], which leaves residual compressive stress in the surface region of the subjected metals or alloys causing its peening through plastic deformation [6].

Mostly LSP is carried out by applying a sacrificial layer on the metal or alloy surface and an overlayer of confinement medium, generally water [7,8]. Even though some researchers prefer to carry out LSP in fs time regime without a sacrificial layer [9], which we have found to

result in compromised surface quality [3]. But even in the former case a confinement layer is invariably used for LSP with conventional nano-second laser pulses because the amount of pressure created due to laser ablation under the confinement medium is several times or even an order of magnitude larger than the pressure created in an unconfined open environment [10]. When the high intensity laser pulse interacts with the target material, a high recoil pressure ablation plume is generated and in the absence of a confinement layer this plume expands and loses most of its energy in the surrounding environment away from the target surface rather quickly. Whereas, in the presence of a confinement medium the expanding plume is confined and thus, the shock energy is concentrated more towards the target surface for longer time, which is beneficial in peening the target material by resulting in enhanced compressive residual stress on its surface region [11]. It has been found that the pressure induced by medium-confined nanosecond laser ablation plume can be as high as an order of magnitude higher and 2 to 3 times longer than pressure induced by the same plume in air or vacuum, for the same laser intensity [12]. It is therefore imperative to choose a good confinement medium to increase the peening efficiency for nanosecond LSP. Even though several new results have been published during 2019–2020, it is still controversially discussed whether the confinement media is needed or not for *ultra-short pulse* laser shock peening (compare e.g., [13,14]).

Choice of a good confinement medium depends on its optical

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transparency for the laser wavelength, chemical inertness to sample material and sacrificial layer, high acoustic impedance and ease of usability. The layer of the confinement medium should also have an optimum thickness. If the confinement layer is very thin, it makes the processing difficult particularly at high laser intensities because it remains ineffective for confinement of the ablation plume produced. And if the confinement layer is too thick, it absorbs, distorts the pulsed laser beam that passes through this layer and attenuates the incident laser energy due to filamentation particularly at shorter pulse durations and higher intensities [15]. That is why it is also advantageous to take a confinement medium with smaller linear and nonlinear refractive indices n_0 and n_2 to increase the self-focusing threshold [16].

To the best of our knowledge, N. C. Anderholm was the first to present the technique of confined laser produced ablation [17]. In this study he investigated a method to generate laser induced stress waves under a transparent overlayer of quartz disc. The experiments were carried out using a ruby laser system at pulse energy of about 7 J and duration of 12 ns. Subsequently several studies have been carried out, which showed increased efficiency of the peening with confinement layer. D. Devaux et al. investigated the characteristics of laser-induced shock wave in a confining environment [18]. Experiments were carried out with a conventional short ns time regime pulsed neodymium glass laser at laser intensities ranging from 10^7 to 10^{11} W/cm². Laser-induced shock pressures were measured using a synthetic piezoelectric quartz gauge under glass, water and without any confinement layer. They found that the shock pressure generated due to the laser ablated plume/plasma under a confinement layer depends on the square root of the combined acoustic impedance of the confinement medium and the target material [18]. They observed the maximum increase in the shock pressure by about an order of magnitude due to the confinement layer in a broad range of the laser intensity [18].

Followed by these studies numerous researchers have studied the conventional LSP under different confinement media in long and short pulse duration regimes. However, to the best of our knowledge, a systematic study on the influence of different confinement media used during LSP in ultra-short laser pulse duration regime of ps - fs is not yet reported in the pertinent literature. We carried out such a study on the role of different confinement media on the mechanical properties of stainless steel subjected to LSP in the ps time regime. We first investigated the problems of laser induced non-linear effects like self-focusing, filamentation and plasma breakdown etc. in the confinement media, which attenuate the laser energy that can be delivered to the surface of the sacrificial layer. Followed by that, under the optimized conditions of LSP, we studied the surface hardness of the steel subjected to ps LSP under different confinement media. The results of these studies are presented and discussed in this paper.

2. Experimental set-up

In this paper we study the effect of different confinement media on the ultra-short pulsed laser shock peening of stainless steel 316 and 316 L. The experimental set-up is shown in Fig. 1. We used a Ti:sapphire laser system (*Spitfire Ace* produced by *Spectra Physics*) operating at wavelength about 800 nm. The pulse duration was stretched by adjusting the compressor of the laser system to about 2 ps and the maximum peak energy was taken about 1 mJ at 5 kHz pulse repetition frequency. As shown in Fig. 1, the laser beam was focused using a telecentric lens of about 128 mm focal length to the spot diameter of about 10 μ m on to the target samples through the glass window of an in-house fabricated processing chamber and the liquid confinement media therein. The target samples were mounted in this confinement media filled processing chamber. Liquids of confinement media were pumped into the processing chamber using a pump to achieve a laminar flow to remove any bubble formed due to the repeated interaction of the laser beam with the target under the confinement media and the

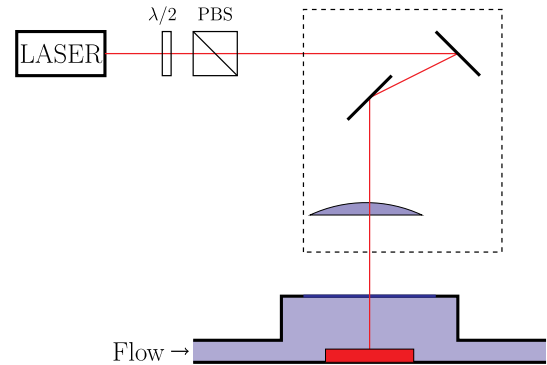


Fig. 1. Experimental set-up for studying the influence of different confinement media on ultra-short pulsed laser shock peening. $\lambda/2$ and PBS are the wave plate and polarizing beam splitter to control the laser fluence. The galvo-scanner consists of two computer-controlled mirrors and focussing optics is marked by the dashed line.

ablated debris that would invariably affect the parameters of the laser light adversely. This chamber (volume of ~ 20 cm³) was flushed with different liquids of the confinement media flowing at a rate of about 4.2 l/min for the aforesaid purpose. The chamber was mounted onto an X, Y-stage for positioning the samples. During the LSP process, a galvo-scanner (*SCANcube10*, manufactured by *SCANLAB*) directed the laser beam on to the target as shown in Fig. 1.

Processing of the samples was carried out at different laser fluences controlled by a $\lambda/2$ wave plate and a polarizing beam splitter. The average laser power was measured prior to the processing using a power meter aligned in the laser beam path. As mentioned, we carried out the LSP of stainless steel 316 and 316 L under different confinement media of Ethanol, Deionized water, aqueous solutions of NaCl and Glycerol separately using a sacrificial layer of a black vinyl adhesive tape. Since pure glycerol is highly viscous, the confinement media in this case was prepared by mixing water in it so that it could flow over the target sample to remove the ablation debris and bubbles etc. at the high repetition rate of the peening laser pulses. Vinyl tape satisfies all the requirements of a good sacrificial layer such as good absorption of laser light, an even surface, chemically inert, good sticking to the target metal, environmentally safe etc., which makes it a better alternative in comparison to layers like black paint, transparent adhesive tape etc. The laser peened samples were investigated for the resulting surface quality using a scanning electron microscope, for chemical analysis using Energy-dispersive X-ray spectroscopy (EDX) and surface hardness using a Vickers hardness tester (*KB30BV7*) with HV 0.1 (equivalent to 0.981 N). The penetration depth of the indenter is larger than the estimated penetration of the shock wave. Therefore, the hardness measured in this manner is convoluted result of the hardness due to the peening and the virgin metal and therefore we expect some under-estimation of the LSP effect.

3. Results and discussions

In the following two subsections the influence of the optical and acoustic characteristics of the confinement media on the LSP process will be analysed.

3.1. Attenuation in confinement media

The main reason why LSP in confinement media is rarely used in combination with *ultra-short* laser pulses, is appearance of complex optical effects specific for ultra-short laser pulses. They are the laser energy attenuating processes, such as non-linear effects of self-focusing, plasma breakdown and filamentation in transparent confinement

media. Due to importance of these processes for LSP in liquids we carried out additional experiments to investigate them in order to optimize our LSP experiments. Schematic of the experimental set-up for investigating the energy attenuation due to filamentation in different confinement media is shown in Fig. 2. The ultrashort laser pulses of duration of ~ 100 fs to 5 ps, wavelength of $800 \text{ nm} \pm 30 \text{ nm}$ and pulse energy of about 1 mJ were focused by a plano-convex spherical lens of focal length of ~ 100 mm into a test cuvette made of BK7 glass containing the confining liquid under investigation. The focal spot size of the laser beam in water, determined by ablation experiments was $\sim 20 \mu\text{m}$. A sharp metal blade was inserted incrementally into the beam path from top to bottom of the cuvette in the focal plane of the laser beam and after the light filament and white-light generation began as shown in Fig. 2 schematically, blocking the initial laser radiation and white light in small steps. The out coming laser and white lights were split to simultaneously pass into a power meter and a spectrum analyzer (USB2000+ made by Ocean Optics). These combined and simultaneous measurements of laser and white light power and spectrum enabled the differentiation between white light and Ti:Sapphire laser radiation through computational deconvolution and acquiring the spatial intensity distributions of the two lights at different pulse durations.

Using the experimental setup shown in Fig. 2, a cross sectional spatial intensity distribution of the laser light and the white light generated through filamentation, which attenuated the laser energy and fluence, were acquired. These spatial intensity profiles of the two lights are shown in Fig. 3. For 800 μJ pulses, transmitting through 40 mm length of water from the focal spot of the laser light, two important inferences were elicited. 1) The focal spot diameter of the Ti:Sapphire radiation increased by a factor of almost 7.5 to about $\sim 150 \mu\text{m}$ reducing the fluence at the target correspondingly. 2) Depending on the pulse length, as shown in Fig. 3, large portion of the laser radiation was found to get converted into white light. The pulse energy loss due to this filamentation and white light generation process was found to be about $\sim 60\%$ for the 100 fs pulses while it was only about 20% in case of the laser pulse duration of 5 ps, as shown in Fig. 3. From this point of view, it is energetically advantageous to use a few ps laser pulses compared to that of 100 fs for shock-wave peening in a confinement medium. Since the durations of the 100 fs and 2–5 ps pulses do not exceed the electron-phonon coupling time in metals, we assume that the amplitudes of the shock waves generated with these pulses are comparable at the constant laser fluence delivered at the target metal. Hence it is imperative to investigate the relative merits and demerits of LSP in these two duration regimes of the laser pulses.

One of the most important issues is the optimization of the confining media thickness. Fig. 4 shows a comparison of the length of the onset and the actual extension of the filamentation produced in deionized water as confinement medium for the laser pulse duration of 100 fs and 2 ps at identical pulse energy of about 1 mJ. As can be seen filamentation and plasma breakdown initiate much later along the path of the laser beam in the deionized water in case of the 2 ps than 100 fs. A similar behavior was also observed in ethanol [15]. This gives a benchmark of the length of the confinement medium, which should be used to locate the target sample so as to zap the laser beam with minimal loss of energy.

3.2. Efficiency of LSP

In our experimental studies we have also investigated the surface morphologies and contamination mainly due to oxidation and carbonization. Our general finding was that there were no significant differences in these aspects due to the different confinement media as long as the laser beam interacted with the sacrificial layer and it was not allowed to interact with the metal surface directly. As stated earlier, different confinement media used in this study were Ethanol, Deionized water, aqueous solution of NaCl at 20% wt./v and glycerol at 60% concentration also in water. The pertinent physical properties of these

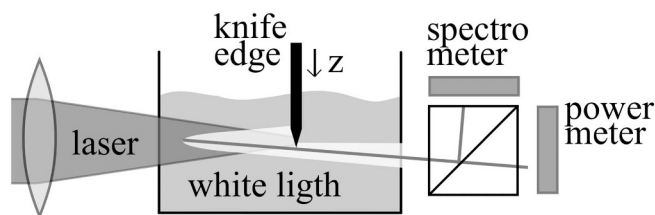


Fig. 2. Schematic of experimental arrangement for the measurements of the incident beam attenuation due to filamentation in different confinement media.

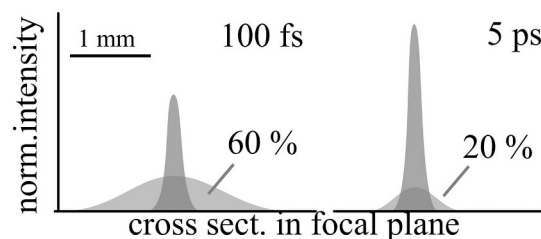


Fig. 3. Comparative spatial intensity distributions of the incident laser radiation (dark-gray sharp peak) and of the white light generated through filamentation (light-gray broad peak) in cases of 100 fs and 5 ps laser pulses.

confinement media are summarized in Table 1.¹ Deionized water was preferred over the normal water, to avoid any mineral deposition in the chamber and on the samples usually caused by the normal water.

To study the effect of different confinement media we used two different samples of steel and measured the surface hardness on Vickers scale at different fluences of the 2 ps laser pulses used for the peening. The reference hardness in the following figures refers to the hardness of the untreated steel samples and the experimental points are the hardness relative to this reference. Throughout this work untreated reference hardness (HV) value was taken as 160 HV for grade SUS316L and 290 HV for SUS316, both measured using Vickers hardness testing equipment. In these experiments the sample surface was covered by overlapping laser pulses. The repetition rate of the laser was 5 kHz, the scanning velocity 10 mm/s, corresponding to 2 μm shift between the centers of two consecutive pulses.

Fig. 5(a) shows the hardness of the stainless steel SUS316 measured at different fluences of the laser beam in the confinement medium of Ethanol. On the x-axis we plot calculated fluence, which is the pulse energy measured before the processing chamber divided by the measured laser spot area. As can be seen in this figure the surface hardness briefly increased with laser fluence and then monotonically decreased before almost saturating at the laser fluence of about 300 J/cm^2 . The initial increase in the hardness is plausibly due to increase in the shock pressure induced by the increasing fluence of the peening laser pulses. Subsequent decrease in the hardness can be attributed to the onset of energy attenuating processes due to non-linear absorption, filamentation etc.

Experimentally measured hardness of the SUS316L stainless-steel as a function of the peening laser fluence in the confinement medium of Deionized water is shown in Fig. 5(c). In contrast to the case of Ethanol stated above, in Deionized water, the surface hardness shows a little decrease in its value with increasing fluence in the beginning, although this decrease is within the measurement uncertainty range and is therefore not significant. It is followed by a drastic increase in the hardness with the fluence preceding the final fall due to onset of the complete ablation of the sacrificial layer on the surface of the target sample. Complete ablation of the sacrificial layer at the laser fluence of

¹ Values marked with * are taken from the NDT Resource Center database *Acoustic Properties for Liquids*, https://www.nde-ed.org/GeneralResources/MaterialProperties/UT/ut_matprop_liquids.php, Accessed: 2019-08-22.

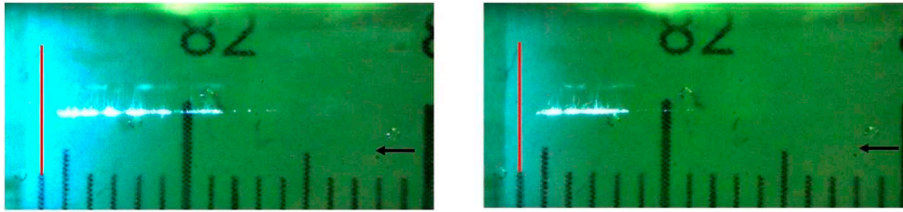


Fig. 4. Longitudinal size of filamentation and its point of initiation observed in deionized water as a confinement medium at laser pulse duration of (a) 100 fs (b) 2 ps. The beam propagation direction is indicated by black arrows. The red vertical line shows the focal point of the focusing lens in the respective image. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Table 1

Pertinent properties of different confinement media used in this study, * are taken from the NDT Resource Center database *Acoustic Properties for Liquids* and ** are from [19].

Confinement layer	Density (kg/m ³)	Sound speed (m/s)	Acoustic impedance 10 ⁶ (kg m ⁻² s ⁻¹)
Ethanol*	789	1180	0.93
Deionized water*	998	1400	1.48
20 % NaCl*	1150	1750	1.57
60 % Glycerol**	1230	1899	2.34

about 650 J/cm² was confirmed by visual inspection of the sacrificial layer and onset of Laser Induced Periodic Surface Structures (LIPSS) on the target metal observed under high magnification of SEM, which are shown in Fig. 6. Earlier we had shown that followed by the complete ablation of the sacrificial layer, whenever there is direct interaction between the laser pulses and the metal surface, LIPSS formation is inevitable [3]. Formation of LIPSS, oxidation of the exposed surface of the target metal and partial ablation at the surface collectively seem to be responsible for the drastic decrease in hardness beyond the laser fluence of 650 J/cm², where complete ablation of the sacrificial black tape occurred.

Dependence of the surface hardness on laser fluence in the confinement medium of aqueous solution of 20% NaCl is shown in

Fig. 5(b). This dependence and the one in the confinement medium of 60% glycerol in water, as shown in Fig. 5(d) are rather similar in trend. First the hardness increases with the fluence due to the increase in the shock pressure generated and beyond the maxima, it decreases putatively due to the onset of laser energy attenuating processes as discussed earlier. However, an interesting feature in Fig. 5(d) is the spike of hardness in the fluence range of 400–700 J/cm² with the exceptionally high peak hardness of about 230 on the Vickers scale. This can possibly be explained by the following mechanism: The linear refractive index n_0 and the nonlinear refractive index n_2 for glycerol are both larger than that of deionized water [20], hence, their product in the water-glycerol mixture is also larger than that in the water. The critical laser pulse power needed for filamentation in transparent media (as shown in Fig. 4) can be estimated [16] as $P_{cr} = \frac{0.15\lambda^2}{n_0 n_2}$, where λ is the incident light wavelength. Moreover, the ionization potential of glycerol is lower than that of water (10.1 eV and 12.6 eV respectively). Hence, the filamentation and the optical breakdown in the water-glycerol mixture must happen at lower laser peak powers (i.e., fluences, because the laser pulse duration and the focusing conditions were the same), than in water. Thus, we suppose that the decrease in the hardness in the Fig. 5(d) is due to onset of the incident laser beam filamentation for fluences above approximately 500 J/cm².

To compare the effectiveness of all the confinement media used in the present study, we have plotted the relative increase in the maximal measured hardness of the stainless steels as a function of the acoustic

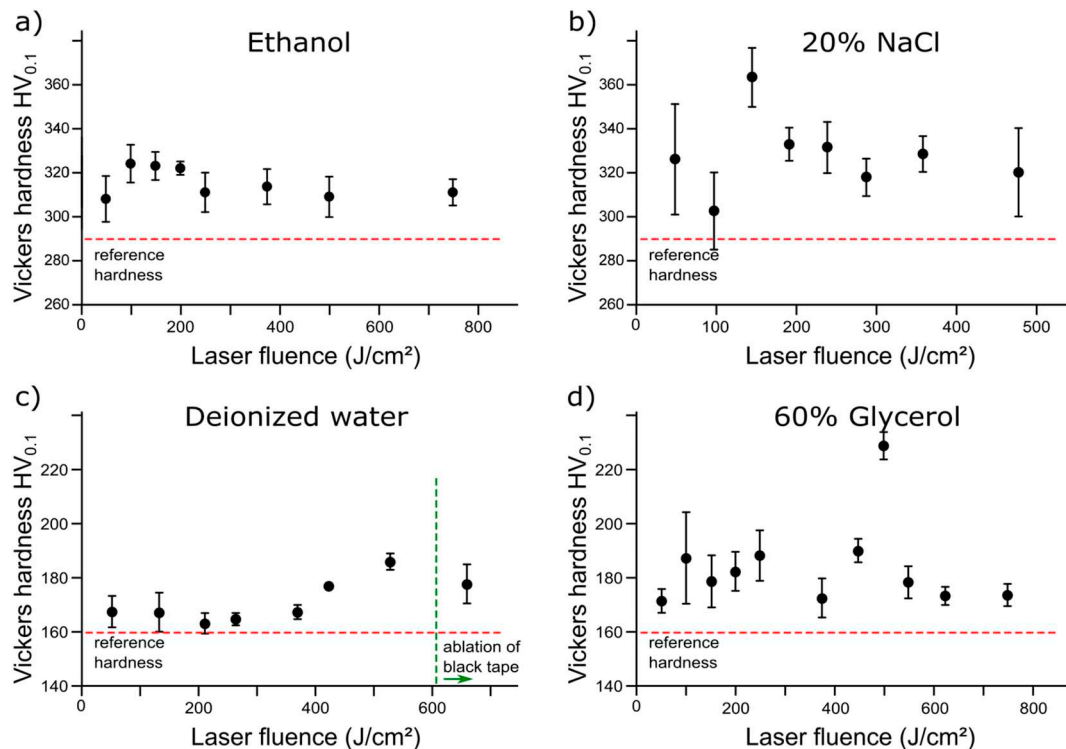


Fig. 5. (a–d). Variation in the surface hardness of stainless steel SUS316L peened at different fluences in (a) Ethanol (b) Aqueous solution of NaCl of 20% concentration (c) Deionized water and (d) 60% Glycerol in water as confinement media.

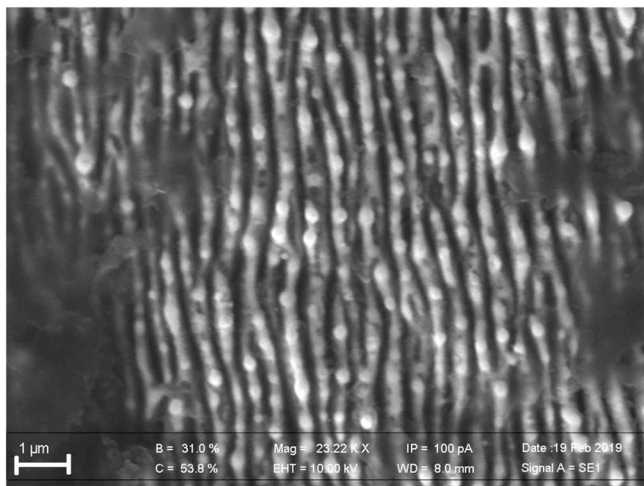


Fig. 6. LIPSS (Laser Induced Periodic Surface Structures) observed on the steel surface after complete removal of the sacrificial layer. The scale bar corresponds to 1 μm .

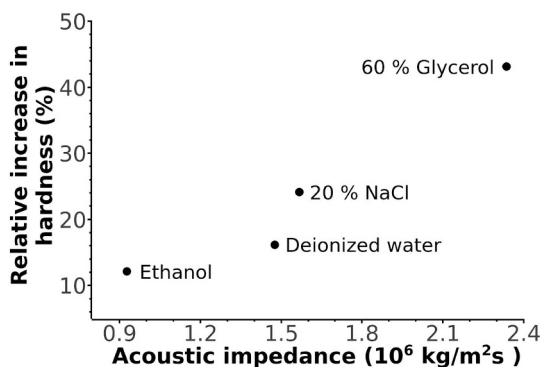


Fig. 7. Comparison of maximum relative increase in surface hardness of stainless-steels laser peened in different media of confinement at different respective acoustic impedances. The points corresponding to Ethanol and 20% NaCl are for SUS316 and the other two points corresponding to Deionized water and 60% Glycerol are for SUS316L.

impedance of the confinement medium, which is shown in Fig. 7. Since acoustic impedance is a product of the density and corresponding speed of sound in that medium it is therefore a very relevant parameter for the confinement effects of the medium. As can be seen in Fig. 7, the relative increase in the hardness rises monotonically with the acoustic impedance in the range of this parameter used in the present study. This is plausible because if the confinement medium is denser and shock wave travels faster in that the resulting containment of the shock energy and its delivery to the target metal will be more effective. Here one may argue that if the speed of sound is higher in a medium, the total duration of the shock exerted on the target might be shorter. But as we know the total duration of the shock applied on the target metal also depends on the thickness of the confinement medium and we expect to find an optimum duration of the shock that would result in the maximum peening effect in the target metal.

4. Conclusions

Two important inferences can be drawn from this experimental study on the fundamental role of the confinement media used during the LSP in the ultra-short laser pulse durations regime. First, in this ultra-short pulse duration regime of LSP, the non-linear mechanisms like self-focusing, filamentation, plasma breakdown and bubble formation at high repetition rate in the confinement media can

significantly attenuate the input energy of the laser pulses. It is therefore of paramount importance to characterize these adverse effects comprehensively so that their influence is minimized while carrying out the LSP in ps - fs pulse regime. Second, it is found that the resulting surface hardness of the peened metal as a function of laser fluence depends significantly on the confinement media and the relative increase in the hardness increases monotonically with the acoustic impedance of the liquid of the confinement medium. Since LSP is a technology of industrial potential, it is imperative to accurately and comprehensively establish the role of different confinement media in this technology. Further experiments are underway to get deeper insight into the role of confinement media in the ultra-short pulsed laser shock peening.

CRediT authorship contribution statement

Kishore Elango: Investigation, Writing - original draft. **Jan S. Hoppius:** Investigation, Methodology. **Lalit M. Kukreja:** Conceptualization, Writing - original draft. **Andreas Ostendorf:** Conceptualization. **Evgeny L. Gurevich:** Conceptualization, Writing - review & editing, Project administration.

Declaration of competing interest

We wish to confirm that there are no known conflicts of interest associated with this publication and there has been no significant financial support for this work that could have influenced its outcome.

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