

Task Title: DETRITIATION OF SOFT HOUSEKEEPING MATERIALS (mainly plastics)

INTRODUCTION

The aim of this task is to study different ways for chemical detritiation of housekeeping materials (at laboratory scale), the processes that seem to be most efficiency were selected to determine whether further experiments are needed to validate this selection.

These dedicated procedures are proposed for the different type of waste as follow:

- parts of gloves (butyl rubber),
- parts of light coveralls (polyester),
- plastic sheet (PVC or polyurethane),
- parts of seals,
- filaments of paper.

The results of the different detritiations must not create other wastes which are difficult and expensive to treat.

2004 ACTIVITIES

A literature review was performed to find different ideas but there is no process actually used. CEA VALDUC, which have the same type of wastes manage them in function of their activities [1].

- introduction in air lock,
- drums emptying,
- counting,
- sorting and control,
- shredding,
- drying (at 90°C, to limit the tritium degas),
- drums filling,
- compacting press,
- closing and control.

Type TFA wastes are evacuated in ANDRA storage.

Type A is stored in hangars.

Type B is stored in ventilated hangars.

Type C with a more important degassing rate is stored in special drums and in more ventilated hangars.

To estimate the degassing rate, all these drums are measured by calorimetry.

Different ways are studied for the housekeeping detritiation without generating other wastes:

- leaching with hot water,
- leaching with acidic solution,
- leaching with basic solution,
- full mineralization in a digester.

With this type of waste, the difficulty of the study is the homogeneity of samples to compare different processes.

An approach with a cryo-grinding treatment with liquid nitrogen shows that it's impossible to transform them as powder. All the samples remain too soft.

So to have comparative decontamination ratio, it is necessary to measure the residual tritium content in the housekeeping material after the detritiation process by full dissolution of samples.

STUDY OF FULL MINERALIZATION SAMPLE TO MEASURE TRITIUM ACTIVITY

In a reactor connected to different traps for tritium measurement:

- a volume of sulfuric acid during one day at 100°C is used for the dissolution of the housekeeping,
- and another volume of sulfuric acid during 3 days and some drops of hydrogen peroxide.

Acidic and basic solutions are distilled and all the traps solutions are measured with liquid scintillation apparatus. The wiring diagram (figure 1) shows the montage.

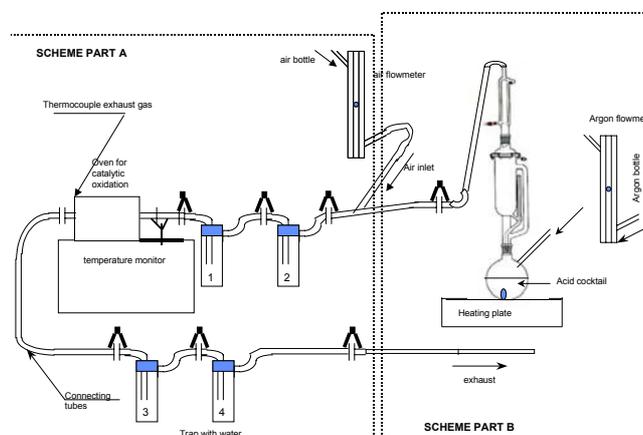


Figure 1 : Wiring diagram

Results : sample 6,4 g of different wastes are treated.

Activity in reactor : ^3H liq. = 260517 Bq.
 Activity in different traps : ^3H gas = 9924 Bq.

Total activity : ^3H = 42256 Bq/g

The results show that more than 90% of the tritium is not under gas form. The tritium comes from the contamination of the materials after the different experiments. In this case, the tritium contamination can be eliminated with different lixiviation processes.

DETRITIATION BY LEACHING WITH HOT WATER (100°C)

Same used montage (figure 1).

After 8 hours : tritium activity = 1289 Bq/g (only in reactor).

After 16 hours : tritium activity still the same as after 8 hours and equal to 1289 Bq/g (only in reactor).

Total activity : ^3H = 1328 Bq/g (reactor+ traps)

More than 90 % of tritium activity is in the reactor and the rest in traps.

Only 100°C
 Tritium is not under gas form

Calculation of the decontamination ratio :

Mineralization of the rest of the lixiviated housekeeping

Total tritium activity = 447 Bq/g
 Decontamination ratio = 75 %

Total tritium activity between first and second experiment is very different. 422256 Bq/g for the first and 1328 Bq/g for the second, which have been done 3 month later. This result shows the heterogeneity of the different wastes and the difficulty of sampling identical samples. The wastes are stored in plastic box, so we have to consider that a lot of tritium is lost by diffusion through the plastic box.

DETRITIATION BY LEACHING WITH HOT NITRIC ACID (2M)

After 8 hours : tritium activity = 1202 Bq/g (only in reactor)

After 16 hours : tritium activity still the same as after 8 hours.

Total activity : ^3H = 1303 Bq/g (reactor+ traps)

More than 90% of tritium activity is in the reactor and the rest in traps

Calculation of the decontamination ratio :

Mineralization of the rest of the lixiviated housekeeping

Total tritium activity = 500 Bq/g
 Decontamination ratio = 72 %

The decontamination ratio with acid solution is quite the same as water.

DETRITIATION BY LEACHING WITH HOT SODA (1M)

After 8 hours : tritium activity = 656 Bq/g (only in reactor)
 After 16 hours : tritium activity still the same as after 8 hours.

Total activity : ^3H = 710 Bq/g (reactor+ traps)

More than 90% of tritium activity is in the reactor and the rest in traps

Calculation of the decontamination ratio :

Mineralization of the rest of the leached housekeeping

Total tritium activity = 308 Bq/g
 Decontamination ratio = 70 %

The decontamination ratio with basic solution is quite the same as water.

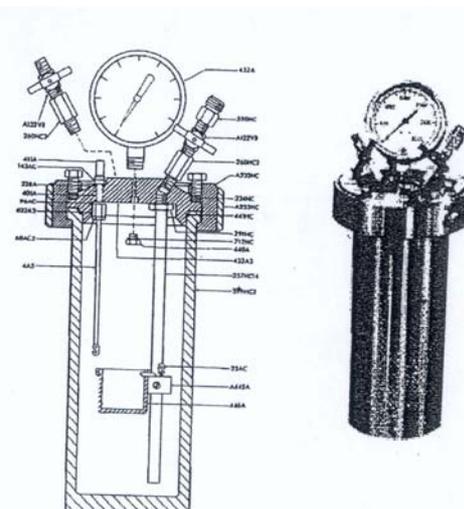
These three leaching types have nearly the same decontamination ratio. They all generate tritiated aqueous solutions. The easiest is the process with water where tritiated water is generated, which have to be stored in polyethylene bottle to avoid corrosion.

For the high activity levels, the water is adsorbed on zeolithe and store in drums.

FULL MINERALIZATION IN A DIGESTER

Under oxygen (25 bars), all the organic structure is burned and destroyed. The tritium is recovered under liquid and gas forms.

The next photo shows the mineralization system.



In this case, all the housekeeping materials are burned and 100 % of the tritium is trapped in the bubbles and rinsed water of the mineralization system as tritiated water.

The total tritium activity measured is 1973 Bq/g.
The decontamination ratio is 100 %.

More than 95 % of the tritium is in the rinsed water phase.
Only tritiated water is created.

CONCLUSIONS

Even the chemical tests would have been done, the extraction rate is around 70 %.

The easiest is the process with water where tritiated water is generated which have to be stored in polyethylene bottle for having no corrosion.

For the high activity levels, the water is adsorbed on zeolithe and store in drums.

In a digester, 100 % of the tritium is transformed with oxygen as HTO form and trapped in bubbles. Only tritiated water is created.

In both cases tritiated water is easy to manage. It must be stored in polyethylene bottle to avoid corrosion.

For the high activity levels, the water is adsorbed on zeolithes and stored in drums.

CEA VALDUC, which has the same problems doesn't detritiate their housekeeping materials. They manage the storage of the wastes pertaining to their activities. They are stored in different hangars with adapted ventilation levels. The degassing rate is controlled by calorimetry measurement on the drums.

REFERENCE

- [1] Compte rendu de réunion : UKAEA/CEA co-operation on fusion decommissioning - S. Rosanvallon DER/STR/LCEP 2001/309.

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Task Title: DETRITIATION OF VACUUM OIL AND ORGANIC LIQUIDS

INTRODUCTION

The aim of this task is to study different ways for chemical detritiation of oil and scintillation liquids (at laboratory scale), the processes that seem to be most efficiency were selected to determine whether further experiments are needed to validate this selection.

These dedicated procedures are proposed for the different type of organic liquids:

- Vacuum pump oil : P3 (PFEIFFER VACUUM).
- Scintillation liquid : Optiphase Hisafe ®.
- Oxysolve T®.

The results of the different detritiation procedures must not create other wastes that are difficult and expensive to treat.

2004 ACTIVITIES

A literature review was performed to find some processes. The only process used is the incineration off-site facility. CEA VALDUC, manage the low activity level oils to an incinerator [1]. The high activity level oils are input in polyethylene drums (to have no corrosion) that are put in stainless steel container which are stored in glove box.

To estimate the degassing rate, all this drums are measured by calorimetry with ³He technique (tritium activity in waste drums correlated to the ³He escape of the drum).

For the low tritium activity levels of scintillation liquid, CEA Valduc collect and send them to ANDRA, which manages them to an incinerator.

At the beginning a method of full mineralization of oil was studied to measure tritium activity. Then different detritiation ways without generating other wastes are studied:

- Chemical extraction:
 - . hot water,
 - . acidic,
 - . basic,
 - . dehydration.
- Mechanical extraction.

A tritium species was performed to understand the phenomena then a thermal process was studied with a digester.

With the scintillation liquid, the tritium activities are measured ; the different studied processes are:

- chemical extraction:
 - . hot water,
 - . acidic,
 - . basic,
 - . freeze drying.
- thermal procedure:
 - . digester.

STUDY OF FULL MINERALIZATION OF OIL OR LIQUID SCINTILLATION SAMPLE TO MEASURE TRITIUM ACTIVITY

In a reactor connected to different traps for tritium measurement:

- a volume of concentrated nitric acid,
- heated under reflux during 6 hours,
- let cool down,
- small quantities of perchloric acid added,
- heat (without reflux) until white smokes and until having clear solution.

Acidic and basic solutions are distilled and all the traps and solutions are measured with liquid scintillation apparatus.

The wiring diagram (figure 1) shows the procedure scheme.

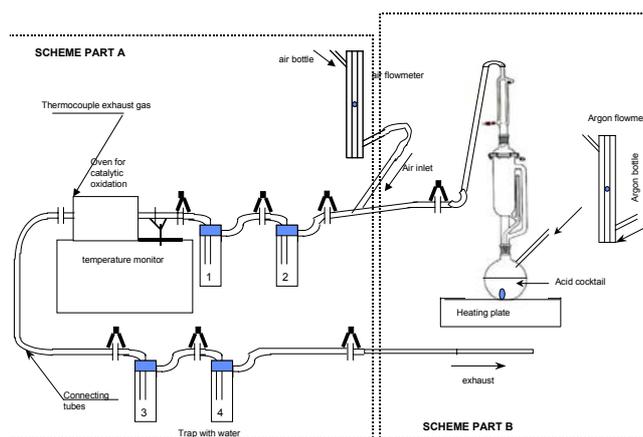


Figure 1 : Wiring diagram

OIL DETRITIATION

Results of the total tritium activity measurement of P3 oil

Estimated or measured activity for the sent oil :
³H activity : 301 kBq/L (unknown date)

Measured activity in our lab :

^3H activity : 8.8 kBq/L (05/05/04)

New measurement 5 month later :

^3H activity : 2.2 kBq/L (10/09/04)

New measurement 1 month later :

^3H activity : 1.8 kBq/L (26/10/04)

The conditioning conditions (plastic bottle) lead to a tritium diffusion through the plastic bottle and a lot of tritium is lost. Oil is self-detririated.

The storage conditions must be the same as it was described for the CEA VALDUC.

Chemical extractions

Experiment with hot water : 30 mL of oil and 30 mL of water under stirring and heated at 140°C 3 times during 5 hours.

Measurements of tritium activity in the oil before and after the experiment give an extraction rate of about 50 %.

Experiment with water heated under reflux :

Extraction rate : ~ 40 %

Experiment with acidic solution (pH=4)

Extraction rate : ~ 30 %

Different experiments with different volumes with different temperatures, pH (basic to acid) lead to the same extraction rate

Less then 50 % extracted

Dehydration experiment

Some dehydration tests are done to trap water.

Soda pellets mixed with oil during 18 hours bring no influence.

With CaCl_2 pellets, there is no influence and CaCl_2 pellets under ultrasound during 12 hours lead to the same results.

Mechanical experiment

To confirm that the quantity of water is very low in this oil, some centrifugation experiments are realized.

30 mL of oil are centrifuged at a speed of 3000 rpm during 30 mn. There is no physic modification. Creaming the surface of the oil, the liquid scintillation measurement shows no difference.

30 mL of oil are centrifuged with 30 mL H_2O at a same speed during 6 hours. The liquid scintillation measurement gives a detririation rate of about 33 %.

Industrial process of hot ultra centrifugation exists for used oil of cars that allow recycling 70 % of oil.

This process is used to separate metallic particles. We don't own such an instrument in our lab to test whether it's possible to adapt it to tritium. The expected results seem to be the same as classical centrifugation because there is no water phase to separate as it was observed with the dehydration test.

Tritium species

Oil is heated in a reactor under argon flow. The outlet of the reactor is connected to air and to the bubbles of MARC 7000 system to trap tritium.

With this type of design, it's possible to define the different species of tritium. In reactor, remains the non volatile tritium (bound tritium), in bubbles 1 and 2 the tritium as HTO and in the bubbles 3 and 4 (after conversion to HTO in the catalytic oven at 450°C), the HT form.

The next photo shows the montage:



Photo 1 : Tritium species montage

The results are after liquid scintillation measurement:

- 25 % of tritium as HTO form,
- 25 % of tritium as HT form,
- 50 % of liquid tritium, which is bound tritium.

Full mineralization in a digester:

Under oxygen (25 bars), all the oil is destroyed. The tritium is recovered as liquid (a little volume of water is needed in the bottom of the digester) and gas form. The gas is transformed as HTO form with oxygen and thus trapped in bubbles.

The calculated detririation rate is 100 % after the different liquid scintillation measurements.

Conclusion of the oil detririation:

Even the chemical tests would have been done, the extraction rate is not higher than 50 %. This extracted tritium is labile.

The tritium, which remains in oil, is considered as to be bound tritium.

In a digester, 100 % of the tritium is transformed with oxygen as HTO form and trapped in bubbles. Only tritiated water is created.

CEA VALDUC, which has the same problem, doesn't detritiate oil. They manage the low activity level oils to an incinerator. The high activity level oils are input in polyethylene drums (to have no corrosion) which are put in stainless steel container which are store in glove box.

SCINTILLATION LIQUID DETRITIATION

Results of the total tritium activity measurement of HISAFE and OXSOLVE T

Optiphase Hisafe : 4.59 kBq/mL (May 15-2004)

Oxysolve : 2.4 kBq/mL (May 25-2004)

Chemical extractions

Experiment with hot water : 20 mL of Hisafe and 30 mL of water under 15 mn stirring then addition of 30mL ultra-pure water for distillation.

Resulting tritium activity of the distillation: 0.05 kBq/mL and a tritium rate of about 1 %.

Same procedure with hot water (close to boiling) leads to an extraction rate of 5 %

Tritium remains in the boiler and a try to continue the distillation is dangerous. A pressure increase has been observed.

Experiment in a vessel with 5 mL of oxysolve plus 5 mL of fresh oxysolve plus 10 mL o water. Heating under air flow with a MARC 7000 downstream. Tritium is mainly recovered as HTO form, but only 30 % of the tritium is extracted.

Experiment with acidic solution or basic solution.

10 mL of Optiphase Hisafe with HCl 6N then a distillation leads to 73 % of tritium extracted. The same operation with soda leads to an extraction rate of 80 %.

Same operation at pH 2 with 10 mL of oxysolve T with 40 mL of acid solution and 48 h of stirring then addition of 100 mL of water and distillation lead to a 96 % rate. However the waste volume increased.

One solution for decreasing the waste volume could be the use of freeze-drying procedure.

Mechanical experiment

The same procedure with water, has been done by heating until 80°C under ultrasound stirring and the obtained extraction rate is only 15.8 %.

Full mineralization in a digester:

Full mineralization in a digester leads to the total destruction of the organic structure and 100 % of the tritium is recovered as HTO form.

In this case, only tritiated water is created.

CONCLUSIONS

Oil detritiation

Even the chemical tests would have been done, the extraction rate is not higher than 50 %. This extracted tritium is labile.

The tritium, which remains in oil seems to be bound tritium. In a digester, 100 % of the tritium is transformed with oxygen as HTO form and trapped in bubbles.

The advantage is that only tritiated water is created. This waste is clearly identified and easy to store.

CEA VALDUC, which has the same problem, doesn't detritiate oil. They manage the low activity level oils to an incinerator.

The high activity level oils are put in polyethylene drums (to have no corrosion), which are put in stainless steel container, stored in glove box.

Scintillation liquid

A long time stirring with acid or base addition improves extraction.

Distillation with concentrated scintillation liquid seems explosive.

In a digester, 100 % of the tritium is transformed (with oxygen) as HTO form and trapped in bubbles.

The same advantage as oil is the generation of tritiated water only.

At CEA Valdudc the low level activity samples are collected and sent to ANDRA that manages them to an incinerator.

REFERENCE

- [1] Compte rendu de réunion : UKAEA/CEA co-operation on fusion decommissioning - S. Rosanvallon DER/STR/LCEP 2001/309.

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Task Title: LASER DECONTAMINATION/TRITIUM REMOVAL Studies on Tokamak wall surfaces decontamination by pulsed repetition rate lasers

INTRODUCTION

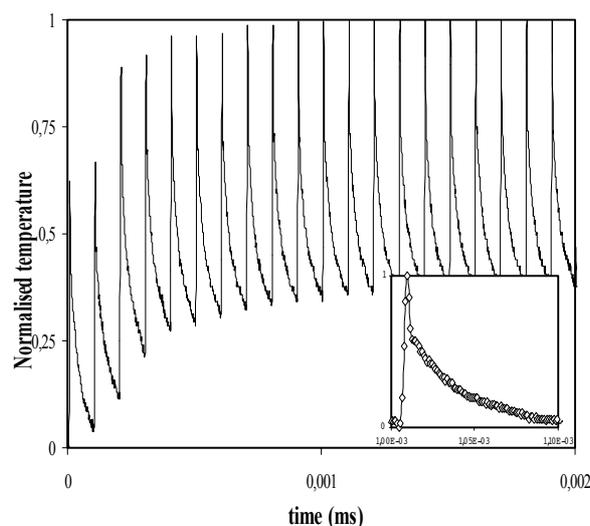
The excessive content of tritium in plasma-facing components is seen as a severe problem for the efficient functioning of a fusion reactor [1-3]. Laser decontamination of the plasma facing component surfaces is of a special interest as it can offer a completely optical method of a surface cleaning by its heating or laser ablation. The possibility to transport the laser beam to the cleaning zone by the optical fiber allows both to remove the laser system away from the contaminated zone and to offer detritiation without direct personnel participation. The absence of the direct contact with the contaminated surfaces, the reduced waste volume, and a possible complete automation of the process that can ensure the personnel safety are regarded as the most attractive features of laser decontamination. The developed and commercially available powerful high repetition rate pulsed Nd-YAG lasers where the radiation transport is carried out with the optical fibers are seen as good candidates for decontamination of the vacuum chamber surfaces in Tokamak thermonuclear installations.

In 2002-2003, our laboratory developed the necessary experimental equipment, and the first investigations on the graphite surface de-hydrogenisation with the pulsed repetition rate Nd-YAG laser systems were made [3]. The development of the experimental equipment (vacuum chamber, sealed cells, pyrometer, plasma imaging with the ICCD camera) and methods for co-deposited layer characterization (GD-OES, optical and electron microscopy, ejected gas chromatography and mass spectrometry) was the first step of a 2004-year programme. The next step of our studies was to validate the laser treatment parameters (defined for graphite samples in 2003) and to make ablation tests on decontamination of diagnostics and tools. Ablation thresholds for different metals (diagnostics and tools) and graphite samples were determined experimentally and compared with the theoretical data. The detailed presentation of the developed experimental equipment and the results obtained are presented in our final 2004-year report.

2004 ACTIVITIES

The design, development, and tests on the new experimental equipment and tools (vacuum chambers and cells, pyrometer, high repetition rate laser on 1.064 μm with ms pulse duration, high speed plasma imaging) were successfully realized. A small sealed cell was applied to analyse ablated matter (gases, micro particles).

The preliminary temperature measurements of the graphite surface (figure 1) heating by a pulsed repetition rate Nd-YAG laser beam were made with a pyrometer with 15 μs time resolution. It was possible to measure the temperature in 280 – 2300°C range with 0.1°C accuracy.



*Figure 1 : Graphite temperature measurement with the pyrometer - Time resolution-15 μs ; space resolution- 0.5 mm - Heating with Nd-YAG laser beam, 10 kHz, 532 nm, 100 ns, 0.6 J/cm², air 1 bar
Insertion- temperature for one laser pulse heating*

Millisecond pulses on 1064 nm wavelength were obtained after modification of the acoustic-optical switcher regime of Nd-YAG laser. With a longer pulse duration, it was possible to apply the same beam transportation system (optical fiber) even for 200W mean laser power (two-fold increase from 100 W at 532nm to 200W at 1064 nm). For detritiation studies, a simple replacement of some optical elements (mirrors, lenses) was sufficient to provide the operation regime of the high repetition rate laser bench.

With pulsed high repetition rate lasers, the surface shielding by ablated matter was particularly pronounced and, consequently, resulted in decrease of laser ablation efficiency. This was observed in our experiments with 10 KHz repetition rate. To study the ablated matter (gases, micro particles, plasma) properties, the imaging system on the basis of the ICCD gated camera was developed. The system may allow the interaction zone imaging with adjustable time resolution up to several nanoseconds. To synchronise the imaging system to a specified pulse, a special electronic system was developed and applied. It allowed also to synchronise the ICCD camera with any specified laser pulse.

Two methods (heating and laser ablation) were applied to characterize the gases released during de-hydrogenisation of graphite samples with a co-deposited layer. TORE SUPRA co-deposited layer characterization was made by the gas release measurements with a hydrogen analyser RH-404 (LECO Corporation) that is used for measuring hydrogen in inorganic matters. Gas release was obtained with the sample heating in a furnace. A low repetition rate laser bench was used to provide ablation of the TORE-SUPRA graphite samples with a thin co-deposited layer. The samples were put inside a developed sealed stainless cell. With 1 J/cm² laser fluence (that corresponds to the ablation threshold of TORE SUPRA graphite with 4 ns pulses), it was possible to obtain only a co-deposited layer ablation.

The cell gas analysis following the co-deposited layer ablation was made with a mass spectrometer. It was possible to make the mass measurements in the range of 1-150 a.u.m. with the ppm accuracy. Approximately the same concentrations of H and D isotopes were obtained. The hydrogen concentration in the ablated layer was comparable with the results obtained with the sample heating in furnace followed by chromatography analysis. The obtained hydrogen contents in TORE SUPRA co-deposited layer is in good agreement with the previous measurements [3] by Glow Discharge–Optical Emission Spectroscopy (GD-OES) method. Thus, the results obtained with three different methods demonstrated a sufficiently good agreement in hydrogen contents in the TORE SUPRA co-deposited layer.

The new results on graphite ablation obtained with a high repetition rate laser bench seem to be very promising. The back side surface of TORE SUPRA graphite samples was ablated with 90 ns pulses of a high repetition rate laser bench. The ablation threshold was determined to be (2.5 ± 0.5) J/cm², that is, 2.5 times higher than the previously determined ablation thresholds of 1 J/cm² for 4 ns laser pulses.

This difference in ablation thresholds might be explained by the longer pulse duration of a high repetition rate laser. The graphite surface heating depth can be described as $L \approx (D \times \tau)^{0.5}$, where D – thermo diffusivity of graphite, τ - laser pulse duration [4-6]. Thus, the longer the laser pulse duration is, the thicker the absorbed energy depth. Our experiments with 4 ns [3] and 90 ns pulse durations confirmed this dependence. For TexTor co-deposited layer, the ablation threshold was determined to be (0.4 ± 0.1) J/cm². In contrast to the backside graphite results, the ablation thresholds for a co-deposited layer were the same for both 5 ns (low repetition rate laser bench) and for 100 ns (high repetition laser rate bench) duration pulses.

Laser ablation thresholds for some metal samples were of particular interest in our studies. Table 1 summarizes the experimental results on the ablation thresholds for different metals (Cu, Al, Fe, Ni, Pb, W) and graphite obtained with 5nsec pulses (532 nm). The ablation thresholds for 100 ns laser pulses were determined by the relation:

$$F_{th}(\tau_1) \approx F_{th}(\tau_2) \times \sqrt{\tau_1 / \tau_2} \quad \text{or} \quad F_{th}(100ns) \cong 4,5 \times F_{th}(5ns)$$

Table 1 : Ablation thresholds for different metal targets with 5 ns and 100 ns laser pulses

Target	Experimental data for 5 ns (J/cm ²)	Estimations for 100 ns (J/cm ²)
Al	1.71	7.7
Cu	2.09	9.1
Fe	1.00	4.5
Mo	2.56	11.4
Ni	0.941	4.2
Zn	0.980	4.4

The ablation thresholds for the metals in our study were in 1-2.5 J/cm² range for 5 ns laser pulses. They were determined to be higher than the co-deposited layer ablation threshold of 0.4 J/cm². For 100 ns laser pulses, the ablation thresholds were determined to be five times higher than for 5 ns pulses.

Different ablation thresholds for a substrate and a co-deposited layer could be applied to ensure self-control of laser surface cleaning. This might be obtained if the laser fluence in the interaction zone is chosen to be lower than the ablation threshold of the substrate. Thus, with 100 ns pulses, laser detritiation of diagnostics and tools can be performed with the laser fluence without the substrates damage.

The ablation threshold was not found to depend on the environmental gas contents. Both in air and in the noble gas (argon in our experiments), the ablation thresholds and ablation rates were determined to be of the same value. A slight difference in laser/surface interaction was observed with the laser fluence lower than 0.5 J/cm². No ablation was observed with $F < 0.5$ J/cm² in Ar. In contrast, the co-deposited layer erosion was observed in air.

This erosion might be associated with the surface oxidation with CO₂ gas release. Environmental condition effect manifested itself as a black circular zone around the crater. The circular zone was observed in Ar, but was not detected with ablation in air.

It is considered to be resulting from the graphite powder re-deposition. Ar was considered to keep hydrogen atoms from oxidation. In air, oxygen gave rise to CO₂ formation and, thus, suppressed the graphite powder re-deposition around the crater.

With the developed plasma imaging system, it was possible to observe the important plasma intensity increase in argon figure 2.

This phenomenon is regarded essential for Laser Ablation – Optical Emission Spectroscopy (LA-OES) method for diagnostics and control of co-deposited layer removal and surface characterization.

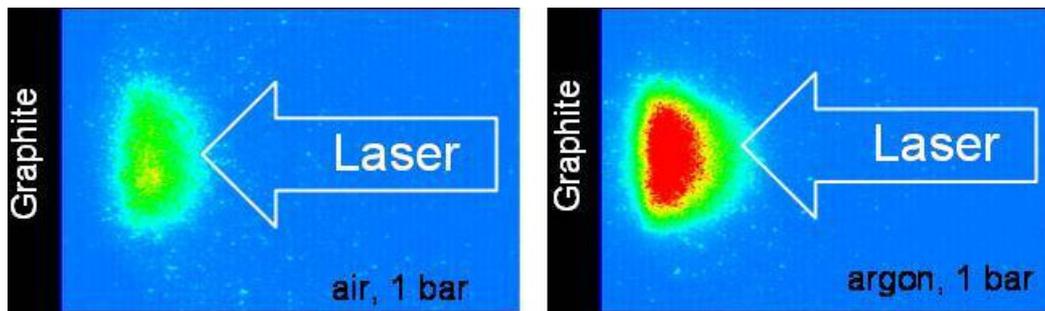


Figure 2 : Plasma images in air and in argon (1 bar at normal temperature). ICCD camera: delay-1 μ s, gate – 20 μ s

The obtained optimal conditions for a co-deposited layer ablation (laser fluence of $F=1-2 \text{ J/cm}^2$, high repetition rate) were applied for TexTor co-deposited layer cleaning. The $10 \times 10 \text{ mm}^2$ zones for cleaning were chosen on the TexTor tile surface where the ablation efficiency had already been determined. Ablation was obtained with a high-repetition rate laser with 20W mean power and 20 kHz repetition rate. The laser beam was focused into a spot of 250 μm diameter (FWe^{-2}) with 2 J/cm^2 maximum fluence and was scanned on the chosen zone of the surface. Figure 3 presents the TexTor tile after laser cleaning. On the right, the decontaminated zone ($10 \text{ mm} \times 10 \text{ mm}$) was obtained by a single scanning over 2 seconds. On the left, the decontaminated zone was obtained with a ten-fold scanning. This zone comprised the surface without a co-deposited layer. It was specially chosen to verify that there was no ablation of graphite surface even with a multiple laser treatment of the surface. The co-deposited layer of 50 μm thickness was almost completely removed with a single scanning of $10 \times 10 \text{ mm}^2$ surface. The graphite substrate surface was protected and did not suffer any damage (the left cleaned zone on figure 3).

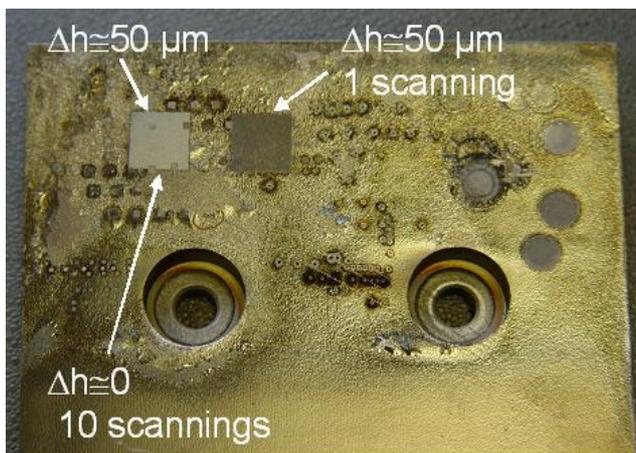


Figure 3 : TexTor tile with co-deposited layer of 50 μm thickness. Interaction parameters: laser fluence - 2 J/cm^2 , laser mean power - 20 W, high repetition rate – 20 kHz, air 1 bar, scanned zones of $10 \text{ mm} \times 10 \text{ mm}$ for 2 sec

Even with a ten-fold laser scanning, the graphite substrate surface was not observed to be damaged. The surface of the cleaned zone was found to coincide with the flaked zone. This was confirmed by TexTor tile backside surface cleaning.

A ten-fold scanning of the backside graphite surface (the same conditions as for the left zone of figure 3) was not determined to damage the graphite surface and resulted only in changing the surface color. Thus, the experiments on TexTor co-deposited layer cleaning by laser ablation gave all the reasons to conclude that the laser surface decontamination should be regarded very promising for plasma facing component surfaces detritiation.

CONCLUSIONS

The design, implementation, and tests of a vacuum chambers and sealed cells were realised. Sealed cells and chambers application allowed to study laser heating and ablation in controlled environmental conditions and also to collect ejected matter and gases for their subsequent analysis. Different analytical methods (ejected gas chromatography and mass spectrometry, GD-OES, optical and electron microscopy) were applied and tested for co-deposited layer and graphite surface characterization.

Validation of the laser treatment parameters (defined in 2003) for graphite samples was performed in the controlled environment (Ar and air). Laser ablation threshold of $(2.5 \pm 0.5) \text{ J/cm}^2$ for graphite without a co-deposited layer was obtained with 100 ns laser pulses. A specially designed imaging system was applied for laser plasma characterization and for environment effect study. The environmental effect was observed as the important plasma intensity increase in argon. The plasma intensity growth resulted from both the intensity and lifetime increase of the spectral lines. This phenomenon is regarded essential for LA-OES method for diagnostics and control of co-deposited layer removal and for surface characterization. The environmental effect manifested itself as the dark matter re-deposition around the crater in Ar and as the surface erosion with the rate of nm/pulse in air at low laser fluence (low than 0.5 J/cm^2).

A specially developed PYROMETER system was applied for laser heating characterization. A new high repetition rate laser system with 1064 nm wavelength and millisecond pulse duration was developed to study surface heating and ablation. Time resolved temperature measurements with the pyrometer will be possible with the millisecond pulses.

Ablation thresholds for different metals (diagnostics and tools) were determined experimentally for 5 ns pulses.

For the samples under study and applied nanosecond pulses, the ablation thresholds were higher than 1 J/cm^2 . Theoretical model of high repetition laser heating of a complex surface (graphite or metal with a co-deposited layer) was developed to explain the experimental results and to obtain laser detritiation optimization [6].

The obtained optimal conditions (high pulse repetition regime, 2 J/cm^2 , ablation rate) were applied for laser cleaning of 1 cm^2 co-deposited layer on TexTor graphite tile without any damage of the graphite substrate. The co-deposited layer of $50 \text{ }\mu\text{m}$ thickness was almost completely removed with a single scanning of $10 \times 10 \text{ cm}^2$ surface during two seconds. Thus, with a laser of 20 W mean power, it was possible to clean 0.2 m^2 co-deposited layer of $50 \text{ }\mu\text{m}$ thickness during one hour. With 100 W mean power, it will be possible to clean 1 m^2 co-deposited layer during the same time (one hour). The comparison of the obtained laser cleaning rate with the data presented in our previous report [3] (1 m^2 per hour of $20 \text{ }\mu\text{m}$ thickness co-deposited layer cleaning with 250 W Nd-YAG laser mean power) demonstrates an approximately six-fold increase in the cleaning rate. Further experimental and theoretical studies to explain the cleaning rate increase and to obtain the subsequent optimization of the laser detritiation performances should be made.

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