

Modelling and Diagnostic of Pulsed Laser-Solid Interactions. Applications to Laser Cleaning.

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ABSTRACT

The present study concerns the cleaning of materials using pulsed laser irradiation and it summarises the most recent results obtained by the collaborative research of different European groups, within the framework of a European program for training and mobility of researchers. A series of pulsed lasers, which emit at various wavelengths (from UV to IR) with short duration of pulse (few nano-, pico- or femto-seconds), is used for the removal of metallic, ceramic and organic pollutants from contaminated solid surfaces of different natures. The scientific results obtained so far are focused on the laser cleaning of silicon wafers from sub-micrometer particles, the theoretical modelling of particles removal mechanism during dry laser cleaning, the removal of oxide layers from oxidised metals and alloys, as well as on the development of laser imaging as a diagnostic tool for the estimation of the efficiency of the proposed cleaning technique.

Keywords: Laser cleaning, decontamination, surface treatment, particles removal, oxide removal, modelling, laser imaging

1. INTRODUCTION

The Training and Mobility of Researchers (TMR) European project, with reference code ERB FMRX-CT98-0188 (1998-2002), is dealing with the laser cleaning of materials. The objective of this project is to investigate the fundamental physical and chemical interactions, which are taking place during pulsed laser irradiation of solid materials, in order to confirm the interest of laser cleaning technique as a reliable tool for industrial applications.

The, widely used until now, chemical and/or mechanical cleaning presents many disadvantages (no selectivity, no good control, stress introduction, etc), which turn these conventional techniques unsuitable for many applications. On the contrary, the non-conventional laser cleaning technique seems to be the adequate one in a large variety of domains: semiconductor and nuclear industry, manufacturing...^{1, 2, 3, 4, 5, 6, 7}. However, the cleaning efficiency of a laser source is strongly dependent on the properties of the pollutant and on the surface to be cleaned, as well as on the nature of the bonding between them.

In the frame of our project, a series of pulsed lasers, which emit at various wavelengths (from UV to IR) with short duration of pulse (few nano-, pico- or femto-seconds), is used for the removal of metallic, ceramic and organic pollutants from contaminated solid surfaces of different natures. The first results obtained by the collaborative research between the groups participating at the ERB FMRX-CT98-0188 network, concern four main topics:

- Laser cleaning of silicon surfaces from sub-micrometer particles. Nano- and pico-second pulsed lasers were used for the removal of particles from Si surfaces and the cleaning fluence thresholds were determined for both dry⁸ and steam laser cleaning^{9, 10}. A theoretical model based on the surface acceleration was also developed, in order to better understand the mechanism of particles removal during dry laser cleaning¹¹.

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- Laser cleaning of ferrous and aluminium alloys from oxide surface layers. In order to increase the laser cleaning efficiency of oxidised iron, the influence of both the environment and of the substrate's polarisation on the optical properties of the surface to be cleaned was investigated^{12, 13}. In the case of Al alloys, the occurred phenomena during cleaning were studied and their dependence on the fluences and the number of pulses applied was determined¹⁴.
- Implications for the laser cleaning of molecular substrates. The photochemical effects during UV laser ablation were investigated in the case of doped polymer systems^{15, 16}.
- Laser imaging as a tool for laser cleaning studies. The developed technique was based on plasmon resonance method and permitted shockwave imaging with high time and spatial resolution¹⁷.

2. RESULTS

2.1. Removal of particles

The removal of sub-micron particles Si surfaces is a permanent problem for the microelectronics industry. Unless removed these contaminants cause high yield losses in the production of ICs. For these contaminant particles Van der Waals-force is the dominant adhesion force¹⁸. To overcome this force, accelerations in the order of $>10^7g$ are needed. Such high accelerations can not be provided by conventional cleaning techniques like ultrasonics¹⁹. Recently it could be demonstrated that laser cleaning is a very promising tool for the removal of such nanocontaminants^{2, 20, 21, 22, 23, 24, 25}. In this context two laser cleaning methods have been developed: "dry" and "steam" laser cleaning. During dry laser cleaning, the particles are ejected from the surface by the thermal expansion of the substrate due to irradiation with a pulsed laser. In the case of steam laser cleaning process, a thin liquid film is condensed onto the substrate and subsequently the system is irradiated with a short laser pulse. The liquid is heated directly by heat transport from the Si substrate. Bubble nucleation at the solid/liquid interface and the subsequent explosive vaporisation of the liquid cause the removal of the contaminants.

2.1.1. Laser cleaning of silicon wafers

Dry and steam laser cleaning was performed to remove colloidal particles (polystyrene (PS), SiO_2) with diameters in the range of 60-800 nm that were homogeneously dispersed as isolated spheres on the surface of Si wafers (figure 1).

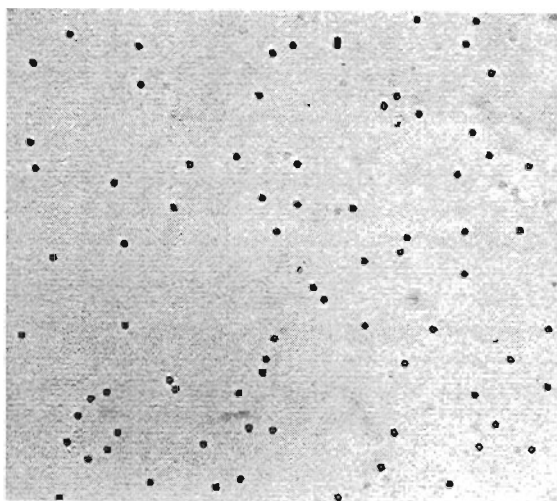


Figure 1. Si wafer with a random dispersion of isolated colloidal PS spheres (480 nm in diameter) (see ref.9).

The efficiency of the process was determined by measuring the particle surface concentration via light scattering with a HeNe-laser before and after cleaning. As cleaning laser we used a Nd:YAG one ($\lambda=532$ nm, 7 ns) and a pulsed dye amplifier seeded either with a cw dye laser beam or with a synchronously pumped mode-locked dye laser ($\lambda=532$ nm, 7 ns)²⁶.

For the steam laser cleaning with ns pulse it was found a universal cleaning threshold of 50 mJ/cm² valid for all the particles (60-800 nm in diameter, material PS and SiO₂, 532, 5 ns). At the wavelength of 583 nm and a pulse duration of 2.5 ns this threshold was also valid for the 800 nm PS spheres examined^{9, 10}. Dry cleaning was found to be less efficient than steam cleaning in the case of ns pulses⁸.

Decreasing the pulse length from 2.5 ns to 30 ps resulted in sufficiently lower cleaning thresholds for both dry and steam laser cleaning⁹. In the case of steam laser cleaning, the dependence of the fluence threshold on the duration of the pulse (figure 2) could be correlated with the absorption of the laser energy by the liquid layer by numerical simulations. From figure 2 it is clearly visible that it is possible to achieve a high efficiency and a very low threshold by using picosecond laser pulses.

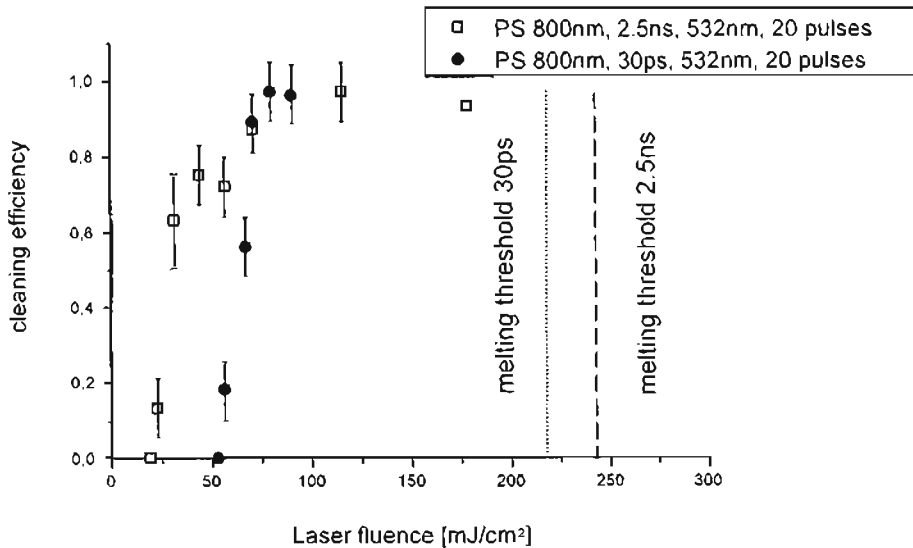


Figure 2. Efficiency of steam laser cleaning as a function of the laser fluence applied for two values of pulse duration: 2.5 ns and 30 ps (removal of PS particles, 800 nm in diameter) (see ref.9).

In the case of dry laser cleaning, the process induced visible and local damage effects on the treated surface mainly due to focusing effects related to the spherical shape of the contaminants. These effects were eliminated when the diameter of the particles was smaller than the wavelength of the irradiating laser applied and/or when the particles were absorbing at that range. Thus, UV irradiation appears to be suitable for dry laser cleaning applications.

2.1.2. Modelling of dry laser cleaning

In order to clarify the dependence of the cleaning efficiency on the energy applied and on the duration of the laser pulse, a theoretical model was developed¹¹. For this purpose industrial silicon samples were irradiated using a frequency doubled Q-switched Nd:YAG laser. The surface displacements were measured using a heterodyne interferometer and recorded with a digital storage oscilloscope (figure 3). For each experimental parameter, several hundreds of displacement curves were averaged and the derived numerically to obtain the surface acceleration.

Experiments showed that the highest accelerations, which are according to the theoretical model directly correlated to the cleaning efficiency, occurred on the time-scale of the laser pulse (figure 4).

It was shown that the maximum displacement depends only on the deposited energy, while the maximum acceleration of the surface was significantly influenced by the temporal shape of the laser pulse. Shorter pulses yield higher accelerations. These two observations will allow to optimise the cleaning process.

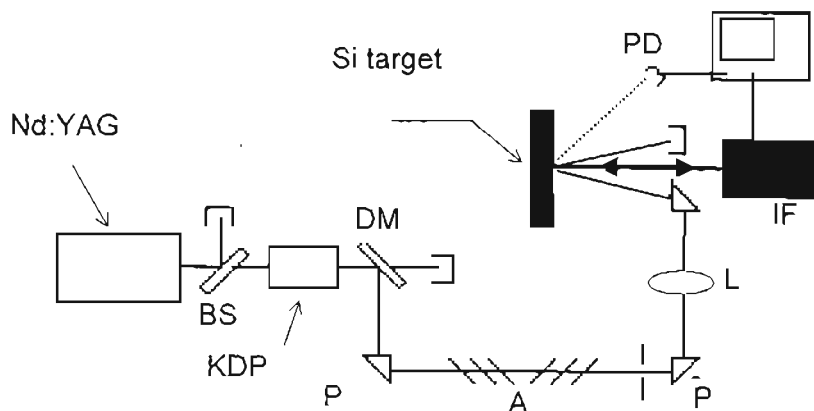


Figure 3. Experimental setup.
 BS: beam splitter, KDP: frequency doubler, DM: dielectric mirror, P: prism, L: lens,
 PD: photodiode, IF: interferometer, A: attenuator, Si: silicon sample, (see ref.11).

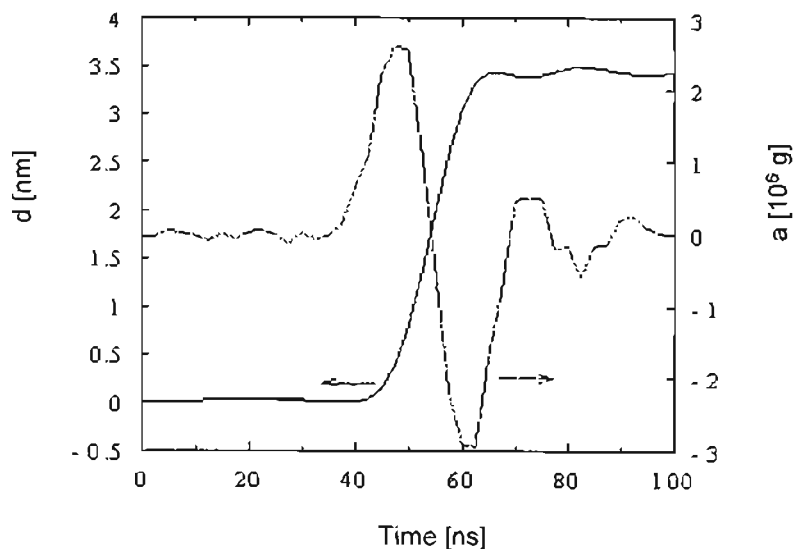


Figure 4. Typical displacement and acceleration dependence on the pulse laser duration (see ref.11).

2.2. Removal of oxide layers

Controlled removal of oxide layers is an important tool for surface preparation of metallic substrates to improve adhesion of coatings or welding. Another domain of application is the decontamination and the decommissioning of nuclear power installations, like the removal of contaminated oxide films formed in vessels or pipes. As metallic substrates are very sensitive to laser interaction in terms of chemical and mechanical changes, detailed studies are necessary to clarify the

different mechanisms inducing the oxide layer removal to choose the most suitable laser regime: i.e. thermoelastic or ablation regime.

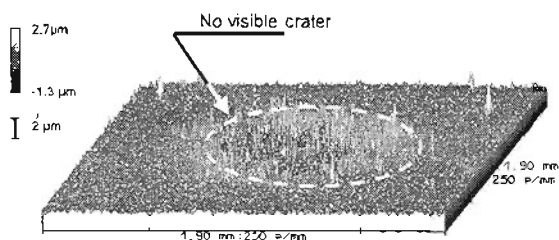
In the frame of this programme, two studies have been started: a basic approach of the behaviour of thermal oxides developed on pure iron and the characterisation of the removal of aluminium oxides grown onto industrial aluminium alloys by anodising.

2.2.1. Laser cleaning of iron

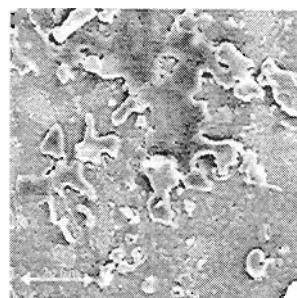
Previous research on the laser cleaning of oxidised iron indicated that the efficiency of the proposed technique is strongly depended on both the environment and of the substrate's polarisation (electrochemical control). In the present study, pure iron samples were oxidised at 500°C, for 48 hours, in a residual pressure of 10 mbar. X-ray analysis showed that the layer developed during oxidation consisted of a 1000 nm thick Fe_3O_4 layer, covered by a 20 nm thick Fe_2O_3 one. Laser cleaning was performed using a Nd:YAG laser ($I=500\text{mJ}/\text{cm}^2$, $\lambda=1064$ nm, FWHM=14.6 ns).

In order to determine the influence of the electrochemical control on the optical properties of the surface to be cleaned and, consequently, on the efficiency of the laser cleaning, three series of experiments were carried out: in air, in basic aqueous solution (pH=9.2) with and without previous polarisation of the substrate.

In the case of specimens irradiated in air or in basic aqueous solution without previous polarisation, their laser treatment resulted in the melting of the oxide layer, but not its removal (figure 5). In the case specimens irradiated in basic aqueous solution after their cathodic polarisation, laser treatment induced the removal of the oxide layer without melting (figure 6).

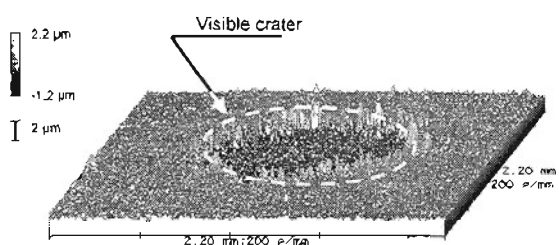


(a)

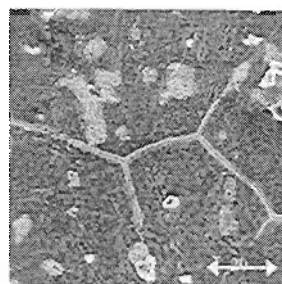


(b)

Figure 5. Feature of the irradiated area at $570 \text{ mJ}/\text{cm}^2$, in air
(a) Laser profilometry : no removal of the oxide layer.
(b) S.E.M. showing partial melting of the oxide layer.



(a)



(b)

Figure 6. Feature of the irradiated area at $570 \text{ mJ}/\text{cm}^2$, in basic aqueous solution with previous polarisation of the substrate
(a) Laser profilometry: removal of the oxide layer.
(b) S.E.M. showing no melting of the oxide layer and grain boundaries of the cleaned substrate, (see ref.13).

For the three series, the optical properties of the surface layer were determined by optical spectrometry at normal incidence, for wavelengths in the range of 450-1100 nm. In order to quantify the changes of the refractive index due to the

electrochemical control imposed, a theoretical model permitting the calculation of the real and imaginary part of refractive index of the oxide layer was developed.

The electrochemical control influenced slightly the real part of the refractive index (<10%), however it resulted in an important reduction of the imaginary part (by a factor 20), leading to the decrease of the optical absorption, in the range of wavelengths examined (figure 7). It can be concluded that the modifications of the optical properties of the surface to be cleaned, when imposing electrochemical control, have a dominant role on the values of the energy absorbed by the treated surface and, consecutively, on the mechanisms of the oxide removal.

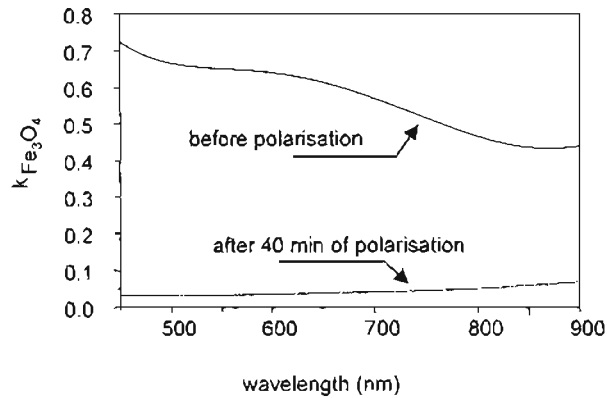


Figure 7. Values of the imaginary part of the refractive index (k) of the Fe_3O_4 layer before and after polarisation (see ref.13)

2.2.2. Cleaning of anodised aluminium alloys

The influence of the wavelength, the fluence and the number of pulses on the interaction phenomena between laser irradiation and Al_2O_3 coated aluminium alloys were investigated. A $20\ \mu\text{m}$ thick Al_2O_3 coating was deposited on an aluminium alloy substrate by the standard anodising technique. By changing the anodising parameters, white and black oxide layers were obtained.

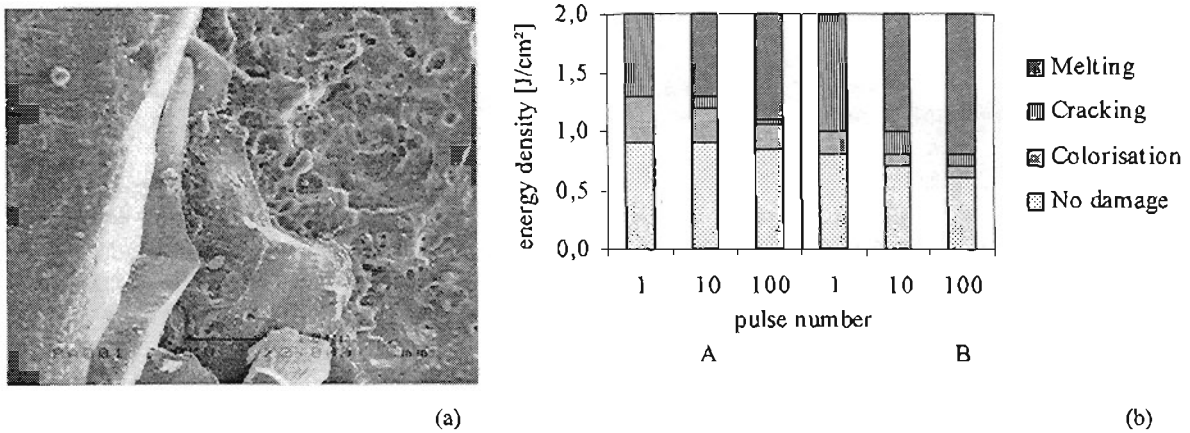


Figure 8.
 (a) SEM micrograph of the borders of the irradiated area on white Al_2O_3 coating (fluence: $1.14\ \text{J}/\text{cm}^2$, 1 pulse).
 (b) Damage diagram for white Al_2O_3 coating, as a function of the fluence and the number of pulses, (A: $\lambda=1064\ \text{nm}$, B: $\lambda=532\ \text{nm}$). See ref (14).

Both white and black coatings presented a similar behaviour, when treated by laser irradiation with wavelength of 532 nm and 1064 nm. In each case, the fluence threshold induced the breaking and removal of the oxide layer (figure 8a) in a single pulse was determined. This critical fluence found depending on both the colour of the oxide and the wavelength used or, in other words, on the absorbance of the surface. Increasing the number of pulses, partial or total melting of the irradiated Al_2O_3 coating could be observed (figure 8b).

Based on the obtained results, it can be concluded that the fluence threshold for the black oxide layer is lower than that for the white one. The better cleaning effect was reached when irradiating at fluences of about 20% higher than the threshold value, with a number of pulses from 4 to 10 and pulse repetition rate less than 0.1 Hz. For the system $\text{Al}_2\text{O}_3/\text{Al}$, the heat transfer to the substrate, which can induce its melting, limits the range of fluences that can be used for cleaning applications.

2.3. Laser cleaning of molecular substrates

Laser ablation cleaning of organic molecular substrates is accompanied by photochemical phenomena, which can compromise the integrity of the substrate. Understanding and therefore controlling of such phenomena is important for the optimization of the UV ablation process in view of its important applications in materials processing, medicine, analytical chemistry and painted artwork cleaning.

Polymer films containing photosensitive dopants constitute good model systems for the study of the photochemical effects in the UV laser ablation of molecular substrates. In this respect polymer matrices (e.g PMMA, polystyrene) doped with a variety of photosensitive molecules (e.g naphthalene, anthracene, pyrene, idonaphthalene, bromonaphthalene) have been examined under UV (248nm, 30ns and 500fs) laser irradiation in an effort to elucidate the nature and extent of photochemical modifications. Fluorescence techniques are being used to monitor the photoproducts formed upon irradiation of the substrate. For example, study of the system idonaphthalene-PMMA under nanosecond irradiation showed increased formation of photoproducts (naphthalene derivatives) above the ablation threshold indicating enhanced mobility of free radical species formed upon photodissociation of the dopant (figure 9).

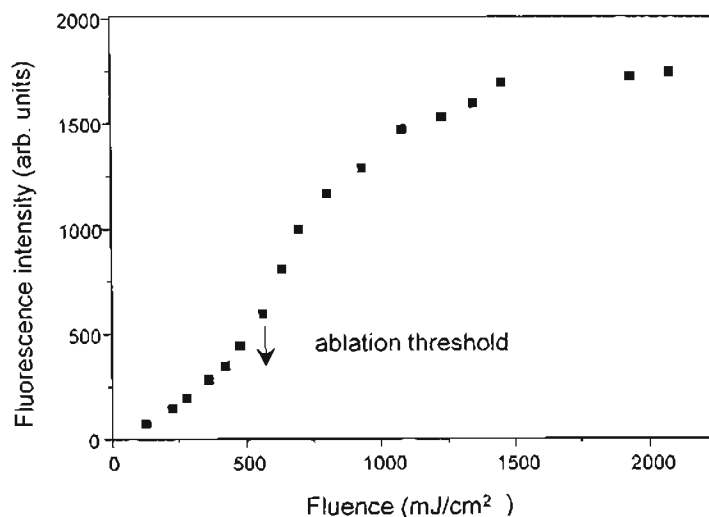


Figure 9. Intensity of the naphthyl-photoproduct fluorescence emission ($\lambda_{\text{exc}} = 248\text{nm}$, $\lambda_{\text{em}} = 355\text{nm}$) following irradiation of a PMMA film doped with idonaphthalene (0.4% w/w) with a single "pump" pulse (248 nm, 30 ns) as a function of the "pump" fluence (see refs. 15-16).

This is an important result, attributed to structural changes induced to the polymer substrate under ablation conditions, and suggests that cleaning of sensitive substrates with nanosecond laser radiation should be done in a way which guarantees that a thin overlayer is left on the substrate, in order to protect it from unwanted photochemical changes. This is indeed among the important criteria followed in demanding laser cleaning applications such as the removal of photodegraded varnish from the surface of paintings.

In sharp contrast to the nanosecond ablation, in the ablation with 500 femtosecond pulses, photoproduct formation is well-defined and limited, indicating the presence of different constraints on the photochemistry in the UV ablation with different laser pulse duration. This finding suggests that the use of short pulse irradiation may be quite important in cleaning of molecular substrates.

2.4. Laser imaging for laser cleaning diagnostic

As it was already mentioned (§2.1), steam laser cleaning is a very efficient technique for the removal of small size particles. The contaminated surface is wetted with a thin film of alcohol and water. When irradiating the wetted surface at a wavelength, at which the liquid is transparent, the rapid superheating of the liquid film at the solid-liquid-boundary induces vapor explosive bubble nucleation and leads to the ejection of the particles from the surface. As this removal mechanism is taking place in a ns time-scale, the development of a ns time-scale observation tool is necessary. For that purpose a laser diagnostic set-up was developed¹⁷. It permits imaging the variations of the refractive index of the liquid induced by heating (thermo-optic), cavitation (phase transition) and explosion shock wave (photo-elastic).

The proposed diagnostic tool is based on the combined application of two techniques involving surface plasmon (electromagnetic surface waves at a metal-dielectric-boundary). A Kretschmann configuration (figure 10a) was used to excite surface plasmons. Due to their dispersion relation, these waves can only be excited at a specific resonance angle for a given wavelength. This resonance angle is very sensitive even to small changes of the refractive index of the dielectric. Thus, for a given angle inside the resonance, a change of the refractive index ($\Delta n/n = 10^{-3}$) leads to a change of the reflectivity (figure 10b).

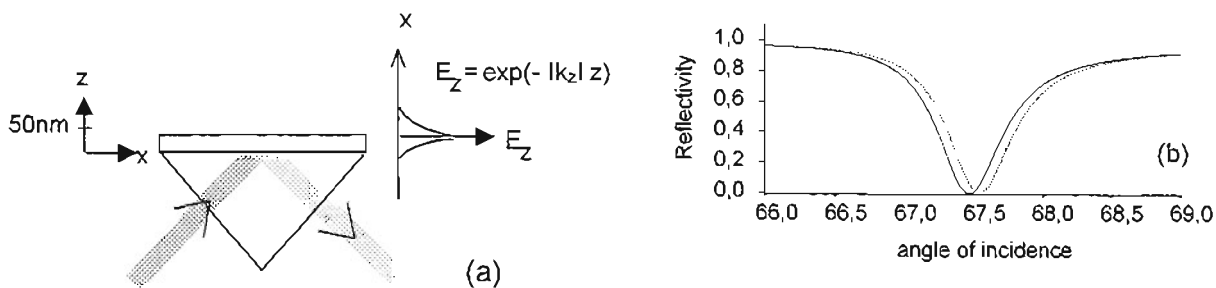


Figure 10. Laser imaging for laser cleaning diagnostic

- (a) Kretschmann configuration
- (b) Change of reflectivity as a function of the resonance angle ($\Delta n/n = 10^{-3}$) (see ref.17)

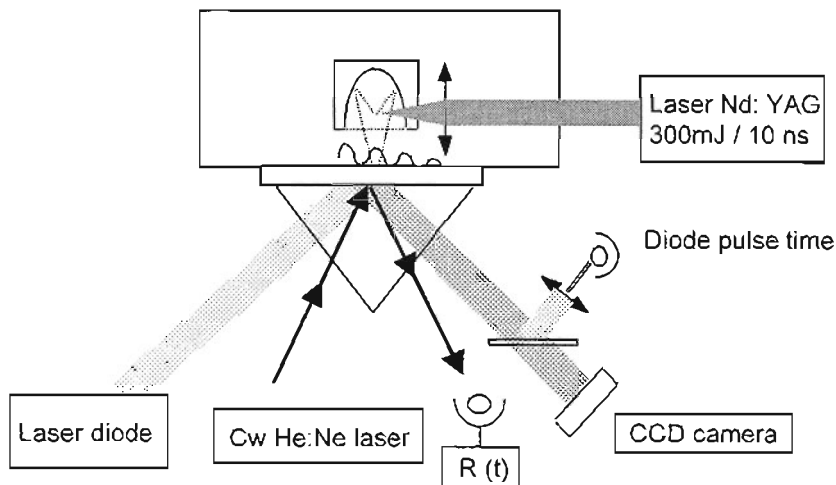


Figure 11. Experimental set-up (see ref.17).

The developed experimental set-up (figure 11) permitted to determine the amplitude of the shock waves on the surface to be cleaned, based on the correlation between the refractive index of the water and its pressure. During the laser pulse leading to explosive bubbles nucleation, the evolution of the reflectivity of the surface was determined by a continuous HeNe laser (10mW, $\lambda=633\text{nm}$). The determination of the lateral pressure distribution at a specific time at the surface was realised using plasmon microscopy. For that purpose, a pulsed laser diode (FWHM 15ns, $\lambda=910\text{nm}$) was used for the irradiation of $1\times 1\text{ cm}^2$ area and the reflected intensity was recorded with the aid of CCD camera. In order to get an intensive shock wave, a frequency-doubled Nd:YAG-laser (FWHM 7ns, $\lambda=532\text{nm}$) was focussed into a water filled cell at the first focal point of an ellipsoidal mirror. The irradiation of the water led to the generation of plasma, which introduced shock waves which are focused to the specimen located at the other focal point of the mirror to increase the pressure amplitude.

During experiments, the shock-waves were developed within 10 ns, reaching pressure values in the order of 1000 bar. The proposed diagnostic permitted the shock-waves imaging with ns time resolution and $50\mu\text{m}$ spatial resolution. This technique is very rapid and sensitive even to slight variations of the refractive index. It has also the advantage to allow temperature field mapping of the interface between a liquid and solid metal, when heated by an intense pulse laser irradiation.

CONCLUSION

This paper reports the most recent results obtained by common research between different European groups on the laser cleaning of various materials. Important experimental results have been obtained on the removal of fine particles from contaminated Si surfaces. A theoretical model based on the surface acceleration was developed, in order to better understand the mechanism of particles removal during dry laser cleaning. In the case of oxidised iron, the influence of both the environment and of the substrate's polarisation on the optical properties of the surface to be cleaned was investigated. Some interesting results were also carried out in the case of laser cleaning of anodised aluminium. Study of the system iodonaphthalene-PMMA under nanosecond irradiation showed increased formation of photo-products (naphthalene derivatives) above the ablation threshold indicating enhanced mobility of free radical species formed upon photo-dissociation of the dopant. Finally, a new laser diagnostic technique, which permits shock-wave imaging with high time and spatial resolution, was developed.

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