# P1-4 The Role of Sub-Target for Soft Sample in Laser-Induced Shock-Wave Plasma

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## Introduction

Laser plasma generated as the result of the interaction between high-power pulse laser and solid target is an interesting phenomenon not only from the point of view of high-temperature hydrodynamics, but also because of its practical applications to certain fields. Recently, in our previous study(1-6), we have shown that the laser plasma induced by the laser irradiation at low pressure is an excellent source for emission spectrochemical analysis. We proved that the laser-induced plasma consists of two distinct regions. First is a small area of plasma (primary plasma) which gives off an intense continuos emission spectrum for a short time just above the sample surface; the second region (secondary plasma) expands with the time around the primary plasma, emitting sharp spectral lines with extremely low background. On the basis of our characteristic technique of time-resolved emission spectroscopy using a TEA $CO_2$ laser, we demonstrated that atoms in the secondary plasma are excited by the shock wave, while the primary plasma acts as an initial explosion energy source. We have referred to this method as laser-induced shock wave plasma spectroscopy (LISPS)(7-9).

Generally speaking, the characteristics of laser plasma depend on many factors involved in the process. Aside from being influenced by the surrounding gas and the various parameters of the laser itself, such as its wavelength, pulse width and power density, the hardness of the target also influences the plasma generation. In fact, in LISPS using TEA $CO_2$ laser, some problems were observed in the case of a soft material, such as a low melting point glass(11) or biological samples, where shock wave plasma could not be generated. We understood this phenomenon by assuming that the soft target absorbs recoil energy and atoms gushing from the primary plasma do not acquire sufficient speed to form a shock wave. If the process is true, we can overcome this problem by setting a sub-target on the backside of the soft sample so as to produce the repulsion force by which the gushing speed of the atom is increased. In order to prove this, the present study was undertaken using mainly silicone grease as a sample and copper plate as the sub-target.

## Experimental Setup

A TEA $CO_2$ laser (Lumonics, multi gas laser, model HE-440B, set at 100 mJ, FWHM 100 ns) was used in this experiment. However, the actual power focused on the target was roughly 50 mJ. During the experiment, the laser was operated shot by shot and power fluctuations were found to be less than 5%.

The laser radiation was focused by a ZnSe lens through a ZnSe window onto the surface of the sample. The radiation of the laser plasma was observed at a right angle to the laser beam with the use of an imaging quartz lens (f = 100 mm). The sample was placed in a small, vacuum-tight metal chamber (75 mm x 75 mm x 75 mm), that could be evacuated with a vacuum pump, and filled with the desired surrounding gas. The chamber pressure was measured precisely with a digital Pirani gauge (Diavac Limited, model PT-1DA). The sample, together with the whole chamber and focusing lens, could be moved in two dimensions relative to the laser beam by means of a stepmotor in the y-direction and a micrometer screw in the x-direction.

When plasma was observed, the plasma light was imaged 1:1 by a quartz lens (f = 100 mm) with an aperture of 10 mm x 10 mm onto the entrance slit of a monochromator (Spex, model M-750, Czerny Turner configuration, focal length 750 mm, grating 1200 grooves/mm blaze at 500 nm). The output of the photomultiplier (Hamamatsu IP-28) was then fed to a digital-sampling-storage scope (HP 54610 B, 500 MHz) after passing through low-impedance circuit.

When the emission spectrum of the plasma and the incandescent light of the droplets' vapor was taken, a gated intensified OMA (Princeton IRY 700) was used and the synchronization signal was also regulated by the external trigger function of the laser system.

For atmospheric experiment, helium gas was flowed inside the chamber at 1 atm and a concave mirror was placed at the side of the chamber to make plasma's image became three times bigger on the slit plane of a monochromator as shown in figure 1.

The sample used in this experiment were high-vacuum silicone grease, which painted with a thickness of roughly 100-micron on the sub-target surface. The sub-target material used in this experiment was copper (Rare Metallic, 4N, thickness 0.2 mm).

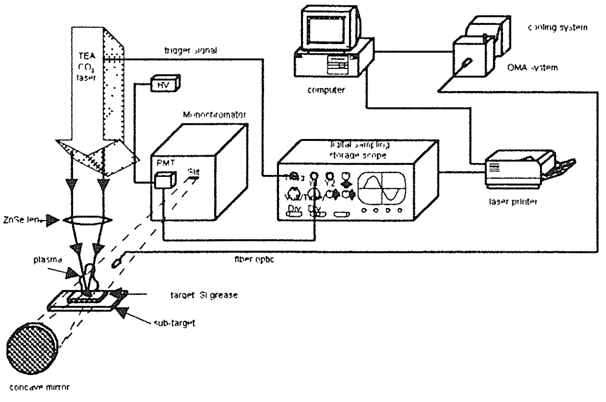


Fig. 1. Experiment Setup for atmospheric pressure

## Results and Discussion

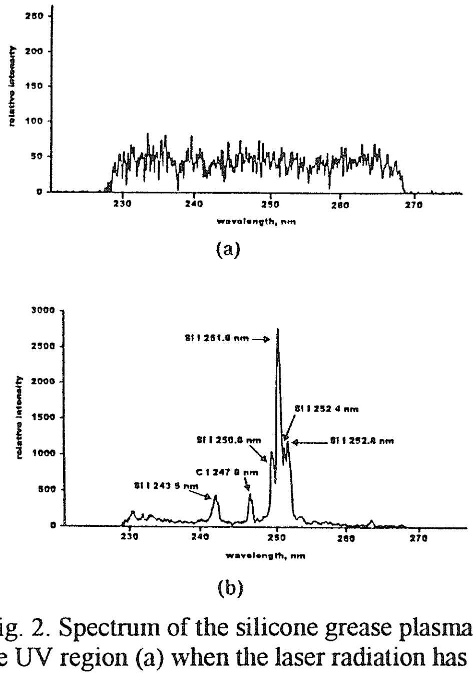
The repeated irradiation of laser light was made on the silicone grease painted on copper plate at fixed position in the surrounding air at 1 Torr. It was observed that when the laser light had not yet reached the hard sub-target, only incandescent emission light took place. It was supposed to be due to the heated particles coming from the grease sample. After the laser light reached the sub-target, the bright emission of the secondary plasma could be seen. However, after more than three shots, no plasma generation took place even though laser irradiation was repeated. This means that the grease layer at the focused point on the surface of the sub-target was completely removed by the laser irradiation.

It is assumed that when the surface of the target is soft, the expulsion of atoms by the surface is weakened because the softened surface absorbs the recoil energy and the atoms gushing from the primary plasma do not acquire sufficient speed to form the shock wave. On the other hand, when the hard sub-target is placed on the back in tight contact with the sample, the forward momentum of the gushed atoms does not weaken without absorbing the energy. We have already proposed the model to explain the generation of shock wave plasma. Namely, by the action of atoms gushing from the target, the induced adiabatic compression of the surrounding gas creates a shock wave. As a result of the compression, the kinetic energy of the propelled atoms is converted into heat energy in the plasma.

Figure 2 shows the spectrum in the UV region obtained (a) at the initial laser irradiation, when the laser radiation has not yet arrived the sub-target and (b) when the laser irradiation has attacked the sub-target. These spectra were collected by single-shot irradiation using the gated mode OMA with an exposure time of 100µs. It can be clearly seen that before the laser light reaches the sub-target, no atomic emission line was observed in the spectrum but only a dark noise. The atomic emission observed in (b) is attributed mainly to Si atom. We also confirmed the detection of a strong Si 288.1 nm emission line ($3p^2\ {}^1D$-$4s^1P^o$) whose excitation energy is as high as 5.08 eV. Considering the strong emission from the excited state of Si, it is assumed that the temperature of the secondary plasma is more than several thousand degrees.

Figure 3 shows the speed of the heated particles and the atoms gushing from the sample. It was found that, in the absence of a secondary plasma, the speed of the incandescent light from the gushing particles was very low, less than Mach 7 and almost constant during transmission in the ambient gas. It was examined that from the incandescent particle, weak continuos emission spectrum took place using the gated intensified OMA in visible region. From these results it is seen that the particles which give emission are not light particles, but rather weight particles or droplets because if they were light particles the speed would soon decrease due to collision with the ambient gas.

When a secondary plasma was generated, the speed of the silicon atoms was measured using Si 298.7 nm and was as high as Mach 55 near the target (3 mm), decreasing to Mach 30 at 6 mm above the sample surface.



(a)

(b)

Fig. 2. Spectrum of the silicone grease plasma in the UV region (a) when the laser radiation has not reached the sub-target and (b) when the laser radiation reaches the sub-target

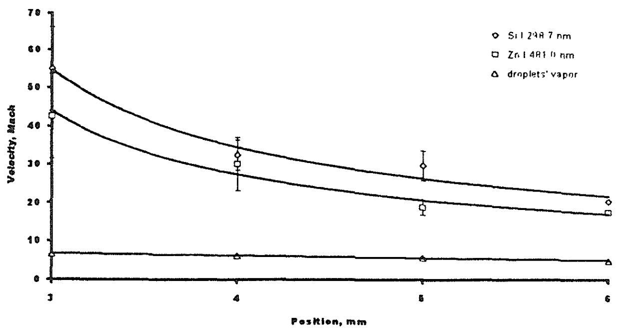


Fig. 3. The relationship between the velocity of the Si I 298.7 nm, Zn I 481.0 nm and incandescent emission light as a function of plasma position

These data were derived from the result of the relationship between the slit position and the rising time, which was obtained by varying the position of the chamber together with the focusing lens. In order to compare the result of Si plasma with ordinary laser-induced metal plasma, we used a brass sample as the target without grease. The curve is also shown in figure 3.

Figure 4 shows the relationship between time and the displacement distance of the front of the emission, which was observed by reading the rising point of the secondary plasma for grease and brass plate. It can be clearly seen that, for both cases, the slope is near 0.4, which is in good agreement with the theoretical result derived by Sedov(14) for a blast wave explosion. We have already proved that the zinc plasma induced by TEA $CO_2$ laser irradiation is excited by shock wave. Therefore it is concluded that shock wave plasma can also be produced even in the soft material when the suitable sub-target is placed at the back of the sample

We also made the experiment at atmospheric pressure in order to prove that even in atmospheric pressure, shock wave is necessary to generate plasma and thus sub-target was needed to help the plasma generation for soft target. Under the surrounding gas Helium at 1 atm, we measured the rising point of Silicone line and Helium line. We noted that the rising point of Silicone line takes place at the same time as Helium, which is shown in figure 5. We also found out that the slope of the curve of the relationship between the time and the displacement distance is almost the same as the case for low pressure.

Figure 6 shows how the emission continues when the laser light was focused repeatedly on silicone grease at a fixed position in Helium surrounding gas at 1 atm. It is noted that the plasma of soft sample still can be detected after 1000 shots. This result is different from the low pressure case. It is probably due to the uncleanness of the target's surface when the sample placed at atmospheric pressure, since the surface remains wet because by the heat due to repeatedly irradiation, the grease layer melted; in case of the low pressure, all the grease layer already gushing out and not remain on the surface. By this method we can make analysis of soft sample in the wide-open area without special vacuum chamber.

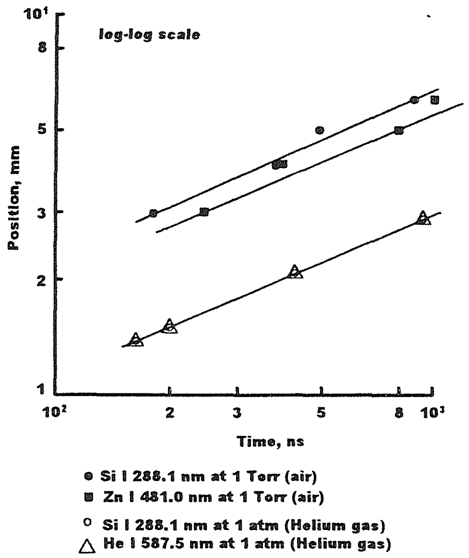


Fig. 4. The relationship between the time and the distance of the rising point of the emission Zn I 481.0 nm at low pressure and He I 587.5 nm at high pressure and also Si I 288.1 nm at both pressure

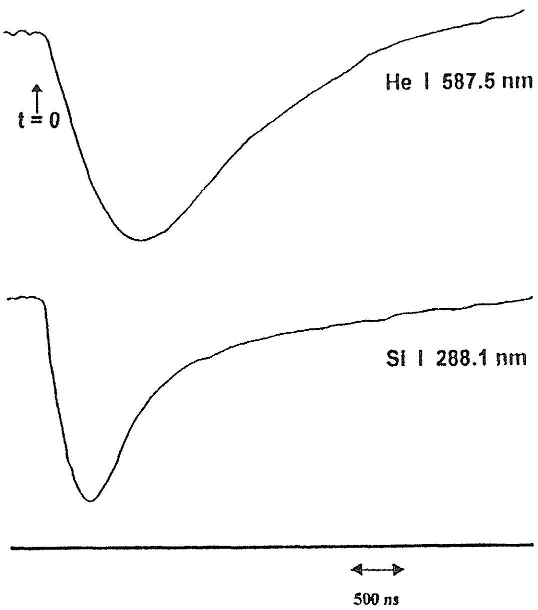


Fig 5. Time profile of the emission intensity observed at 1.3 mm in Helium at 1 atm

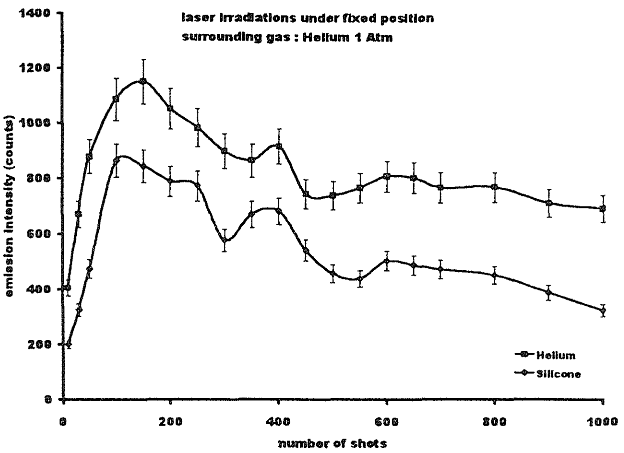


Fig.6. Relationship between intensity and number of laser irradiation at high pressure

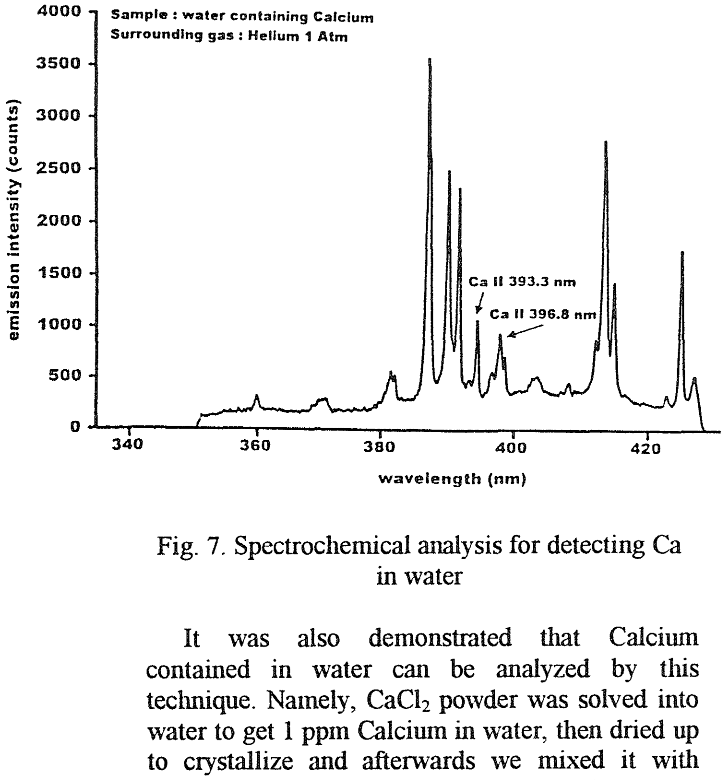


Fig. 7. Spectrochemical analysis for detecting Ca in water

It was also demonstrated that Calcium contained in water can be analyzed by this technique. Namely, $CaCl_2$ powder was solved into water to get 1 ppm Calcium in water, then dried up to crystallize and afterwards we mixed it with silicone grease. The grease containing Calcium was painted on the copper sub-target, and irradiated in Helium 1 atm. As the result, a very bright secondary plasma was observed with a nearly hemispherical shape after a few shots of laser irradiation and the emission of Calcium and Helium detected as shown in figure 7. Therefore, it can be said that this sub-target technique can be applied to the spectrochemical analysis of liquid sample; in general, such liquid sample can not be used as a sample for laser ablation atomic emission spectrometry.

Therefore, we can say that the sub-target effect can be effectively employed for practical application in the quantitative analysis for soft and liquid samples.

## Conclusion

It has been proved that in the case of soft samples, such as silicone grease, shock wave plasma can not be produced. However, when we place a sub-target on the back of a sample, shock wave plasma is generated. It is believed that in the absence of a sub-target, the expulsion of atom is weakened because the soft surface absorbs recoil energy, and atoms gushing from the primary plasma do not acquire sufficient speed to form shock wave. The main role of the sub-target is to produce repulsion force for atom gushing with high speed. Those experimental results obtained in this study also become strong evidence that support our model, which explains the mechanism of laser-induced shock wave plasma. It should be noted that this sub-target method could be successfully utilized to realize highly sensitive and rapid quantitative analysis for soft samples.

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