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Temporal Evolution of the LIBS Spectrum of Aluminum Metal in Different Bath Gases

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14. ABSTRACT

The spectral emission of gas phase aluminum (Al) and Al oxide was measured during and immediately after exposure of a bulk Al sample to a laser-induced spark produced by a focused, pulsed laser beam (Nd:YAG, 10 ns pulse duration, 35 mJ/pulse, $\lambda = 1064$ nm). The spectral emission was measured as a function of time after the onset of the laser pulse, and was also measured in different bath gases (air, N_2, O_2, and He).

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1. Introduction

Aluminum (Al) is a common ingredient of explosives and propellants. In explosives, Al is used to augment air blast, raise reaction temperature, and create incendiary effects (*1*). In rocket propellants, Al is used to increase thermal energy and elevate the flame temperature (*2*). A proposed mechanism for the combustion of Al in O_2 follows(*3*):

$$Al\ (l) \rightarrow Al\ (g) \qquad \Delta H = 317.7\ kJ \tag{1}$$

$$AlO + Al\ (l) \rightarrow Al_2O \qquad \Delta H = –224.0\ kJ \tag{2}$$

$$Al\ (g) + O_2 \rightarrow AlO + O \qquad \Delta H = 6.53\ kJ \tag{3}$$

$$Al\ (g) + O + M \rightarrow AlO + M \qquad \Delta H = – 492.1\ kJ \tag{4}$$

$$AlO + O_2 \rightarrow AlO_2 + O \qquad \Delta H = –78.6\ kJ \tag{5}$$

$$AlO + AlO_2 \rightarrow Al_2O_3\ (l) \qquad \Delta H = –1557.2\ kJ \tag{6}$$

$$Al_2O + O_2 \rightarrow Al_2O_3\ (l) \qquad \Delta H = –1562\ kJ \tag{7}$$

For some explosive materials, Al may be used to tailor performance to specific needs. Measurements of relative amounts of Al metal and Al oxide (AlO) during explosions of energetic materials may provide insight into increasing the performance of Al-containing explosives. The experiments described here are a preliminary study of the application of laser-induced breakdown spectroscopy (LIBS) to this problem.

Although best known for high selectivity for metals analysis (*4*), LIBS has also been used to detect energetic materials (*5*), trace elements in liquids (*6*), organic compounds in ambient air (*7*), and some biological materials (*8*). In LIBS, a pulsed laser focused onto a target material converts some of the material into a plasma of ions and electrons, with temperatures that may approach 20,000 K (*9*). As the plasma cools, some of the energy is radiated as light. When measured using a spectrograph, the wavelengths of the emitted light are characteristic to the elemental components of the target, while the intensity of light over a given wavelength range may yield the proportion of that element within the target material (*10*). Additionally, the time evolution of the emission following the laser pulse may be used to identify certain chemical reactions occurring in the plasma as it cools.

In this report, we measure the emission from a laser-induced spark produced by focusing a pulsed Nd:YAG laser onto the surface of an Al rod. The emission is spectrally and temporally resolved, the effect of different bath gases (air, O_2, N_2, and He) on the emission is measured, and temperature and electron density are calculated.

2. Experimental

A schematic of the simple LIBS system used in this work is shown in figure 1. Briefly, a light pulse (~10 ns, 35 mJ per pulse) from an actively Q-switched Nd-YAG laser (Big Sky Laser Technologies Inc., Bozeman, MT) emitting at a wavelength of 1064 nm was focused by a 50-mm convex lens onto the surface of an Al rod. A Si-Si optical fiber (600-µm core diameter) collected the emission from the plasma spark. A lens was placed in front of the fiber so that the plasma spark was sufficiently defocused to eliminate any spatial effects. An echelle spectrometer (Catalina Scientific Corp., Tucson, AZ) fitted with a gated, intensified CCD camera (Andor Technology Com., Model DH 734-18-03) was used to measure the emitted light. The entire experiment, including background measurement, laser control, data acquisition, and data processing, was controlled by a laptop computer (Dell).

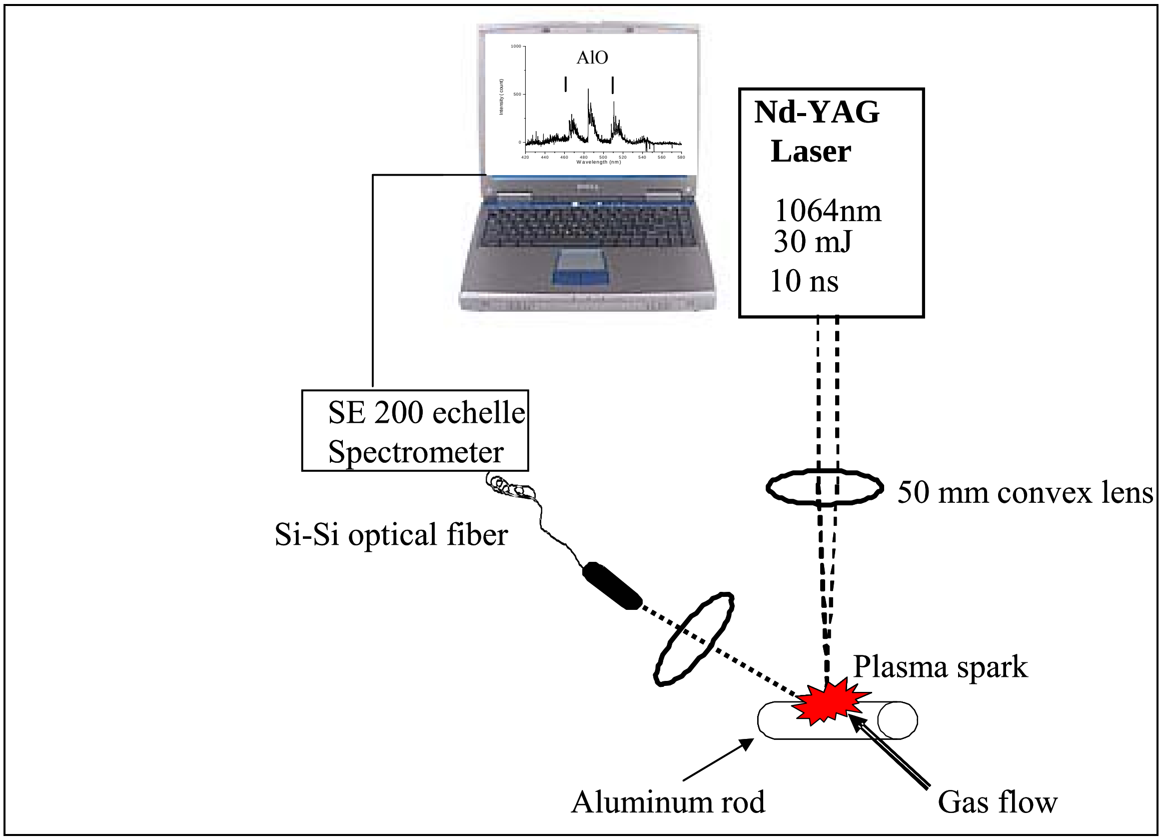

Figure 1. Schematic of the experimental setup used to measure LIBS spectra.

Prior to the measurement of each LIBS spectrum, a background spectrum was measured and subsequently subtracted from the sample spectral data. In an attempt to minimize errors due to shot to shot variations in the laser output power (~5%), each spectrum used in the data analysis is the average of 50 “single shot” spectra. For each LIBS spectrum measured, the Al rod was repositioned so only a new sample was exposed to the laser-induced spark. To enable comparison with previous LIBS studies of Al (*11*), a detector gate width of 2 µs was used for these experiments. Detector gate delays (relative to the Q-switch of the Nd:YAG laser) ranged from 0 to 30 µs. The composition of Al rods used in this study was Al 91.4%, Cu 5.67%, Fe 1.28%, Li 1.11%, and minor constituents (Mg, Mn, Ti, and Zn percentage <0.5% by weight). Bath gases (N_2, O_2, and He) were obtained from Matheson and were used without any further purification. Typical flow rates were ~2 L/min. The gas flow was delivered via 4-mm I.D. Tygon tubing. The exit port of the tubing was ~5 mm from the location of the plasma volume.

3. Results and Discussion

3.1 Emission Spectra of an Al Rod in Air

The most intense regions of the Al rod LIBS spectrum (bath gas = air [ambient], gate width = 2 µs, gate delay = 20 µs) are shown in figures 2, 3, and 4. The first spectral window (figure 2) from 300 to 420 nm includes emission from gas phase aluminum (Al I) at wavelengths of 308.34, 309.44, 394.56, and 396.26 nm. The second spectral window (figure 3; 420–580 nm) includes emission from the gas phase molecular species AlO, with the most intense emission near 484.58 nm. The third spectral window (figure 4; 740–760 nm) includes emission from gas phase aluminum (Al II) at a wavelength of 747.14 nm. For Al combustion in air, previous investigators have suggested that above the melting point of Al_2O_3 (2315 K) (*12*), the Al species with highest partial pressures are Al and AlO.

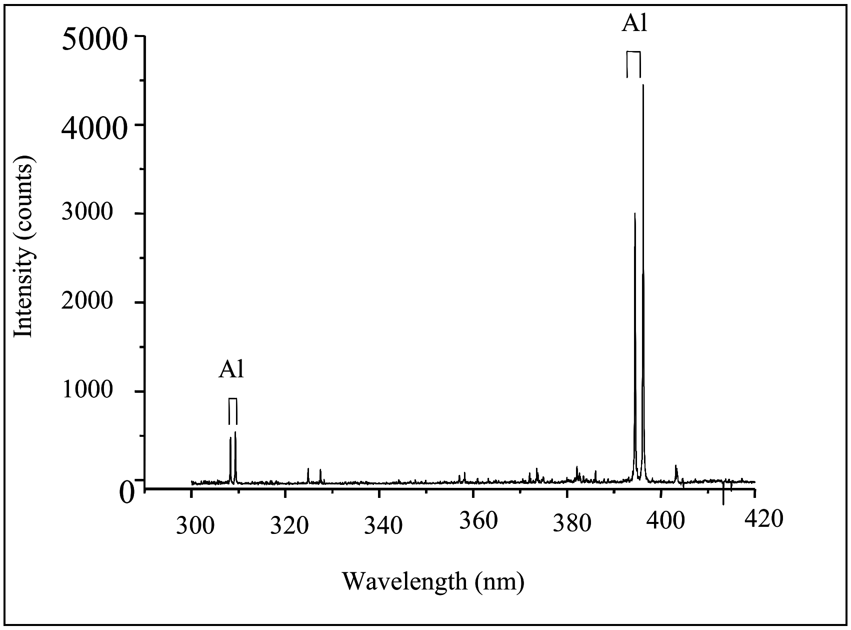

Figure 2. A portion of LIBS spectrum of an Al rod in air with a 20-µs gate delay and 2-µs gate (300–420-nm region).

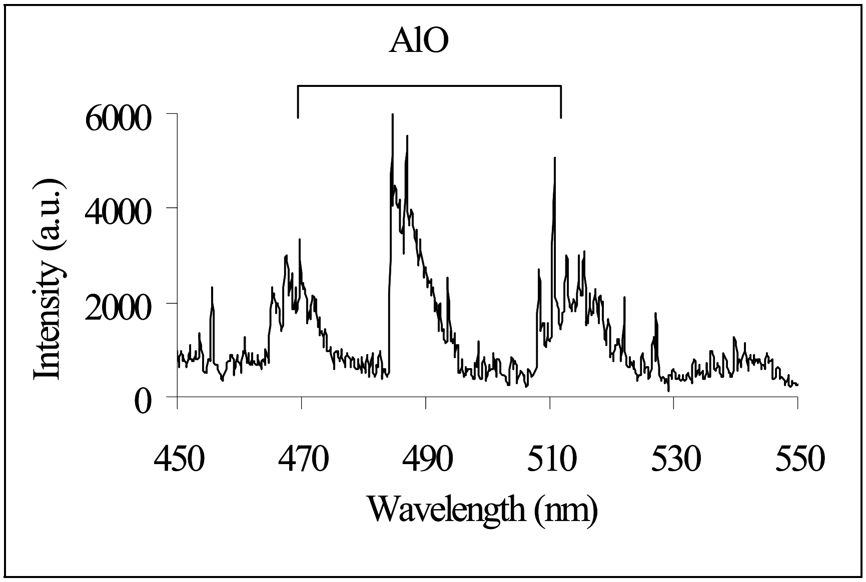

Figure 3. A portion of LIBS spectrum of an Al rod in air with a 20-μs gate delay and 2-μs gate (450–550-nm region).

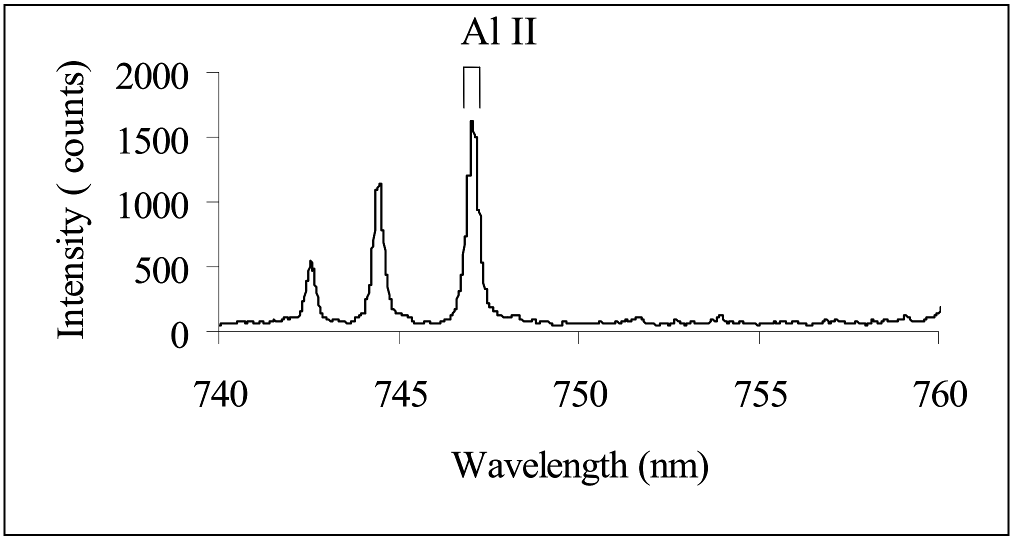

Figure 4. A portion of LIBS spectrum of an Al rod in air with a 20-μs gate delay and 2-μs gate (740–760-nm region).

3.2 Temporal Evolution of Al and AlO Emission

Figure 5 shows the LIBS spectrum of an Al rod in air at various gate delays (gate width = 2 μs). As seen in figure 5, the Al I line (396.2 nm) reaches its maximum intensity in air ~5 μs after the laser pulse. The band from AlO emission (484.4 nm) reaches its maximum intensity ~20 μs after the laser shot. This is qualitatively consistent with the combustion mechanism for Al in oxygen, outlined in reactions 1–7 earlier.

LIBS spectra of the Al rod (measured from 350 to 580 nm) at various gate delays (gate width = 2 μs) for the bath gases O_2, He, and N_2 are shown in figures 6, 7, and 8, respectively. For comparison, the peak intensities of the Al I line in each figure have been normalized. It is worth noting that the emission near 484 nm (from AlO) in figure 7 (He bath gas) and figure 8 (N_2 bath gas) is vanishingly small compared to the emission near 484 nm in figure 5 (air bath gas) and figure 6 (O_2 bath gas).

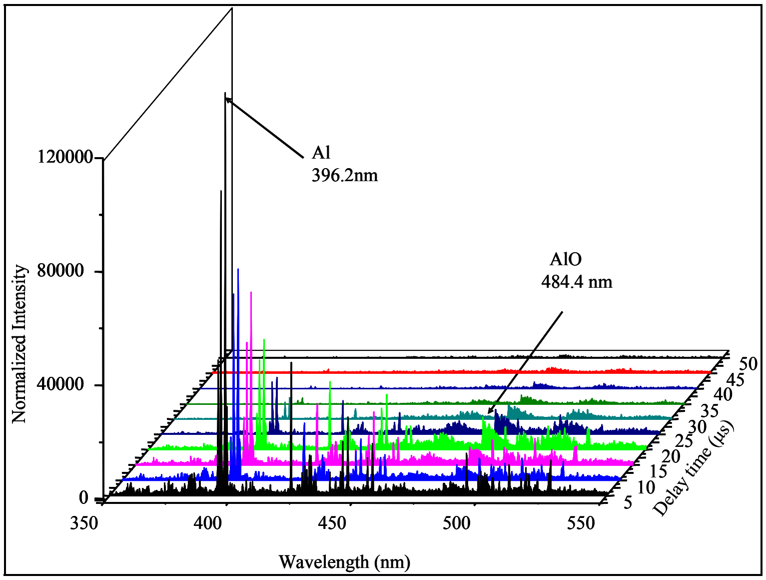

Figure 5. LIBS spectrum of an Al rod in air, at various gate delays. The gate pulse width is 2 μs.

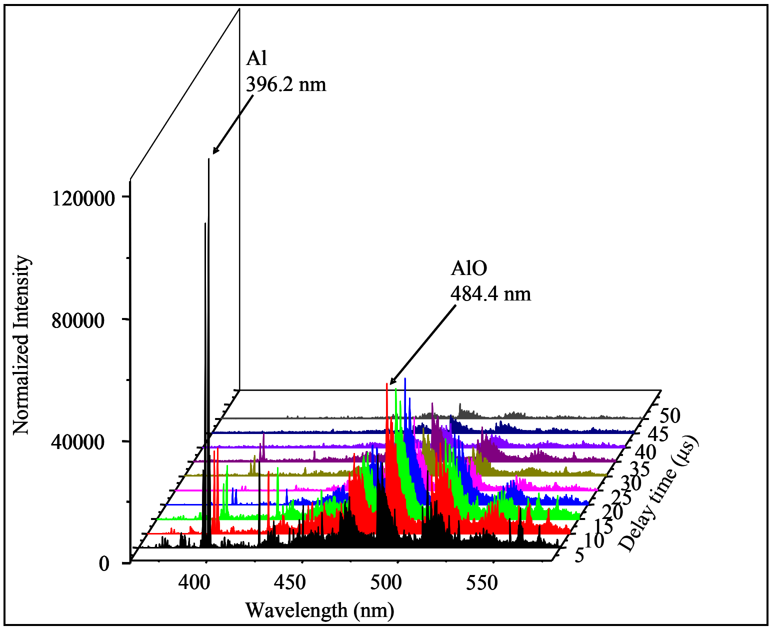

Figure 6. Temporal emission evolution of Al LIBS in O_2. The gate pulse width is 2 μs.

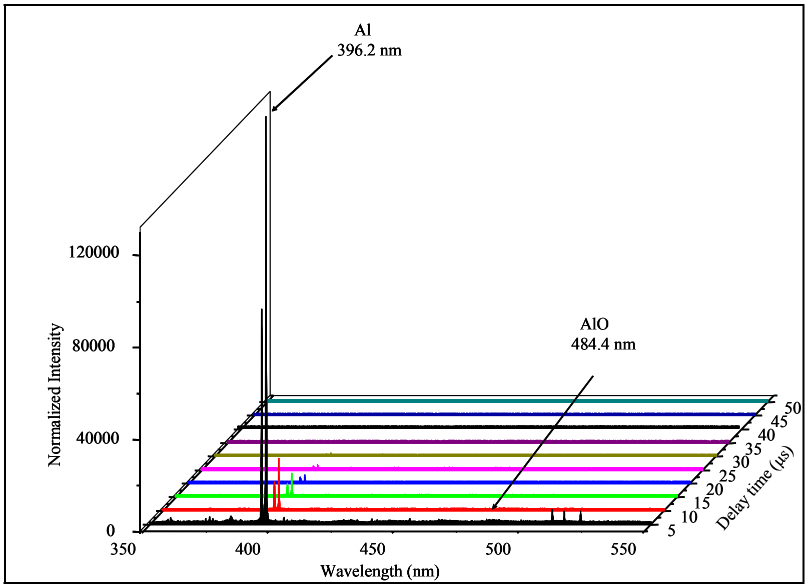

Figure 7. Temporal emission evolution of Al LIBS in He. The gate pulse width is 2 µs.

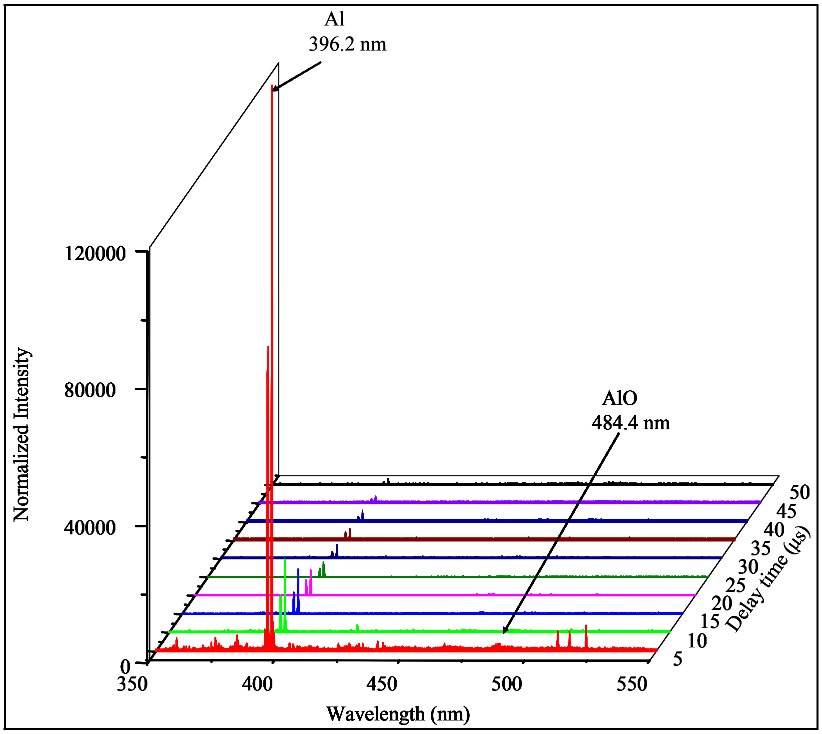

Figure 8. Temporal emission evolution of Al LIBS in N_2. The gate pulse width is 2 µs.

Figure 9 shows that the decrease in Al emission (396 nm) with time appears exponential. Figure 10 shows a pseudo-first-order plot of logarithm of intensity at 396 nm vs. time. From this plot, the deactivation of Al (fastest to slowest) as a function of bath gas is O_2~He>air>N_2. Figures 11 and 12 (expanded by a factor of 10,000) show the maximum emission intensity of the AlO band near 484 nm as a function of time for reactive (air and O_2) and nonreactive (He and N_2) bath gases, respectively. Figure 11 shows that the maximum emission from AlO occurs 10 µs after the laser pulse in the pure O_2 atmosphere, while the intensity in air reached a maximum 20 µs after the laser pulse. Therefore, we believe the main source of AlO emission in bath gases of O_2 and air is AlO formed by the reaction of Al (g) with ambient O_2, analogous to reactions 3 and 4 for the combustion of Al in O_2. This is also supported by the increase in AlO emission as the bath gas is changed from air (figure 5) to O_2 (figure 6).

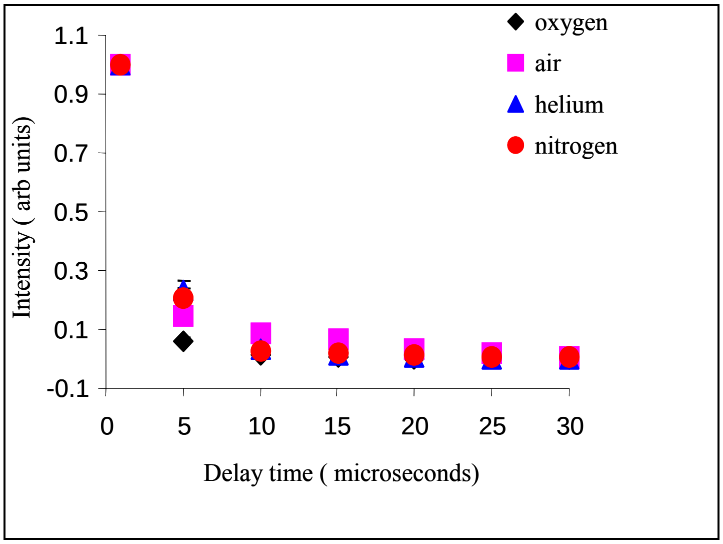

Figure 9. Comparison of time evolution of emission intensity for Al (396-nm) LIBS signal in air, He, N_2, and O_2 with a 2-µs gate pulse width.

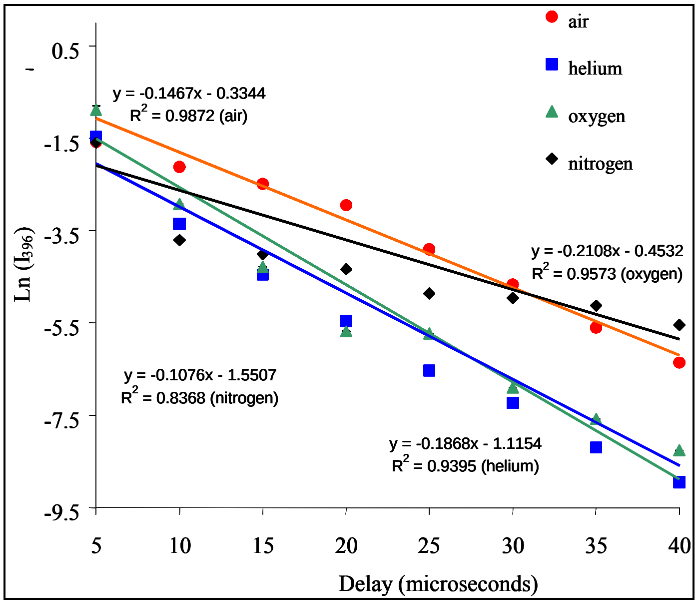

Figure 10. A plot of logarithm of intensity at 396 nm vs. time for the different bath gases used in these experiments.

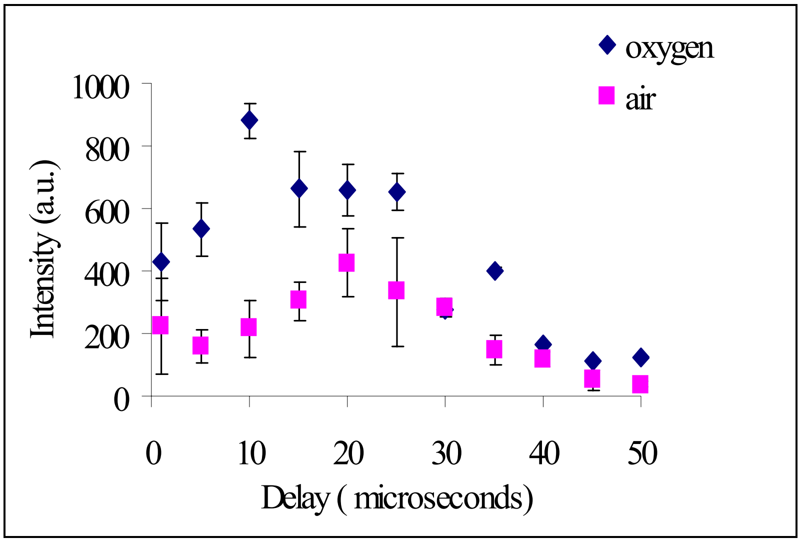

Figure 11. The maximum emission intensity of the AlO band near 484 nm as a function of time for the bath gases air and O_2.

Figure 12 shows that, in the absence of ambient O_2, the temporal behavior of the AlO emission is similar to that of the Al emission; i.e., the temporal behavior of the AlO emission in the unreactive bath gases is similar to emission from material (Al) native to the Al rod. Therefore, we believe the source of the AlO emission in the absence of ambient O_2 is the Al_2O_3 layer on the Al metal.

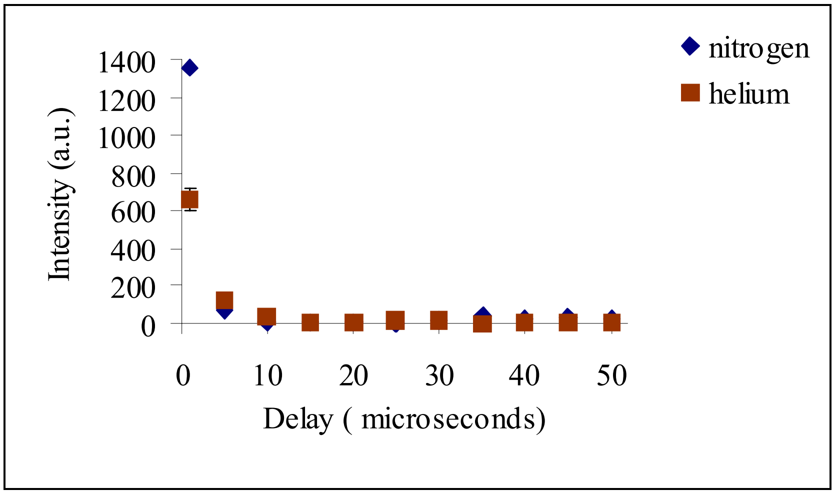

Figure 12. The maximum emission intensity of the AlO band near 484 nm as a function of time for the bath gases He and N_2.

3.3 Temperature Calculations

For the temperature calculations reported, we assume that for the gate width used (2 µs), the time rate of change of the plasma temperature is small, and that light emission collected and analyzed is emitted from a gas region that is approximately homogeneous in temperature and composition.

This assumption of "local thermodynamic equilibrium" is necessary when calculating temperatures using a Boltzmann distribution. The intensities of Al I spectral lines at wavelengths of 308.34, 309.44, 394.56, and 396.26 nm were used to calculate temperatures at different gate pulse delays according to the following equation:

$$\mathrm{Ln}\,(I/\,(g_i\,A_{ki})) = -\,(E_k\,/\,kT) + \mathrm{Ln}\,(C_\alpha F/\,U_\alpha(T))\,, \tag{8}$$

where I is the peak line intensity of atomic species α with concentration C_α, E_k is the upper energy level, T is the plasma temperature, $U_\alpha(T)$ is the partition function of the species α, k is the Boltzmann constant, F is a constant depending on experimental conditions, A_{ki} is the transition probability, and g_i is the statistical weight for the upper level. Spectroscopic data (table 1) were obtained from the National Institute of Standards and Technology database (*13*). A plot of Ln $(I\,/\,(g_i\,A_{ki}))$ as a function of E_k will have a slope equal to $-1/kT$. A typical Boltzman plot using equation 8 is shown in figure 13.

Table 1. Spectroscopic parameters for Al I and Al II investigated lines.

	Wavelength (nm)	**A_{kj} (10^8 s^{-1})**	**E_k (eV)**	**g_i**	**ω (nm)**
Al I	308.34	0.63	4.021485	2	—
	309.44	0.74	4.021650	4	—
	394.56	0.49	3.142721	2	—
	396.26	0.98	3.142721	2	—
Al II	466.30	0.53	13.25646	3	6.85×10^{-3}
	747.14	0.94	15.30840	7	1.26×10^{-2}

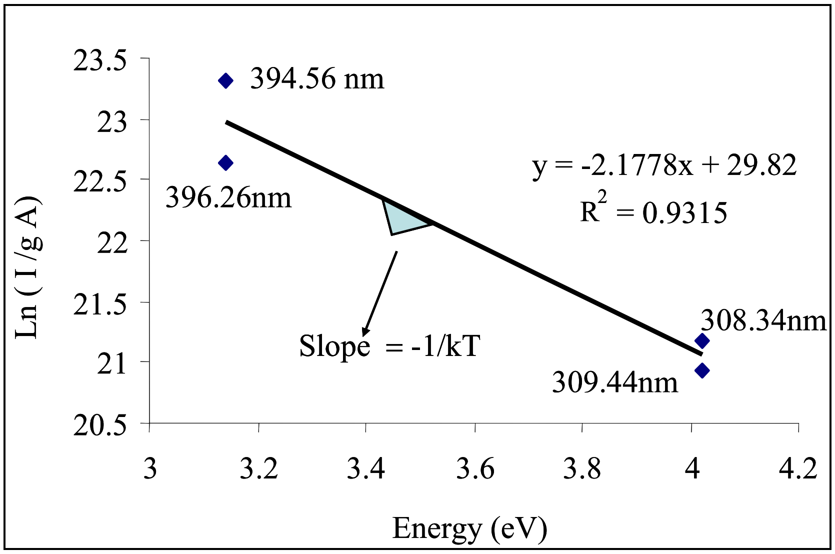

Figure 13. A Boltzman plot for 308.34-, 309.44-, 394.56-, and 396.26-nm Al I lines in O_2. The gate pulse width is 2 μs. The gate pulse delay is 15 μs.

Temperatures calculated using equation 8 and spectral line intensities from LIBS spectra measured in different bath gases are shown in figure 14. These calculated temperatures are in good agreement with previously reported calculated temperatures for similar systems (*14–16*). In general, the calculated temperature exhibits an approximately exponential decay over the emission lifetime.

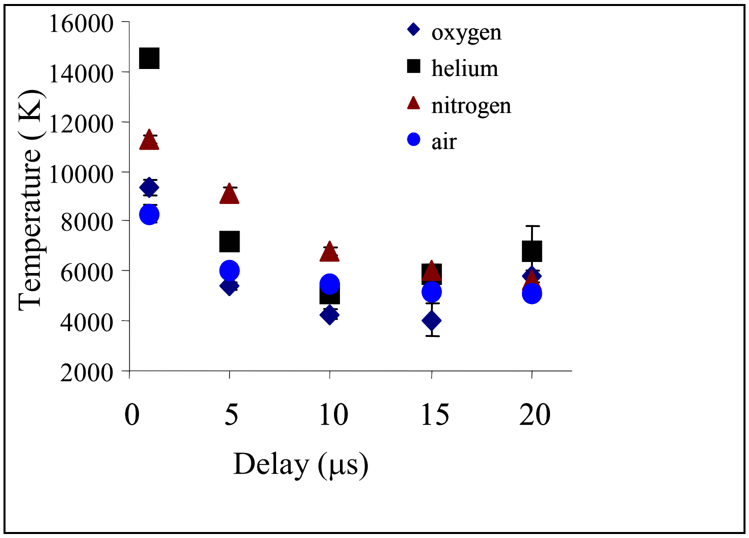

Figure 14. Excitation temperature vs. gate pulse delay with a 2-μs gate width.

3.4 Electron Density

The electron density (N_e) was determined using the Stark broadening effect (*17*) and assuming the plasma to be optically thin (negligible self absorption) for the Al II emission line at 747.14 nm. Stark broadening parameters are available for the lines at 747.14 and 466.3 nm (*18*). The Al II line at 466.3 nm was not used because this line is partially obscured by the AlO band near 484 nm. The relation between the line width (full width at half maximum [FWHM]) of the Stark broadened line and the electron density is given by equation 9:

$$\Delta\lambda_{1/2} = 2\omega\,(N_e / 10^{16}) + 3.5\,A\,(N_e / 10^{16})^{1/4}\quad(1 - BN_D^{\;-1/3})\,\omega\,(N_e / 10^{16}), \tag{9}$$

where $\Delta\lambda_{1/2}$ is the line width (FWHM), ω is the Stark broadening parameter, A is the ion broadening parameter, N_D is the number of particles in the Debye sphere, and B is a coefficient equal to 1.2 for ions and 0.75 for neutral lines. The values of ω were taken from Coloa et al. (*18*).

The measured line width was corrected to first order by subtracting the contribution of the instrumental line broadening. The instrument line broadening was found to be 0.1 nm, as determined by measuring the emission lines from a calibrated mercury lamp. The first term on the right side of equation 9 is the contribution of electron broadening. The second term on the right side of equation 9 is the quasistatic ion broadening contribution, which can to be neglected in this analysis (*19*). Equation 9 then reduces to

$$\Delta\lambda_{1/2} = 2\omega\ (N_e / 10^{16}). \tag{10}$$

In order to make a determination as to whether the local thermodynamic equilibrium conditions were satisfied for the selected spectral lines, the critical value of electron density distribution (N_e) was evaluated by following the procedure described by Aragon et al. (*20*). The critical limit of electron density distribution was determined from equation 11:

$$N_e \geq 1.6 \times 10^{12}\ T^{1/2}\ (E_k - E_i)^3\ . \tag{11}$$

For the experiments reported here, the critical electron densities varied from 3×10^{15} to 9.5×10^{15} cm^{-3} for the temperature range from 4000 to 14,500 K. The lowest calculated electron density value exceeded these critical values by a factor of 20 for the range of temperatures calculated using the Boltzmann equation (equation 8). As seen in figure 15, there is a general trend toward lower electron densities at later decay times (also see figure 16) as the plasma cools.

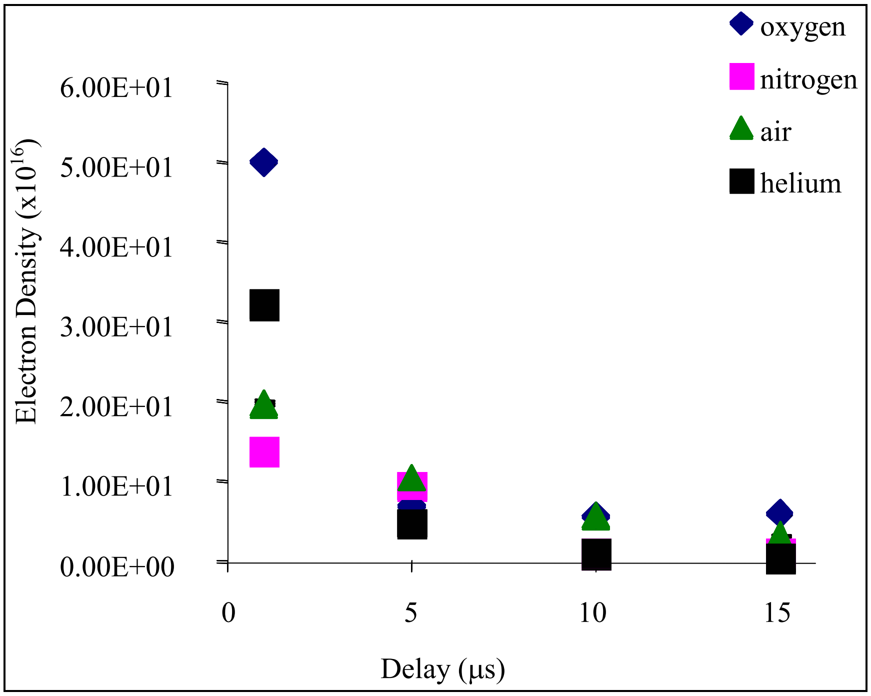

Figure 15. Electron density of Al vs. gate pulse delay with a 2-μs gate width.

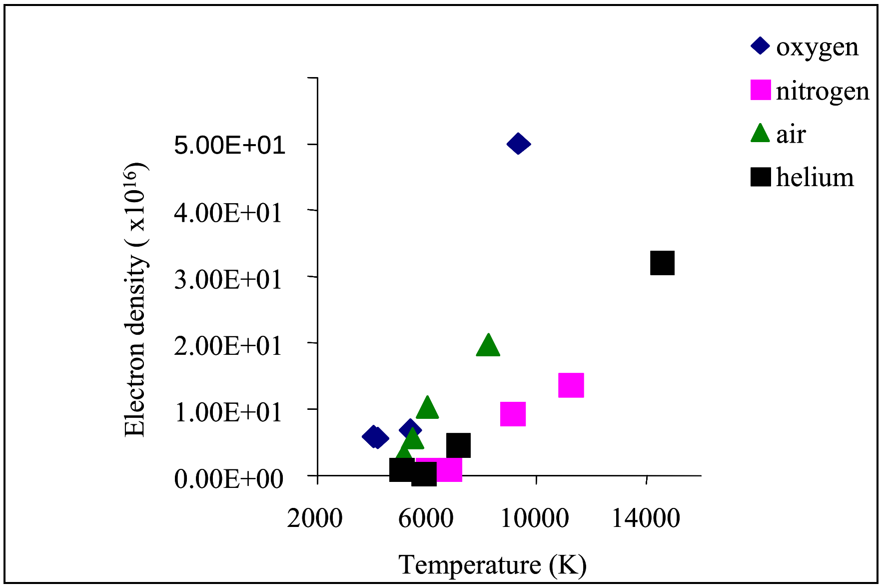

Figure 16. Excitation temperature vs. electron density profile in different atmospheres.

4. Conclusions

Measurements of the emission of AlO following exposure of an Al metal surface to a laser-induced spark have been carried out for bath gases of air, O_2, N_2, and He. Results of these experiments indicate that virtually all of the AlO emission is from AlO formed by the reaction of Al vapor with O_2 from the bath gas (if present). Emission from AlO initially present as an Al_2O_3 oxide layer on the metal sample was vanishingly small for emission spectra measured in bath gases of N_2 and He, when compared to the AlO emission measured in air and in O_2 bath gases. However, it is possible to distinguish the AlO emission from the Al_2O_3 oxide layer from AlO formed by reaction with ambient O_2 by examining the temporal behavior of the emission. The temporal behavior of Al and AlO emission following Al metal exposure to a laser-induced spark (in air and O_2) is consistent with known chemical mechanisms for Al combustion in O_2. Finally, calculations of temperature assuming a Boltzmann distribution of Al emission lines gives results in good agreement with calculations by previous investigators.

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