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# Effect of distances between lens and sample surface on laser-induced breakdown spectroscopy with spatial confinement

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## Abstract

Spatial confinement can significantly enhance the spectral intensity of laser-induced plasma in air. It is attributed to the compression of plasma plume by the reflected shockwave. In addition, optical emission spectroscopy of laser-induced plasma can also be affected by the distance between lens and sample surface. In order to obtain the optimized spectral intensity, the distance must be considered. In this work, spatially confined laser-induced silicon plasma by using a Nd:YAG nanosecond laser at different distances between lens and sample surface was investigated. The laser energies were 12 mJ, 16 mJ, 20 mJ, and 24 mJ. All experiments were carried out in an atmospheric environment. The results indicated that the intensity of Si (I) 390.55 nm line firstly rose and then dropped with the increase of lens-to-sample distance. Moreover, the spectral peak intensity with spatial confinement was higher than that without spatial confinement. The enhancement ratio was approximately 2 when laser energy was 24 mJ.

**Keywords:** laser-induced breakdown spectroscopy, LIBS, spatial confinement, distance of lens-to-sample, signal enhancement

(Some figures may appear in colour only in the online journal)

## 1. Introduction

The laser-induced breakdown spectroscopy (LIBS) technique was first proposed by Brech and Cross in 1962 [1]. The laser is focused onto the sample to make it atomize, and plasma is generated. The formation of plasma only occurs when the focused laser reaches a certain threshold, which generally depends on the environment and the target material. In principle, LIBS can analyze any matter, whether its physical state is solid, liquid or gas [2, 3]. Since all elements can emit light

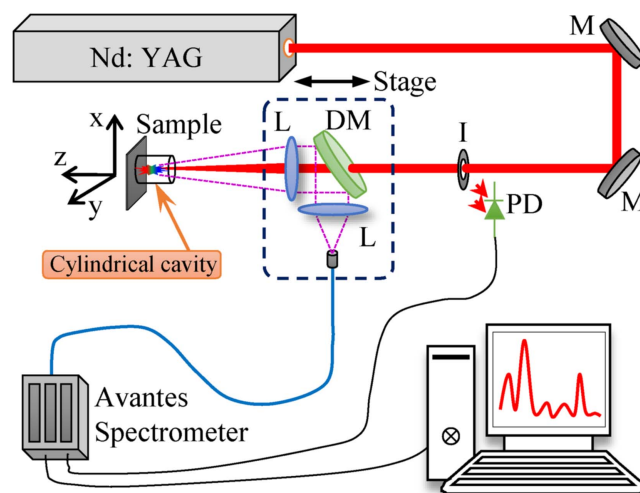
with characteristic wavelength when they are excited to a high enough temperature, all elements can be detected by this technique, which is limited by many factors, such as the laser energy, the wavelength range of spectrograph, and the sensitivity of detector.

LIBS technology is favored by researchers because of its many advantages, such as real-time analysis, multi-elemental analysis, simple or no sample preparation requirements, and remote analysis [4–7]. In recent years, this technology has been widely used in many fields. However, the problem of low detection sensitivity of LIBS technology is becoming more and more obvious [8, 9]. Therefore, improving spectral

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intensity has become the focus of researchers. A large number of previously published reports have showed that spectral intensity is affected by several parameters [10–18]. For instance, researchers reported the effect of distance between focusing lens and target surface on laser-induced plasma [19, 20]. Multari *et al* studied the effect of lens-to-sample on laser-induced plasmas in air using spherical and cylindrical lenses. It was found that atomic emission intensities, plasma temperature and ablated material mass depended strongly on the distance between lens and sample surface for both types of lenses [21]. The fundamental reason why the distance can change the spectral intensity is the change of the spot size. Li *et al* investigated the influence of spot size on the expansion dynamics of laser-induced copper plasma expansion in air. It was found that when the laser energy was fixed, the same spot size would produce similar expansion dynamics, no matter whether the target sample was located in front or behind the focal point [20]. Harilal *et al* investigated the spot size effects on the extreme ultraviolet conversion efficiency (CE) of CO<sub>2</sub> laser-produced Sn plasmas. This result showed that CE was to be lower at the focal position. The maximum emission intensity and CE could be obtained by selecting the optimum lens-target position [22]. Aguilera *et al* investigated the dependence of the distributions of Fe and Ar lines on the laser focusing distance, they found that a significant variation was obtained, regardless the shape of the distributions and the magnitude values [23].

In addition, it is well known that spatial confinement can significantly enhance the optical emission intensity. A powerful laser beam focuses on solid, liquid, or gas targets to produce a luminous hot spark, this is called laser-induced plasma. With the generation of the plasma, a shock wave is produced. Spatial confinement is actualized when the plasma is confined to a small region by a certain obstacle, the shock wave encounters obstacles in the process of expansion, and it will be reflected back into the plasma center to compress the plasma plume, producing an enhanced spectral signal. Spatially confined laser-induced plasma has been studied by many researchers. Shen *et al* investigated the spatial confinement effect in laser-induced plasma of aluminum sample by optical emission spectroscopy and fast photography in air [24, 25]. An obvious enhancement in the spectral intensity of Al (I) line was observed. Guo *et al* studied the accuracy of the quantitative analysis improvement of low concentration elements from LIBS measurement using spatial confinement [26]. They improved the enhancement effects by the spatial confinement and dual-pulse in LIBS [27], and investigated the enhancement effects by applying combined spatial and magnetic confinements in LIBS [28]. Gao *et al* investigated the role of spatial confinement by using fast imaging and optical shadow imaging [29]. The results showed that the shape of the plasma plume became narrower and longer with spatial confinement. The spectral enhancement was attributed to the reflection of the shock wave by the wall. Wang *et al* presented an experimental and computational study on LIBS for both unconfined flat surface and confined cavity cases [30]. Li *et al* investigated the spatial confinement of plasma by using time resolved spectroscopy, fast imaging,



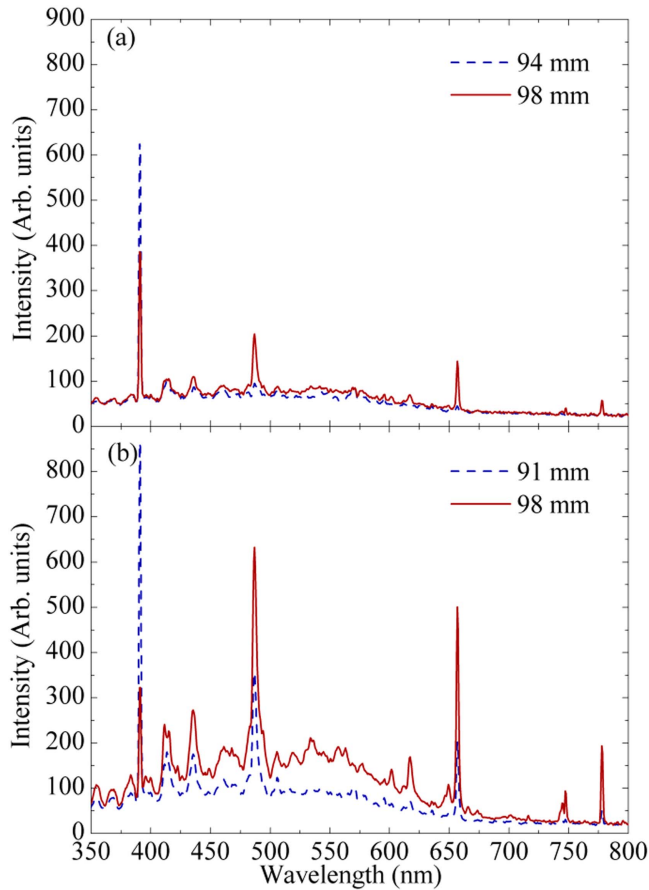
**Figure 1.** Experimental setup for spatially confined laser-induced Si plasma with cylindrical cavity. The components include mirror (M), iris (I), lens (L), DM (dichroic mirror), and photodiode (PD). Cavity diameter is 2 mm, height is 2 mm.

interferometry, and numerical computation. Experimental results confirmed that the plasma was constricted by the reflected shockwave associated with a temperature and density gradient. Li *et al* studied the dependences of spectral signal enhancement on relative permittivity by varying materials to confine the plasma [31]. They found that higher relative permittivity rings induce stronger enhancement ability, which restricts the energy dissipation of plasma better. These works investigated the effect of the spatial confinement of the LIBS signal, these studies mainly focused on the understanding plasma characteristics and improving the enhancement effect [24, 25, 32–34].

In this paper, we carried out this experiment to investigate the effect of distance between lens and surface sample on the laser-induced silicon breakdown spectroscopy with spatial confinement for different laser energies. It was found that the intensity of spectral line first increased and then decreased with the distance of lens-to-sample, when the laser energy was fixed. And an optimized spectral intensity at a certain distance of lens-to-sample could be obtained. Moreover, the optimized spectral intensity with spatial confinement was much higher than that without spatial confinement.

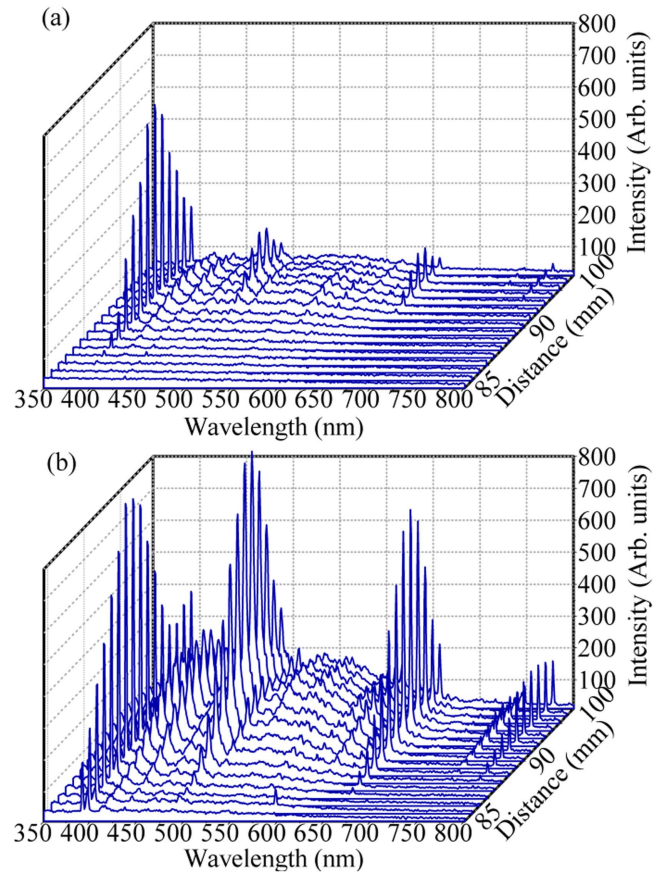
## 2. Experimental setup

The schematic drawing of experimental setup used in this study for optical emission spectroscopy is shown in figure 1. The used laser system was a Q-switched Nd:YAG laser (Continuum, Surelite III). The full-width at half maximum (FWHM) of the pulse is 10 ns, the maximum output energy is approximately 800 mJ, the wavelength is 1064 nm, and the repetition rate is 10 Hz. The sample (Si(100), MTI KJ Group,  $500 \pm 10 \mu\text{m}$  thickness) was mounted on a computer-controlled X-Y-Z stage (Thorlabs, PT3/M-Z8), which guaranteed that the sample location was renewed before each laser shot. The surface of the sample was placed at an angle of 90° with



**Figure 2.** Emission spectra of laser-induced Si plasmas for two laser energies at different distances of lens-to-sample. Distances of lens-to-sample are 91 mm, 94 mm, and 98 mm. Laser energies are 12 mJ (a), and 24 mJ (b).

respect to the direction of the laser beam. The laser beam was focused onto sample surface by using a plano-convex quartz lens with the focal length of 100 mm and the diameter of 25.4 mm. The surface of the lens is parallel to the surface of the sample. The cylindrical confinement cavity was placed tightly on the surface of the sample, and the plasma plumes were generated in the center of the cylindrical cavity. The diameter of the cavity used for this experiment is 2 mm, its material is the aluminum. The height of the cavity is 2 mm. The emission spectra were reflected by a dichroic mirror between the sample and focusing lens, which was placed at an angle of  $45^\circ$  to the laser beam. The dichroic mirror was transparent to the laser light of 1064 nm but reflective to the visible light. The reflected spectra were focused into a fiber using a lens (BK7), which were orientated parallel to the laser beam. The fiber tip was positioned using a manual 3D translation stage. The spectra were guided to a spectrometer (Avantes, AvaSpec-ULS2048) through the fiber. A photodiode received scattered light of iris to trigger the spectrometer, which guaranteed time synchronization between laser signal and spectral signal. The final measured data was recorded by a computer. By moving the position of the focusing lens, the distance of lens-to-sample is changed. The



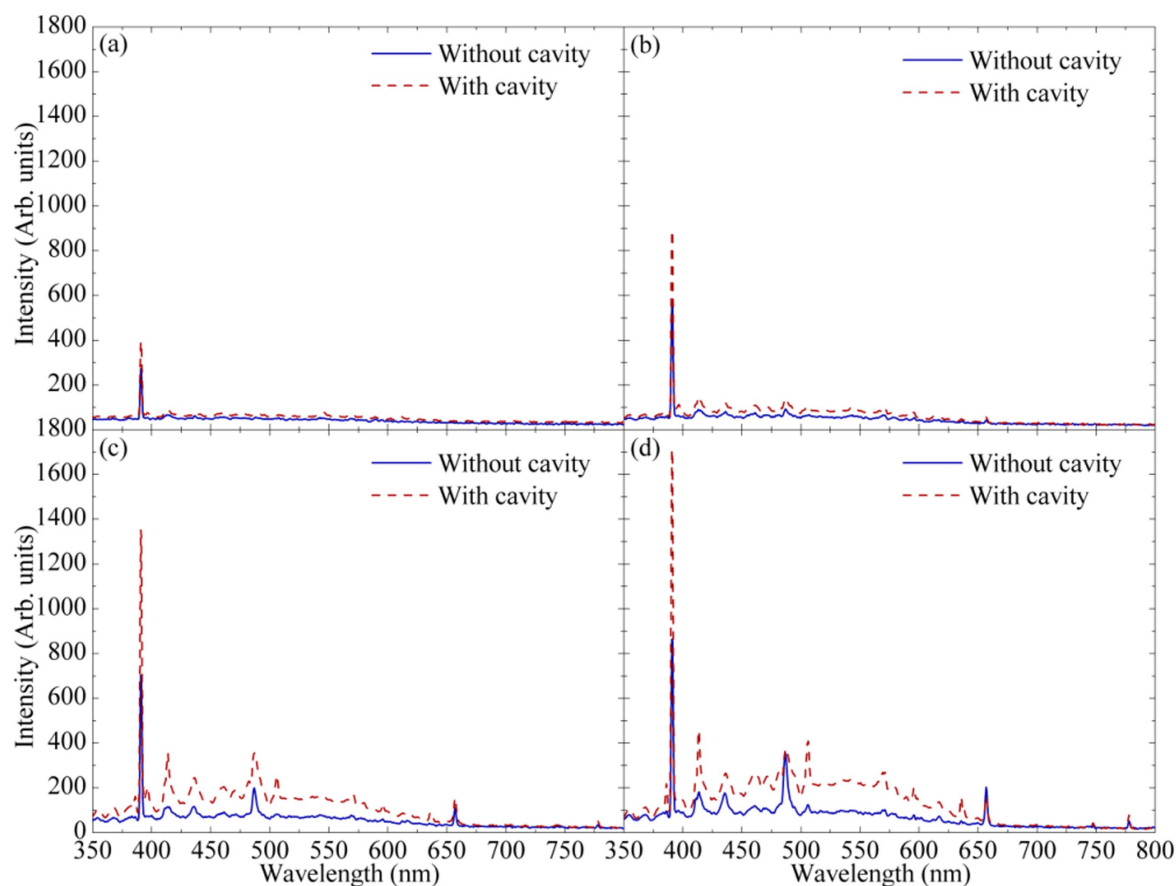
**Figure 3.** Distribution of spectral intensity with distance and wavelength. Laser energies are 12 mJ (a), and 24 mJ (b).

entire experiment was carried out in air. Each data point was typically an average of 10 shots.

### 3. Results and discussion

Optical emission spectroscopy technique is widely used to study the behavior of different kinds of atomic lines in processes of laser ablation. Each atomic emission line represents an electron excitation of a particular component in plasma plume. By analyzing the emission lines of luminous plasma, it is easily to deduce the characteristics of components (excited atoms and ions).

Spectral emission intensity can be affected by the distance of lens-to-sample [35]. In order to understand the influence of distance between the focusing lens and sample surface on emission intensity of LIBS, typically three distances of 91 mm, 94 mm and 98 mm were selected to compare spectra of laser-induced Si plasma, as shown in figure 2. For figure 2(a), the spectral emission intensity of neutral atomic line with the wavelength of 390.55 nm at the distance of 94 mm is higher than that at the distance of 98 mm. In figure 2(b), when the energy increases to 24 mJ, similar trend can also be observed. It can be seen from these results that the distance between lens and sample surface can change the emission intensity of spectral line. We have already mentioned that this experiment is carried out in the atmospheric



**Figure 4.** Comparison of spectral intensities with and without spatial confinement. The laser energies are 12 mJ (a), 16 mJ (b), 20 mJ (c), and 24 mJ (d). Lens-to-sample distance is 91 mm.

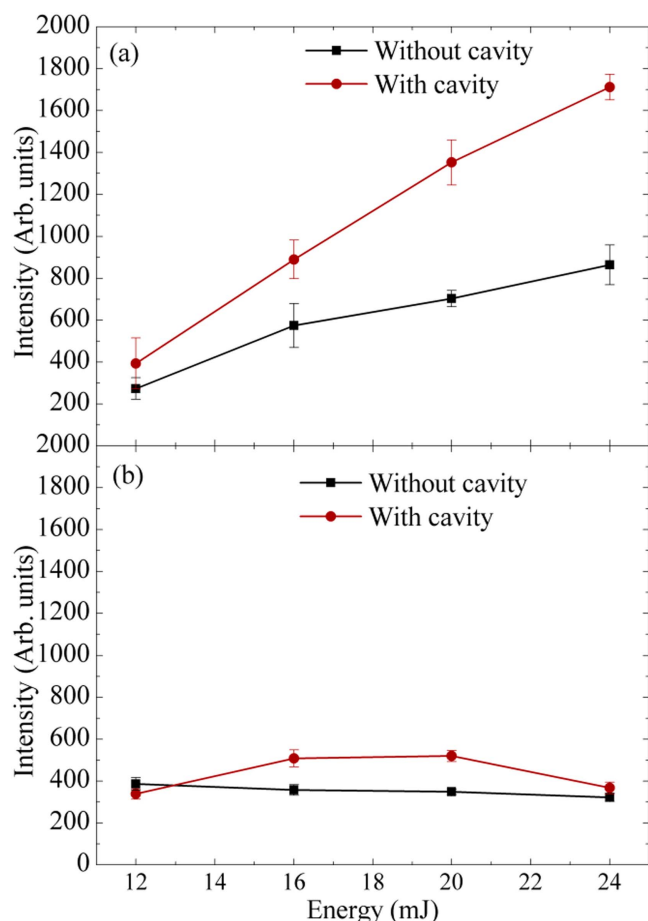
environment. A beam of high energy laser pulse irradiates the sample, and the beam is focused on a small analytical point on the sample surface. In the laser spot region, the material in the sample is ablated due to the light absorption and multi-photon ionization processes. Many atoms, molecules and other particles are formed over the sample, the plasma is gradually formed [12]. The optical emission intensity depends strongly on the produced plasma.

We measured the distribution of the plasma emission intensity with the distances between the lens and sample surface in the range from 350 nm to 800 nm, as shown in figure 3. The purpose is to investigate the influence of the lens-to-sample distance on optical emission spectrum in LIBS. The laser energies are 12 mJ and 24 mJ. From the distribution of emission intensity, it can clearly be seen that the spectral emission intensity first increases and then decreases with the increase of distance, no matter what the wavelength is. Therefore, it is further indicated that the lens-to-sample distance can significantly affect the spectral emission intensity. In addition, the spectral line intensity has the maximum value at a certain lens-to-sample distance. In essence, the change of lens-to-sample distance is equivalent to changing the spot size [36]. For a shorter distance between lens and sample surface (large spot size), the plasma plume exhibits a hemispherical structure along the lateral and axial directions. When the distance increases, the spot size

decreases. Small spot size gives the plasma plume a stream-like structure [20]. In the current experiment, when the laser energy is fixed, the lens-to-sample distance is shorter, the spot size is larger, while laser fluence is lower. Therefore, spectral intensity is weaker when sample surface is away from the focal point. With the increase of lens-to-sample distance, laser fluence becomes larger leading to the increase in spectral emission intensity. Continue to increase the distance (close to the focal point), the laser fluence is growing stronger and stronger, this process is accompanied with the enhancement of the plasma shielding effect [20]. This effect becomes more and more obvious, the coupling of the laser energy and the target is reduced leading to the decrease of the ablation mass. Finally, spectral intensity is also decreased.

In addition, laser energy plays an important role in plasma emission intensity. With the increase of laser energy, the ablation area of the interaction between laser and target material can be obviously increased, the ablated mass is increased, eventually the emission intensity will be increased [37]. From figure 3, the emission intensity with laser energy of 24 mJ (figure 3(b)) is higher than that with laser energy of 12 mJ (figure 3(a)). Due to the increase of laser energy, the target material will absorb more laser energy. The spray diameter on the surface of the sample will become larger, and ablation mass of target material increases, leading to a stronger emission in the spectral intensity. In nanosecond





**Figure 5.** Evolution of spectral intensities of Si (I) 390.55 nm with and without spatial confinement as functions of laser energies. Lens-to-sample distances are 91 mm (a), and 98 mm (b).

laser ablation, laser pulse is divided into two edges: the leading edge and the trailing edge of the nanosecond laser pulse in LIBS. The leading edge interacts with the target material producing the plasma, and the trailing edge is used to heat the generated plasma [38]. In the processes of laser ablation, after the leading edge of the nanosecond laser pulse, the atoms, ions and electrons constitute the ultra-dense cloud clusters over the sample. They formed silicon plasma on the target surface, which consumes a small fraction of the energy. The trailing edge of nanosecond laser pulse can heat the plasma with the remaining laser energy. Therefore, when laser energy is larger, more energy will be used to heat the plasma, leading to the increase of the collision rate of particles in plasma, leading to the increase in the emission intensity.

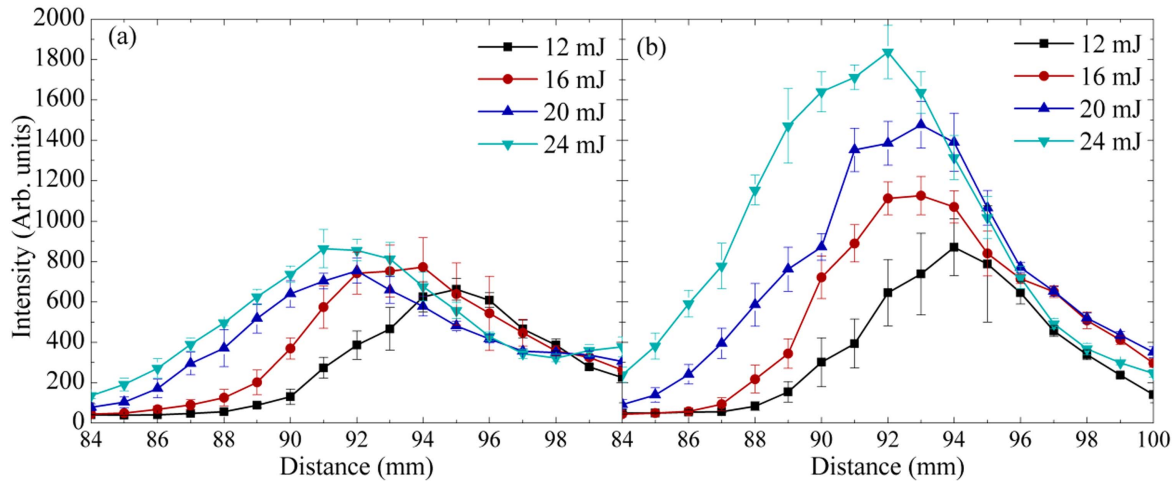
The spatial confinement can significantly enhance emission intensity of LIBS. Figure 4 shows the comparison of spectral intensities with and without spatial confinement at the lens-to-sample distance of 91 mm. The laser energies are 12 mJ, 16 mJ, 20 mJ, and 24 mJ. It can be seen that emission intensity with spatial confinement is higher than that without spatial confinement. For the spatial confinement, the intensity of Si (I) 390.55 nm line increases significantly with the increase of laser energy. At the laser energy of 24 mJ, the enhancement ratio is approximately 2. The reason is that

spatial confinement can enhance spectral intensity due to the compression of plasma plume by reflected shockwave [29, 30, 39, 40]. The ambient air pressure around the surface of laser ablated sample increases rapidly, therefore, a shock-wave is generated. The reflected shockwave compresses plasma plume causing the increase in plasma temperature and electron density, so that the emission intensity is enhanced [41–44].

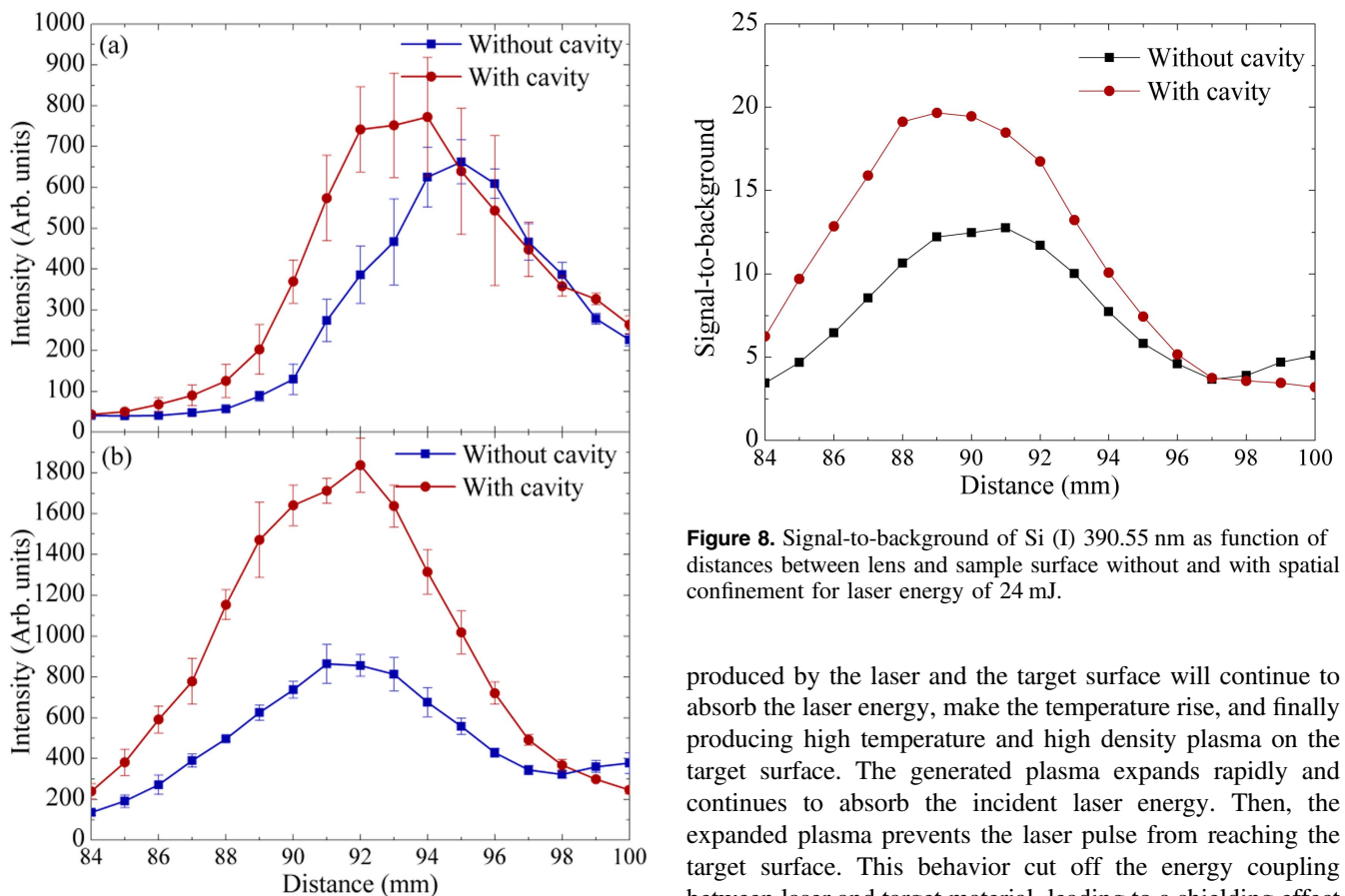
Figure 5 shows the evolution of spectral intensities of Si (I) 390.55 nm with spatial confinement and without spatial confinement. The distances are 91 mm, and 98 mm. As can be seen from figure 5(a), the spectral line intensity increases with the increase of laser energy. And, the increasing trend is more obvious when cylindrical cavity is presented. Since, laser pulse with higher energy produces stronger plasma, and the stronger interaction between laser and target is accompanied by stronger shockwave. Therefore, the stronger shockwave can compress the plasma more effectively. This produces higher temperature and higher density plasma resulting in stronger optical emission. However, as shown in figure 5(b), when the distance of lens-to-sample is 98 mm, the intensity of spectral line almost keeps constant with the increase of laser energy. This distance is almost close to the focal point, so that laser fluence is high enough to appear plasma shielding effect [45].

Figure 6 shows the evolution of spectral emission intensities of Si (I) 390.55 nm with the distance between lens and sample surface without and with cylindrical cavity. The laser energies are 12 mJ, 16 mJ, 20 mJ, and 24 mJ. From figure 6(a), the intensity increases with the increase of laser energy. At the same time, the distance corresponding to the maximum intensity moves towards the opposite direction of the laser beam with the increase of laser energy. In other words, when the energy is 12 mJ, the maximum emission intensity appears at the distance of 95 mm; at the energy of 24 mJ, the distance is 91 mm. When the cylindrical cavity is added (figure 6(b)), the emission intensity increases significantly with the increase of laser energy. Similarly, as the laser energy decreased, the distance corresponding to the maximum intensity moves towards the direction of focal point.

Moreover, the curves after appearing the maximum values begin to drop, which may be caused by two reasons. One reason is the increase in the emission intensity of the ionic spectral line. The energy of the incident laser is sufficient to affect the ionization process and subsequent plasma generation [46–48]. Firstly, the cascade ionization contributes to the initial ionization process. When the sample surface is irradiated by a high-energy laser pulse, the atoms absorb photons and then are ionized, leading to generation of initial free electrons in the focusing region of laser pulse. With the increase of laser energy, free electrons accelerate and accumulate energy continuously. Once the energy is large enough, they will bombard atoms. These atoms are ionized and further produce new electrons. These newly generated electrons also undergo accelerated, storing energy and bombarding the atoms, leading to the atoms continue to be ionized, the ion content increases continuously, and eventually forming the



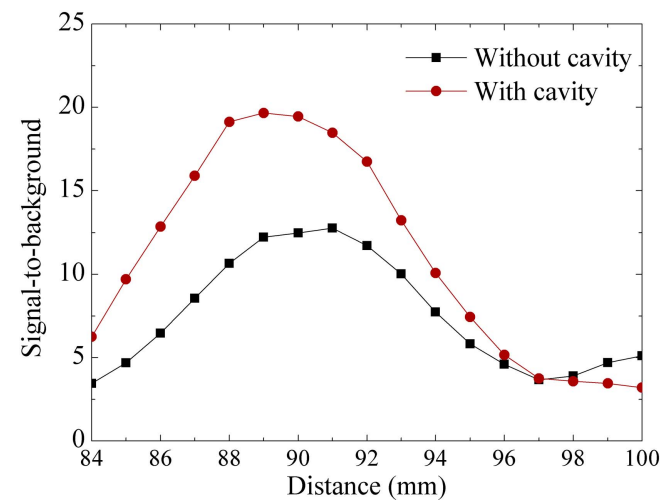
**Figure 6.** Spectral intensities of Si (I) 390.55 nm as function of distances between lens and sample surface without spatial confinement (a) and with spatial confinement (b) for different laser energies. Laser energies are 12 mJ, 16 mJ, 20 mJ and 24 mJ.



**Figure 7.** Spectral intensities of Si (I) 390.55 nm as function of distances between lens and sample surface without and with spatial confinement for two laser energies (12 mJ (a) and 24 mJ (b)).

cascade ionization. The free electrons generated by multi-photon ionization are more energetic, especially in the case of higher laser energy, which leads to more ions in the plasma plume.

Another reason is the shielding effect (usually, people call it the plasma shielding effect) for explaining the variation of the emission intensity with the distance. The plasma



**Figure 8.** Signal-to-background of Si (I) 390.55 nm as function of distances between lens and sample surface without and with spatial confinement for laser energy of 24 mJ.

produced by the laser and the target surface will continue to absorb the laser energy, make the temperature rise, and finally producing high temperature and high density plasma on the target surface. The generated plasma expands rapidly and continues to absorb the incident laser energy. Then, the expanded plasma prevents the laser pulse from reaching the target surface. This behavior cut off the energy coupling between laser and target material, leading to a shielding effect [23, 49]. As mentioned above, with the increase of the distance, the spot size becomes smaller and the laser energy density is larger. This process is accompanied by the enhancement of plasma shielding effect. The plasma shielding effect increases gradually after the maximum emission intensity value appears. With the increase of focused laser beam (the increase of the distance), this effect becomes more and more obvious, which leads to the decrease of ablation mass. Finally, the coupling of laser energy and target decreases, and the emission intensity of high temperature plasma decreases. Therefore, the decrease in emission

intensity can be attributed to the enhancement of the plasma shielding effect.

In order to further compare the maximum emission intensities with and without spatial confinement, spectral intensities of Si (I) 390.55 nm as functions of distances between lens and sample surface without and with spatial confinement at the same laser energy are shown figure 7. When the energy is 12 mJ (figure 7(a)), spatial confinement effect is not obvious on the line intensity. However, when the energy is changed to 24 mJ (figure 7(b)), the spectral line intensity is remarkably enhanced by using spatial confinement. And, under the optimal distance, line intensity is increased by 2 times. The condition of maximum spectral intensity with the spatial confinement is that the distance between the lens and sample surface is 92 mm at the laser energy of 24 mJ. Compared with lower laser energy, higher energy laser pulse can produce larger size plasma plume, and the accompanied shockwave is stronger. The interaction between these two factors leads to stronger emission. In addition, when the distance of lens-to-sample is close to focal point, the enhancement effect becomes weaker. In particular, laser energy is low (figure 7(a)), spectral enhancement can hardly be observed in the range from 95 mm to 100 mm. However, the distance is away from the focal point, the enhancement effect is better. The reason for the results is described as follows: when the distance of lens-to-sample is small, the spot size of the incident laser beam converging to the target surface through the lens is relatively large, the laser-induced plasma plume expands in both lateral and axial directions with a hemispherical structure [20], the lateral width of plasma plume is large. And, under the current experiment, the used confined cavity is cylindrical, the reflected shockwave compresses the plasma plume in lateral direction. So, the laterally expanded plasma plume can be effectively compressed. The enhancement effect is stronger. When the sample surface is getting closer and closer to the focal point, the converging effect of the focusing lens to the incident laser pulse is gradually enhanced, the spot size decreases. The laser-induced plasma plume expands rapidly in axial direction, the plasma plume forms a narrow cylindrical structure [50]. Similarly, the reflected shockwave can only compress the plasma plume in lateral direction, while the expansion of the plasma is mainly in the axial direction. The compression effect will be very weak. Especially, laser energy is low, the compression effect can almost be ignored. Therefore, the enhancement effect becomes weaker. In addition, figure 8 shows the signal-to-background of Si (I) 390.55 nm as function of distances between lens and sample surface without and with spatial confinement for laser energy of 24 mJ. It can be clearly seen from this figure that the signal-to-background with spatial confinement is higher than that without spatial confinement.

#### 4. Conclusions

In this paper, we experimentally investigated the effect of the distance between focusing lens and target surface on spatially

confined laser-induced Si breakdown spectroscopy with a cylindrical cavity. We clearly found that the lens-to-sample distance could obviously change the magnitude of the emission intensity. The spectral emission intensity first increased and then decreased as the lens-to-sample distance progressively increased. The curve decline may be due to the shielding effect. At the same time, the spatial confinement could also significantly improve the spectral emission intensity. When the laser intensity is 24 mJ, the enhancement factor of Si (I) 390.55 line from plasma plume was approximately 2 by using confinement cavity. And, the condition of obtained maximum spectral intensity with the spatial confinement was that the distance between lens and sample surface was 92 mm. This provided a better understanding of the influence of the distance between lens and sample surface on spatially confined LIBS.

#### Acknowledgments

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