Characterization of ablation craters associated to Laser Induced Breakdown Spectroscopy (LIBS) measurements

The purpose of this study is dual: 1) to characterize the ablation craters associated to Laser Induced Breakdown Spectroscopy (LIBS) measurements on a fresco sample (Figure 1); and 2), to compare the craters on a 2-cent euro coin associated to a portable instrument (EasyLIBS) and a laboratory instrument (Figure 2).

Figure 1. *Analyzed fresco sample.*

PACEA is a research laboratory for the French National Centre for Research (CNRS), the University of Bordeaux, and the French Ministry of Culture and Communication. Our research mostly focuses on Paleolithic cultures in Europe and Africa and their environments, biological anthropology, funerary practices, and rock art. The authors of this study are Lena Bassel and Alain Queffelec, with the contribution of Maud Mulliez (Archéovision), Bruno Bousquet and Julian Guezenoc (CELIA laboratory), Rémy Chapoulie (IRAMAT laboratory), and Clément Melkebeke (CELIA and IRAMAT laboratories).

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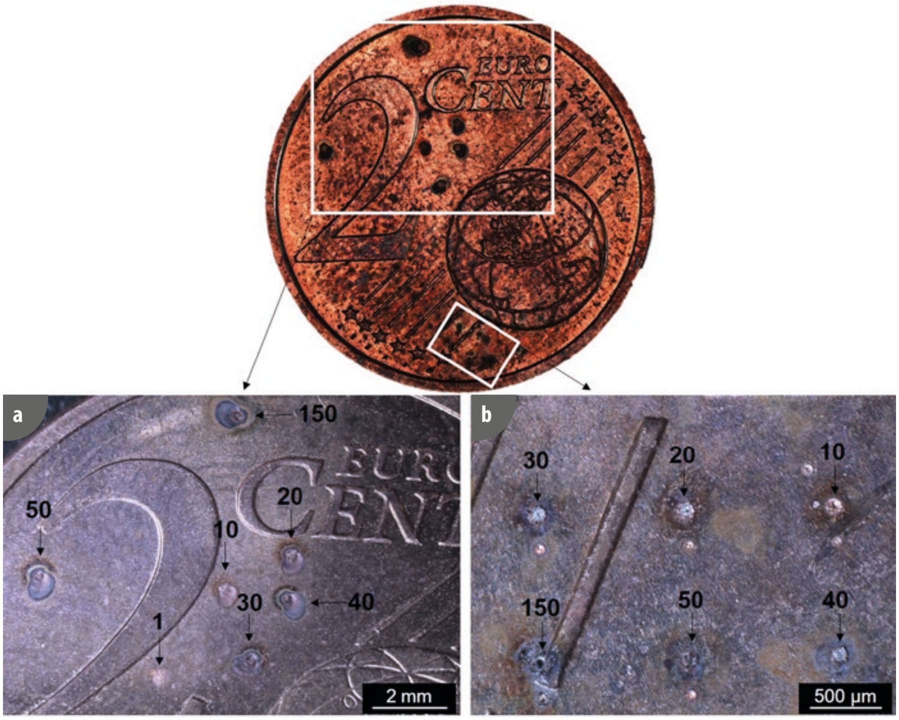


Figure 2. *Overview of the coin* **a)** *Zoom-in on the area analyzed using the portable instrument* **b)** *Zoom-in on the area analyzed using the laboratory instrument.*

Measurements

Part 1: Analyses of ablation craters on a fresco sample

A study on the dimension and depth of the ablation craters associated with the LIBS measurements was conducted. The craters were analyzed using the 3D optical profilometer S neox in Confocal mode. For this purpose, six ablation craters were formed following 1, 3, 8, 10, 15 and 20 laser shots (Figure 3). An initial qualitative observation established that the crater is accompanied by a dark aureole, which demonstrates that the matter was highly heated upon impact. This is termed the heat-affected zone or HAZ, which is well known in laser ablation.

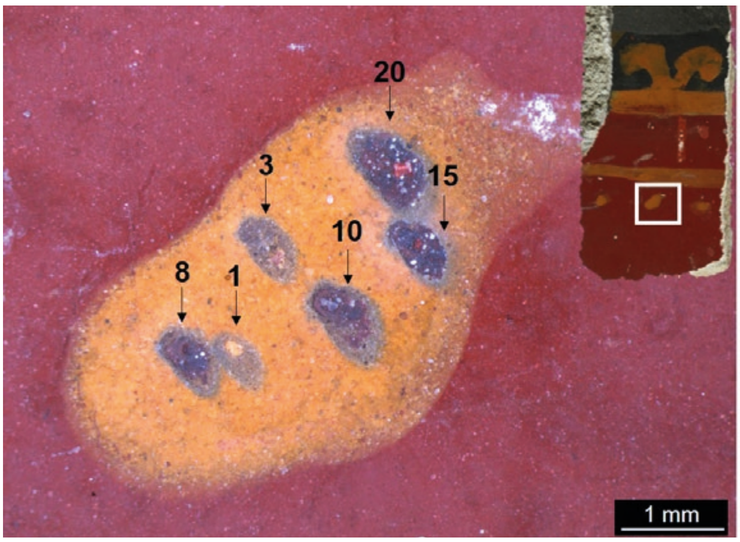
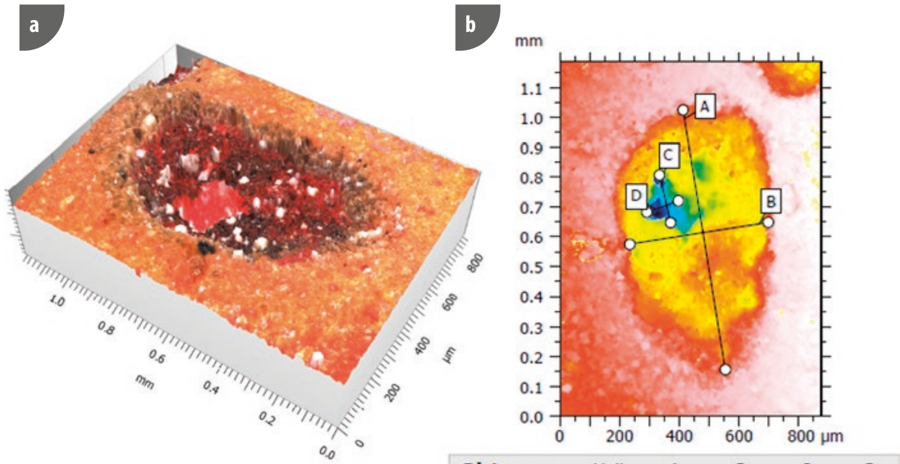


Figure 3. *Location of the points analyzed at the level of the yellow decor overlaying the red pigment.*

The average size of the craters is approximately 800 µm x 400 µm on all of the craters formed. The crater formed by 20 shots was subject to a more in-depth study (Figure 4). Indeed, within the crater, a hollower area is observed, with dimensions of approximately 160 µm x 100 µm. The same observation was made for each of the ablation craters.



Distances	Unit	A	B	C	D
HDist	µm	880	470	164	113

Figure 4. *Crater formed by 20 shots.* **a)** *3D view of the crater formed by 20 shots;* **b)** *Dimensions of the crater in µm and observation of a hollower zone (shown in blue).*
A profile taken along each of the craters demonstrates that the depth of the craters increases with the number of shots (Figure 5). The sample was hollowed out over around 100 µm in the deepest area after 20 shots.

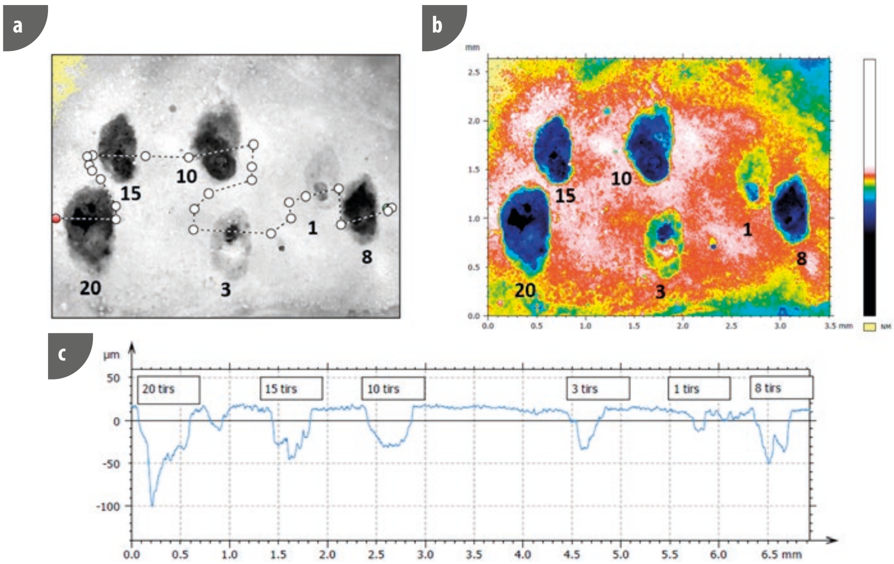


Figure 5. *Depth of the craters in µm.* **a)** *and* **b)** *top-view topographies, c) profile.*

Part 2: Comparison of ablation craters associated to two different LIBS instruments (EasyLIBS and a laboratory Instrument).

In the case of the EasyLIBS intrument, a broad even ring can be observed demarcating the craters formed (Figure 6). Overall, the craters are very flat and shallow: it appears that a molten mass has been formed that has remained at the crater site and has not been ejected.

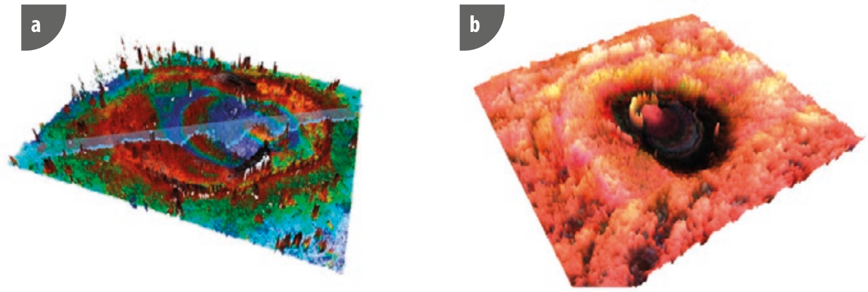


Figure 6. a) *3D view of the crater obtained after 150 shots with the portable instrument;* **b)** *3D view of the crater obtained after 500 shots with the portable instrument.*

For the measurements carried out with the laboratory instrument, matter ejections are visible all around the crater (Figure 7). In addition, the craters formed using the laboratory instrument all have "edges" where the matter accumulates. The height of these edges in relation to the surface of the coin varies between 4 µm and 20 µm depending on the number of shots fired.

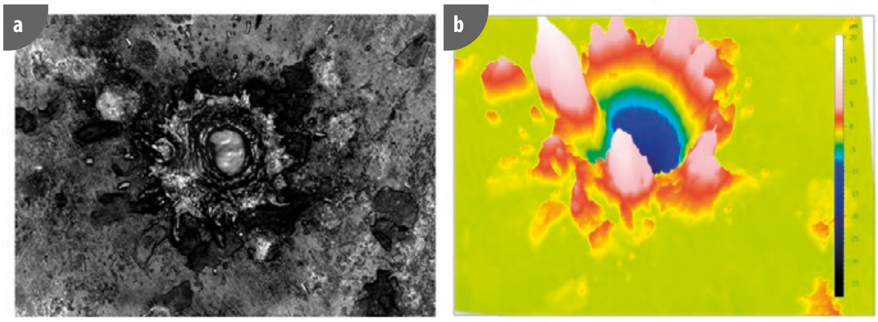


Figure 7: *Crater obtained after 20 shots with the laboratory instrument, the matter ejections are visible* **a)** *Image taken with the confocal microscope S neox;* **b)** *3D view of the crater.*

The comparison of the depth of the craters between the EasyLIBS and the Laboratory Instrument for different amounts of laser shots is shown in Table 1. In addition, Figure 8 and Figure 9 show 2D pictures of the resulting craters for both LIBS instruments after 50 and 500 shots, respectively.

Depth of craters

	EasyLIBS	Laboratory instrument
10 shots	Non-measurable as merged with the roughness of the coin	~21 µm
20 shots	Not measured	~35 µm
30 shots	Not measured	~46 µm
40 shots	Not measured	~55 µm
50 shots	The gradient of the coin is greater than the depth of the crater. ~0,5 µm	~63 µm
150 shots	Not measured	~160 µm but difficult to measure as the basi is not very well lit
500 shots	~8 µm	Non-measurable as too deep

Table 1. *Comparison of the depth of the craters based on the number of shots for the portable instrument (EasyLIBS) and the laboratory instrument.*

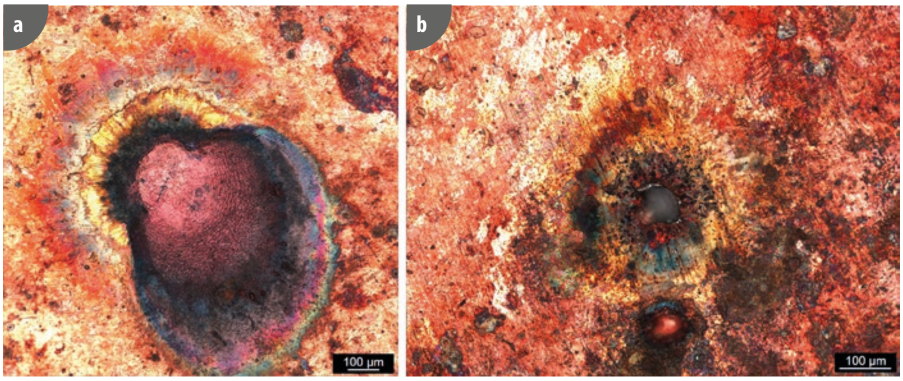


Figure 8. a) *Crater obtained after 50 shots with the portable instrument (EasyLIBS);*
b) *Crater obtained after 50 shots with the laboratory instrument.*

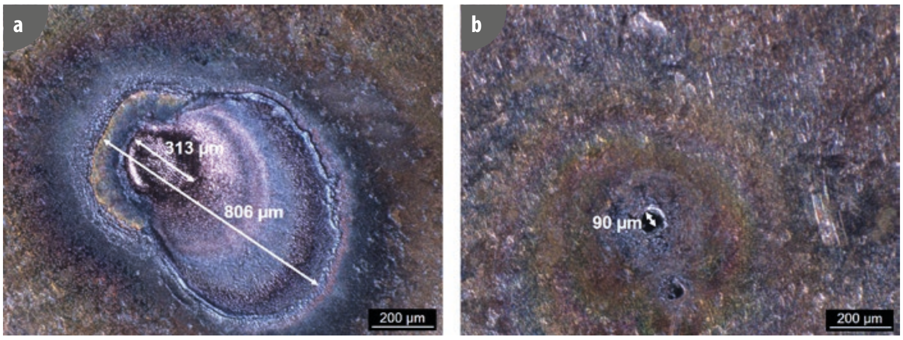


Figure 9. *Shape and size of the craters; a) Crater obtained after 500 shots with the portable instrument (EasyLIBS);* **b)** *Crater obtained after 500 shots with the laboratory instrument.*

Conclusions

The 3D optical profilometer S neox has been shown to be an accurate, fast and easy-to-use tool to investigate ablation craters associated to Laser Induced Breakdown Spectroscopy measurements. The 3D optical profilometry allowed qualitative and quantitative analysis, in particular, Confocal technology has proven to be an effective technique to investigate and characterize the size, diameter and depth of the craters of different LIBS instruments. This is important to archaeologists, curators, since they need to assess the benefits/risk before applying micro-destructive methods on patrimonial artifacts.

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