Comparison of transient absorption of laser ablation plasma with fundamental plasma absorption relations

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Received: 24 February 2021 / Accepted: 30 May 2021 / Published online: 10 June 2021
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Abstract
Nanosecond pulsed laser ablation plasmas were studied by time resolved shadowgraphy coupled with normal imaging, followed by laser probing and plasma spectroscopy in the 5-25 J/cm² fluence regime. We describe methods for imaging and probing that allow us to determine variations in the distribution of ejecta in the plume and monitor the optical absorption using a probe laser to obtain a measure of the linear absorption coefficient of the plasma. Experimental determination of absorber distribution also corresponds well to the theoretical prediction of density increase near the emitted shockwave edge. We finally demonstrate that fundamental plasma correlations can accurately describe the absorption of light by the plasma near the ablation wavelength. We observed good agreement in peak attenuation, directly measuring 65% peak absorption and compared to a calculation of 57% using a simple model of the plasma, but a 10 ns shift in peak attenuation time. The shift in dip times is explained both by experimental error and a fundamental imprecision in the model proposed for the expansion.

Keywords Nanosecond laser ablation · Laser plasma · Time resolved imaging · Plasma shielding · Plasma spectroscopy

1 Introduction
Pulsed laser ablation is well-studied and broadly applied, both scientifically and industrially, yet it still proves to be one of the more thinly understood processes across the broad array of micromachining techniques. This is concerning, since laser ablation remains extremely commercially relevant and of significant scientific interest. In order to design processes that take advantage of laser ablation kinetics and enhance material removal rates, the dominant mechanisms must be well known. Previous studies of laser ablation consider thermal models that are simplified to enable predictions. While these models are useful in providing correlational evidence for the importance of various processes, they have lacked ability to completely capture the dynamics of material removal. The rapid advances in computational power and simulation have now rendered high powered laser processing within reach of precise and thorough computations. This has created a need for more detailed measurements, especially of transient intensive properties, to feed the computation. Additionally, for laser drilling applications, it is very important to have a qualitative understanding of the evolution of the crater morphology. To this end, high quality time resolved imaging of the ablation process is extremely important. There are some precise values that can be obtained by imaging of the ablation process, including plume radius, transmission, and surface reflectivity. The visualization of the structures themselves, however, can be practically more important, allowing easier identification of the important mechanisms.

Many measurements of optical properties of laser plasmas and particle plumes have been attempted, but the primary difficulty in interpreting these measurements is the broad set of possible physical processes at work. Within the crater, fluid motion, superheated nanoparticle generation, plasma emission, and absorption are contributors to the ablation process. We used flash imaging techniques to aid in accurate identification of mechanisms present in the laser ablation process. By using time resolved images, we can pick and choose where these processes are spatially segregated and probe them. Studies attempting to quantify plasma behavior have typically utilized probe beams in a parallel to the sample surface configuration. This technique has generated useful data [1–4], however it has limitations that we
have addressed in the present work to yield a better signal by perpendicular probe beam alignment.

Plasma shielding, where plasma is emitted from the surface during the pulse itself, absorbing some of the incident light [5], is known to affect laser processing rates. Understanding the timescales of the plasma absorption and its spectral properties is critical for process design to minimize this effect. The development of laser-induced breakdown spectroscopy (LIBS), [6, 7], has yielded good understanding of the spectral characteristics of the plasma plume emission. The absorption, more relevant to the plasma shielding, has been more difficult to quantify. Although fundamental relations yield accurate results for inverse Bremsstrahlung (IBA) in X-ray generation or steady state plasmas, the broad array of uncertain parameters in transient laser ablation processes has kept such relations from accurate application to laser-induced plasmas [8, 9]. One consideration is accounting for the scattering effects accompanying phase explosive particle ejection, which have long been theorized [10, 11]. Many attempts have been made to quantify the plasma onset, threshold, and intensity magnitude, in correlation with various models [1, 12–15], yet there remains significant uncertainty in applying them. Another confounding process stems from the fluidic effects that have been modeled theoretically and computationally [16–18] with good, albeit qualitative flow similarity. Modeling the plasma alone requires extensive spectral measurements. Due to these difficulties, we will focus on separating possible effects of phase explosion and fluid flow in our detailed measurements of the plasma. Instead of considering detailed dynamic models of the phase explosion process, we will use our imaging to identify key regimes of expected spinodal breakdown and avoid them in our probing.

In this study, we determine the plasma plume geometry via time resolved imaging from two viewing directions. Then, we use a laser probe to obtain a measure of the linear absorptivity close to the processing laser wavelength in conjunction with spectroscopic methods to determine the state of the plasma. Finally, using knowledge of the thermodynamic state of the plasma, as well as its spatial extent over time, we apply a simple model to estimate the expected attenuation of the probe beam by the plume. Their agreement provides evidence that fundamental plasma relations can accurately predict linear absorption near the processing laser wavelength.

2 Experiments and results

2.1 Normal imaging

We performed nanosecond resolution imaging from the normal direction using a homogenized profile, 3 ns, 532 nm illumination beam and a 1064 nm, 7 ns/170 µm FWHM processing beam at 17 J/cm² (Fig. 1) in bulk silicon. All images were captured with a Thorlabs DCU223M high resolution CCD camera. We passed the beam through a beam expander that was slightly defocused, causing a divergent processing beam. This widened the focused beam spot while maintaining a more highly focused image and matching the ablation crater diameter to the field of view. This effort was done in order to obtain a clear image of the crater, following works employing a similar technique done with femtosecond lasers in a standard microscope [19, 20]. The referenced studies, however, were performed with ultrashort pulses and were not utilized to detect plasma expansion. The band pass filter completely eliminated plasma emission from the image.

In the early stages of ablation, we can see immediate darkening of the crater. There are observable structures at negative times because the pulses are gaussian in time, indexed by rising half maximum times. For clarity, we have included energy deposition percentages for the first ten images calculated via the gaussian fit of the processing pulse. Previous studies have identified such early dark structures as indicative of expanding solid–liquid mixtures [19, 20]. In our studies, side imaging and plasma spectroscopy evidence that they are the result of IBA [21], proof of which is given in the ensuing paragraph. By 1.6 ns, the silicon in the region surrounding the crater lightens to a bright halo surrounding the crater exhibiting transition to a reflecting molten phase. This melt region expands, reaching a maximum radius at around 17 ns, then upon cooling and solidification forms a jagged solid/liquid interface edge. During the time light is irradiating the sample, there is a deep darkening of the ablation crater. We consider three possibilities as causes for this darkening: (1) the silicon plasma plume expelled from the sample directly absorbs the illumination beam, (2) nanoparticles ejected from the surface due to phase explosion or hydrodynamic spallation absorb or scatter the illumination beam, and (3) high spatial frequency ripples and roughness in the crater liquid bottom scatter the incident light. We can readily eliminate high crater roughness causing diffuse scattering by examining the timescale of fluid flow. The crater is clearly brightened by only 7.6 ns, yet in the next 66 ns, there is no observable evolution of the crater fluid floor, implying significantly different timescales between darkening and fluid flow. Additionally, we can see the darkness propagate radially over the solid area.

2.2 Side imaging

Although we know there will be some solid and liquid particles in the plume, they are not expected to contribute substantially to absorption and scattering on these timescales and create the dark cloud over the crater. In order to eliminate particle absorption and scattering as a cause for
the observed dark region, we performed side transmission imaging at 532 nm (Fig. 2). We can see the plume expanding hemispherically away from the surface, with the first absorption visible even before the rising half maximum of the processing pulse. The shockwave is barely visible at 66 ns. Between 10 and 17 ns, there is very rapid lightening and disappearance of the plume. Previous studies have also shown rapid decrease in the IBA effect in silicon over this timescale. Thus, we propose plasma cooling as the reason for the rapid increase in transmission through the plasma. If this increase in transmission of the plume was caused by particle absorption or scattering, the contrast would be expected to scale only with plume volume, yet the difference in plume radius is small between these two images. Therefore, some intensive property of the plasma cloud not directly related to density must be changing. However, temperature change and associated ionization stage change altering absorptivity could occur solely through radiative cooling, without rapid volume change, and therefore, it is our candidate cause. Further evidence will be presented later in the discussion of CW laser probing in Fig. 4. Additionally, ejected particles should have to travel at over 5 km/s. Although this velocity is reasonable for plasma, it is unlikely for larger emitted particles, as previous investigators have observed order of magnitude
slower particle ejection [12]. Based on this rationale, we determined that in the early nanoseconds of ablation, plasma must cause the crater and surrounding region to darken.

Following identification of the plume as a plasma, the distribution of absorbers through the plume must be determined. The Sedov solution for blast-induced shockwaves predicts mass accumulation at the edges of the expansion plume according to shockwave radius progression of the form of Eq. (1a) and density distribution near the plume core of the form of Eq. (1b) [8]:

\[ R = \xi \left(\frac{E}{\rho}\right)^{1/2} t^{2/5} \]  
\[ \rho \sim r^{-1/2} \sim t^{-6/5} \text{ as } r \to 0 \]

In the above, \( \gamma \) is the specific heat capacity ratio, and \( \xi \) is a dimensionless similarity variable of a value close to 1 and with a weak dependence on spatial dimensionality and specific heat capacity ratio. \( \rho \) is the density as a function of radius, \( r \). For \( \gamma = 1.4 \) in air, and the dimensionality, \( n = 3 \), \( \xi \) can be shown to be equal to 1.115 by solving mass, momentum, and energy conservation equations. These conservation equations also lead to the dependence indicated in Eq. (1b), which points to a hollow plume core as the density sinks to 0 for \( r \) approaching 0. Compressive shockwaves, in fact tend to have mass accumulation at the shockwave edge. To check if this Sedov blast wave approximation is applicable, we fit the solution to the \( R - t^{2/5} \) dependence expected and achieve good agreement (Fig. 3a). From the Sedov fits, we have velocities of over 10 km/s, far past the speed of sound in air of about 330 m/s, further intimating a plume shell type density distribution. Examining the side imaging, a density of absorbers of about 55 μm in thickness is evident along the edge as outlined in Fig. 3b. By normal probing, we can also time the duration of the probe signal dip due to absorption. Using the Sedov-Taylor fits, we calculate the thickness of the shell independently. This is done by timing the falling half maximum and the rising half maximum of the transmission drop. At said times, we calculate the expected radius, and subtract the two. This gives the expansion of the plume over the dip time interval. This process is diagrammed in Fig. 4b and yields a 60 μm shell thickness, agreeing closely with the imaging. We considered using the Sedov idealized shock-wave model to predict the density distribution as a continuous density distribution rather than the binary shell model indicated above. Jeong and colleagues previously indicated that transmission data do not achieve good agreement with the Sedov solution in laser plasmas [4], indicating deviations in the density distribution from the ideal case. Secondly, visual data qualitatively exhibit the binary shell absorption

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Fig. 2 Side transmission imaging of silicon laser ablation plasma under 17 J/cm² incident laser fluence. a: Diagram of the experimental setup. Two CCD’s are used, one for imaging, one for alignment.
behavior by simple examination. We took these images at 17 $J/cm^2$, however the plume must behave very similarly for a broad range of fluences based on dimensional analysis. Given the 0.2 power dependence of radius on kinetic energy, the large changes in fluence will yield a very small change in the plasma distribution. For example, a 100% increase in energy yields only 14% increase plume radius at any time. Therefore, this inferred plume shell thickness will be very similar across a broad range of fluences.

### 2.3 Laser probing

To obtain an indicator of linear absorption by the plasma, we must next probe the plume. The most useful probe wavelength will be near the processing pulse in order to evaluate the effects of plasma shielding on the processing beam, but different enough to spectrally separate them using standard optical filters. We therefore probe at 980 nm with a CW beam, as diagrammed. The probe was irradiated through a defocused lens system as diagrammed in Fig. 4d, allowing the probe to be 30 $\mu m$ in diameter while the processing beam is 170 $\mu m$. We tried three locations for the probe, directly in the center of the crater, adjacent to the crater, or 300 $\mu m$ away from the crater center as shown in Fig. 4c. As we discussed, the probe signal in the center of the crater (Fig. 4c, (i)) is confounded by effects of melt, particle ejection, and plasma, which we cannot easily separate. Moving to the outer crater edge (Fig. 4c (ii)) yields a more comprehensible result, yet there is still interference from the melt induced reflection increase. Although we see both a brief dip from plasma absorption and a rise due to melting, we cannot determine what causes these effects individually, which is necessary to properly diagnose the plasma. To completely avoid these effects, we moved the probe further away from the center, 300 $\mu m$ away (Fig. 4c (iii)). This results in a sharp drop and recovery, which correlates well to the leading edge of the plume shell passing by the probe. We must now revisit our hypothesis of a radiative cooling related increased transmission. If the attenuation was based on the dispersion of solid particles without a change in absorption cross-section, we would expect to see a sharp dip, and partial recovery, as the laser will still pass through the top of the absorbing plume. This type of dip would be much wider and show a much longer recovery time than dip time. In the experiment, the dip and recovery times are very similar, so the additional temperature decrease must play a significant role, quickly lightening the plume.

Now that we have a precise idea of the plasma geometry, we can formulate a straightforward calculation of the probe attenuation based on the plume radius, linear absorption coefficient of the plasma, and probe beam. The probe has a circular gaussian cross-section intensity distribution. The beam is in fact convergent, however beam waist calculations accounting for the hyperbolic widening of the beam show that at 350-$\mu m$-height, the widening of the beam is less than 1 $\mu m$, compared to the 30 $\mu m$ beam full width half maximum, so this effect is ignored. The depth of field of the imaging system is 38 $\mu m$, however the depth of focus of the probe is 2.8 mm due to the smaller initial beam diameter. Thus, after normalizing to the baseline reflectivity of silicon, the reflected energy can be directly calculated at any point. The domain is divided into 3 regions labeled in Fig. 4a: Region 1, closest to the center of the crater, where the beam passes through the plume shell, then an empty region, and afterward through the plume again, Region 2, where the probe passes through the approximately vertical region of the plume shell, and Region 3, where the incident light is unperturbed. Therefore, the total transmission through the plume is calculated via the following integral over the 2D surface:

$$T = \frac{\int_{R_1} I_r \exp \left( -\frac{2k_x(z_{out} - z_{in})}{\sigma^2} \right) dA}{2\pi \sigma^2}$$

$$+ \frac{\int_{R_2} I_r \exp \left( -2k_x z_{out} \right) dA}{2\pi \sigma^2} + \frac{\int_{R_3} I_r dA}{2\pi \sigma^2}$$

$$I_r = \exp \left( -\left( \frac{x^2 + y^2}{2\sigma^2} \right) \right)$$
In the above, $x$ is the offset distance of 300 $\mu$m, $w$ is the plasma shell thickness of 60 $\mu$m, and $\epsilon$ is the 1/e radius of the probe. At this point, we have everything necessary to calculate expected probe attenuation over time, with the exception of the plasma absorption coefficient, which will be discussed in the following section.

### 2.4 Plasma spectroscopy

To verify the absorption mechanism of IBA, we determined the linear absorption coefficient $k$ to feed into Eq. (2a). With the time resolved plasma plume radius data, we can directly compare laser probe attenuation measurements to the prediction from plasma IBA. A number of works recommend relations used to estimate the plasma shielding. We follow the work of Liu et al. to calculate the electron density and electron temperature in the plasma [7]. To summarize, by obtaining time resolved laser-induced plasma spectra, we can measure in fine detail electron density and temperature. Neutral lines have been well documented for Stark broadening measurements, where lines with the necessary intensity and broadening at the expected electron densities must be chosen. If the broadening is too great, the line will be smeared too far to measure above the noise, and if the line is narrow its broadening will be below the spectrometer resolution. The Si 390.55 nm line was found to have the necessary characteristics. Although the continuum emission background will be much stronger than any neutral spectral lines, the spectral emission must have a much sharper distribution than the continuum. The two mechanisms can therefore be easily separated in our case, as the line emission is not close to falling below the level of experimental noise. The spectral peak was also fitted with a double gaussian deconvolution fit to eliminate the effects of the Si III peak at 394.75 nm. The number density of the plasma can then be estimated by examining the peak broadening of the Silicon plasma emission lines via the following relations:
\[ \Delta \lambda_{1/2} = 2W \left( \frac{n_e}{10^{16}} \right) \left[ 1 + 1.75A \left( \frac{n_e}{10^{16}} \right)^2 \left( 1 - \frac{3}{4}N_D^{-1/3} \right) \right] \]  

\[ N_D = 1.72 \times 10^9 \times \frac{T^{3/2}}{n_e^{1/2}} \]  

\[ E(x) = \frac{2A w}{\pi} \left( \frac{w}{4(x - x_c)^2 + w^2} + \frac{2A^2 w_2}{\pi} \frac{w_2}{4(x - x_{c2})^2 + w_2^2} + \gamma_0 \right) \]  

In the above, \( \Delta \lambda_{1/2} \) is the full width half maximum of the plasma peak, \( W \) is the broadening parameter, \( A \) is the ion correction parameter [22], and \( N_D \) is the number of particles in the Debye sphere. Equation 3c is a Lorentzian distribution where \( A \) is the integrated peak intensity, and \( w \) is the FWHM used to fit and interpret plasma spectra. Subscript two represents the second peak parameters and are not used. Additionally, the integrated peak intensity to continuum ratio gives a measure of the electron temperature as shown below:

\[ \frac{E_{\text{i}}}{E_{\text{c}}} = C_r \frac{A_{21} \xi}{U_i T_e} \exp \left( \frac{E_{2} - E_{i}}{kT_e} \right) \]  

\[ U_i(Y) = 5.33707 + 1.05984Y - 0.87284Y^2 + 0.32479Y^3 + 0.02802Y^4 - 0.00578Y^5 \]  

\( C_r \) is the continuum emission coefficient[23], \( A_{21} \) is the Einstein transition probability, \( U_i \) is the partition function (where \( Y = T/10000 \)), \( g_2 \) is the electron degeneracy, \( E_i \) is the ionization energy, \( E_2 \) is the upper energy level [24], and \( \xi \) is the continuum correction factor. The values of terms in Eqs. 3a and 4 are listed in Table 1. The error in temperature and density from the given calculations is below 30% [25] and should contribute no systematic errors.

Curve fits against data are shown in Fig. 5a, and calculated electron temperature and density are shown in Fig. 5b. We obtained all spectra with an intensified charge-coupled device (ICCD) camera (PI-MAX, Princeton Instruments) collected by a Mitutoyo 10X NUV lens. Both temperature and density exhibit a generally decreasing trend and are

| \( A_{21} \) \( (s^{-1}) \) | \( C_r(sK) \) | \( E_i(eV) \) | \( E_2(eV) \) | \( g_2 \) | \( \xi \) | \( W (nm) \) | \( A \) |
|------------------|----------|-----------|---------|------|------|---------|------|
| 1.33 \times 10^7 | 2.005 \times 10^{-5} | 8.151     | 5.083   | 3    | 1.4  | .156 \times 10^{-2} | .027 |

Fig. 5  
(a) Time resolved spectral measurement (blue) with fit overlain (red) for 25 J/cm² of the Si I 390.55 nm line. (i) 15 ns; (ii) 20 ns; (iii) 25 ns; (iv) 30 ns; (v) 35 ns; (vi) 40 ns; (b) Spectral calculation results for spectra shown to the left.
within expected ranges of values expected for this process from previous study [7]. Once these values are calculated, we use Kramers' theory for IBA absorption with Maxwellian electron velocity distribution. This equation is recommended [8, 21, 26, 27] to estimate the linear absorption:

$$a = 3.69 \times 10^8 \times \frac{Zn_e^2}{T^{(1/3)}e^3} \text{[cm}^{-1}]$$

(5)

where Z is the average ion charge number. To determine Z, we performed spectral measurements in high vacuum \((2.5 \times 10^{-5}\text{Torr})\) to eliminate the possibility of atmospheric spectral lines, acquiring the first 100 ns of emission. After comparing with the NIST spectral database, we labeled the spectral lines (Fig. 6). Given the strength of the Si III lines and the fact that higher ionization stages will contribute more significantly to IBA, we assume Z=2 for the calculations. We have determined a vacuum experiment is the best way to estimate the degree of ionization, however it should be said that there may be different ionization conditions in high vacuum vs in atmosphere. The principle effect the atmosphere should have is confining the plasma more strongly, and therefore increasing the density. The plasma must cool mainly due to radiation, so all effects of the atmosphere on temperature should be considered of second order, therefore little affecting the degree of ionization. Further study will be needed to fully distinguish atmospheric vs silicon spectral lines in an ambient pressure environment.

Using the relations for absorptivity (Eq. 5), the model proposed in Fig. 4a and Eq. (2a), and the electron temperature and densities calculated in Fig. 5b, we can directly calculate the expected absorption by the plasma of the laser probe as a function of time matching the conditions of the actual probing experiment very closely. Plotted together in Fig. 7, we compare the calculated absorption with the experiment. Small fluctuations in reflectivity above 1 are due to laser source noise. Based on the calculation, we
see a dip corresponding to 57% attenuation, compared to 65% attenuation in the direct experiment. Both show similar steep transmission drops. The two profiles are shifted in time by about 10 ns. Examination of plume radius data shows a standard deviation of 18 μm in from shot to shot in plume radius, corresponding to 5 ns temporally according to the Sedov-Taylor fits. Given the two independent measurements (spectral and absorptive), shot to shot variation could account for much of this shift. The Lorentzian profile fit the spectral line well, so noise in the spectrum is not expected to contribute significantly to experimental error. The highest experimental uncertainty is expected to originate from the spatial variation in the plasma density. In our modeling, we had to assume a binary shell-core model as described at length following Fig. 4. This assumption places all scatterers right at the edge of the plume. In reality, this distribution will be smoothly varying and not binary with sharp transitions. Therefore, our assumptions place the absorbers somewhat further toward the edges of the plume than they would be in the real case, therefore pushing the time delay for the start of the dip earlier. As we see, the calculated absorption dip does come earlier than the experimentally measured dip. This spatial variation, in addition to random variations, is believed to account for the separation in dip times. Spatial mapping of the spectra may allow for closer matching between predicted and measured absorption, but it is out of the scope of this paper due to the difficulty of this kind of mapping. Importantly though, the two curves exhibit similar dip shapes and dip depths, providing strong evidence that the simple plasma relations apply under such laser ablation conditions. This validation also gives us good reason to suggest that the relations may continue to apply for a broad parameter set, especially as such relations are fundamentally derived. The condition of local thermal equilibrium is the most important assumption for IBA to hold, and it is well known what physical conditions will allow for this.

### 3 Conclusion

We show that Kramers’ theory for inverse Bremsstrahlung absorption with Maxwellian electron velocity distribution, derived directly from classical first principles, describes transient laser-induced plasmas with accuracy. To do this, we provide acquisition of spectra, spatially and temporally resolved probing, and imaging results from multiple viewpoints to determine the linear absorption of the plasma plume. Up to now, intensive plasma properties associated with laser ablation have mostly been estimated by the Sedov-Taylor solution fits of the shockwave energy, plasma peak broadening, and plasma peak-continuum ratio measurements that are subject to uncertainty in the distribution of plasma and scatterers. By considering the plasma as a thin shell on the periphery of the ablation shockwave, we eliminate much of this uncertainty. Accordingly, we develop a methodology to calculate physical properties involved in the laser ablation. First, normal and side imaging give a complete picture of the actual geometry of plasma expansion. As laser diagnostics rely rather extensively on optical probing, determination of the shape and distribution of ejecta is critical. Since optical probing methods inherently integrate along a line of sight, a certain degree of deconvolution is always necessary, requiring density distribution data. Then, probing and calculation allows us to accurately calculate plasma linear absorption. Concurrence of IBA relations and experimental results to within experimental error evidences the applicability of detailed fundamental relations to plasma absorption in laser ablation.

The spectroscopic method employed here for determining the plasma absorption can be employed to estimate the plasma shielding in a wide array of laser ablative material removal processes. The method of normal imaging can also be directly employed for analysis of industrial processes to yield a better understanding of the laser ablation. Additionally, this work confirms mechanisms of plasma absorption under these conditions. Computational models of the laser ablation process will necessarily continue to rely heavily on modeling of the plasma. Verification of the dominant physical mechanisms is both necessary and informative. Based on this work, we propose that future computational modeling should have strong rationale for using the IBA as a means to estimate plasma absorption.

### Acknowledgements
This work was partially supported through a grant by the Applied Physics Laboratory of Johns Hopkins University to the University of California, Berkeley. The authors acknowledge discussions with Drs. Kaushik Iyer, Xun Zhu of APL/JHU, and Dr. Darren Luke of AFRL.

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