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INVESTIGATION OF LASER INDUCED DEPOSIT FORMATION UNDER SPACE CONDITIONS

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ABSTRACT

In this paper comprehensive investigations of laser induced deposit formation are reported. In a high vacuum chamber ($p < 10^{-6}$ mbar) different space relevant materials containing epoxy, silicone and polyurethane based components were tested under space conditions. The experiments were performed with a pulsed Nd:YAG laser with peak fluences up to 2.5 J/cm² at 355 nm wavelength and 3 ns pulse width. Additional tests were performed with an UV cw laser diode at 375 nm and 10 mW mean power. The onset and growth of the deposits was monitored in-situ and online by UV induced fluorescence imaging. The influence of roughness, temperature and chemical composition of the optical surface on the deposition process was investigated. Time-of-flight secondary ion mass spectroscopy (ToF-SIMS) was used for chemical characterization of the deposits. Furthermore the influence of deposits on the UV-transmission of the optics was estimated.

1. INTRODUCTION

The European Space Agency (ESA) plans to launch several LIDAR systems in space within the next five years (ADM-Aeolus, EarthCare, Bepi-Colombo). The stated aims of these missions are detailed earth atmosphere observations for improved climatology and meteorology predictions as well as planetary surveying. The suited LIDAR systems comprise high energy pulsed lasers in the IR and UV spectral range. Under the vacuum conditions of space especially the outgassing of organic materials is inevitable, in spite of using only materials with low outgassing rates (total mass loss (TML) < 1%, collected volatile condensable material (CVCM) < 0.1%). The interaction of intense laser radiation with volatized material's constituents gives rise to a high risk of deposit formation on the optics and consequently to a drastically reduced lifetime.

2. EXPERIMENTAL

The arrangement of the experiment is shown schematically in Fig. 1. A detailed description can be found elsewhere [1].

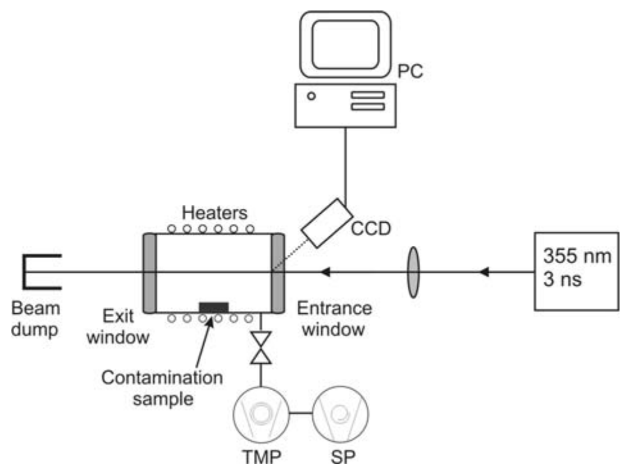

Fig.1: Experimental setup for laser induced contamination testing (TMP: turbo-molecular pump, SP: scroll fore pump)

Space conditions were simulated using an oilfree pumped vacuum chamber with a base pressure better than 10^{-6} mbar. Coated and uncoated fused silica windows (diameter 1", thickness: 3mm) served as optical test specimens. The total amount of the contamination material was in the range of 0.3 – 0.5 g, coated on 5 x 5 cm² aluminium sheets. Heating ribbons are mounted on the outside of the chamber for indirect heating of the contamination samples. Before each new run the chamber was baked out at 165°C for 24 hours to prevent cross-contamination between consecutive tests. Prior to each new material test blank tests without any contamination sample were performed, using the same temperature, pressure and laser parameters. In case of detecting any fluorescence or depositions on the windows, the bake-out was repeated until the blank test was successful. In case of deposit formation under UV irradiation, the organic compounds in the deposit are excited to fluorescence. The fluorescent deposits were recorded in-situ and online by a high sensitive electron multiplying CCD (EM-CCD) with a Peltier-cooled sensor (-20°C) and two pre-amplifiers (Andor, Luca DL-6588M-TIL). The signal to noise ratio is very high. The quantum efficiency reaches 50% in the 400 nm - 700 nm wavelength range. After each test the surface structure and thickness of the deposits was analyzed by white light interference microscopy. By comparison of the surface contour maps with the EM-CCD fluorescence images a very good correlation between deposit thickness and fluorescence intensity was found [2]. The total amount of deposit in dependence of laser exposure time was estimated in the following way from the fluorescence pictures: the gray scale values in a circular area at the beam position were integrated and recorded online. The diameter of the integration circle was chosen five times larger than the beam diameter.

3. DYNAMIC OF DEPOSIT FORMATION

The evaluation of in-situ recorded fluorescence images offers the possibility to gain insight into the dynamic of deposit growth. Fig. 2a shows a Nomarski micrograph of a typical deposit grown on an uncoated fused silica optic after a test with a polyurethane based contaminant. The irradiation parameters were the following: laser fluence: 0.4 J/cm², wavelength: 355 nm, pulse width: 3ns, repetition rate: 100 Hz, shots: 10 Mio. The beam profile of the laser is Gaussian. The contaminant temperature was 40°C. Fig. 2b shows the linescan of the surface profile of the deposit. The crater or doughnut like structure is very typical for laser induced deposits at high fluences. Fig 3. shows a series of fluorescence pictures, taken at the onset of a deposition process. By comparison of the surface profile of the deposit after the test with the corresponding fluorescence picture a calibration of the fluorescence intensity to deposit thickness was performed. At the beginning a steady growth of the deposit is observed with a pancake-like structure. Then a competing process of deposit removal starts at the center of the deposit, i.e. the region of highest laser intensity. After this the removal of material dominates and the "doughnut" structure develops with an increasing central hole. The removal process is probably caused by increasing absorption and thus increasing temperature with rising deposit thickness. Potentially the absorption is further enhanced by material decomposition and formation of carbon rich

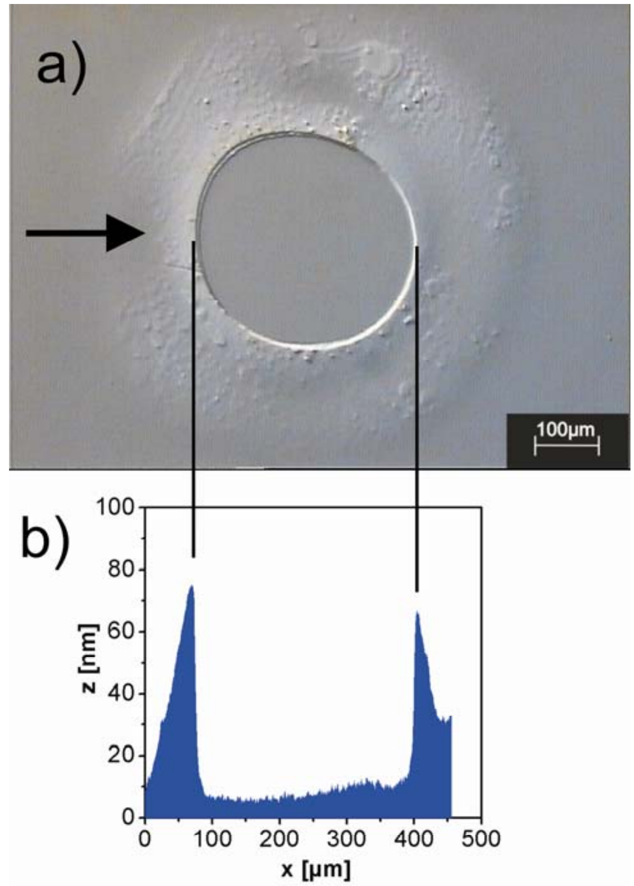

Fig.2: Nomarski micrograph (top) and surface line scan (bottom) of a typical laser induced deposition at high fluence

residues. We found in our tests that the transition from "pancake" to "doughnut" growth occurred earlier when applying higher laser fluences. In case of very low fluences (< 0.1 J/cm^2) "pancake" growth dominates for most contaminant materials. The size of the central hole in the doughnut like deposits also increases with increasing laser fluence (Fig.4). From a comparison of deposit surface profiles with the corresponding beam profiles (1/e² beam diameter 360 µm) we estimated the threshold value above which no deposit occurs in the center of the beam to approximately 0.12 J/cm^2 for a test series with a silicone based glue.

For an unambiguous comparison of different contaminants in terms of deposit formation activity it is favourable to investigate pure pancake growth without the competing removal process. As already mentioned, this can be realized in case of a pulsed laser by irradiating with low fluence. An alternative is to use a cw laser diode. Fig. 5 shows the appropriate deposit growth dynamic calculated from the corresponding fluorescence pictures. The wavelength of the laser

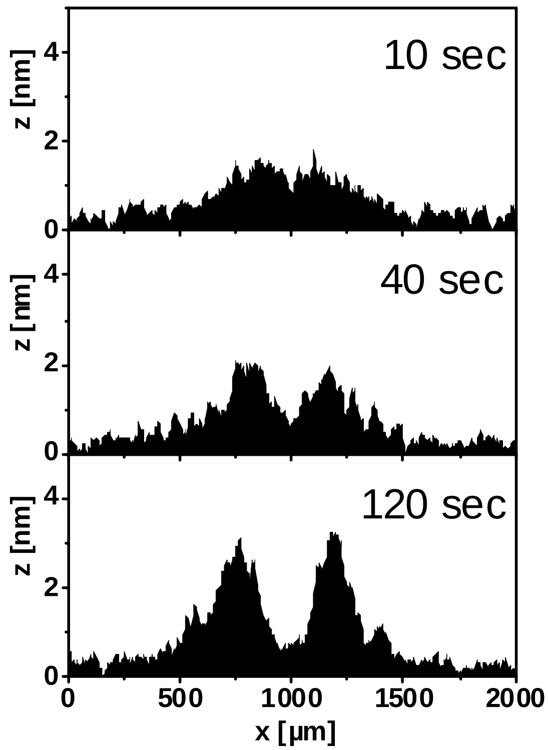

Fig. 3: Dynamic of deposit formation for doughnut like growth. (Peak fluence: 2.5 J/cm², wavelength: 355 nm, pulse width: 3ns, repetition rate: 100 Hz, epoxy based contaminant, temperature: 100 °C)

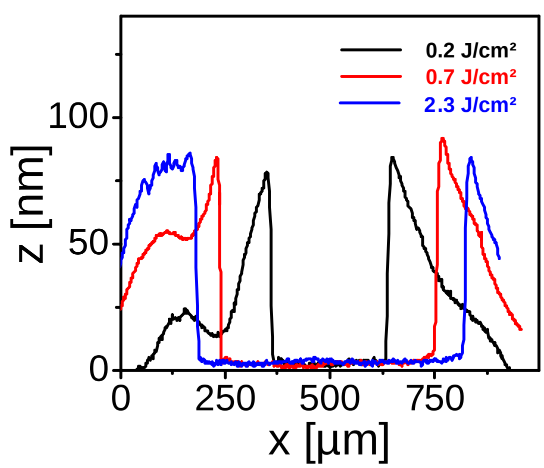

Fig. 4: Influence of peak fluence on the deposit structure (silicone based contaminant, T = 40°C, 10^7 pulses, pressure: $p<10^{-5}$ mbar, optic: uncoated fused silica)

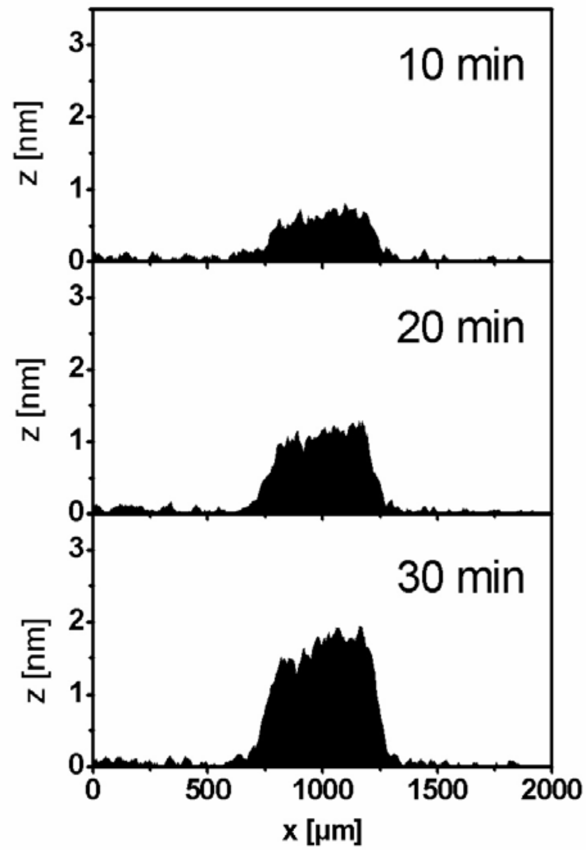

Fig. 5: Dynamic of deposit formation for pancake like growth (cw-laser diode, 375 nm, mean power: 10 mW, epoxy based contaminant, temperature: 100 °C)

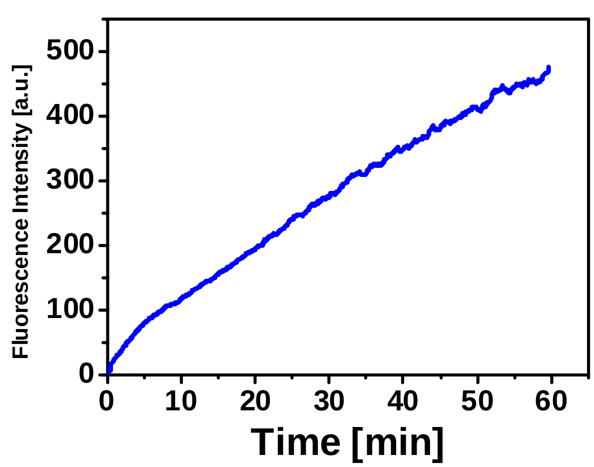

Fig. 6: Integrated fluorescence intensity in dependence on growth time as measure for total deposited material for pancake like growth

diode was 375 nm, the mean power 10 mW. The mean laser intensity was similar to the test with pulsed irradiation, shown in Fig. 3. Fig. 6 shows the integrated fluorescence gray scale values of the deposit as a measure for the total amount of deposited material. A nearly linear growth as a function of laser exposure time is observed.

4. INFLUENCE OF OPTICAL SURFACE PROPERTIES ON DEPOSIT FORMATION

The interaction of volatized molecules with the surface of the optical sample is one of the initial processes for deposit formation. We therefore investigated the influence of different surface properties (temperature, roughness and chemical composition) on this interaction. MgF_2 and SiO_2 are typical materials used in optical coatings. Fig. 7 shows the results of contamination tests with these compounds as top layers for AR coated samples. The tests were performed with peak fluences between 0.2 and 1.3 J/cm^2. As contaminant a two part polyurethane based glue was used. The contaminant temperature was 40°C. The

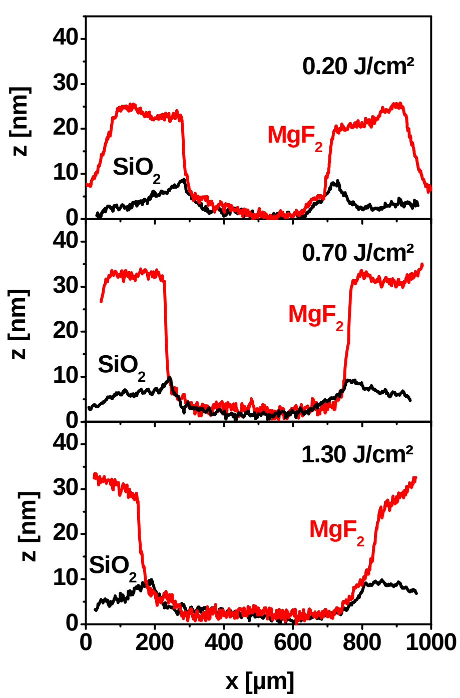

Fig.7: Surface profiles of deposits built-up on different optical surfaces (red: SiO_2, black: MgF_2) Irradiation wavelength: 355 nm, pulse width: 3ns, repetition rate: 100 Hz, 10^7 pulses, contaminant: polyurethane based glue, temperature: 40 °C)

surface profiles of the deposits show, that the growth rate on MgF_2 is clearly higher than on SiO_2. Similar results were obtained with a silicone based contaminant.

To investigate the influence of surface roughness on deposit growth, tests with uncoated fused silica samples with mean roughness R_a = 0.1, 0.3, 0.5 and 1.5 nm were performed. There was no significant difference in deposit growth rate observable.

Fig. 8. shows the influence of surface temperature on the deposition process. The optical samples were uncoated fused silica windows, which were cooled resp. heated to 17, 30 and 50°C. The contaminant, an epoxy based glue was heated to 100°C. The deposit growth rate clearly increases with decreasing surface temperature. The adsorption of volatized atoms and molecules on surfaces is a temperature dependent process and seems to play an important role in the laser induced deposition process.

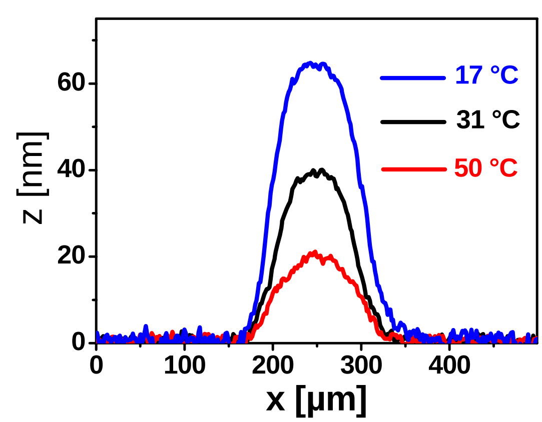

Fig. 8: Influence of surface temperature on deposit growth (cw laser diode, wavelength 375 nm, mean power 10 mW, contaminant:epoxy based glue, temperature 100°C)

5. DEPOSIT COMPOSITION

The chemical composition of the deposits was investigated by ToF-SIMS. Fig. 9 shows a distribution mapping of a deposit grown on a MgF_2 coated fused silica optic. The contamination sample was a silicone based material heated to 40°C. The laser parameters were the following: wavelength 355nm, pulse length 3 ns, repetition rate 100 Hz, peak fluence 1.5 J/cm^2, 10^7 shots. The distribution of the chemical components reproduce the crater-like structure found in the surface profile analysis. In the ring hydrocarbons are dominant (dark-blue). In the non-irradiated zone unaffected MgF_2 coating was detected (green). At the outer edge of the central hole degraded and non-degraded silicone from the contamination sample is dominant (red). Obviously silicone can exist at higher fluences than hydrocarbons. A similar behaviour was found for a deposit produced in a test with IR-irradiation at 1064 nm.

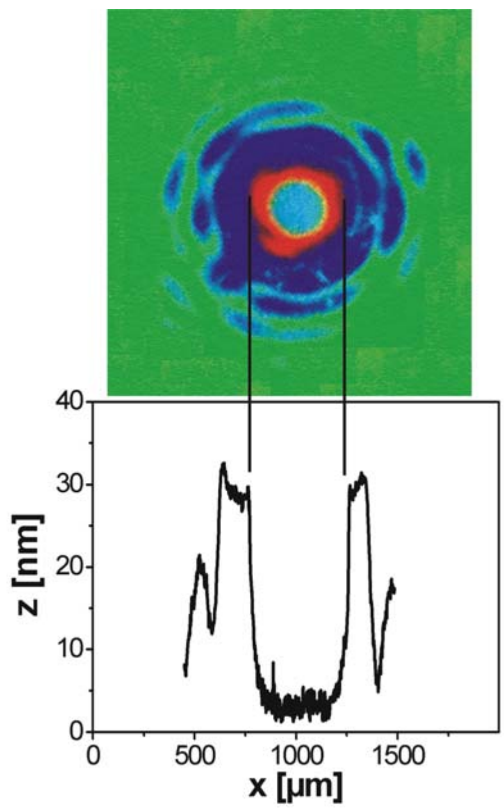

Fig. 9: Chemical composition mapping (top) and surface profile of a deposit (green:MgF_2, red: silicone, blue: hydrocarbons)

6. INFLUENCE OF DEPOSITS ON OPTICAL PROPERTIES

The quality reduction of optical components through deposit formation was estimated by transmission measurements. Fig. 10 shows a comparison between an unaffected area and a 50 nm thick deposit on an uncoated fused silica sample. The transmission in the UV wavelength range is reduced by 8%. Even for a 15 nm thick deposit a transmission reduction of 5% was found.

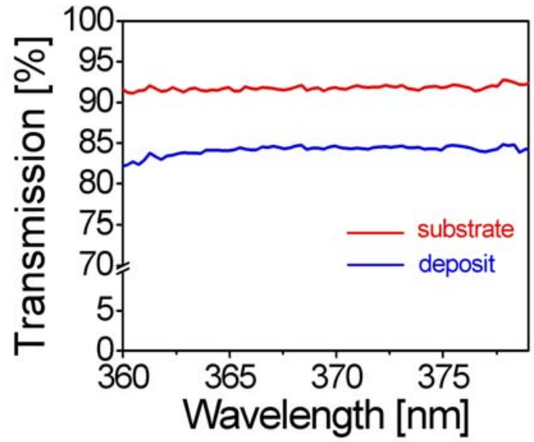

Fig. 10: Transmission reduction by a deposit (thickness 50 nm, contaminant: epoxy based glue)

6. CONCLUSIONS

Laser induced deposit formation was investigated under space conditions. Epoxy, silicone and polyurethane based contaminants were tested. Different growth modes were observed. At high fluences, a deposit removal in the center of the beam is evident, leading to characteristic crater-like structures. At low fluences a homogenous growth was found. The formation of depositions depends on the surface composition of the optical samples. For MgF_2 coated optics the deposition growth rate is 2 - 3 times larger than on SiO_2 coated ones. The deposition rate increases with decreasing surface temperature. The surface roughness has no significant influence on the deposition process. Surface analysis of the deposits showed that silicone constituents can exist at higher laser fluences than hydrocarbons.

5. ACKNOWLEDGEMENTS

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6. REFERENCES

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