

## Sub-micrometer coloration depth of linens by vacuum ultraviolet radiation

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### Abstract

We present experimental results of excimer laser irradiation (wavelengths 193 nanometers and 308 nanometers) of raw linen fabrics, seeking for a coloration similar to that of the body image embedded onto the Shroud of Turin. We achieved a very superficial Shroud-like coloration of linen yarns in a narrow range of irradiation parameters. We also obtained latent coloration that appears after artificial aging of linen following laser irradiations that at first did not generate any visible effect. Most importantly, we have recognized distinct physical and photo-chemical processes that are responsible of both coloration and latent coloration. These processes may have played an important role in the generation of the body image on the Shroud of Turin.

**Keywords:** Excimer Laser, Latent image, Coloration depth, Shroud of Turin, Photo-chemistry

### 1. INTRODUCTION

The faint yellowed body image embedded into the linen cloth of the Turin Shroud has peculiar chemical and physical characteristics [1] that at the moment cannot be replicated all together in laboratory [2 - 20]. The only in-depth analysis of the properties of the body image on the Shroud was performed under the auspices of the Shroud of Turin Research Project, Inc., (STURP) [4 - 11]. STURP researchers found no evidence for pigments (paint, dye or stains) or artist's media on the Shroud. They concluded that the two faint body images are not painted, printed nor produced by heating the cloth. Moreover, the image color resides on the topmost fibers in the cloth weave, and recent results [18] show the depth of coloration is very thin, down to 200 nm ( $1 \text{ nm} = 10^{-9} \text{ m}$ ), i.e. the thickness of the primary cell wall of the single linen fiber. Up to date, attempts to reproduce an image with the same characteristics have been unsuccessful. Some researchers obtained images having a similar macroscopic aspect [6, 17, 19, 20] but none of them matches all the microscopic features of the Shroud image. In this respect, the origin of the body image is still unknown. In this paper, we summarize the main results of the experiments of linen coloration performed at the ENEA Research Centre of Frascati in the years 2005 - 2010, seeking for physical and chemical processes apt to generate a coloration similar to that of the Shroud image.

### 2. THE MAIN IDEA

Several independent works [13 - 17] have shown that a burst of electromagnetic energy may account for the main

Shroud image characteristics, e.g., color superficiality, distance coded information, image in non-contact linen areas, absence of pigments. An attempt to reproduce the face of the Shroud irradiating a linen with a CO<sub>2</sub> laser (infrared emission wavelength  $\lambda = 10.6 \mu\text{m}$ , where  $1 \mu\text{m} = 10^{-6} \text{ m}$ ) gave a similar macroscopic result, presently exposed at the Shroud Museum in Turin. However, the microscope analysis revealed a too much thick coloration depth and many linen yarns burned [20], which are substantial differences with respect to the Shroud image characteristics [1]. It was clear that one of the main causes of the unwished burned yarns observed in [20] was the too long wavelength radiation emitted by the CO<sub>2</sub> laser. In fact, long-wavelength (infrared) radiation excites vibrational energy-levels of the target matter that relax in far-infrared energy, instantaneously heating the irradiated linen. On the contrary, it is well known that the energy carried by short wavelength radiation breaks the chemical bonds of the irradiated matter, almost without secondary heating effects. As a consequence, in 2005 we have considered the ultraviolet (UV) radiation as one of the best candidate for obtaining two of the main characteristics of the Shroud image: a thin coloration depth and a low-temperature image-formation process [1, 9, 11]. We have first irradiated linen cloths with two XeCl excimer lasers (UV emission wavelength  $\lambda = 308 \text{ nm}$ , i.e. 34 times shorter than the CO<sub>2</sub> wavelength) emitting different pulse durations [21, 22]. The encouraging but improvable results, summarized in the section 3, pushed us to repeat irradiations using a shorter wavelength radiation in the vacuum UV (VUV), namely the  $\lambda = 193 \text{ nm}$  emitted by an ArF excimer laser [23, 24] as discussed in the section 4.

### 3. EXPERIMENTAL RESULTS WITH UV LASER RADIATION

When irradiating linen cloths with a sequence of laser pulses (corresponding to an hypothetical burst of energy correlated to the Shroud image) emitted by a 5J/pulse, 120 ns XeCl laser ( $\lambda = 308$  nm), we were not able to obtain any coloration. In fact, at large intensity values linens were burned, and at lower intensity values linens were not colored at all. (We remind the intensity is the laser energy per unit time per unit surface incident on the linen fixed on a frame. The intensity on linen is varied by moving the linen along the optical axis of the 1-m focal length lens).

Then, we repeated the irradiations with a different XeCl laser emitting a 30-ns pulse duration (4 times shorter than before) and 0,4 J/pulse scanning a range of intensity values centered around the same intensity values tested with the previous longer pulse duration laser. In this way, we have obtained the permanent coloration of linens that can be achieved in a narrow range of pulse duration, intensity and time sequence of laser shots. Experimental setup and results are detailed in [21]. However, the hue of color (light brown) was darker than the yellow-sepia of the Shroud image, and the thickness of coloration of linen yarns was still larger than the topmost ( $\approx 0.2$   $\mu\text{m}$  thick) fiber coloration of the Shroud body image [18].

To obtain a thinner color penetration depth, we used an ArF laser emitting a wavelength much shorter than XeCl lasers. In this way we obtained a reduction of the coloration depth and a better overlap with the features of the Shroud image.

### 4. EXPERIMENTAL RESULTS WITH VUV LASER RADIATION

The ArF excimer laser emits pulses at  $\lambda = 193$  nm, in the VUV spectral region, with 0.08 J/pulse in a 12 ns pulse duration and 1 Hz repetition rate. As in the previous experiments, we investigated a wide range of different combinations of laser intensity and number of shots. Table 1 reports a summary of naked-eye observation of irradiated linens vs. the number  $N$  of consecutive shots, the spatially averaged single-shot intensity  $I$  and the total spatially averaged intensity  $I_T$  respectively defined as

$$I = (1/A) \times \iint_{\sigma} I(x,y) dx dy, \quad (1)$$

and

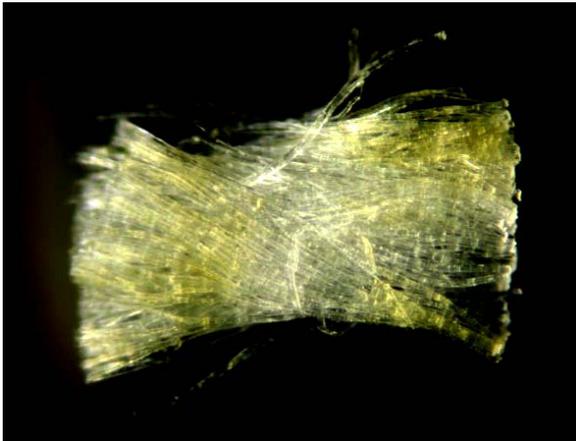
$$I_T = (N/A) \times \iint_{\sigma} I(x,y) dx dy, \quad (2)$$

where  $A$  = area of the laser spot and  $I(x,y)$  = local intensity value at the point with coordinates  $x, y$  within the irradiated spot  $\sigma$ . Table 1 clearly shows that the effects of laser irradiation on linens are proportional to the total intensity  $I_T$  and not to the intensity  $I$ . A yellow coloration like that shown in figure 1 is achieved when the combination of single-shot intensity (see eq. (1)) and number of shots produces a total intensity (see eq. (2)) in the range  $I_T \approx (2 - 4) \times 10^9$  W/cm<sup>2</sup>. When  $I_T > 4.6 \times 10^9$  W/cm<sup>2</sup> linen is ablated, and when  $I_T > 6 \times 10^9$  W/cm<sup>2</sup> it is holed.

An interesting property of the irradiated linen is that the hue of color can continuously change from light yellow to yellow-sepia by increasing  $I_T$ .

**TABLE 1.** Summary of the main results observed on linen as a function of the ArF laser irradiation parameters.

| <b>I (MW/cm<sup>2</sup>/shot)</b> | <b>N</b> | <b>I<sub>T</sub> (MW/cm<sup>2</sup>)</b> | <b>Macroscopic results on linen</b>                      |
|-----------------------------------|----------|--|--|
| 35                                | 30       | 1050                                     | unchanged  |
| 14                                | 100      | 1400                                     | small surface change observed at grazing incidence light |
| 36                                | 50       | 1800                                     | light yellow coloration                                  |
| 10.5                              | 200      | 2100                                     | yellow coloration  |
| 11.2                              | 200      | 2240                                     | yellow coloration  |
| 6.6                               | 402      | 2645                                     | yellow-sepia coloration                                  |
| 6                                 | 600      | 3600                                     | yellow-sepia coloration                                  |
| 13.3                              | 500      | 6650                                     | ablated  |



**Figure 1.** Microphotograph of a linen yarn irradiated with a total laser intensity  $I_T = 2.2 \times 10^9 \text{ W/cm}^2$ .

In other words, we can easily adjust the RGB value and chromatic coordinates [25] just changing the total laser intensity (i.e. the number of laser pulses).

As an example, let us consider the third row of table 1. In this case we obtain a very light yellow coloration after 50 laser pulses. Each laser pulse changes a little the coloration achieved by the previous shot. Namely, each shot changes the contrast and RGB value of the linen coloration by a quantity  $1/50 \approx 2\%$ , thus allowing a very accurate control of the chromatic coordinates. In fact, a change of 2% cannot be appreciated by naked eye observation, because in this case the coloration after 50 shots (i.e. the 100% color change) is close to the threshold for visual inspection. Similar arguments can be stretched to the second – to seventh rows of table 1.

Equations (1) and (2) show the intensity values in table 1 are “averaged” across the laser spot size. This means that due to the “bell-shaped” spatial intensity profile of the laser beam, the local intensity value  $I(x,y)$  may substantially differ from the average intensity  $I$ . As a consequence, coloration was not uniformly distributed across the laser spot at the yarn level. In some cases we observed all the possible effects on linen within the same laser spot. As an example, figure 2 shows linen yarns ablated in the middle of the laser spot (where the intensity is higher), while one yarn away, at an intermediate intensity level, there are yellowed yarns. At lower intensities, close to the spatial wings of the laser spot, linen yarns are unaltered. In this respect, the range of ArF laser parameters suitable to achieve a permanent coloration is much narrower than the range of XeCl laser parameters reported in [21].

Concerning the depth of coloration, microphotographs show the average color penetration depth in many different linen yarns irradiated with different  $I_T$  values ranges between  $7 \mu\text{m}$  and  $26 \mu\text{m}$  [24] that is, a factor 11 to 3 times smaller than the penetration depth achieved after irradiation at  $\lambda = 308 \text{ nm}$  [21, 22]. This experimental evidence confirms that a shorter laser wavelength

produces a thinner depth of coloration. Our linen yarns have an average diameter of  $300 \mu\text{m}$ , this means the  $\lambda = 193 \text{ nm}$  ArF laser pulses penetrate 2% to 9% of the yarn diameter, in average, depending on the specific irradiation condition.

Most importantly, among the available microphotographs of colored yarns/fibers (we analyzed only few thousands fibers over half million irradiated fibers) we found at least one irradiated fiber showing a colorless medulla, see figure 3, and in this case the coloration may reside in the primary cell wall of the same linen fibers, which has an approximated thickness of  $0,2 \mu\text{m}$ , a property that closely resembles the very thin coloration depth of the image fibers of the Shroud [18].



**Figure 2.** The linen area irradiated by ArF laser pulses shows different characteristics corresponding to the local laser intensity value  $I(x,y)$ . 1) colored area; 2) ablated area; 3) area irradiated below threshold for coloration. From Ref. [24].



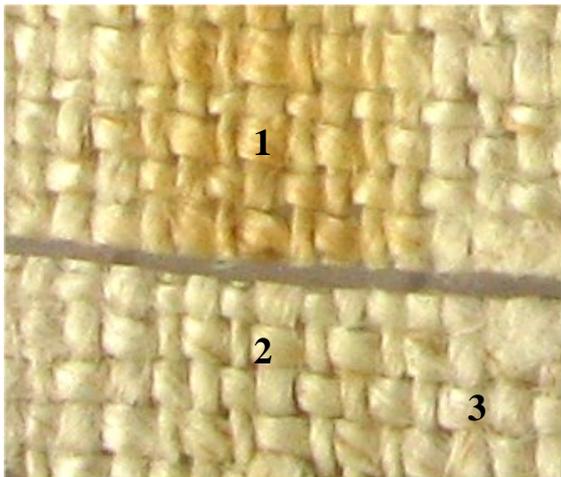
**Figure 3.** Microscope image of a single linen fiber colored with ArF laser. The mechanical damage in the middle shows a colorless inner medulla. Contrast-enhanced detail of figure 8 in Ref. [24].

## 5. LATENT COLORATION

A suitable aging technique can color the irradiated area of yarns even when no visible results are obtained by laser irradiation, as described in the following.

We cut half of the laser spots on linen irradiated at  $I_T = 1.4 \times 10^9 \text{ W/cm}^2$ , i.e., below threshold for coloration (see table 1). One of the two parts was heated 10 seconds by an iron at the temperature of  $190 \pm 10 \text{ }^\circ\text{C}$ , and a visible coloration of the heated part of the fabric appeared in the area corresponding to the laser spots, as shown in figure 4.

Figure 4 shows that the heating process, which simulates aging, colored only the linen area irradiated just below threshold.



**Figure 4.** Linen fabric cut after laser irradiation below-threshold for coloration. 1) Irradiated region after heating. 2) Irradiated region not heated. 3) Non irradiated region. From Ref. [24].

Moreover, when heating the laser spot on linen corresponding to the first row of table 1, latent coloration does not appear. This means we are below the threshold for latent coloration. As a consequence, we can fix the range of the total intensity suitable to obtain latent coloration as  $I_T \approx (1.1 - 1.7) \times 10^9 \text{ W/cm}^2$  corresponding to a range of total fluence (total radiation energy per unit surface)  $F_T \approx (13 - 20) \text{ J/cm}^2$ .

By the way, when heating a colored spot (i.e. a linen irradiated above-threshold for coloration) the yellow color becomes more visible as it shows a higher contrast with respect to surrounding not irradiated areas.

In section 7 we will discuss the physical and chemical processes possibly involved in the coloration and latent coloration results described above.

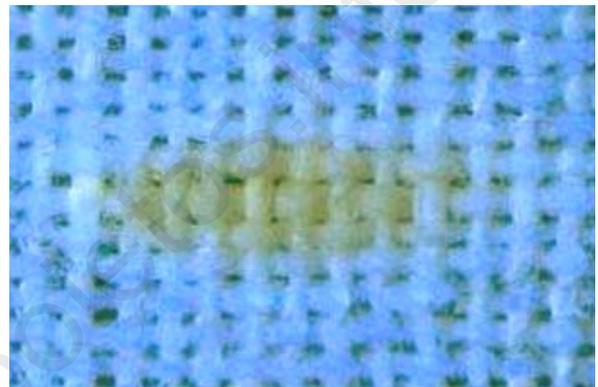
## 6. ULTRAVIOLET FLUORESCENCE

One of the peculiar properties of the Shroud image is the lack of fluorescence of the image fibers under UV illumination [1, 6, 11].

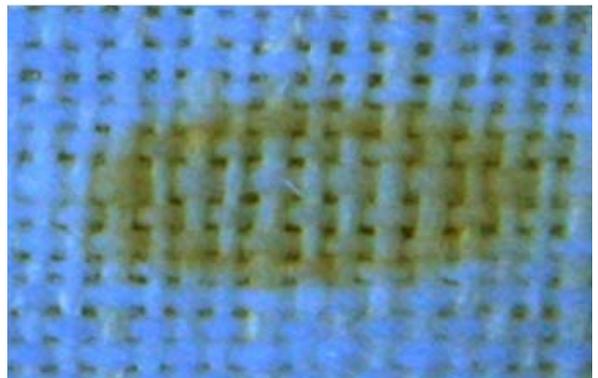
Figure 5a shows the UV-induced fluorescence of the linen after laser irradiation. Note the region irradiated by the ArF laser in the middle is not fluorescent under UV illumination, like in the case of the Shroud image fibers.

Figure 5a suggests that the VUV laser photons modify the electronic structure of linen in a way that allows the quenching of the fluorescence of the background linen.

As it happened in the coloration process, the lack of fluorescence of the irradiated fibers under UV illumination appears only in a very narrow range of irradiation parameters. As an example, figure 5b shows that a too much intense laser irradiation produces the lack of fluorescence only in a spatial ring, corresponding to the “right” range of intensity across the laser spot (which has a “bell-shaped” intensity profile). Outside this ring the intensity is too large (in the middle) and too weak (in the external part) to allow the quenching of fluorescence.



**Figure 5a.** Ultraviolet induced blue fluorescence of linen after irradiation with ArF laser in the working point of the third row of table 1. The irradiated area is recognized by the lack of fluorescence. From Ref. [24].



**Figure 5b.** Ultraviolet induced blue fluorescence of linen after irradiation with ArF laser in the working point of the seventh row of table 1. In this case, the lack of fluorescence is not uniform across the laser irradiated region.

Note that the laser spot of figure 5b gives an apparently uniform yellow-sepia coloration when observed at sunlight (cf. seventh row of table 1). This means the UV induced fluorescence gives a more accurate and selective response than the naked eye. In fact it allows to recognize linen regions irradiated by a intensity too large to obtain the lack of fluorescence, but still giving a yellow-sepia coloration.

## 7. ANALYSIS AND DISCUSSION

The experimental results detailed in the previous sections are quite exciting, as it seems we found a way to obtain a hue of color and a very thin penetration depth inside the linen fibers that closely resemble those of the image embedded onto the Turin Shroud. We carefully checked the reproducibility of the results to make sure that everybody can replicate them, provided a sufficient attention is paid to match the narrow conditions of irradiation suitable to obtain the above Shroud-like linen coloration.

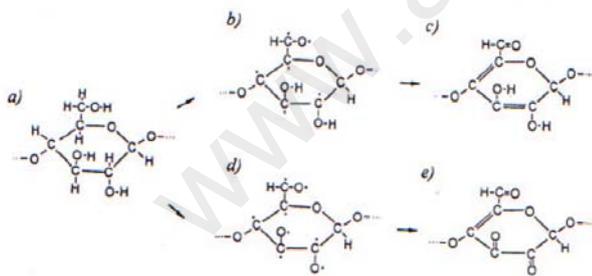
Obviously, nobody can claim the body image of the Shroud was made by a burst of VUV flashes emitted by an excimer laser. Rather, the excimer laser can be considered a powerful tool to investigate the physical and chemical processes experienced by the Shroud to finally give such a peculiar coloration. To gain a deeper insight into these processes, let us give a closer look at the chemical and physical properties of linens.

### 7.1 Chemical processes.

Flax fibers spun to produce linen yarns from which Shroud was woven are made by an inner part of nearly pure cellulose and by an external thin layer (the so called primary cell wall) basically made by hemicellulose [26, 27].

The very superficial coloration of the Shroud image was formed by an unknown process that caused oxidation, dehydration and conjugation of the polysaccharide structure of the flax fibers, to yield a conjugated carbonyl group as the chromophore, i.e. a kind of premature aging process of the linen [9, 10, 28].

Figure 6 shows different chemical steps possibly involved in the linen coloration of the Shroud.



**Figure 6.** Main sugar kernel of both cellulose and hemicellulose (a) and steps (b)  $\rightarrow$  (c), and (d)  $\rightarrow$  (e) transforming it into a chromophore made by conjugated carbonyl groups after experiencing dehydrative oxidative processes. The double bonds C=C in (c) and (e) ultimately lead to the coloration of the image fibers of the Turin Shroud. From Ref. [28].

The different coloration depths obtained with XeCl and ArF lasers (see sections 3 and 4) may be due to the

different wavelength. In fact, the shorter  $\lambda$  allows a smaller penetration depth and then a greater energy absorbed for unit volume. However, in [23, 24] we have experimentally shown that there is only a 11% difference in linen absorption between 193 nm and 308 nm. As a consequence, an additional and more specific mechanism is necessary to explain the different depth of coloration as well as the different color, namely a yellow-sepia after 193 nm irradiation and a light-brown after 308 nm irradiation.

This additional mechanism might be triggered by the absorption peak below 260 nm of the ketonic carbonyls that promote yellowing of the less stable hemicellulose in the primary wall cell [26, 28, 29]. In other words, the VUV 193-nm photons are absorbed by ketonic carbonyls and bring photolytic degradation of hemicellulose, causing molecular bonds dissociations that promote Shroud-like chromophoric changes as shown in figure 6, finally leading to the yellowish coloration.

Note that the UV 308-nm photons do not match the absorption band of ketonic carbonyls. Rather they can be absorbed by aldehyde groups [29]. As a consequence, 308-nm radiation cannot start the above described multi-step process that lead to the yellowing of cellulose and hemicellulose. In fact, our UV 308-nm photons produce a light brown coloration of linens.

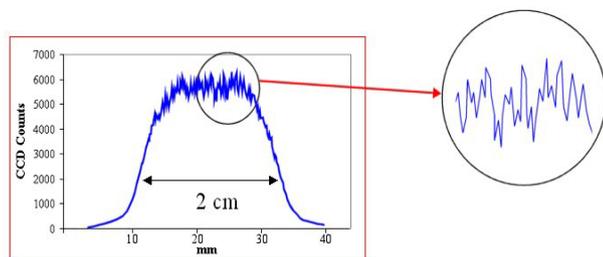
Concerning latent images formation described in section 5, they can be explained by the oxidation of cellulose (and thus production of conjugated unsaturated structures) induced by heat. In fact, the coloring process triggered by an initial exposure of UV light is accelerated and strengthened by heat, as detailed in [30].

### 7.2 Physical processes.

A basic question is whether the intensity or the energy density (fluence) of laser pulses is the key parameter for the coloration of linens. We have shown in section 3 that two XeCl laser pulses having the same fluence and different pulse durations give much different coloration results, thus suggesting the intensity is the key parameter. However, Table 1 shows that subsequent laser pulses sum their effect, suggesting on the one hand that the total intensity  $I_T$  is the relevant parameter, and on the other hand that once fixed the pulse duration, the total number of photons for unit surface (i.e., the total fluence) is the key parameter. This apparent dichotomy testifies we are facing an intricate photochemical process, where the intensity and the fluence play a dominant role by turns, depending on the range of pulse duration, number of photons, number of consecutive shots and repetition rate of the laser pulses.

Let us now discuss why we obtained the Shroud-like coloration of the primary wall cell of linen fibers (see figure 3) only locally. As mentioned in section 4, the intensity profile of excimer laser beams is not flat-top. In particular, the excimer laser intensity profile shows high-spatial frequency fluctuations, that can be revealed and

measured by a high-resolution CCD camera as shown in figure 7.



**Figure 7.** Two-dimensional intensity shape of the laser beam measured by a CCD camera Andor model DV-430UV, having a single pixel resolution  $11 \mu\text{m} = 0.011 \text{ cm}$ . The inset shows a zoom of the high-frequency intensity fluctuations.

Figure 7 shows the intensity fluctuations have a period randomly fluctuating in the sub-mm scale, with intensity gradients as high as  $350 \text{ MW/cm}^2/\text{cm}$ . A so huge spatial gradient of the intensity may explain why we can achieve the “right” intensity value for a sub-micrometer coloration only in a very small spatial region.

We devote a final comment about the main difference that still exists between our linen coloration and the Shroud image. Photomicrographs and samples show that the fading of the Shroud image is a result of concentrations of yellow to light brown fibers [1]. Moreover, the color of the image-areas have a discontinuous distribution along the thread of the Shroud, and striations are evident [31].

Obtaining the same characteristics with a laser is difficult, although possible. We would need a laser beam having a very peculiar spatial shape of the intensity, similar to a profile saw-toothed with variable period. The state of the art technology of diffractive optics can modulate the intensity shape of laser beams at easy, producing a shape able to reproduce striations and discontinuous distribution of colored fibers across the linen textile. However, in our opinion this effort would be meaningless and beyond our aim. Our goal cannot be the perfect reproduction of the whole Shroud image by a VUV excimer laser. Our goal is obtaining a deeper insight on the physical and chemical processes experienced by the Shroud to generate the image embedded onto.

## 8. CONCLUSION

In this paper we have summarized five years of experiments apt to show that nanosecond-duration VUV laser beams are able to color the outermost portion of the linen threads (color penetration depth down to  $7 \mu\text{m}$ ). We also obtained at least one fiber colored across the sub-micrometer depth on the primary cell wall of linen fibers, see figure 3, comparable with the thinnest coloration depth observed in the Turin Shroud fibers image [18].

The permanent coloration is a threshold effect and it can be only achieved in a very narrow range of photons parameters:  $I \times N = I_T \approx (2 - 3.6) \times 10^9 \text{ W/cm}^2$ . Above this range linen is ablated, while below this range irradiations bring latent images that appear only after artificial aging. When  $I_T \leq 1.1 \times 10^9 \text{ W/cm}^2$  linen is not colored at all. Even when  $I_T$  is above threshold for coloration, not all the irradiated fibers are colored (see figure 2) due to spatial intensity fluctuations across the laser beam (see figure 7).

Compared with previous results [21, 22] it appears that the shorter the radiation wavelength, the thinner the color penetration depth and the narrower the range of laser parameters suitable to obtain a linen coloration.

The hue of color depends on the laser  $\lambda$  and on the number of shots. Linens irradiated at  $\lambda = 308 \text{ nm}$  are light-brown, while at  $\lambda = 193 \text{ nm}$  photons induce a yellow coloration (see figures 1 and 2) similar to the color of the Shroud image. In both cases the image contrast increases with the number of laser shots, also allowing a fine and accurate control of the RGB value and of the chromatic coordinates by varying the laser  $I_T$  (see table 1 and figures 1, 2).

The different colors are due to different chemical reaction chains triggered by the 308-nm and by the 193-nm radiation. The 193-nm radiation, thanks to the absorption peak of ketonic carbonyls, induces a photolytic degradation of linen cellulose that promotes the formation of chromophores (see figure 6) having double bonds  $\text{C}=\text{C}$  leading to the yellow coloration of the linen fibers [9, 11, 28, 29].

The local spatial gradient of the radiation intensity may play a role in the color penetration depth, see figure 7.

Following laser irradiation that at first does not generate a clear image, latent images appear after artificial aging of the linen (see figure 4) or one year later by natural aging [21 - 24, 30].

The lack of UV-induced fluorescence observed in the irradiated spot is an additional characteristic of our coloration that resembles the Shroud image, see figure 5a. The UV-induced fluorescence has also shown the capability to selectively recognize the quality of coloration, cf. Figures 5a and 5b.

By using a petrographic microscope we have observed some UV- and VUV-induced defects in the crystalline structure of irradiated linen fibers, showing analogies to those observed in image fibers of the Shroud. This is not discussed in this paper, see [21 - 24] for details.

In summary, our results demonstrate that a short and intense burst of directional VUV radiation can provide a linen coloration having many peculiar features of the Turin Shroud image, including the hue of color, the coloration of the outermost fibers of the linen threads and the lack of fluorescence. However, the total VUV radiation power required to color a linen surface corresponding to a human body, of the order of

# Proceedings of the International Workshop on the Scientific approach to the Acheiropoietos Images, ENEA Frascati, Italy, 4-6 May 2010

$$I_T \times \text{body surface} = 2 \times 10^9 \text{ W/cm}^2 \times 17 \times 10^3 \text{ cm}^2 = 3.4 \times 10^{13} \text{ W}$$

makes impracticable the reproduction of the whole Shroud image by using a single laser, as this power cannot be delivered by any VUV source built to date. Rather, we have shown that a VUV laser is a powerful tool to gain a deeper insight into the physical and chemical processes that generated the body image embedded onto the Shroud, independently of the radiation (or energy) source that possibly generated this image.

The enigma of the origin of the body image of the Turin Shroud is still “a challenge to our intelligence” [32].

## ACKNOWLEDGMENTS

Authors would like to thank the Referee for making suggestions that improved the quality of the paper.

Authors gratefully acknowledge Prof. G. Fanti for his help in the analyses of yarns and fibers, and for a critical reading of the manuscript.

## PHOTO CREDITS

Figures 1 and 5b: Giulio Fanti.

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