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14. ABSTRACT

A high-speed megapixel camera capable of taking 22,000 frames-per-second (fps) at 1280 × 800 resolution and 1,000,000 fps at 128 × 32 resolution was purchased to investigate dynamic phenomena in DOD-related research, along with augmenting educational activities at Rutgers.

The purchased equipment allows for the overall high-speed capture of nano-energetics combustion, specifically aspects related to ignition and propagation speed. Research also encompasses utilizing the camera as part of an

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Report Title

Final Report: DURIP: High-Speed Camera for Characterization of Flame-Synthesized Nano-Energetics and Flame Spray Pyrolysis

ABSTRACT

A high-speed megapixel camera capable of taking 22,000 frames-per-second (fps) at 1280 × 800 resolution and 1,000,000 fps at 128 × 32 resolution was purchased to investigate dynamic phenomena in DOD-related research, along with augmenting educational activities at Rutgers.

The purchased equipment allows for the overall high-speed capture of nano-energetics combustion, specifically aspects related to ignition and propagation speed. Research also encompasses utilizing the camera as part of an optical emission spectroscopy (OES) system for assessment of temperature and local species. The camera can be used concurrently with an existing spectroscopic system.

Additionally, in research involving investigating single droplets comprised of precursor/solution, whereby their combustion produces metal-oxide nanoparticles to be used in nanoenergetic compositions, fundamental understanding will aid in optimizing and scaling processing conditions for flame spray pyrolysis (FSP) of metal-oxide nanopowders. The high-speed camera is being employed to image directly the transient behavior of such droplet combustion, which involves droplet explosion and disruption.

Finally, the camera is being used in an ARO project to study the various parameters involved in the forced ignition of spray/energetic combustion in high electric fields where spark discharges are triggered by the presence of the fuel/energetic aerosol itself. For this investigation, high-speed imaging and spectroscopy are the key diagnostics to better understand the fundamental mechanisms involved.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

Received Paper

TOTAL:

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

Received Paper

TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

Number of Presentations: 0.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Received Paper

TOTAL:

Number of Manuscripts:

Books

Received Book

TOTAL:

Received Book Chapter

TOTAL:

Patents Submitted

Patents Awarded

Awards

Graduate Students

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Post Doctorates

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Faculty Supported

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Under Graduate students supported

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: 0.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:...... 0.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:...... 0.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):...... 0.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:...... 0.00

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 0.00

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields:...... 0.00

Names of Personnel receiving masters degrees

NAME

Total Number:

Names of personnel receiving PHDs

NAME

Total Number:

Names of other research staff

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

See Attachment.

Technology Transfer

DURIP: High-Speed Camera for Characterization of Flame-Synthesized Nano-Energetics and Flame Spray Pyrolysis
(Grant W911NF-14-1-0412)

Final Report
PI: Stephen D. Tse, Rutgers University

Summary

A high-speed megapixel camera capable of taking 22,000 frames-per-second (fps) at 1280 × 800 resolution and 1,000,000 fps at 128 × 32 resolution was purchased to investigate dynamic phenomena in DOD-related research, along with augmenting educational activities at Rutgers.

The synthesis of unique metal-oxide nanostructures, which have nano-energetic applications involving thermite reactions with nano-Al, has been investigated in a previous and proposed ARO projects. Nanowires of tungsten oxide, molybdenum oxide, copper oxide, and iron oxide have been grown using a flame synthesis method, and then coated with Al nanolayers using ionic-liquid electrodeposition. The nanocomposite geometry not only presents an avenue to tailor heat-release characteristics due to anisotropic arrangement of fuel and oxidizer, but also eliminates or minimizes the presence of an interfacial Al_2O_3 passivation layer. Upon ignition, the energetic nanocomposite exhibits strong exothermicity, thereby being useful for fundamental study of aluminothermic reactions, as well as enhancing combustion characteristics. The heat-release behavior of these novel nano-thermites needs to be characterized.

The purchased equipment allows for the overall high-speed capture of the combustion phenomenon, specifically aspects related to ignition and propagation speed. Research also encompasses utilizing the camera as part of an optical emission spectroscopy (OES) system for assessment of temperature and local species. The camera can be used concurrently with an existing spectroscopic system.

Additionally, in research involving investigating single droplets comprised of precursor/solution, whereby their combustion produces metal-oxide nanoparticles to be used in nanoenergetic compositions, fundamental understanding will aid in optimizing and scaling processing conditions for flame spray pyrolysis (FSP) of metal-oxide nanopowders. The high-speed camera is being employed to image directly the transient behavior of such droplet combustion, which involves droplet explosion and disruption.

Finally, the camera is being used in an ARO project to study the various parameters involved in the forced ignition of spray/energetic combustion in high electric fields where spark discharges are triggered by the presence of the fuel/energetic aerosol itself. For this investigation, high-speed imaging and spectroscopy are the key diagnostics to better understand the fundamental mechanisms involved.

Acquired Equipment

This DURIP grant (W911NF-14-1-0412) was used to purchase a Phantom v2011 one megapixel digital high-speed camera, able to record over 22,000 fps at full 1280 × 800 resolution, and, with the FAST option, up to 1,000,000 fps at reduced resolution. The 35.8 mm × 22.4 mm CMOS sensor is composed of 1280 × 800 pixels at 28 µm pixel size, with 12-bit depth. Its electronic shutter is capable of 1 µs minimum exposure standard, and 300 ns minimum exposure with

FAST option. There is a shutter-off mode for PIV. The operating software comes with motion analysis, including basic measurements for distance, speed, acceleration, angles, and angular speed, as well as manual and automatic point collection for target tracking.

In conjunction with existing instrumentation for advanced laser-based spectroscopy, the high-speed camera forms a diagnostic system that can characterize various dynamic phenomena. Figures 1 and 2 show images of the high-speed camera, along with the laser-based spectroscopy system that it complements.

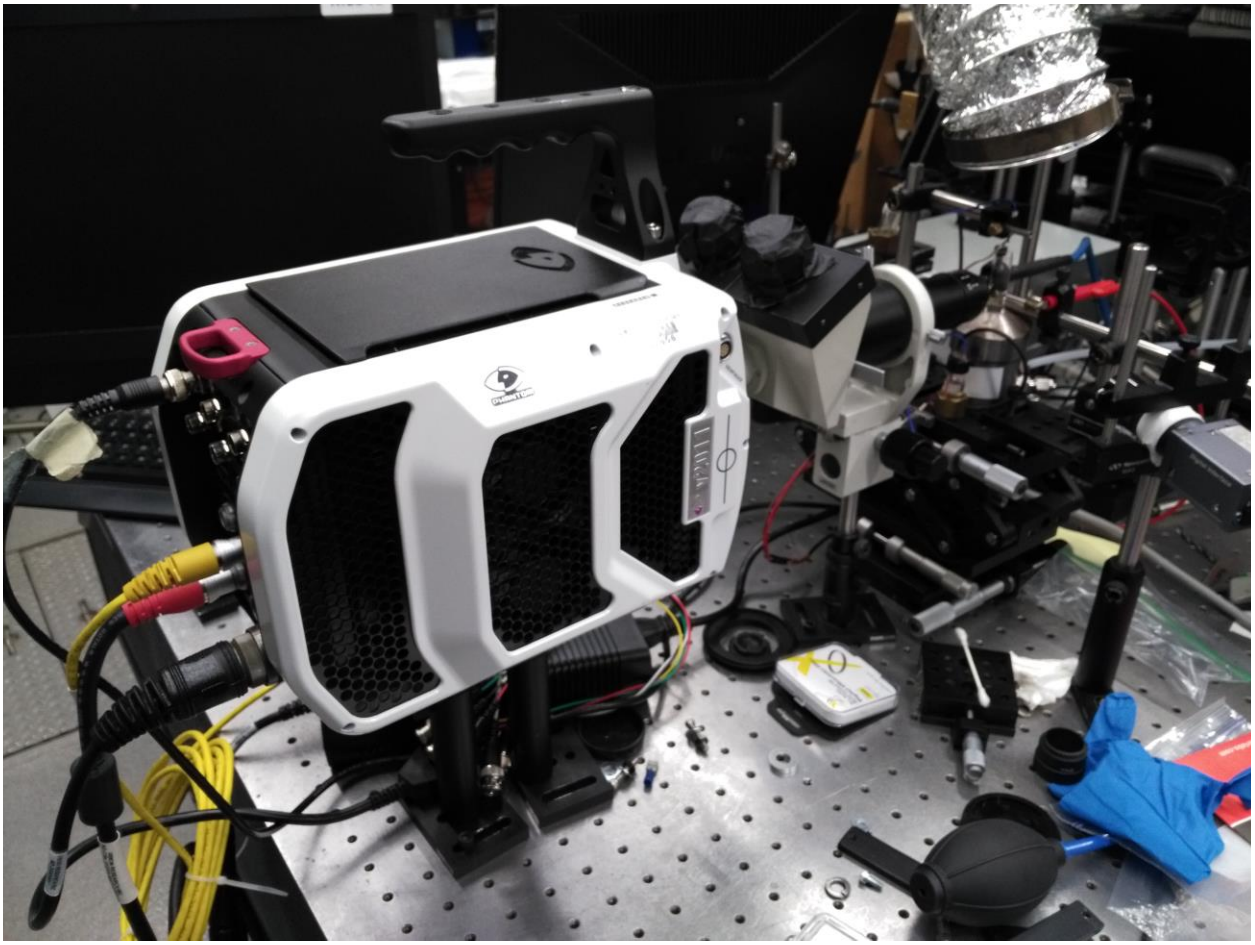

Figure 1. High-speed camera being used in an experimental setup to examine micro-droplet dynamics and induced electrical arcs.

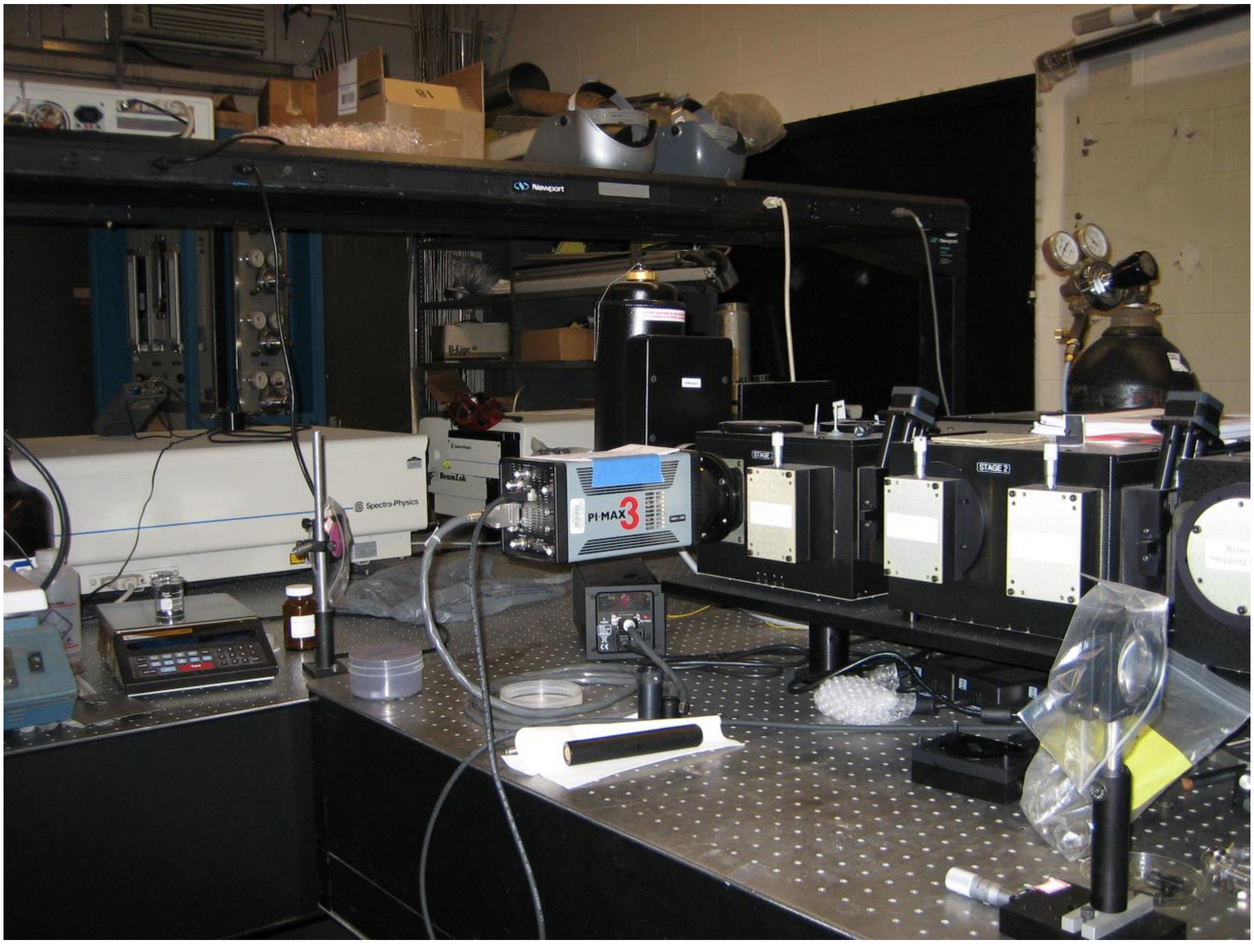

Figure 2. Complementary laser-based spectroscopy diagnostics benches. ICCD (PI MAX3) is shown attached to a triple spectrometer. Laser excitation is from a Nd:YAG laser, which can be used directly for scattering measurements, as well as used to pump an optical parametric oscillator (OPO) for fluorescence, absorption, and photoluminescence measurements.

As result, the the high-speed camera can support several research projects involving dynamic phenomena. Simultaneous utilization of advanced spectroscopic diagnostics enables temporal measurement of chemical species and temperature. Such knowledge gives insight into the fundamental mechanisms involved in the transient processes. In terms of educational use, the equipment and experimental facility will be incorporated into the curriculum on nanomaterials science and engineering (which is being offered as a joint program between several departments) and into the graduate curriculum on advanced experimental methods.

Research involving Acquired Equipment

<u>Combustion of Novel Co-axial Nanowire Thermites</u>

Our research has combined flame and ionic-liquid electrodeposition synthesis to produce arrays of composite nanostructures, including metal-oxide nanowires coated with Al nanolayers. See Fig. 3. Such thermite geometry not only presents an avenue to tailor heat-release characteristics due to anisotropic arrangement of fuel and oxidizer, but also eliminates or minimizes the presence of an interfacial Al_2O_3 passivation layer. Upon ignition, the energetic nanocomposite exhibits strong exothermicity, thereby being useful for fundamental study of aluminothermic reactions, as well as enhancing combustion characteristics.

The strong exothermic behavior and combustion dynamics of these nano-thermites is being characterized. The reaction propagation speed of nano-energetics is a function of the energy release rate, which is of critical interest to the energetics community. Direct and OES high-speed imaging (using a microscope objective) of the reaction propagation allow the reaction propagation speed to be extracted. For ignition studies, temperature and/or chemical species evolution can be assessed both spatially and temporally. The arrays (milligram quantities) are on a "long" substrate (e.g. tungsten wire) and ignited at one end using laser-energy deposition. For example, vertically-well-aligned Al-coated metal-oxide nanowires sticking out from a tungsten substrate/wire (see Fig. 3) can be ignited at one end of the substrate/wire, with high-speed camera capturing the overall reaction propagation along the length of the substrate/wire. The images from the high-speed camera is being correlated to temporal (PMT-based) optical emission spectroscopy (OES) to assessment better ignition point and propagation speed. This knowledge in turn allows us to optimize the structural details of the thermite composites, e.g. metal oxide nanowire diameter, array density, Al coating thickness, etc., in order to tailor the nanocomposite heat release characteristics.

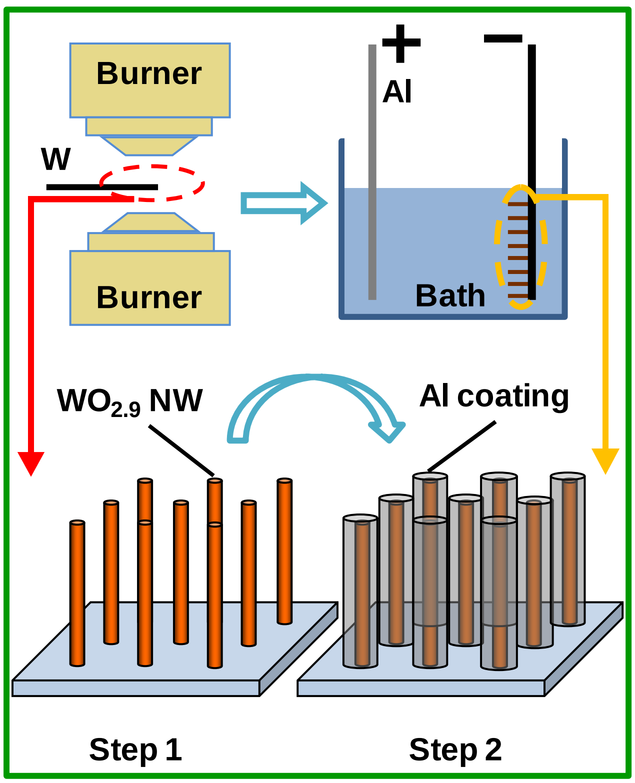

Figure 3. Schematic setup of the two-step synthesis of coaxial $WO_{2.9}$/Al nanowires (NWs). Step 1: Flame synthesis of $WO_{2.9}$ nanowires. Step 2: Ionic-liquid electrodeposition of nano-scale Al coatings on $WO_{2.9}$ nanowire arrays.

<u>Droplet Combustion Studies of FSP Production of Metal-Oxide Nanoparticles</u>

Combustion synthesis has demonstrated a history of scalability and offers the potential for high-volume commercial production, at reduced costs. Therefore, we are also examining flame spray pyrolysis (FSP) as a highly-promising and versatile technique for the rapid and scalable synthesis of metal-oxide nanopowders, which can include nanorod and nanodisc geometries, to be used in nanoenergetic compositions. As a fundamental investigation to determine the appropriate processing conditions for large-scale FSP, we are conducting experiments using single droplets comprised of precursor/solution, whereby their combustion produces metal-oxide nano-particles, rods, and plates. The high-speed camera is being utilized to image directly the transient behavior of such droplet combustion, which involves droplet regression, but also also possible explosion and disruption. Monitoring of the droplet reaction and droplet temperature by rainbow spectroscopy is being explored.

We have conducted experiments using the acquired high-speed camera. The results, given below, demonstrate the capability of the equipment in elucidating governing mechanisms in the combustion dynamics of the droplets. The experiments are conducted in a $10 \times 10 \times 40$ mm^3 cuvette as shown schematically in Fig. 4. Isolated monodisperse droplets are produced with a piezoelectric generator (piezodropper) at a frequency of 4 Hz. The investigated solvents are supplied from a small reservoir to the piezodropper, which ejects the droplets upward through a 2mm gap between two 100μm diameter tungsten electrodes, which ignite the droplets by means of a synchronized spark. The co-flow consists of oxygen, which is regulated with a mass flow controller and is made uniform with a porous frit before entering the cuvette, preventing fluctuations in the droplet trajectory and establishing plug flow conditions.

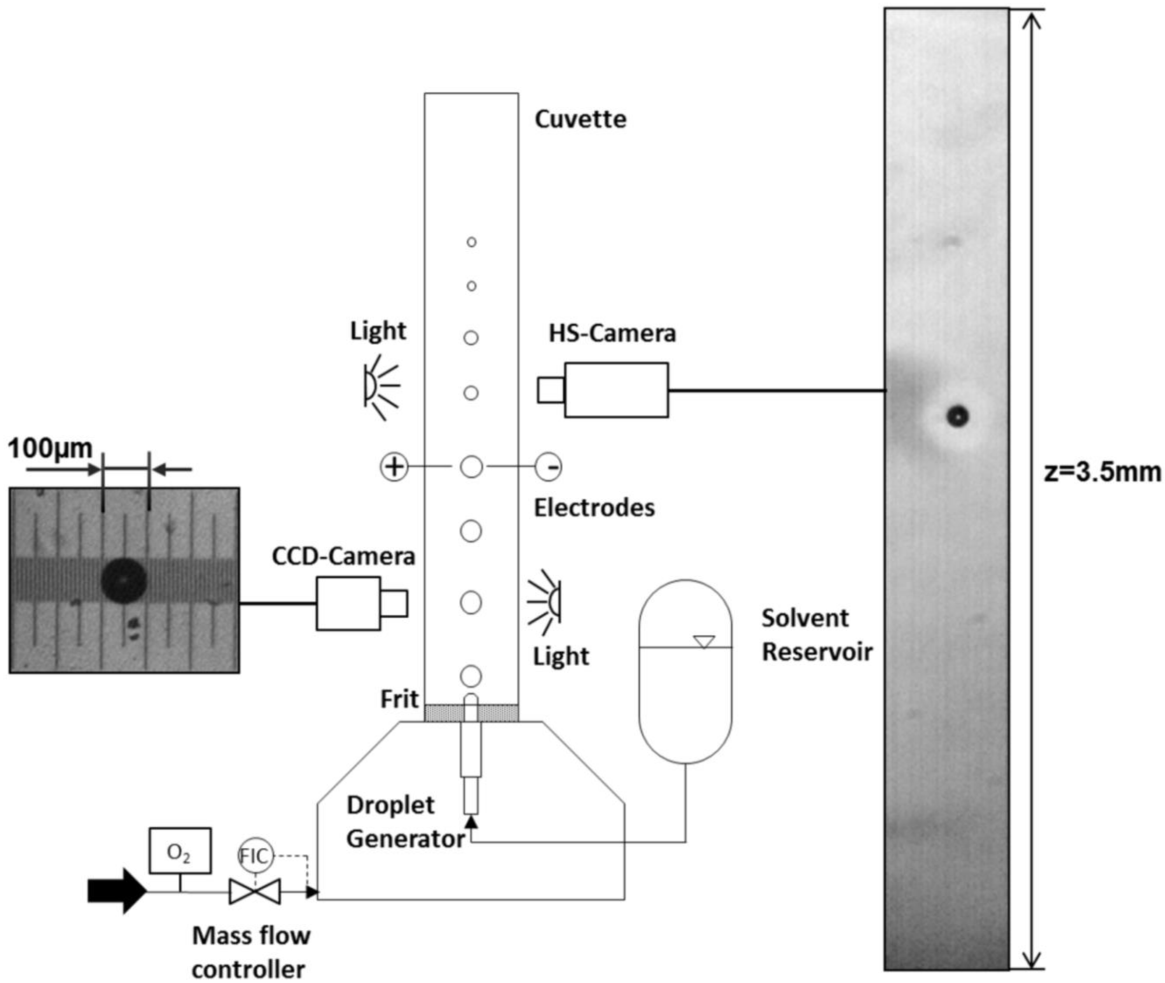

Figure 4. Experimental setup showing droplet generator supplied by a solvent reservoir with xylene, ethanol, heptane, dodecane, or precursor solutions. The droplet (e.g. 100 μm) is ejected into the co-flowing oxygen and traverses the field-of-view of the CCD and high-speed camera. (in collaboration with the Prof. Lutz Mädler at the University of Bremen, Germany).

Figure 5 shows images from the acquired equipment of a single droplet (xylene with 0.5 mol/L tin etheylhexnoate) that burns without disruption. The sequence is captured at 67,065 fps with 12.5μs exposure time.

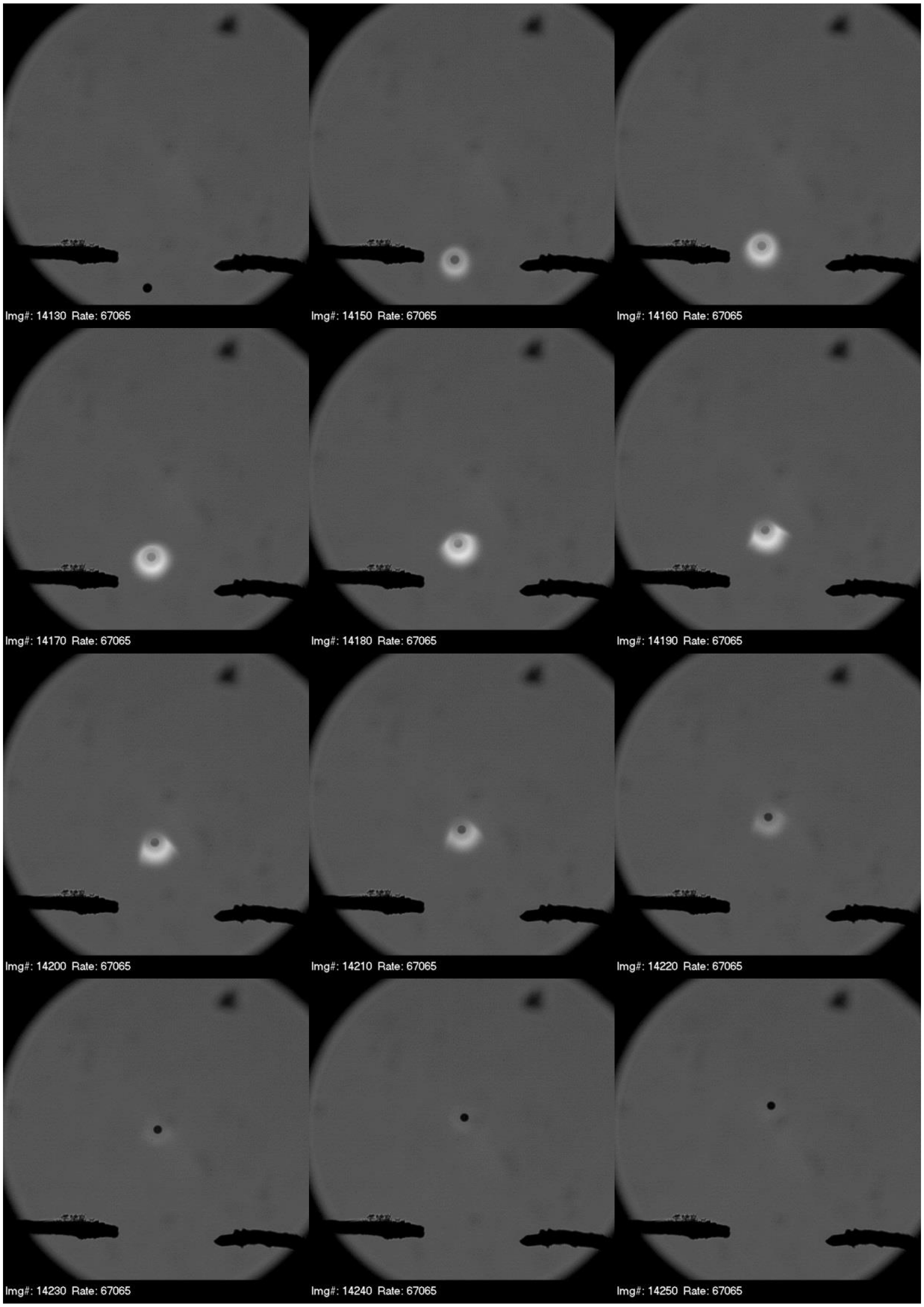

Figure 5. Image sequence of a droplet (xylene with 0.5 mol/L tin etheylhexnoate) burning in co-flowing oxygen.

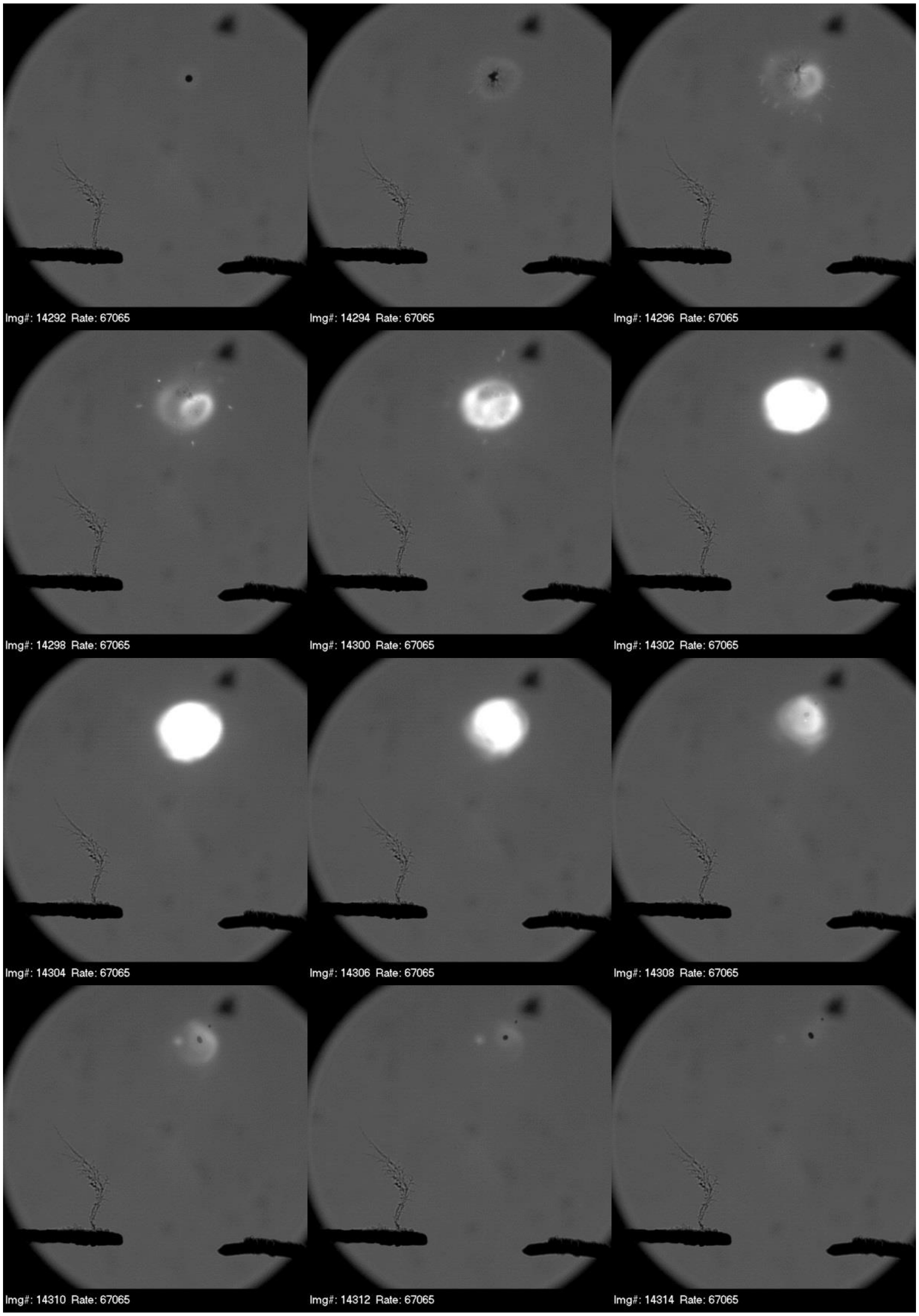

Figure 6. Image sequence of a droplet (xylene with 0.5 mol/L tin etheylhexnoate) burning with disruption and explosion, in co-flowing oxygen.

Figure 6 shows images from the acquired equipment of a single droplet (xylene with 0.5 mol/L tin etheylhexnoate) that burns with disruption and explosion. The sequence is captured at 67,065 fps with 12.5μs exposure time.

Electric Arc Triggered by Non-conductive Droplets

Droplets and particles are known to disturb electric fields, initiating breakdown and spark onset, while the fundamental mechanisms are not well understood. Interesting results have been obtained on *non-conducting* fuel aerosols which can trigger their own electrical discharge and thus induce their own ignition and combustion. Depending on the aerosol composition and characteristics, some sprays induce voltage breakdown below the threshold for air, while others raise the threshold for spark initiation. Examination of the phenomena using the high-speed camera reveals interesting processes at play. Figure 7 shows a sequence of images of droplets initiating arc over between electrodes.

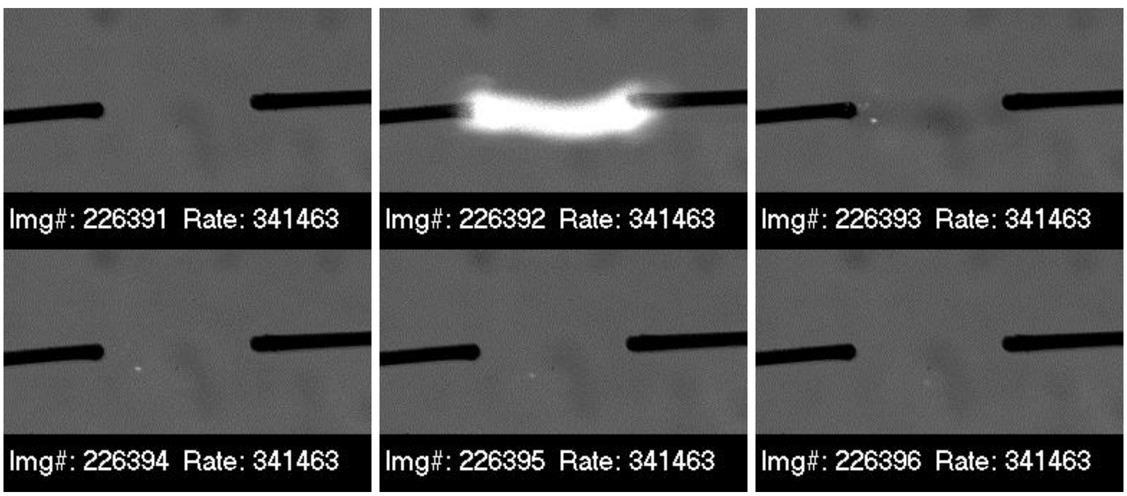

Figure 7. Images of droplets sprayed at high-voltage electrodes at 341,463 fps at 0.66μs exposure time. The spark lasts for less than 3μs.