Investigation for Ignition of ADN-based Ionic Liquid with Visible Pulse Laser

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(Received June 23rd, 2017)

We evaluated the ignition probability of high-energy ionic-liquid droplet due to breakdown from visible pulse laser. The high-speed camera result, pressure profile, temperature trace from an R-type thermal couple, and radical measurement results using an image-intensifier charged-coupled device camera revealed the ignitability of this system. In addition, we simulated a simple thermal-diffusion model and investigated the temperature distribution for comparison with the experimental results. As a result, the probability of ignition due to a single pulse is low; however, potential for gasification and atomization of the ionic-liquid droplet is probable.

Key Words: Ammonium Dinitramide, Ionic Liquid, Laser Ignition, Pulse Laser, Breakdown

Nomenclature

α	: temperature diffusivity
λ	: wavelength
ρ	: density
τ	: time resolution
C_p	: specific heat capacity
D	: diameter
f	: focal length, position
H	: repetition frequency
I	: laser intensity
I_{sp}	: specific impulse
k	: thermal conductivity
M	: mean molecular weight
Nu	: Nusselt number
T	: temperature
t	: time
V	: volume
x	: distance, position

Subscripts

b	: beam (laser)
c	: combustion
d	: droplet
f	: focal length, position
g	: gas
gr	: glass rod
L	: laser
max	: maximum
p	: pressure
pl	: pulse
tc	: thermocouple
SH	: bandpass filter SH0500
LV	: bandpass filter LV0550

1. Introduction

Hydrazine is the most popular liquid propellant for a reaction-control system in satellites because it easily reacts and decomposes with N_2O_4 or a catalyst. However, it is highly toxic and carcinogenic¹⁾; therefore, we must pay attention in treating and preparing the protective equipment against hydrazine. Furthermore, the European Union created a regulation named Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH)²⁾ that addresses chemical treatment.

According to REACH, we will be not allowed to manufacture and treat hydrazine. From this information, we need to consider another liquid propellant. One of these liquid propellants is the green propellant (GP). GP is a type of propellant with low toxicity and high energy density. Almost all GPs consist of high energetic salts and some liquids such as water. The typical high energetic salts are hydroxylammonium nitrate,³⁻⁵⁾ ammonium dinitramide (ADN),⁶⁻⁸⁾ and hydrazinium nitroformate.^{9, 10)} These species are classified as ionic materials; thus, they are hygroscopic. In other words, they can be better used as a solution with some liquids than solid. Nevertheless, the energy density of energetic materials decreases with their dissolution into solvent. Burning temperature may also decrease, resulting in the reduction in performance, for example, specific impulse I_{sp} .

$$I_{sp} \propto \sqrt{\frac{T_c}{M_c}} \quad (1)$$

Matsunaga et al.^{11,12)} reported a non-solvent liquid propellant, which is a eutectic ionic liquid. This ionic liquid includes ADN, other high energetic salt, and fuel. It is characterized by higher potential than conventional liquid propellants. An ionic liquid possesses some chemical characteristics. One of them is the extremely low volatility at room temperature, which means safer storage characteristic in a propellant tank, and this propellant is suitable for longer-term operation such as deep-space exploration. However, the combustibility of this liquid is low due to low vaporization.¹³⁾ Furthermore, the combustion

Fig. 1. Diagram of ignition test equipment for ionic liquid droplet with visible pulse laser.

temperature increases to 2000 K owing to the high energy density of high energetic salts. A conventional ignition system, for example, catalyst, heater, or spark plug, contacts the target to add ignition energy. Such temperature causes rapid deactivation of the ignition system. The lifespan of an ignition system is directly related to the thruster life, and the thruster life greatly affects the operation of a satellite. In other words, knowing how to properly ignite ionic-liquid propellants is very important. Our group focuses on breakdown ignition using a pulse laser. Breakdown generates a light plasma by electron acceleration through photon absorption and electronic avalanche when the laser energy density exceeds a threshold.¹⁴⁾ The breakdown ignition uses a light plasma through a pulse laser. Since the 1960s, some researchers have studied this ignition system, but almost all of the solid pulse lasers were large. Hence, laser ignition was not realized for practical usage. Recently, Taira *et al.* have downsized a solid pulse laser to a palm size.^{15,16)} This laser allows application of breakdown ignition to some combustion systems. In fact, reports have been presented on the application of breakdown ignition on internal combustion engines.^{17,18)} The target of breakdown ignition is gas at more than 1 atm. In an actual thruster actuation, we can separate the steps into three phases to obtain an impulse: 1) injection of propellant into the chamber and atomization of the propellant, 2) ignition of the atomized and gasified propellant by an injector, and 3) spreading of combustion. In particular, in phase (1), this ionic-liquid propellant through the injector is almost in a liquid state due to low volatility. Therefore, if a pulse laser is irradiated into the thruster chamber, a high probability exists that breakdown of the ionic-liquid droplet can result. Few reports about breakdown ignition that focused on

the droplet as an ignition target have been presented. We need to investigate the phenomena in this liquid propellant droplet when a pulse laser irradiates the droplet.

In the present study, we evaluate the ignition probability of an ADN-based ionic-liquid propellant droplet using laser breakdown ignition to measure the droplet distribution due to one laser pulse shot. We also demonstrate a suitable thruster system that combines an ionic-liquid propellant with a pulse laser.

2. Experimental Setup

Figure 1 shows a simple system map used in this study. In this experiment, we implemented four methods to evaluate the combustion of ionic-liquid droplet, namely,

- I. High-speed camera recording
- II. Pressure profiling
- III. Self-luminous radical tracing
- IV. Droplet-temperature measurement

An ADN:monomethylammonium nitrate:urea sample with a ratio of 4:4:2 was used, and the volume was 1 μL ($D_L = 1.2$ mm) in all experiments. A pulse laser light (PIV-400-8-10, Spectra Physics, $\Delta t_{pl} = 8$ ns, $H = 10$ Hz, $\lambda = 532$ nm, and $I_{max} = 600$ mJ/pulse) was passed through two kinematic mirrors (TFMHP-30C05-532, Sigma Opt., Inc.). The focus lens (NADL-30-100PY, Sigma Opt., Inc., $f = 100$ mm) converged the laser into a handmade chamber ($V = 40 \times 40 \times 40$ mm³) in the xyz-stage (TSD-605S, Sigma Opt., Inc.). The focal positions were set on three conditions, namely, $f = 100$, 90, and 82. The beams diameters at $f = 100$ and 90 were 100 μm and 1.2 mm, respectively, and $D_{L,f=82}$ was 2.0 mm. The beam line was

Fig. 2. Calculation model of the thermal diffusion equation on the droplet.

adjusted by a second mirror with two irises (IH-30, Sigma Opt., Inc.) to the laser shield set on the rail (OBT-500SH, Sigma Opt., Inc.). A droplet was suspended on the cross point of two glass rods (micro glass rod, Fujirika Co. Ltd., $D_{gr} = 0.1$ mm). In the droplet temperature measurement, we placed the ionic-liquid droplet at the junction of an R-type thermocouple (Niraco Co. Ltd., $D_{tc} = 25$ μ m). A vacuum condition was created using a vacuum pump (NVP-1000, Eyela), and the atmospheric condition consisted of N_2 gas (N_2 99%, Tomoe Shokai). The pressure distribution was recorded using pressure sensors (XTL-140, XT-140, Kulite Co., Ltd.) and an amplifier (AM32AZ, Unipulse). We used two types of cameras with a fiber light (KTL-1001, Tokina Co., Ltd.): one is a high-speed camera (AX mini 240, Photron Co. Ltd.), and the other is an image-intensifier charge-coupled device (ICCD) camera. During the camera recording, we adopted two filters (SH0500, LV0550, Asahi Spectra, $\lambda_{SH} < 500$ nm and $\lambda_{LV} > 550$ nm). Both cameras were deployed on the same place. A delay generator (DG535, Stanford Research Systems) controlled the laser flash timing and ending of the camera recording. The one-pulse shoot was adjusted using a handmade switch, which was set between the laser and DG535. A data logger (NR-600, Keyence) simultaneously recorded the laser flash signal, camera recording results, droplet temperature, and pressure profile.

3. Numerical Setup

Gas breakdown ignition is classified as a thermal ignition by non-equilibrium plasma from a pulse laser. Meanwhile, a shock wave is simultaneously generated with the plasma generation. The shock wave can possibly affect the ignition or other phenomena. In the present study, we evaluated the flammability and gasification of an ionic-liquid droplet from thermal distribution by simulating a simple thermal-diffusion model. The generation and deactivation of a plasma may be too fast, and the generation time is close to the pulse width of the laser. The recorded plasma temperature was approximately 30000 K.¹⁹⁾ We set this temperature as the initial condition. Figure 2 shows a model of this simulation. Similar to the experiments, we assumed a 1- μ L droplet where $D_d = 1.2$ mm. This droplet was fixed at $x = 0$ without moving.

We formulate the basic thermal-diffusion equation as

Fig. 3. Curve of temperature diffusivity of the ionic liquid [C₄min][BF₄] by calculating k and C_p using the least square methods.

$$\frac{\delta T}{\delta t} = \alpha \frac{\delta^2 T}{\delta x^2}. \quad (2)$$

Learning temperature diffusivity α is necessary, which can be expressed as

$$\alpha = \frac{k}{\rho C_p}. \quad (3)$$

This parameter depends on the temperature. Knowing the thermal parameters of this ionic liquid is necessary; however, no studies about them are available. In our calculation, we used the parameter of [C₄min][BF₄], which is one of the ionic liquids. Thermal conductivity $k^{(20)}$ and specific-heat capacity $C_p^{(20)}$ were fitted using the least square method to obtain the fitting equation. With regard to the density, fitting was difficult because the density rapidly decreased beyond a certain temperature and reached a critical value. Thus, we set a constant volume for the density of the ionic liquid. Figure 3 shows the fitting curve using the least square methods. In this simulation, we chose the explicit method to solve the Eq. (2) and set $\Delta t = 0.00001$ s as the time step. For the distance step, we decided to satisfy the Newmann stable conditions. We set $T_{x>0.6mm, t} = 300$ K as the Dirichlet boundary condition.

4. Results and Discussion

4.1. High speed observation and pressure profiles

Because this ionic liquid has a low pressure deflagration limit at low pressure, as obtained from the strand burner experiments²¹⁾ for measurement of solid's and liquid's linear burning rate, the ionic liquid cannot be possibly continuously burned under vacuum conditions. In our experiment, we performed investigations under approximately 1 atm and vacuum condition (20 kPa). The droplet exhibited a lensing effect in ambient environment due to the different refractive indexes. Therefore, under the condition of complete condensation, the focal point may move to the front of the laser path due to the lensing effect. For effective energy introduction, controlling the focal point is important. To identify the effect of focal control, we set three focusing conditions: two defocusing conditions ($f = 82$ and 90) and a focusing condition ($f = 100$). In all conditions, the laser intensity was adjusted to approximately 150 mJ/pulse. Figure 4(a) shows the result of the ignition test under 1-atm N_2 gas, and Fig. 4(b) shows the

(a) N₂ condition under 1 [atm] **(b) Vacuum condition under 20 [kPa]**

Fig. 4. Results of the high-speed record tests under $f = 82, 90$, and 100 conditions. (a) Under 1-atm N₂ condition. (b) Vacuum condition (20 kPa).

vacuum-condition result. The laser irradiation time into the droplet was set to $t + 0$ ms. We can observe the plasma generation from the strong luminescence at $t + 0$ ms. After the plasma inactivation, the ionic-liquid droplet started to spread to many directions. The spreading speeds among these three conditions were different. According to the droplet distribution at 166 ms, the speed at $f = 90$ was the fastest, and slow diffusion was observed at $f = 100$. The difference in the atmospheric condition created spreading-speed gaps in all focusing settings. In the $f = 100$ results at 1 atm, the breakdown appeared to be generated on the surface of the ionic-liquid droplet and not at the center of the droplet. This result shows that the droplet acted as a lens in this experiment. Meanwhile, under vacuum condition, the spreading speed of the droplet was slower than that at 1 atm. Therefore, breakdown was generated not only at the surface of the droplet but also in the gas surrounding the droplet. Pinnick reported that the breakdown threshold of a droplet is lower than that of gas,²²⁾ which means that the breakdown of the droplet is easier under a gas condition. If the laser intensity does not reach the threshold of the gas breakdown and is higher than one of the breakdown values in the droplet, breakdown on the surface of the droplet can possibly be achieved. We need to adjust the focusing conditions, including the droplet effect as lenses. Comparing the results at $f = 90$ and $f = 82$, the spreading speed at $f = 90$ was faster than

that at $f = 82$. These are the defocal conditions, and beam diameters $D_{b,f=90} = 1.2$ mm and $D_{b,f=82} = 2.0$ mm were bigger than the completely focused beam size $D_{b,f=100} = 100$ μ m. From the measurement, the beam diameter at $f = 90$ was 1.2 mm at the position of the droplet, and that at $f = 82$ was $D_{f=82} = 2.0$ mm, which was slightly bigger than the 1.2-mm-diameter droplet. At $f = 82$, a difference in the diameter and droplet size increased the energy loss, which could have caused the spreading-speed difference between $f = 90$ and 82. Between $f = 90$ and 100, the speed of the droplet spreading was clearly different. The energy conditions were the same. The aforementioned energy loss at $f = 100$ condition may have possibly caused these results. We performed the same experiments at $f = 95$ and between $f = 90$ and 100. The results show that the spreading speed at $f = 95$ was lower than that at $f = 90$ and 82. If the irradiated laser density decreases with the expansion of the laser with focus control, the cross-sectional area of the breakdown of the initial generation point on the droplet could increase with the decrease in the breakdown threshold from gas to liquid. Therefore, increasing the breakdown volume promotes droplet spreading. We also measured the pressure profiles under all analysis conditions. At the 1-atm condition, shock wave generated by the plasma appeared on the pressure profile in all conditions, resulting in complications and noise, as shown in Fig. 5(a). On the other hand, under vacuum condition, we could see that the

Fig. 5. Pressure profile of the laser irradiation test at $f=90$. (a) 1-atm N_2 , (b) Under vacuum condition (20 kPa).

pressure increased due to laser irradiation at $f=82$ and 90 . Figure 5(b) shows as an example the pressure profile for $f=90$ condition under vacuum. The noise profile due to the shock wave is the same as that shown in Fig. 5(a). The maximum pressure increment was very low compared with that under an ideal pressure volume, which reached to approximately 1 to 2 kPa. This result implies that the gasification of the laser pulse of approximately 5% was incomplete and inefficient.

4.2. Temperature measurement of ionic-liquid droplet

We tried to investigate the temperature profile of the droplet under all focusing conditions at the 1-atm N_2 condition. Figure 6 shows the result at $f=90$. In this measurement, $t=0$ was set at the onset of the temperature rise, which clearly showed the rapid increase in the droplet temperature. Thereafter, the temperature slowly dropped from laser irradiation to droplet spreading. The maximum temperature in this profile reached approximately 530 K. Matsunaga *et al.* reported that the initial decomposition temperature of an ADN-based ionic liquid is approximately 410 K,²³⁾ which is lower than this maximum temperature. Furthermore, the droplet reached the decomposition temperature and was transformed to gas due to the pulse laser shot. However, the resolution of the temperature measurement was not sufficient to indicate a clear profile because the temperature rise was too rapid compared with the maximum sampling rate of the data logger (10 kHz). The issue on the thermocouple resolution is important, which depends on the size of the junction and diameter of the thermocouple. We can express the resolution time of the thermocouple as follows:²⁴⁾

$$\tau_{tc} = \frac{\rho_{tc} c_{tc} D_{tc}^2}{4Nuk_g} \quad (4)$$

Fig. 6. Temperature profile of the laser irradiation on the ADN-based ionic-liquid droplet under $f=90$ condition.

When we set the droplet regression speed from 0.01 to 10 mm/s, τ_{tc} was calculated to be from 0.28 to 0.36 ms, respectively. From the above-mentioned measurement using a high-speed camera, the spreading was found to be fast. The scale of this phenomenon was below 0.01–0.1 ms, which is the reciprocal to the 1–10-kHz sampling rate. This calculation shows that this measurement is not sufficient for tracing the temperature profile using laser irradiation. We need to perform analysis using a non-contact method, for example, the two-color thermometry that uses a high-speed camera system, to more accurately monitor the temperature.

4.3. Radical distribution recorded by ICCD camera

Measuring the combustion flame is the simplest and easiest test to evaluate flammability. Some of the combustion flame spectra come from radical generations, and these wavelengths depend on the radical species. Radical luminescence of a radical type contains some wavelengths because it is in the excited and ground states. In this study, we chose $A^2\Sigma^+ \rightarrow X^2\Pi$ (approximately 306 nm) of OH^* and $A^2\Delta \rightarrow X^2\Pi$ of CH^* (approximately 431 nm) as main targets. When breakdown occurs, plasma luminescence also occurs. If the plasma luminescence, which is a strong flash, is introduced into an ICCD, it may result in complication of the recorded pictures. For this reason, we set the gate opening to 10 μs after laser irradiation of the droplet. In addition, we attached band-pass filter SH0500, which protects the 532-nm scattering light. The gate opening was adjusted to 100 μs because the lifetime of a hydroxyl radical is reported to be approximately a few hundreds of microseconds with many reaction paths, including the OH^* consumption and generation.²⁴⁾ We performed measurement under the 1-atm N_2 condition and show the results of the ICCD measurement in Fig. 7. Compared with the ICCD pictures, we can see that some pictures are flashed in all focusing conditions; however, they are smaller and closer to the droplet size. One possibility is that these flashes come from laser scattering. If it is a part of the combustion flame, the flammability would be low. To summarize the above-described experiments, igniting an ADN-based ionic-liquid propellant droplet with one shot of a pulse laser could be difficult. In other words, adjusting the parameters will be an effective process to gasify and atomize a droplet.

4.4. Thermal-diffusion model of an ionic-liquid droplet

The calculation result is shown in Fig. 8. The droplet diameter is represented by the x axis, and the y axis represents the time. Location near the red mark indicates higher

Fig. 7. Radical distribution through laser irradiation of an ADN-based ionic liquid droplet under all focusing conditions. Yellow circles indicate the droplet position.

temperature and that near the blue mark indicates lower temperature. This ionic liquid decomposes at around 530 K and ignites for dropping at around 680 K. From this simple calculation, the thermal-diffusion speed is quick due to the high temperature of plasma generation. Thus, igniting this ionic-liquid droplet using breakdown plasma is possible. In fact, we have presented the increase in temperature in Section 4.2. Some correlations exist between this calculation and the actual condition. In the actual experiment, heat generation is followed by combustion phenomena. Therefore, the actual speed of this phenomenon is more rapid. The shock wave is popularly believed to be caused by the exponential change in temperature and volume due to plasma inactivation; thus, the flame kernel belatedly appeared. This calculation did not include the shock-wave effect. Energy loss due to the shock wave decreased the ignition efficiency of this droplet. Considering how to prevent shock-wave generation by ignition using the non-equilibrium plasma is important to efficiently ignite the propellant droplet. On the other hand, shock waves may affect the efficiency of the droplet atomization. If one laser pulse is used as an atomizer for an ionic-liquid droplet, increasing the shock-wave effect would be better. For both purposes, increasing the plasma volume is important. A laser beam has some confocal parameters: one is the confocal depth. The breakdown threshold of a droplet is 100 to 1000 times lower than that of gas. Thus, its breakdown into a droplet is easier than into gas. When a laser irradiates a droplet, the cross-sectional area of the breakdown plasma depends on the beam diameter at a specific focal position. Plasma decomposes and atomizes the

surrounding liquids. The remaining laser is scattered by the decomposed gas and atomized droplets. Immediately thereafter, however, the non-reacted liquid works as a lens, and the breakdown shape becomes cylindrical. The plasma volume depends on the confocal depth, which is the same as the focal position, and the laser intensity. The atomization and gasification effect further increases as the beam diameter becomes bigger because thermal diffusion is enhanced with the increase in the plasma volume and not because of the shock wave.

4.5. Double laser-induced breakdown ignition method

On the basis of the results presented in Sections 4.1 to 4.4, we discuss the next approach to ignition of this ionic-liquid propellant droplet. We can decompose and atomize the ionic-liquid droplet using one laser pulse, which means that the droplet is changed to a decomposed gas and smaller droplets. Gas-breakdown ignition has been recently reported in large number in the field of internal combustion engine. We expect that we can ignite the decomposed gas from an ionic-liquid droplet using laser breakdown. To perform this, we need to evaluate the ignition ability and flame stability of the decomposed gas. If we can ignite it using laser breakdown, the effective thruster model would appear to consist of two laser pulse systems. The first pulse acts as an atomizer to the droplet, and the second pulse generates a laser-induced breakdown in the decomposed gas and ignites it. Figure 9 shows the system diagram. The experimental approach to evaluate the ignition

Fig. 8. Thermal diffusion results under simulated ionic liquid condition.

ability and flame stability of the decomposed gas is difficult. As a next step, we will simulate it using the chemical reaction simulator CHEMKIN.

5. Conclusion

In this paper, we have presented the investigation of the flammability of an ADN-based ionic-liquid droplet by breakdown ignition using a pulse laser to investigate the phenomena by laser-irradiation experiments and validate a simple thermal-diffusion model. According to the high-speed camera results, the lensing effect of the droplet affected the droplet distribution. We could measure the temperature profile when the laser irradiated the droplet, and the temperature reached the decomposition temperature of an ionic liquid. The ICCD records showed a radical distribution through the laser irradiation test. This result indicates the low flammability of the droplet. The thermal-diffusion calculation indicated that the focusing parameters affected the plasma volume and promotion of thermal diffusion from the plasma. From these results, the flammability using our method was lower, but the effect of the gasification and atomization was better. In summary, generating breakdown on gas species such as N_2 or noble gases from ionic liquid or other substrates could be a reasonable and effective method. One example of a possible method to ignite this propellant droplet is shown in Fig. 9. As our next step, we will research on the flammability of decomposed gases from an ionic liquid. In addition, Ide²⁵⁾ reported a CHEMKIN calculation using a chemical reaction model of this propellant. Using this model, evaluating the ignition ability of the decomposed gases from an ADN-based ionic-liquid propellant is important.

Acknowledgments

This research was supported by the high-energy material research group in Japan Explosives Society. For the radical

Fig. 9. An example of igniting the propellant droplet using a pulse laser. The first shot breaks and atomizes the droplet, and the second shot breaks down the decomposed gas from this droplet and creates ignition.

measurement by ICCD, we were helped by T. Sakae and H. Musasa in Tokyo Instruments, Inc. We would also like to thank Prof. Saito in Meisei University for his helpful discussion.

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