Update On CO_2 Laser Ablation Of Polyoxymethylene At 101 kPa

John E. Sinko[a], Stefan Scharring[b], Naoya Ogita[c], Akihiro Sasoh[c], Hans-Albert Eckel[b], and Hans-Peter Röser[d]

[a]*Micro-Nano Global Center of Excellence, Nagoya University, Nagoya, Japan 464-8603*
[b]*Studies and Concepts Group, German Aerospace Center (DLR), D-70569 Stuttgart, Pfaffenwaldring 38-40, Germany*
[c]*Department of Aerospace Engineering, Nagoya University, Nagoya, Japan 464-8603*
[d]*Institute of Space Systems, University of Stuttgart, D-70569 Stuttgart, Pfaffenwaldring 31, Germany*

Abstract. Recent work has brought about a renewed interest in CO_2 laser ablation studies of polyoxymethylene, due to its potential as a test target for enhancing modern understanding of the laser ablation process. In this paper, new results taken in air at atmosphere pressure are reported, including data measured at institutions in Germany and Japan, which increase the body of literature data on CO_2 laser ablation of polyoxymethylene. The results are discussed in terms of aerospace parameters such as the momentum coupling coefficient and specific impulse, and are compared to a previous literature study. The threshold fluence is specified for ablation of polyoxymethylene by CO_2 laser radiation. Fluences higher (and lower) than previously tested for CO_2 laser ablation were studied herein, and record specific impulse values for CO_2 laser ablation of flat polyoxymethylene are also reported here.

Keywords: Laser ablation, laser propulsion, ablation threshold, polyoxymethylene, CO_2 laser
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INTRODUCTION

Two recent papers [1,2] surveyed a wide variety of literature results describing the CO_2 laser ablation of the polymer polyoxymethylene (POM). Although many studies have investigated mass removal and impulse generation properties of this interaction, various aspects of the ablation process remain mysterious. In the current paper, we unite new results taken at the German Aerospace Center (DLR Stuttgart, Germany) and Nagoya University (NU, Nagoya, Japan) with literature data from the University of Alabama in Huntsville (UAH, Huntsville, Alabama, USA) to achieve a closer understanding of the ablation behavior during CO_2 laser ablation of POM.

A photochemical model has often been applied to laser ablation, including CO_2 laser ablation of polymers. This model is based on the Bouguer-Lambert-Beer law [3], and may be expressed in terms of ablated mass areal density as:

$$= \frac{\rho}{\alpha} \ln \frac{\chi \Phi}{\Phi_a}, \qquad (1)$$

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where ρ is the density, α is the absorption coefficient, Φ is the fluence of the ablating laser beam, Φ_a is the threshold fluence for ablation, and χ (explained more fully in [2]) is a transmission term including effects from plasma and surface reflectivity. Unfortunately, the validity of the photochemical approach is in question for this interaction, partly because the C-O bond energy linking adjacent monomers in polyoxymethylene (≈3-4 eV) is so much greater than the energy per laser photon (≈0.12 eV at 10.6 m). In such a case, a thermal approach may be more appropriate. A photothermal model has also been suggested; for instance, a representation of 1-dimensional thermal diffusion into a surface has been given by Bäuerle [4], which may be related to the ablated mass, assuming a top-hat beam. Again, we express the relationship in terms of :

$$\approx 2\rho\sqrt{D\tau\ln\left(\frac{\chi\Phi}{2\rho c_p\sqrt{\pi D\tau}T_b}\right)}, \quad (2)$$

where c_p is the specific heat, D is the thermal diffusivity, T_b is the boiling or vaporization temperature, and τ is the laser pulse length. This expression implies the relationship:

$$\Phi_a = \frac{2\rho c_p T_b\sqrt{\pi D\tau}}{\chi}, \quad (3)$$

which successfully predicts the experimental ablation threshold of POM [5]. However, (2) is not linearizable (cannot be expanded to first order) at the threshold, which raises questions about its validity, since experimental data seems to support such linearity. In addition, the fit of (2) to experimental data over a wide range of fluence is inferior to that of (1) in terms of correlation, and in fact, neither (1) nor (2) satisfactorily describe the data over typical ranges of fluence for laser propulsion when physical parameters are used (versus arbitrary fitting parameters), as shown in [1,2]. In addition, the models above do not account for the presence of an ambient atmosphere, and so would be most valid in a strong vacuum. To date, not a single analytical model can account simultaneously for ambient pressure, fluence, spot area, and laser pulse length - even against a flat target. Improved modeling would facilitate advances in industry and academia; thus, as the basic analytical description of laser ablation remains an open topic, the accumulation of an adequate data set to support such development should be a prime target of the laser ablation and laser propulsion communities.

METHODS AND TECHNIQUES

New results will be reported later for ablation experiments at two institutions, each with its own facilities and equipment. A CO_2 laser was used at each of these institutions, one at the German Aerospace Center (DLR) and one at Nagoya University

(NU). The two lasers are significantly different in operation and output energies. NU uses an industrial TEA CO_2 laser (Selective Laser Coating Removal GmbH, Germany) with up to about 10 J per pulse, 90 ± 10 ns main pulse and 3-5   s tail. DLR uses a specially made electron beam-sustained CO_2 laser currently operating at up to about 150 J per pulse, 300 ± 40 ns main pulse and 7-10   s tail. In practice, the useful beam energies are a little lower than stated above; after sending the pulse through an experimental setup to the target, the energy is reduced; *e.g.*, due to reflection and absorption losses from optical components in the beam path.

For this collaborative effort, NU supplied a number of 25.8 mm-diameter, 10 mm-tall cylinders which were used in their original manufactured condition. The ablation studies at NU reported here exclusively used these cylindrical targets. Such targets were also used in Stuttgart, but DLR also provided square POM plates of 127.5 mm × 127.5 mm × 5 mm (5" × 5" × 3/16"). The experiments described in this work were performed at atmospheric pressure (101 kPa) or (at NU) in a slight vacuum of 100 kPa.

Force measurements described in this work were all made using piezoelectric force sensors (PCB Piezoelectronics, Inc., models 208C01, 208C04 and 200C20). Mass measurements for the experiments at DLR were made using a scientific balance (PCE group, model LS500) with 1 mg readability, and at NU using a different balance (Shimadzu, model AW320) with 0.1 mg readability. In practice, the measurement accuracy of both balances is at best 2-3 times the readability. At DLR, spot areas were measured with a Vernier caliper, based on the visibly ablated areas on the surface of the target material, and using thermal paper to confirm beam location and centering. This method was also used at NU, but in addition, explicit surface profilometry (*e.g.*, see [5]) was conducted to aid in determination of spot areas. Energy was measured using thermopile-type joulemeters (at NU, Gentec EO, Inc. models QE50LP-H-MB-DO and ED-500LIR; and at DLR, Ophir model PE50BB-SH-V2). At DLR, the output pulse energy was often measured by applying a calibrated scaling factor to the energy emitted from a $\phi = 5$ mm aperture at the rear of the laser cavity.

Additional details about the experimental conditions are provided, *e.g.*, in [6].

RESULTS

Results measured with the laser systems at DLR and NU are organized below in Tables 1 and 2, respectively. The symbols P, E, a, I, and m denote ambient pressure, laser pulse energy, ablated spot area, imparted impulse, and ablated mass, respectively, and N_E, N_I, and N_m denote the number of energy, impulse, and mass measurements in the sequence, respectively. Each row represents a particular experiment, including the date on which the experiment was conducted and various experimental conditions and measurements. Several record experimental conditions were achieved in this study, including the largest area sizes and fluences ever tested for CO_2 laser ablation of POM. Several record measurements were also achieved, including the smallest impulse and mass measurements for CO_2 laser ablation of POM. The joint experiments at DLR tested pulse energies from about 0.1-150 J at the target and area sizes from about 0.1-50 cm^2. Previously, the largest spot areas known to the authors had been tested by AVCO, as described in [7]. The measurements recorded at NU used net delivered pulse energies of about 1-9 J and spot areas from about 0.01-3 cm^2. The highest fluences previously measured in ablation of POM by a CO_2 laser are described in [8,9].

TABLE 1: Joint Experiments at DLR using 150 J CO_2 laser (n.d. denotes 'no detection')*

Date	***P* [kPa]**	***E* [J]****	N_E	***a* [cm²]**	***I* [Ns]**	N_I	***m* [mg]**	N_m
7/14/2009	101	21.9±0.4	3(1)	37.2±0.7	220±50	3(1)	2.0±0.9	3(1)
7/14/2009	101	16.7±0.2	3(1)	27.4±0.6	140±20	3(1)	1.3±0.8	3(1)
7/14/2009†	101	12.0±0.1	4(1)	22.2±0.6	115±3	4(1)	5±4	3(1)
7/14/2009	101	7.98±0.08	4(1)	13.9±0.4	50±5	3(1)	0.5	2(2)
7/14/2009†	101	4±2	4(1)	5.4±0.2	14±3	3(1)	5.0±0.7	2(1)
7/14/2009	101	2.51±0.02	3(1)	1.7±0.1	2±2	3(1)	0.5	1(2)
7/14/2009†	101	0.90±0.01	3(1)	0.11±0.01	n.d.	n/a	n.d.	n/a
7/15/2009†	101	2.55±0.02	3(1)	4.1±0.4	9±4	3(1)	n.d.	n/a
7/15/2009	101	6.9±0.1	7(1)	7.2±0.4	301±5	4(1)	1.1±0.6	7(1)
7/15/2009	101	13.6±0.2	3(1)	14.4±0.6	810±30	3(1)	3±1	3(1)
7/15/2009	101	22.7±0.3	3(1)	22.2±0.7	1500±60	3(1)	6±2	2(1)
7/15/2009	101	33.2±0.3	3(1)	29.0±0.7	2210±60	3(1)	8±4	3(1)
7/15/2009	101	46.2±0.5	3(1)	41.1±0.9	3020±50	3(1)	10±6	3(1)
7/15/2009	101	61.4±0.9	3(1)	51±1	3420±150	3(1)	13±1	3(1)
7/15/2009	101	11.9±0.3	5(1)	8.8±0.5	920±130	5(1)	3.0±0.9	3(1)
7/15/2009	101	23.5±0.4	3(1)	16.4±0.7	2110±50	3(1)	6±1	3(1)
7/15/2009	101	39.0±0.7	5(1)	24.0±0.8	4000±200	4(1)	10±1	4(1)
7/15/2009	101	58.0±0.7	3(1)	33.2±0.9	6020±90	3(1)	17±4	3(1)
7/15/2009	101	82.2±0.3	3(1)	43.4±0.9	8300±500	3(1)	21±2	3(1)
7/15/2009	101	110±2	3(1)	53±1	9100±400	3(1)	27±7	3(1)
7/15/2009	101				8100±900	3(1)	29±2	3(1)
7/15/2009	101	4.5±0.1	3(1)	3.8±0.4	271±9	3(1)	0.6±0.5	2(≈1)
7/15/2009†	101	0.49±0.01	3(1)	1.4±0.4	n.d.	n/a	0.3	1(3)
7/15/2009	101	1.93±0.06	4(1)	1.4±0.2	50±7	3(1)	0.3	2(2)

* The format means: "number of measurements(laser shots per *each* measurement)"; in a few cases (*e.g.*, due to laser misfire or an oscilloscope error), a rigid number of shots was impossible in a given experiment. Thus, for instance, 5(≈6) means 5 measurements (about 6 shots per measurement).
**The ± here denotes experimental jitter in pulse energy; in addition, known error components include 4.5% from the calibration factor (558 ± 25) used for energy measurement at the rear aperture, and a systematic error of the particular Ophir PE50BB detector of about 5%. A full error value may be computed by adding these terms in quadrature.
†These specific 5 rows of data were excluded from the linear fit in mass removal as outliers

By explicitly providing the data from the experiments in the form of tables, we hope to enhance its usefulness to current and future laser propulsion researchers. Below, we will consider several common laser propulsion parameters including the momentum coupling coefficient C_m, the specific impulse I_{sp}, and the ablated mass per input pulse energy ξ. More explicitly,

$$C_m = \frac{I}{E}, \tag{4}$$

$$I_{sp} = \frac{I}{mg}, \tag{5}$$

where $g \approx 9.8$ is the constant of gravitational acceleration at sea level, and

$$\xi = \frac{m}{E}. \tag{6}$$

Note that the inclusion of g is only due to the definition of specific impulse, an aerospace engineering parameter. Otherwise, g has no physical significance in this expression.

TABLE 2. Supporting Experiments at NU using 10 J CO_2 laser (n.d. denotes 'no detection')

Date	*P* [kPa]	*E* [J]	N_E*	*a* [cm^2]	*I* [Ns]	N_I*	*m* [g]	N_m*
1/28/2009	101	4.51+0.03	10(1)	2.49±0.04	404±17	22(1)	730±160	23(≈1)
1/29/2009	101	5.92±0.03	5(1)	3.23±0.05	742±21	10(1)	1020±380	10(1)
2/8/2009	101	1.54±0.01	3(1)	0.914±0.003	82±12	5(1)	270±60	5(1)
2/8/2009	101	1.92±0.01	5(1)	0.86±0.02	208±30	4(1)	313±70	5(≈1)
2/8/2009	101	1.54±0.01	5(1)	0.71±0.02	186±17	4(1)	320±150	5(1)
2/8/2009	101	1.60±0.01	5(1)	0.16±0.03	240±12	4(1)	245±100	5(≈1)
2/8/2009	101			0.09±0.02	293±44	6(1)	260±60	5(1)
2/12/2009	101	2.97±0.02	5(1)	0.37±0.04	494±39	3(1)	n.d.	n/a
2/12/2009	101			0.37±0.04	560±100	5(1)	550±60	4(1)
2/13/2009	101	3.25±0.02	5(1)	0.10±0.01	465±45	5(1)	320±100	4(1)
2/13/2009	101			0.096±0.005	471±59	5(1)	310±120	6(1)
2/19/2009	101	2.57±0.01	5(1)	0.024±0.002	248±18	8(1)	72±33	5(1)
2/25/2009	100	2.56±0.05	5(1)	0.018±0.001	276±25	22(1)	70±30	5(≈5)
2/26/2009	100	2.56±0.03	3(1)	0.454±0.008	428±46	24(1)	530±30	5(5)
2/27/2009	100	2.69±0.04	6(1)	0.140±0.005	440±20	24(1)	400±40	5(5)
3/4/2009	101	1.97±0.04	5(1)	3.9±0.7	13±1	20(1)	116±27	5(5)
3/4/2009	101	1.70±0.02	6(1)	3.2±0.8	12±2	22(1)	86±35	5(5)
3/4/2009	101	1.44±0.02	5(1)	2.9±0.3	10±1	22(1)	82±28	5(5)
3/4/2009	101	1.19±0.02	5(1)	2.1±0.5	9.1±0.9	25(1)	88±47	5(5)
3/4/2009	101	0.956±0.009	5(1)	1.6±0.6	7±1	21(1)	44±16	5(5)
3/4/2009	101	0.749±0.007	5(1)	1.2±0.4	5.0±0.7	22(1)	38±15	5(5)
3/4/2009	101	0.572±0.005	5(1)	0.9±0.4	4.7±0.7	20(1)	32±19	5(5)
3/4/2009	101	0.396±0.004	5(1)	0.6±0.3	3.7±0.5	21(1)	22±4	5(10)
3/4/2009	101	0.251±0.002	5(1)	0.3±0.2	1.8±0.5	21(1)	20±14	5(10)
10/8/2009	101	1.46±0.01	5(1)	3.1±0.3	4.0±0.2	8(1)	n.d.	n/a
10/8/2009	101	1.34±0.01	5(1)	3.1±0.3	0.9±0.2	8(1)	n.d.	n/a
10/8/2009	101	1.14±0.01	5(1)	3.0±0.3	0.6±0.1	2(1)	n.d.	n/a
11/16/2009	101	8.45±0.04	5(1)	0.016	352.2	1(1)	0.047	1(1)

*The format means: "number of measurements(laser shots per each measurement)"

For consideration of ablated mass, we are particularly interested in the value of the threshold fluence, therefore we shall focus on the data below 2.5 J/cm^2 for consideration of the ablated mass areal density. The other data relevant to laser propulsion is better represented in terms of commonly-used aerospace parameters including the momentum coupling coefficient C_m, specific impulse I_{sp} and the mass removal per pulse energy ξ. Returning to the topic at hand, the results for near the ablation threshold may be plotted in terms of fluence and fit by linear regression. In that case, we find the results as displayed in Figure 1.

In Fig. 1, the linear regression fit for the data taken at DLR is distorted somewhat by two outliers at low fluence. Excluding these two points, there is very good agreement between the three sets of measurements, pinning down the threshold fluence to around 0.4-0.6 J/cm^2. Thus, an additional linear regression analysis was carried out on this particular data set with the outliers removed. The results of this analysis are provided in Table 3.

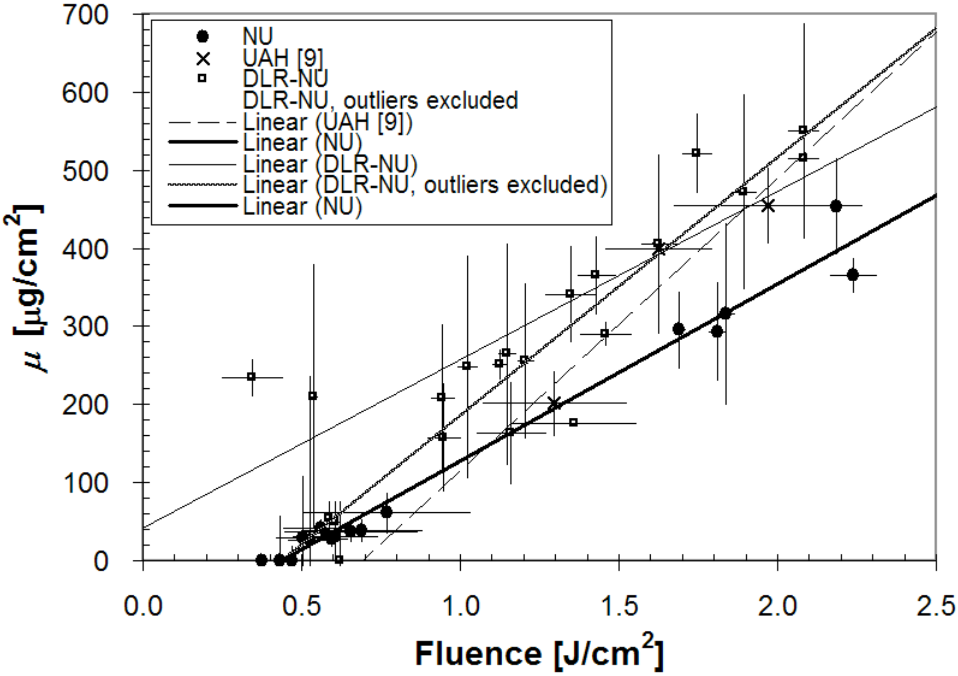

FIGURE 1. (Φ) data fit by linear regression in the threshold region. DLR-NU denotes 150 J CO_2 laser data taken at DLR-Stuttgart during the DLR-NU collaboration, NU denotes 10 J CO_2 laser data taken at Nagoya University, and UAH denotes data taken with the 20 J CO_2 laser at the University of Alabama in Huntsville.

Table 3. Fitting results for (Φ) data

Data set	Regression equation for [g/cm^2] near Φ_a [J/cm^2]	r_{adj}^2	Φ_a [J/cm^2]
NU (<10 J)	= 220.3 Φ - 97.3	0.972	0.44
UAH (<20 J)	= 374.4 Φ - 259.1	0.896	0.69
DLR-NU (<150 J)	= 215.6 Φ + 42.2	0.272	0.20
DLR-NU* (<150 J)	= 330.6 Φ - 143.3	0.907	0.43

*This data set specifically excludes 5 data points (outliers) as noted in Table 2.

In fact, the trend lines from both the DLR (outliers excluded) and NU data sets are in close agreement, placing the ablation threshold fluence slightly above 0.4 J/cm^2.

Using (3) and the typical thermal and physical values for POM from [5], we would expect the threshold to lie between 0.33-0.40 J/cm^2 for the 7-10 s pulse at DLR-Stuttgart, and between about 0.22-0.28 J/cm^2 for the 3-5 s pulses at NU and UAH.

The area values used to compose the fluence in the UAH data set were measured by a Vernier caliper from brown marks left on flat surfaces of gray polyvinylchloride (PVC) placed in the same plane as the POM surface. It was originally assumed that the brown marks were patterns of char, but it is now known that the PVC samples used were in fact a fiber-filled variety of PVC (confirmed by optical microscopy); the brown color is merely representative of the color of the underlying fibers. In any case, PVC is now known to have a significantly different ablation threshold compared to POM. It is therefore unsurprising that the UAH result deviates slightly from the recent, more accurate efforts.

Figure 2 graphically displays the corresponding fluence-dependent results for ξ, the ablated mass per input laser pulse energy.

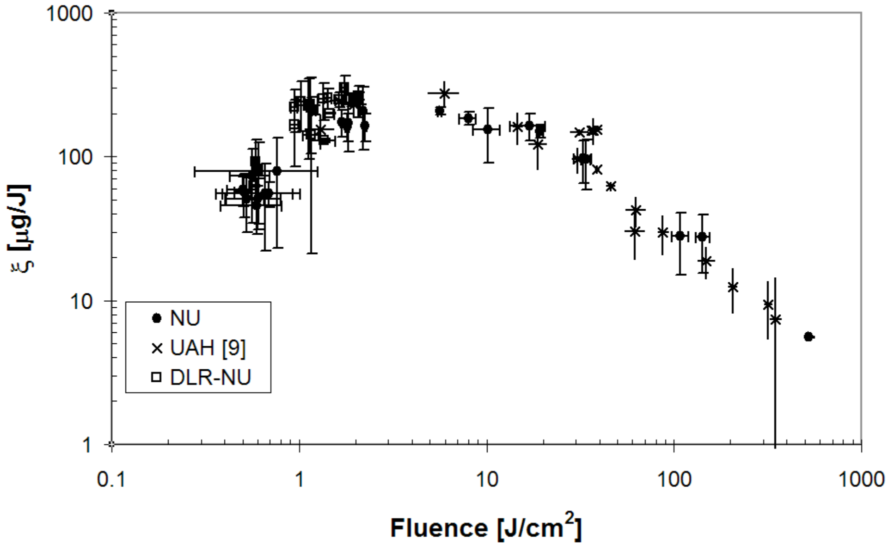

FIGURE 2. Experimental data for $\xi(\Phi)$; further explanation of the legend is given in Fig. 1.

Figure 2 shows moderate increase in ξ above the ablation threshold until about 2-5 J/cm^2, when a plateau is reached. At around 10-20 J/cm^2, this is followed by a tail-off in ablated mass per pulse energy. Although the illustrated maximal ξ region is less interesting for laser propulsion, it is important for industry, and basically specifies the optimized regime for CO_2 laser processing of POM in atmosphere. The tail-off to the right is due to plasma shielding; *i.e.*, the increase in plasma absorptivity as the fluence is increased above the plasma threshold.

Experimental results for C_m are shown in Figure 3. In Figure 3, the data generally confirm previous results for momentum coupling. The fluence region for maximum C_m (about 150-190 Ns/J) is between about 5-15 J/cm^2. These maximum values are slightly higher than (but consistent with) the results in [9]. At the highest fluence measured, above 500 J/cm^2, $C_m \approx 40$ Ns/J. Here, we also report some of the lowest fluence values ever measured for CO_2 laser ablation of POM, well-below 1 Ns/J, at a fluence very close to the ablation threshold. These sensitive measurements are an indication of the quality of the experimental apparatus.

Figure 4 shows the measurements of specific impulse at the three institutions. In Figure 4, we have measured the highest recorded I_{sp} for CO_2 ablation of a flat POM surface, over 750 s, at a fluence of over 500 J/cm^2, where the latter area measurement was confirmed by profilometry. The curve of I_{sp} appears to consist of three major regions: (1) a strong increasing region from the ablation threshold until about 2-5 J/cm^2 fluence; (2) a plateau region from about 5-30 J/cm^2 fluence characterized by little to slight growth in I_{sp} and (3) a return to an increasing upper bound on I_{sp} above 30 J/cm^2 fluence as the plasma regime is reached, accompanied by increasing scatter in the data due to plasma shielding of the ablation surface.

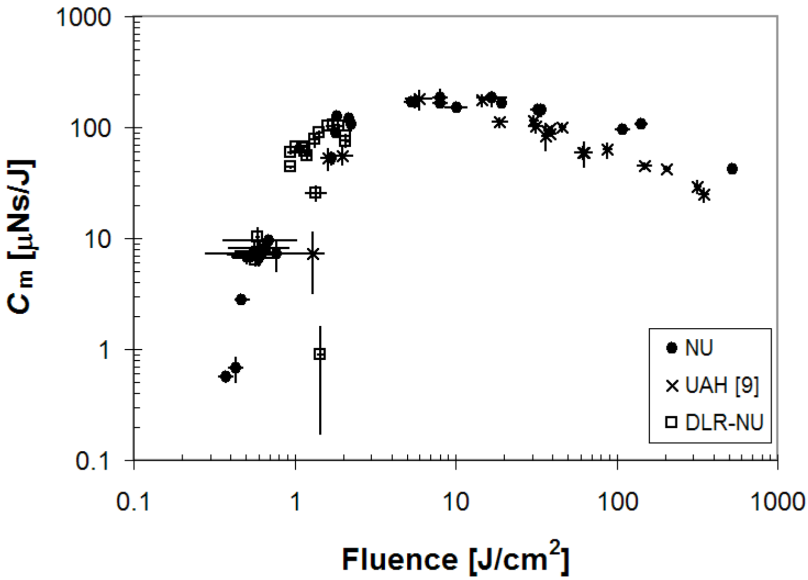

FIGURE 3. Experimental data for $C_m(\Phi)$; further explanation of the legend is given in Fig. 1.

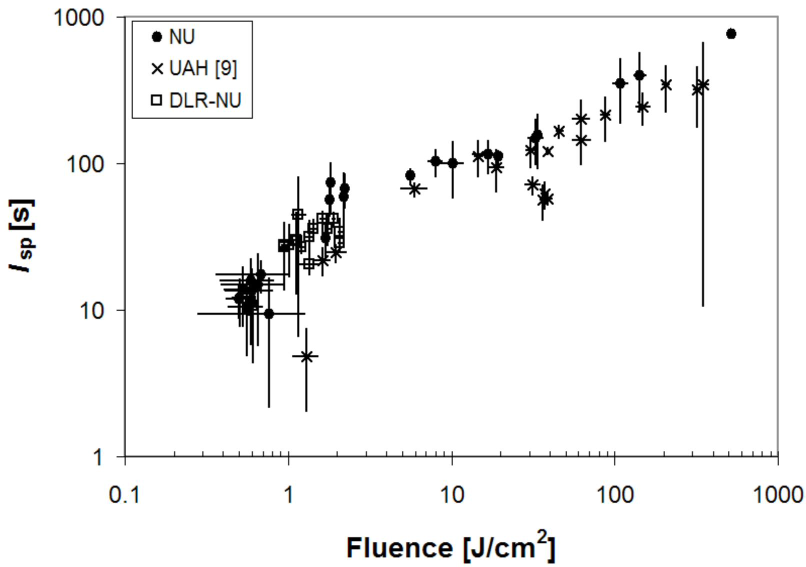

FIGURE 4. Experimental data for $I_{sp}(\Phi)$; further explanation of the legend is given in Fig. 1.

CONCLUSIONS

In this paper, we reported results for ablation of POM in atmospheric conditions from experiments conducted at the German Aerospace Center (DLR-Stuttgart) and Nagoya University. The experiments in the ablation threshold region allowed a more rigorous confirmation of the ablation threshold fluence value, and confirmed other recent work. The wider range of fluences used in the experiments as a whole extended the upper and lower boundaries of reported fluence and area conditions for measurements for CO_2 laser ablation of polyoxymethylene, as well as representative parameters such as the momentum coupling coefficient and specific impulse. Additional experiments and analytical work are necessary to improve the understanding of ablation models, and in particular, more study in vacuum conditions is needed.

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