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Laser decontamination of surface impregnated with radioactive material

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Abstract
Pulsed lasers can evaporate (ablate) metals and polymers, carrying away radioactive contaminants. Several studies found in the literature lack information or cannot be compared due to the different methodologies, thus the current study aimed to test a wide range of samples using the same method. A Nd:YAG nanosecond laser operating at 1064 nm was used to process four different types of polymers and six metals, using fluences ranging from 1 to 10 J/cm^2. Samples were contaminated with an acid solution containing ^{137}Cs. Weighting the effectiveness and yield, 5 J/cm^2 was shown to be the most viable irradiation condition.

Article highlights
- Due to the absence of byproducts laser ablation is an interesting approach to decontaminate waste with radioactive material on the surface;
- Decontamination of ^{137}Cs was achieved in 6 different metals and 4 different polymers applying 3 fluences: 1, 5 and 10 J/cm^2 using a Nd:YAG laser (1064 nm 5ns pulse duration);
- We proposed an exponential decay model to represent the decontamination process as a function of the number of irradiations;
- Decontamination in low and high fluences cannot achieve optimal results; Overall, 5 J/cm^2 delivered good results, surpassing that of 10 J/cm^2 in some tests.

Keywords Laser ablation · Decontamination · Solid radioactive material

Introduction

The management of surface contaminated radioactive waste (SCRAW) materials is still a challenging issue. SCRAWs are generally generated in large volumes, with varying geometries, in all types of materials [1, 2] impregnated with a wide variety of radionuclides (mixed or not), which generally have low to intermediate activity, but which can often be long-lived and highly radiotoxic. In contrast to other radioactive wastes, SCRAWs require intensive screening and great volume reduction among other precautions. In addition, SCRAWs are often made of valuable materials that can be reused, providing economic and environmental benefits if recycled. Partial or total decontamination of the surfaces is the treatment that best suits the objective of reducing waste amounts and waste management overall costs.

A variety of methods to decontaminate SCRAWs are reported in the literature [9], which can be performed by physical and chemical processes or a combination of both, they are intermediated by a substance/material as the decontamination agent, which will end up as a secondary radioactive waste. Dry ice blasting is perhaps the more efficient solution in this matter, as the resulting CO_2 gas can be easily filtered, resulting in the lowest volume of secondary radioactive waste. Nonetheless, it is a rough approach since the operation pressure is typically 43 to 174 psi [10].

There are occasional efforts to understand and demonstrate the viability of laser cleaning for SCRAWs

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Table 1 Material with the respective radionuclide that was decontaminated by 1064 nm nanosecond laser ablation and the decontamination efficiency (DE) obtained in works consulted in literature

Material	Radionuclide	Reported Laser Nd:YAG 1064 nm specifications	DE	Ref
Metallic	^{137}Cs,	50 Hz, 10 ns	76%	[12]
	^{60}Co,		70%	[12]
	^{152}Eu		85%	[12]
Austenitic and Ferritic Steel	^{137}Cs	105–230 ns, fluence 0 to 6.1 J/cm^2	98.7%	[18]
	^{60}Co		68%	[18]
	^{90}Sr		96%	[18]
Stainless steel	^{60}Co	-	~99%	a[8]
	^{60}Co	6ns, 10 Hz	~80% (12 J/cm^2)	[19]
	^{137}Cs	1.6 J, 8 ns	~99%	[20]
		450 mJ/pulse and 14ns pulse	---------	[3]
		450 mJ/pulse, fluence 6.4 to 229.3 J/cm^2	95% (57 J/cm^2) ~70% (6.4 J.cm^{-2})	[19]
		6ns, 10 Hz	~80% (12 J/cm^2)	[21]
		8 ns, 1,6 J	----------	[22]
Carbon steel	^{60}Co	-	~99%	a[8]
zircaloy substrate	UO_2	6–8 ns, irradiance 1.2 to 6.2x10^7 W/cm^2	~90%	[23]
	ThO_2	6–8 ns, irradiance 1.2 to 6.2x10^7 W/cm^2	~90%	[23]

[a]Ref [8]: Use the laser associated with a gel

decontamination [3–13]. Laser decontamination is primarily based on the ejection of the superficial layer to which the contaminant is adhered, through the vaporization process. Laser technique has interesting advantages over the aforementioned methods such as being easy and quick to apply and dispensing with chemical or abrasive inputs, reducing the generating secondary waste and reduction of the exposure of operating personnel to radiation. Laser ablation has been applied to decontaminate surfaces [14, 15] in the decommissioning of nuclear facilities, such as contaminated paint [16, 17], and contaminated fuel pins [6]. Table 1 shows the results of a shortlist of related studies. Many of them do not specify the fluence or the laser intensity, just the pulse energy. Some even did not specify the sample characteristics, making it difficult to extract relevant information.

The references listed in Table 1 describe unique laser parameters and samples, not allowing comparisons of how a broad variety of materials respond to a fixed methodology. The present work aims to, using a set of different sample materials, submit them to the same methodology, thus evaluating the effectiveness of the laser for each, plus associate a mathematical model to represent the process.

Each laser technology finds its application niche according to a set of parameters (wavelength, beam quality, spectral width, etc.). The pulse duration is a key parameter since it will set the thermal dynamic that will process the material, which nowadays goes from continuous operation (CW) to femtosecond (fs) pulses. To exemplify, CW lasers are used to cut and weld metals (substantial deposition of energy as heat), and fs lasers are largely used for ophthalmic surgery. In between, the nanosecond (ns) pulsed laser, is the most suitable to process the surfaces of opaque materials.

Nanosecond lasers have been largely used for marking surfaces, and the technological advances in such equipment are leading to increased peak power, repetition rate (τ^{-1}) and operational reliability at the same time that costs are becoming more accessible.

The combination of these advances is promoting the application of nanosecond lasers to surface cleaning, which uses similar parameters to laser marking, but demands larger areas to be processed.

The laser-matter interaction for nanosecond pulses [24, 25] begins with the energy being absorbed and converted into electronic excitation, then transferred to the lattice as vibrations (thermal heat) [24]. Depending on the thermal properties of the material, the energy amount, and the speed with which it is delivered to the substrate, laser irradiation can cause a series of thermal effects: heating, melting, vaporization, phase explosion, and plasma generation. There are also other interesting phenomena that are worth mentioning as a consequence of the thermal effects [24, 25]: crystalline phase changes, shock waves, and melted material convection due to the Marangoni effect.

In practice, many of these processes are taking place at the same time. The proportion of each one will be ruled by the laser intensity/fluence and the material properties. For deep cleaning: ablation, melting, vaporization, phase explosion, and droplet ejection may occur depending on laser parameters. There are more delicate types of laser cleaning in which the goal is only to detach small particles adhered to the surface by weak forces (van der Waals). In general, this is not enough for the decontamination of SCRAWS. Nonetheless, there were interesting initiatives for cleaning nuclear fuel elements in which particles of fissile material end up on the exterior of the fuel tubes [26] after fuel assembly.

Laser ablation only occurs at an appreciable level and if the intensity trespasses a certain value, which depends on the material properties. Some of these are:

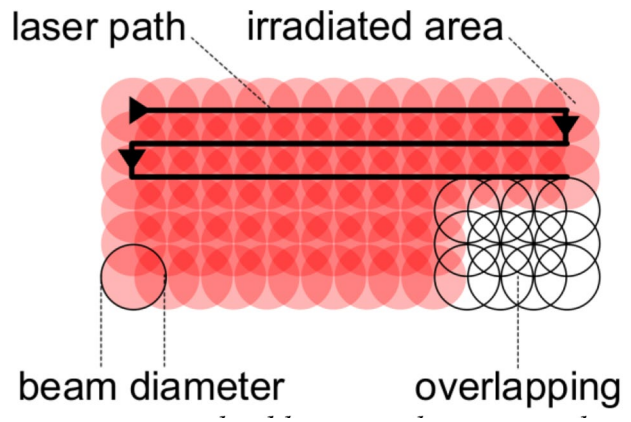

Fig. 1 Covering an area using a pulsed laser implies an overlap of spots where the laser beam hits the surface

- The absorptivity for the wavelength used, which is important because it will determine how much energy will effectively be transferred to the surface.
- The thermal conductivity, also a key parameter, because the lower the value, the easier it will be to heat an area and promote vaporization and phase explosion. At high conductivity values, the heat tends to spread far away from the point where the laser is processing material, impairing the ablation.
- The melting and evaporation points of the target material, are major parameters as well, with lower values tending to help the ablation process.

Regarding the wavelength in nanosecond lasers, visible and UV usually offer a better coupling, i.e., absorption of the pulse energy occurs in a thinner layer, reducing the volume which will be heated, thus favoring the ablation. Near-infrared lasers (1064 nm, for instance) are famous for their robustness and equipment costs, compensating for the advantages of VIS and UV wavelengths. Ultra short pulses (femtosecond) interaction with matter follows different physical mechanisms; the ablation is referred to as "non-thermal". As a consequence of the pulse duration, the pulse energy is lowered, decreasing the potential damage to the surrounding areas, nonetheless the ablation for this kind of laser uses a very tight focus impairing the productiveness.

Typically in nanosecond laser ablation of metals, the surface temperature reaches ~ 10^3 K, melt thickness of 1 µm and a removal depth of about 100 nm [27].

Finally, when dealing with a pulsed laser source that needs to process a given area, it is necessary to sweep the beam over an area, forming dots over the surface, Fig. 1. The size of the displacement will promote an overlap of the dots if the magnitude of the movement is smaller than the diameter of the affected area. Usually, a certain level of overlap is desired to ensure that all the surface has received the minimum amount of energy.

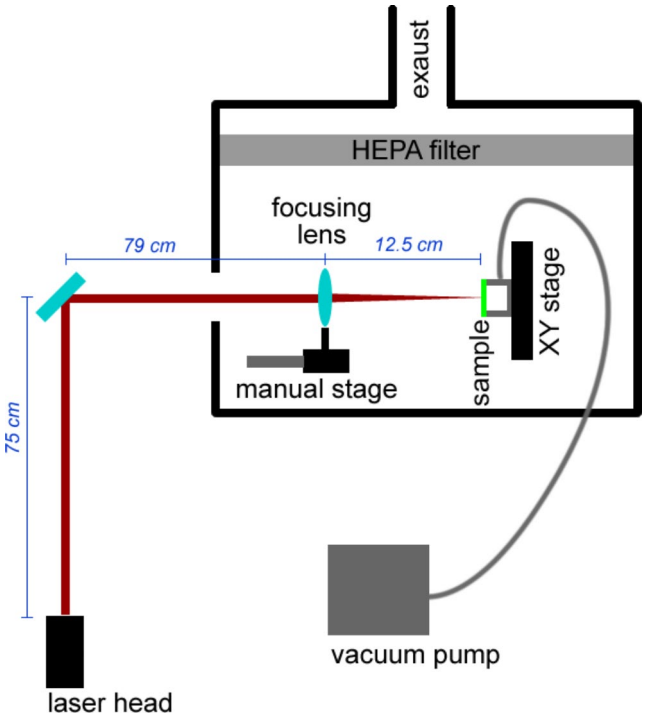

Fig. 2 Schematic illustration of the experimental setup. The vacuum pump was used to hold the sample in place without touching the borders and also without the use of adhesives or mechanical devices to attach the sample to the sample holder. The manual stage was used to compensate for the sample thickness, so the focal condition was maintained

Materials and methods

For the present study a Q-Switched Nd:YAG laser (Quantel, Ultra 100) operating at 1064 nm with a 5 ns pulse duration and a 20 Hz repetition rate was used. Two motorized translation stages (Thorlabs, LTS300/m) were coupled in an XY configuration, which moves a vacuum-operated sample holder. A focusing lens (Thorlabs, LA1986) with an effective focal length (EFL) of 125 mm was mounted on a manual linear stage to account for different sample thicknesses. Figure 2 shows a schematic of the experimental arrangement used. At the sample surface position, the beam diameter was measured following the Kapton® method [28] and for $1/e^2$ criteria (where e denotes the Euler's number), the beam presented 0.98 mm in diameter. The pulse energy was measured at the sample position using a power meter (Coherent Field Max II and J-50 MB-YAG sensor), which allowed us to set any given fluence by dividing the pulse energy by the spot area. An overlap of 50% was chosen pondering a good

covering and productivity, resulting in a raster velocity of 0.49 mm/s.

The following materials were selected as sample surfaces for the ablation tests: PVC, acrylic, polyethylene, vinyl, plated steel, copper, stainless steel, carbon steel, lead and aluminum. The selection was made considering the volume of SCRAWs stored at IPEN's repository - a nuclear institute where this work was carried out.

For each material listed above, plates with a thickness of a few millimeters were used to prepare 10 samples with a size 2.5 cm x 2.5 cm. Nine of the samples were contaminated on one surface by dripping 0.2 ml of an HCl solution (0.1 Mol/L) containing ^{137}Cs with (5.06 ± 0.30) kBq.mL^{-1} of activity concentration and dried under an infrared lamp. Each sample was contaminated with an estimated activity of (1.01 ± 0.05) kBq. The dripping was performed using a micropipette in the sample's central area. The 9 samples of each material were divided into 3 groups and irradiated under different laser fluences: 1, 5, and 10 J/cm^2. These fluences were chosen according to the maximum energy of our laser for a focal spot of 1 mm diameter. A low fluence of 1 J/cm^2 was of interest to observe the decontamination efficiency for sub ablation conditions in metals. For each fluence the decontamination runs were done in triplicate, and the standard deviation was used to evaluate the statistical fluctuations.

Before and after irradiation with the laser beam, each sample was counted for 1 min in the alpha and beta counter Ludlum model 2929 coupled with the model 43-10-1 detector. The activity counting (before and after ablation) was used to assess the decontamination efficiency. The process was repeated with each sample until background counting was achieved or four irradiation scans were performed.

The total surface of the contaminated surface of the sample was scanned by the laser beam, independently of the size of the area where the radioisotope was deposited.

Assuming that the decontamination in each run has a constant removal rate, an exponential decay could represent the data as a function of the irradiation runs. Based on this hypothesis plots of the normalized counting as a function of the number of irradiations were fitted with an exponential decay function, expressed by the Eq. (1).

$$y(x) = y_0 + Ae^{-xk} \quad (1)$$

where y(x) is the activity remaining in the sample after x decontamination runs, y_0 represents the residual activity not removed by the laser irradiation, after an arbitrarily large number of irradiation runs, A is the initial activity, x is the irradiation run number and k a decontamination 'constant'.

To present the results of the irradiation runs, both y(x), the remaining activity in the sample, and A, the initial activity, were represented by the 5-minute counting of the sample in the alpha and beta counter, converted to a normalized value by dividing all counting results by the initial counting.

The count difference is defined as the radiation counting in a given instance (irradiation) subtracted by the counting at the prior instance, this value will show in which irradiation the process is removing more contaminants:

$$CD_{f,i} = C_{f,i-1} - C_{f,i}$$

where f is the fluence (1, 5, and 10 J/cm^2), and I is the irradiation number (from 1 to 4). For $i = 0$ (in other words, no irradiation), $C_{f,-1} = C_{f,0}$, *i.e.*, $CD_{f,0} = 0$.

The uncontaminated samples were used to characterize the interaction of the laser beam with the substrate surface. A single laser pulse was used to assess the damage caused by the three different fluences, as observed using a scanning electron microscope (Hitachi, TM3000). The samples were also photographed after each laser irradiation, using a macro lens.

Results

Decontamination results of polymers are displayed in Fig. 3, further images (photographs and scanning electron microscopy are available in the supplementary material).

PVC: The data points to an almost ideal scenario for decontamination with 10 or 5 J/cm^2 when looking for the fitting constants, $y_0 \rightarrow 0$ and $x \rightarrow 1$, means that residual decontamination is not present, and high values (> 2) for the decontamination constant k, denote an efficient decontamination process. On the other hand, the fluence of 1 J/cm^2 did not achieve good results and did not achieve a plateau of radiation counting even after 4 rounds of irradiation. The results also failed to fit an exponential decay, since the data is almost in a straight line. When analyzing the counts' difference it is noted that the second irradiation was more effective than the first in contrast with the other results.

Polyethylene: The graph shows that for 10 and 5 J/cm^2 the decontamination was effective, with fitting values y_0 and x tending to 0 and 1, respectively, For fluence of 1 J/cm^2, the fitting suggests a plateau of residual radioactivity of 47% of the initial value ($y_0 = 0.47$).

Vinyl: the plot for 1 J/cm^2 shows poor decontamination occurred, the fitting points to a plateau of 21% of the initial activity will remain in the sample. For 10 and 5 J/cm^2, the decontamination process was effective ($y_0 = 0$ and x = 1) and efficient (k > 5), only one irradiation removed all the contaminants.

Acrylic: Transparent acrylic was used as sample, due to its weak interaction with the laser (especially in the used

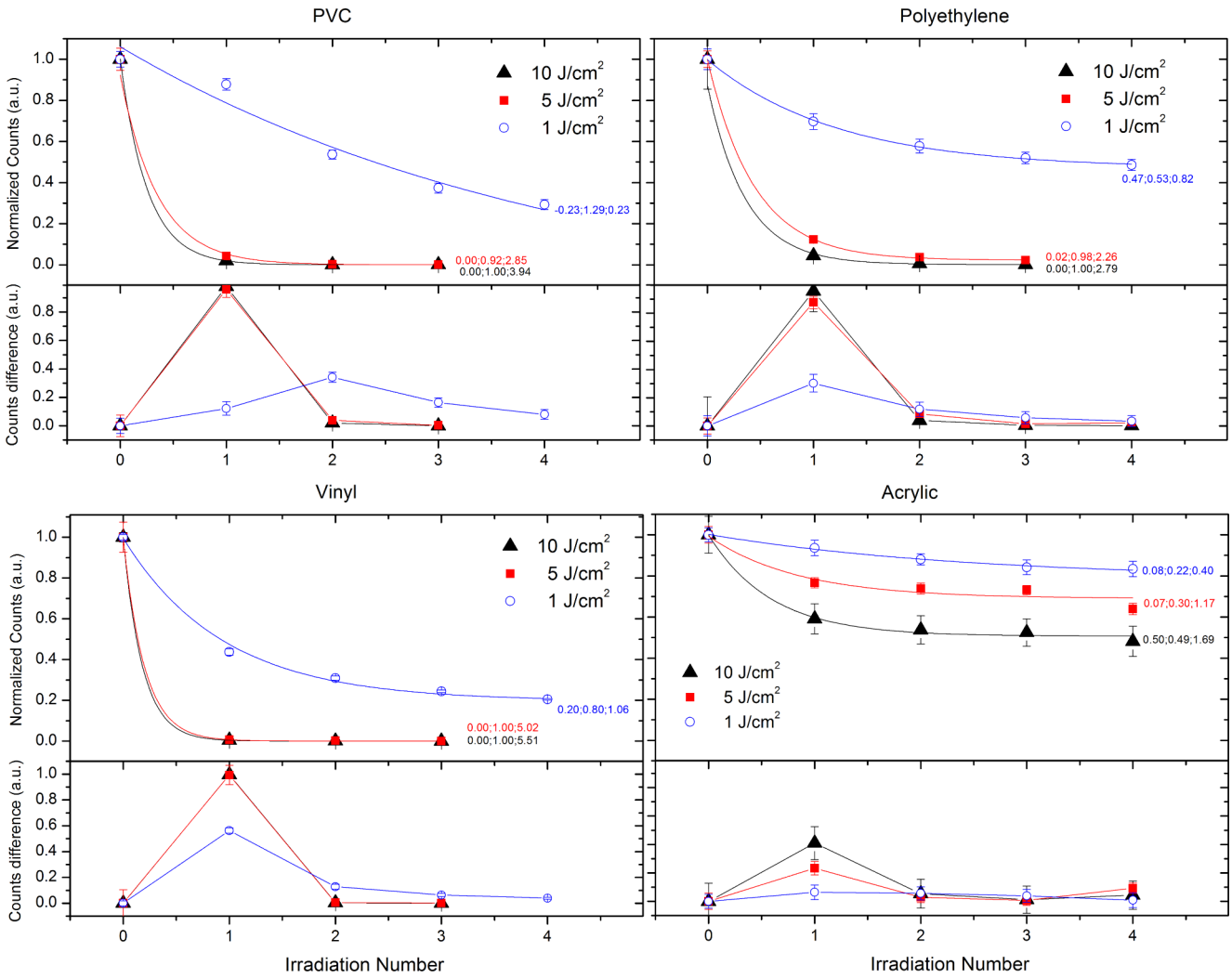

Fig. 3 Normalized counting and counts difference. Next to each fitting line, three parameters are shown (y_0, A, k), according to Eq. 1

wavelength) there is no perceived alteration on the surface appearance, suggesting no ablation processes. The decontamination analysis corroborates the photographs (see supplementary material); the best result left ~ 50% of the initial activity when using 10 J/cm^2.

Decontamination results of metals are displayed in Fig. 4 further images (photographs and scanning electron microscopy are available in the supplementary material).

Stainless steel: The data plotted presents an interesting dynamic: 10 J/cm^2 presented a good decontamination value on the first irradiation (counts difference), however, after that the laser could almost not remove any contamination from the sample (y_0 pointing to a remaining activity of 16%). The 1 J/cm^2 had more subtle decontamination on the first round but was able to eject the contamination from the sample in the subsequent rounds. 5 J/cm^2 presented greater efficiency and effectiveness.

Galvanized steel: The decontamination process occurred effectively for the three fluences, for 5 and 10 J/cm^2 single irradiation removed more than 90% of the initial activity (approximately 92% and 96%, respectively). After 4 irradiations all samples presented activities below 1% of their initial activity.

Lead: The fittings for the data show the same $y_0 \rightarrow 0$ and A→1, but the velocity of the decontamination is slightly different for each fluence, growing as the fluence increases.

Copper: The collected data and fittings, for 5 and 10 J/cm^2 presented very similar results, and 1 J/cm^2 also presented satisfying decontamination values, achieving 97% of radioactivity removal in the first irradiation.

Aluminum: Good efficiency for 10 and 5 J/cm^2 with almost the same performance, 1 J/cm^2 left 13% of initial activity, not an ideal scenario, however, it kept the visual aspect of the sample which for specific applications could be a requirement.

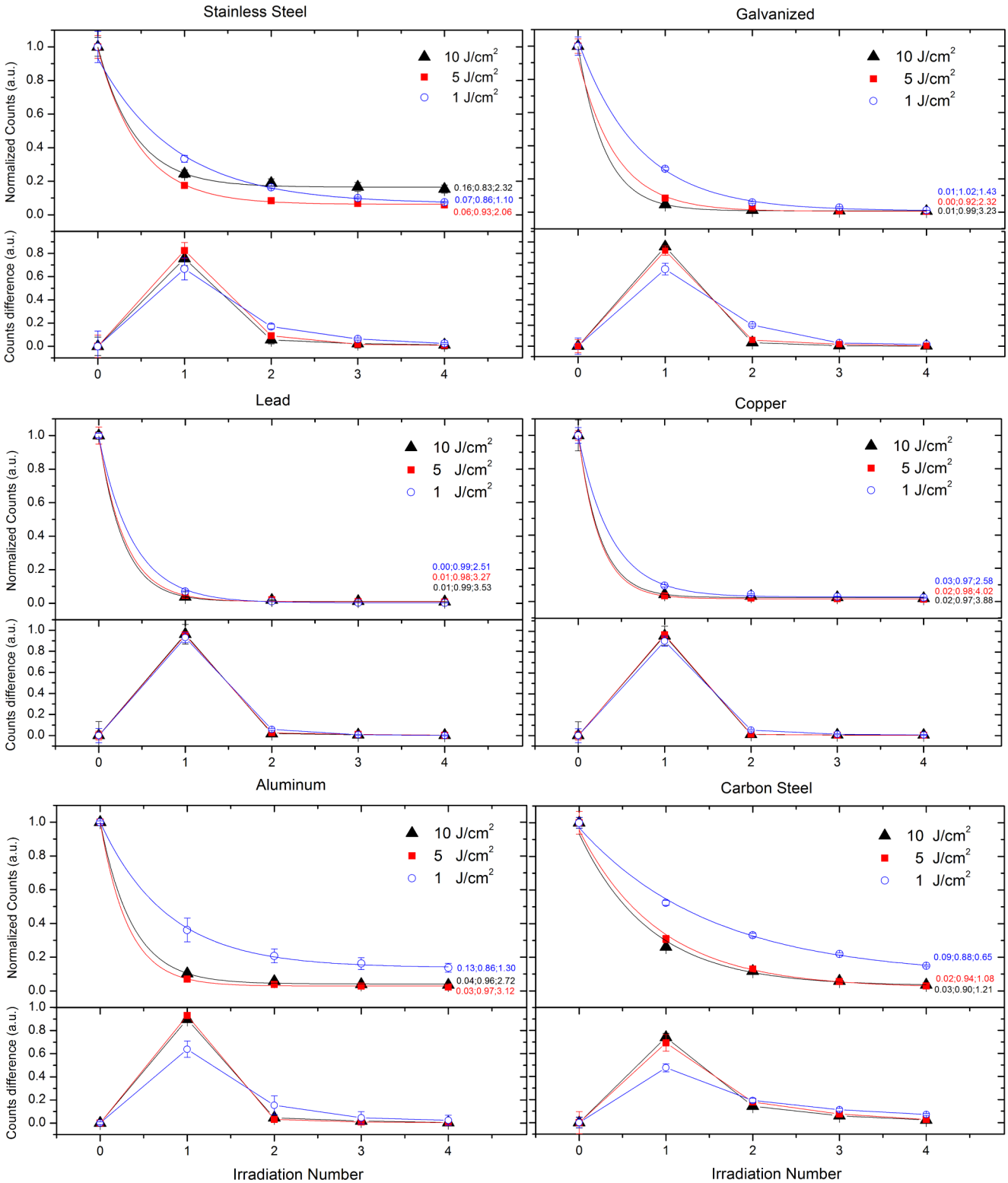

Fig. 4 Normalized counting and counts difference. Next to each fitting line, three parameters are shown (y_0, A, k), according to Eq. 1

Comparative analysis and general discussion

A difference in behavior, see Fig. 5, between polymers and metals was perceived for the 1 J/cm^2 (especially on the first irradiation), the removal of contaminants was well below the average of the metals, even when ignoring the acrylic

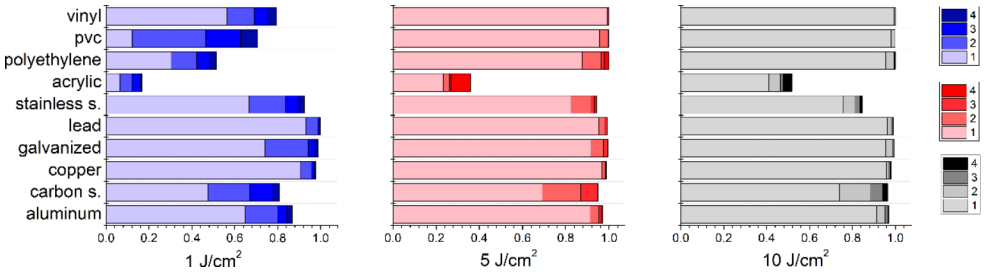

Fig. 5 Cumulative decontamination as a function of irradiation number (from light to dark color), and as a function of the fluence. The first four samples are polymeric, and the last six are metallic

sample (which is challenging due to its transparency). The polymers, in general, seem to have a threshold between 1 and 5 J/cm^2, above which the decontamination efficiency soars. On the other hand, metals were shown to be more progressive in decontamination, increasing the efficiency gradually through the fluences and irradiation runs.

Even though part of the contaminant had been removed for the acrylic samples, ablation played a minor role in the process, as the SEM images showed. The process that could explain the partial decontamination is described by A. C. Tam et al. [29] and Kelly et al. [30] as a particle and/or substrate ejection by thermal expansion, or even humidity on the surface being evaporated and carrying the contaminant away. In our case, the contaminant is within salt crystals formed by the HCl.

The decontamination in the present study occurred mainly by the ejection of the substrates themselves.

As metals present free electrons, they absorb the laser energy in a thickness of about 10 nm [31] (extinction coefficient of aluminum is ~9@1064 nm[32]), on the other hand, polymers (extinction coefficient for PVC is $\sim 6.5 \times 10^{-7}$@1064 nm[33]) absorbs the laser energy in a greater volume. In other words, the laser energy for metals is deposited in a smaller region, concentrating the heat and thus promoting the ablation more easily.

In respect to the stainless steel and aluminum results which had better results for 5 J/cm^2 than 10 J/cm^2, we hypothesized that the contaminant has been internalized through the movement of the molten metal by the Marangoni current [34] due to the high energy delivered by the laser pulse.

In this study in many cases, there was no ablation (melted and expelled material), with poor decontamination results. And since there is no need to maintain surface integrity SCRAWS, ablation proved to be the more efficient way to approach the subject.

Conclusion

Despite the present study is not the first to test laser as a tool for decontaminating surfaces impregnated with radioactive material, we demonstrate the application of a standardized methodology for a variety of 10 different materials using 3 different fluences. This broad experimental study showed that the majority of samples behave slightly differently for low laser fluences (metals and opaque polymers) but are very similar for fluences greater than 5 J/cm^2. The present work helps, together with the previous studies found in the literature, to support the potential application of lasers in SCRAWS treatment.

The results obtained present predictable behavior for the majority of the samples, with the activity of the samples decreasing as a function of both fluence and number of irradiations, pointing out that the proposed methodology, from sample preparation to radiation counting, can be considered reliable and consistent.

Perhaps, the only point in the methodology that deserves a review for future studies was the use of HCl solution (as the ^{137}Cs carrier). The acid, even quite diluted, may have attacked some samples, altering their original characteristics, however was not an impairment issue for the evaluation proposed.

The fluences applied were also shown to be well chosen for the laser parameters (wavelength and duration), as for the lowest fluence the majority of the samples had been poorly decontaminated, and for the maximum fluence in most of the cases reaching more than 95% of the activity removal. Therefore, the range from 1 to 10 J/cm^2 reached both extremes.

The 5 J/cm^2 fluence was shown to be a great choice between effectiveness and efficiency. For all the samples this fluence delivered results very similar to 10 J/cm^2 applying half of the pulse energy. For field applications using highly energetic laser systems, it may be more productive to increase the beam area while maintaining the 5 J/cm^2, than use higher fluences.

The proposed exponential decontamination model, to represent the decontamination process, agreed consistently with the data behavior in all cases in this study, indicating that the decontamination of each irradiation depends on the initial activity. Therefore, the adjusted parameters provided quantitative information to evaluate the process. The k factor represents the rate at which the contaminants are removed from the sample over the irradiations and y_0

provides information about the residual activity despite the increased number of irradiations.

For all samples, excluding the transparent acrylic, 5 J/cm^2 was shown to be a feasible fluence for decontamination using up to 3 irradiation runs and 50% of beam overlap. If the materials are not heavily oxidized (as the carbon steel), 2 irradiation will remove more than 90% of the initial activity.

The initial concern that the non-ejected laser molten material could incorporate and retain the radioactive material does not seem to be a major mechanism, and the ejected part of this molten material seems to be an efficient process for removing the contaminating material. A system capable of capturing and retaining the removed contaminants still needs to be developed to make this approach viable.

Supplementary Information The online version contains supplementary material available at https://doi.org/10.1007/s10967-022-08525-5.

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