

Investigation of Gold-bars

by Laser Induced Breakdown Spectroscopy (LIBS)

**Report prepared by: AEMAS analytical laboratory,
Oosterhout
Author: A.A. van der Hoeff
Date: 23-01-2003**

Introduction

3 small gold bars (10, 10 and 20 grams each) were made available for investigation of brown-black small spots on the surface.

The laboratory has tried to identify the elemental content of the colored spots.

The equipment used was a LIBS (laser induced breakdown spectroscopy) spectrometer (SOLAR TII LEA-S500).

Technique used

With LIBS a laserpuls with certain energy and diameter of the laserbeam is aimed at the surface of the sample which can be viewed at the same time by a built-in solid state camera.

The laserpuls evaporates a small amount of sample from the surface that results in short-lived plasma that emits light from the elements present in the sample.

During the investigation laser energy of 12 mJ and a spot of 300 micron was used.

Various spectra were taken from the surface, using single laserpulses at the same spot, both on "clean" surfaces and on "spots".

The results are shown in figures 1, 2 and 3

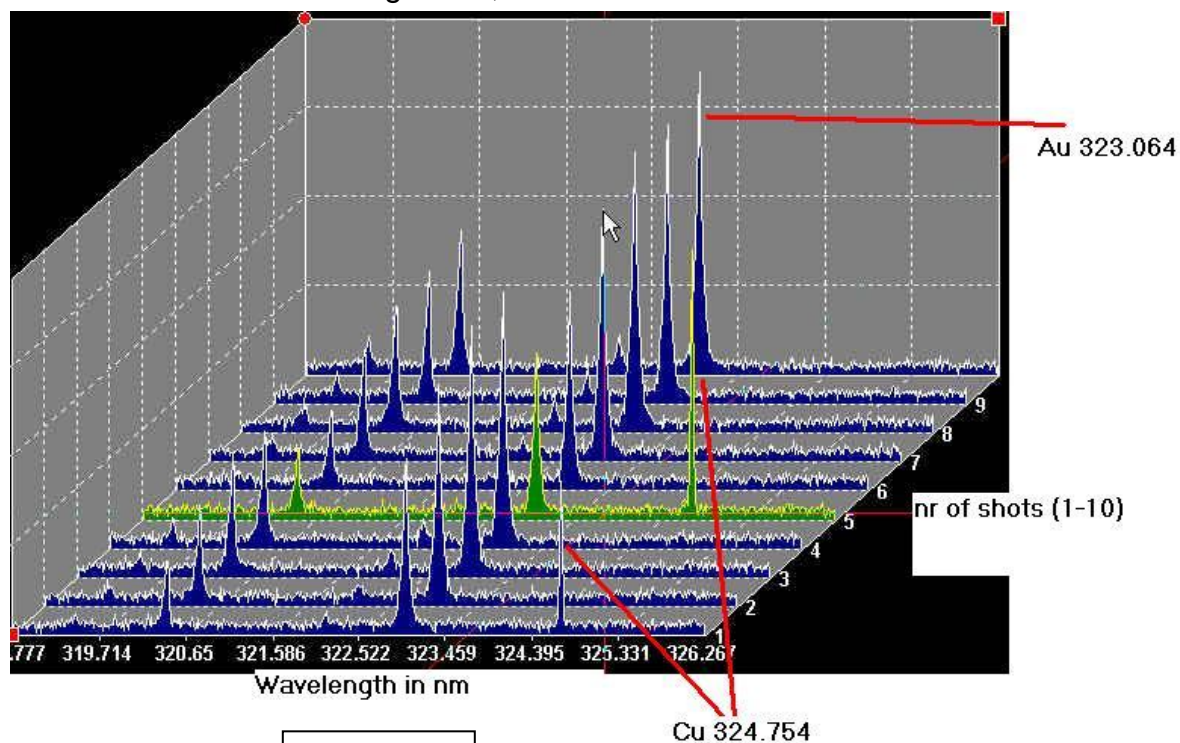


Figure 1

The shots (spectra) 1 through 4 were taken from a clean surface from the same clean spot. (the first spectrum was the first shot)

It can be seen that the peaks for Au are more or less similar, indicating a constant Au-concentration.

The spectra 5 through 10 were taken on a colored spot.

In the 1st and 5th spectrum (the first shot on a clean surface and the first shot on the colored spot) clearly a peak for Cu can be seen.

The height of the Cu-peak in the 5th spectrum is clearly higher compared to the first spectrum on the colored spot, indicating a higher Cu-content at that location.

This is confirmed by the fact that the Au-peak of the 5th spectrum is lower than the following Au-peaks.

In the spectra 2,3,4, 6, 7, 8, 9 and 10 Cu can not be determined anymore, leading to the conclusion that there is a very slight Cu-contamination on the surface (possibly coming from the mould or machines that were used to produce the bars).

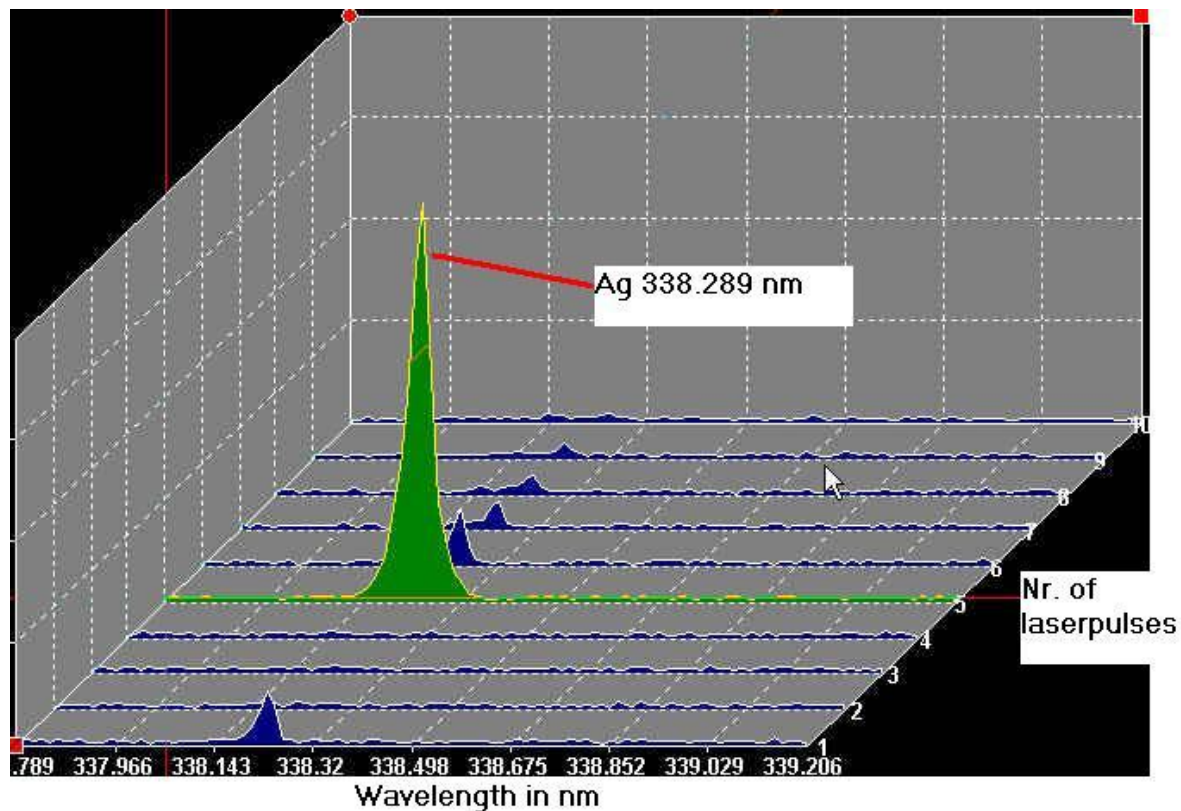


Figure 2

Figure 2 shows the spectra taken in the same way as the spectra shown in figure 1. It can be seen that on the surface a very small Ag-contamination is present (1st spectrum).

From spectrum 5 through 10 (taken on a colored spot) a significant higher Ag-concentration is measured, that requires several additional laserpulses to be taken away.

In the first spectrum a very small Ag-concentration can be seen.

This leads to the conclusion that the colored spot consists mainly out of Ag.

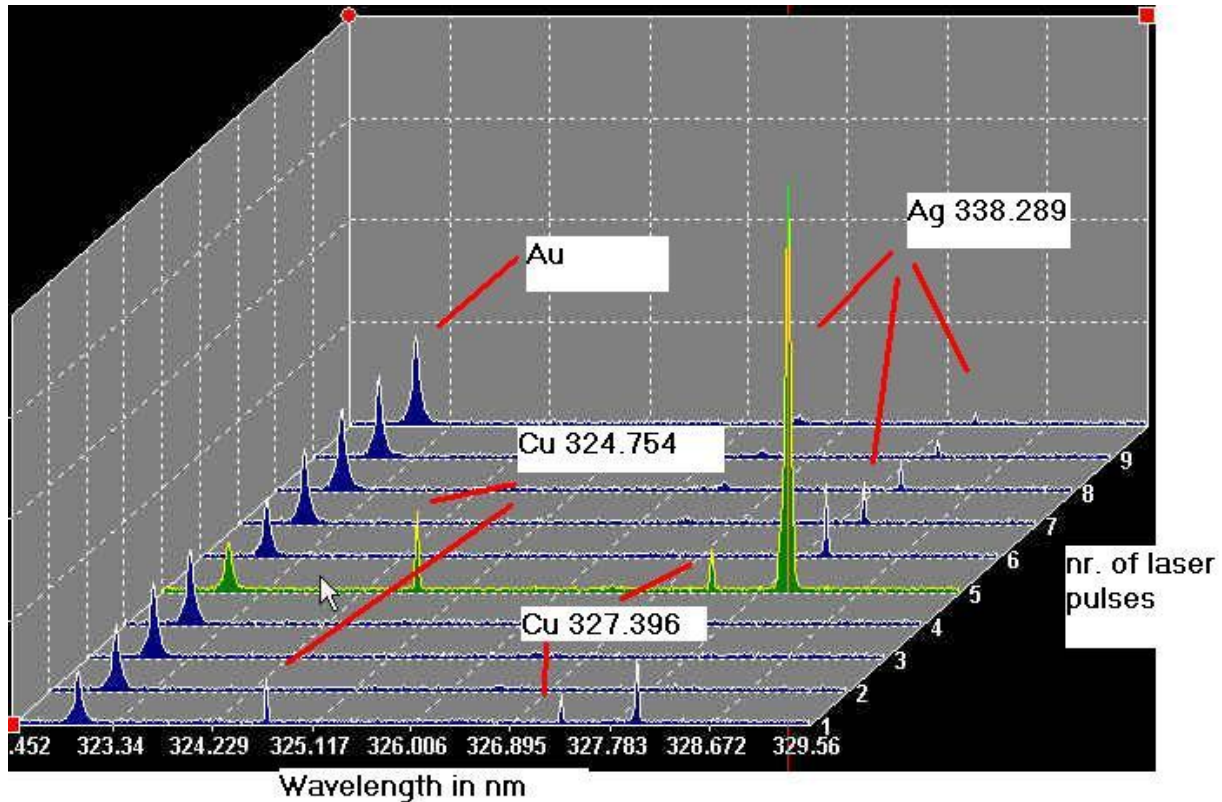


Figure 3

The spectra in figure 3 again were taken in the same way as shown in figure 1.

The Au concentration is constant for the spectra 1-4.

However the Au-concentration in spectra 5, 6 and 7 is increasing and becoming constant from 7-10. This indicates that on the surface the Au concentration is relatively low.

The peaks that can be seen from the Ag 338.289nm line confirm this (spectra 5-10).

Also the small Cu contamination on the surface (both on clean and colored spots) can be noted.

Microscopic inspection

Diameters of the spots vary, but are in the range of 0,3-0,6 mm.

Careful examination of the samples under a microscope shows that the centers of the spots are darker colored than the area around the center.

The centers are more blackish and the outsides are more brownish.

The center seem to consist of a spec of silver that may have been deposited on the surface of the bar.

A resulting silversulfide (AgS) formation will "bloom" away from the deposited silver spec, thus causing the colored spot on the surface of the bars.

Discussion

Other elements were tested (Fe, V, Co, Mn, Al and Ca) but could not be seen in the spectra, with the exception of Ca, that was seen in 1 spectrum: this Ca could not be confirmed in other tests. The measured Ca was most likely due to a surface contamination caused by the manual handling of the samples.

In all the tests that were performed, a very small Cu and Ag contamination on the surface was noted.

Conclusion

The colored spots consist of Ag (silver).

In case the surface of the bars would be totally covered with the blackish-brownish coloration, the estimated concentration of the silver is in the order of 2 - 5 mg/m².

On all the samples a very small surface contamination is present from Cu (copper) and Ag (silver) (the estimated concentration of silver and copper on the overall surface is in the order 0 - 1 mg/m²).

Oosterhout, the Netherlands

30-01-2003