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Evaluation of laser cleaning progress and quality

Ocena postępu i jakości metod czyszczenia laserowego

Keywords: Laser cleaning; Colourimetry; Laser spectroscopy; Artwork diagnostics

Słowa kluczowe: metoda laserowa, kolorometria, spektroskopia laserowa, ocena stanu dzieł sztuki

1. INTRODUCTION

Conventional methods of surface cleaning in conservation of art works are based mainly on mechanical or chemical techniques which are individually selected by experienced conservator. Cleaning of delicate objects, diverse from the point of view of materials composition needs not only extended expert appraisements of used substances, but also minimization of possible damages, always present in the case of mechanical cleaning. These traditional methods are very difficult to control. Chemical reagent shows similar interaction in conservation of paintings, where chemicals penetrate technological painting layers and causes permanent, difficult in analysis cross-sectional alterations. Conservation practice shows necessity of frequent treatments of sophisticated objects with complex technological structure and individual preservation state, resulting from influence of diverse external factors as well as changes in original building material itself. Every detail requires individual, predetermined cleaning parameters, which causes that application of conventional conservation methods is limited and difficult.

Laser technique gives possibility of almost full control of encrustation removal process at the surface of art works. Selective and precise interaction of light beam is fundamental advantage of non-invasive treatment of more or less tight unwanted surface layers. Specific properties of lasers, decreasing of systems costs, and reduction of dimensions of laser cleaning systems caused rising application of lasers in conservation [1-3]. Laser cleaning must be considered as an advanced tool applied in cases where traditional techniques may be inadequate. However, even if the last generation of laser systems has improved the comprehension of their effects and their engineering, laser cleaning is not yet a mature technology for earlier restoration tests, there is also lack of in-depth knowledge of the basic laser-artwork interaction mechanisms. There is still also a lack of diagnostic devices providing qualitative and quantitative information during the laser cleaning intervention.

Application of laser radiation in physico-chemical surface analyses and structural objects investigations, have started simultaneously with development of laser cleaning systems. In the face of increasing interest in laser cleaning and diagnostic systems, important is acquaintance of the conservation community with the fundamental advantages and shortcomings of laser radiation in treatment and analysis of matter.

In the present paper, authors describe and discuss selected areas of applications of lasers and optoelectronics in conservation of monuments and works of art, with particular attention paid to noninvasive, physico-chemical and structural analytical methods. Presented data are based on almost twenty years experience in laser cleaning and diagnostics of dozens priceless objects in Poland and abroad [4-6].

2. ENVIRONMENTAL POLLUTION AND CLEANING

Increasing pollution levels of monuments and sculptures exhibited in the open air inside built-up areas are the result of environmental pollution, composed mainly of soot and dust emitted by industrial objects or rising into the air from the earth surface. Next pollution groups originate from motor exhaust gases and substances generated by modern industry. Their influence on environment, including biological effects, is by far stronger than in the case of particulates. Effective detection, counteraction and removal are respectively much more difficult, in some cases unknown are efficacious restoration procedures.

Fast degradation of human natural environment lasts over 200 years, but main changes were the result of last seventy years of neglected control of industrial development. The percentages of artwork surface associated with corrosion damage (see e.g. Figure 1) reaches yearly 3 to 5% in dependence on monument localization and atmosphere pollution [7]. The complex nature of the formed unwanted superficial layers and limited versatility of any diagnostic

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method requires a combination of several surface analytical techniques for the complete characterization of deposits. This procedure should always be developed prior to the conservation treatment.

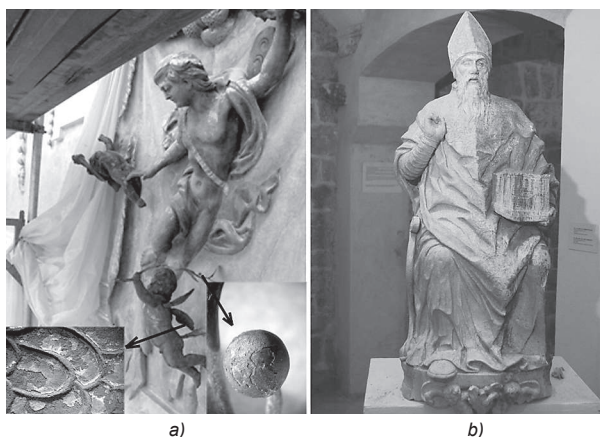


Fig. 1. a) Pair of bronze putti with gilding at the garden's façade of the Wilanów Palace in Warsaw. Black arrows indicate microphotographs of damage details; b) Limestone figure of St. Vlah, patron of Dubrovnik, Croatia. Left side of figure shows the result of laser cleaning

Beside the artistic and aesthetic depletion of the artwork, deposits and encrustation can cause further degradation processes of both a physical and a chemical nature, requiring prompt restoration interventions before the artistic content of the stonework is irretrievably lost [8]. Such an active conservation include surface cleaning which is often one of the first actions to be undertaken. It represents a crucial step in the whole restoration procedure, as the effects of this operation are irreversible and influence the future conservation of the restored artworks. Cleaning itself is also an important part of the artwork stabilizing process and is one of the most important processes in the active conservation of artifacts, preparing possible further treatments if needed: consolidation, coating of a surface or reconstruction of totally damaged elements.

3. PHYSICO-CHEMICAL AND STRUCTURAL DIAGNOSTIC METHODS

The main aim of analysis of monuments and works of art is identification of artwork structure, its important chemical components as well as characterization of its preservation state, including determination of influence of external factors. It is well known, that nature of a given conservation problem prescribes future utilization of specified technology or a combination of several techniques. Therefore, many powerful analytical methods developed as a result of technological progress in optoelectronics are addressed now to solve different and complex problems, arising in art conservation. It applies to different conservation studies, from identification of substrate and top layer materials (pigments, painting media, varnishes), through mapping of structural defects and mechanical discontinuities, up to dating, authenticity studies and careful removal of unwanted layers (encrustation, overpainting, old varnish etc.). Obviously, the most safe for objects are noninvasive techniques *in situ* (if possible to apply).

Main laser and optoelectronic techniques involved in physico-chemical and structural studies of artworks are following:

- Object chemical structure and surface analysis:
 - reflection/scattering;
 - absorption/transmission;
 - laser spectroscopy: induced fluorescence (LIF), induced breakdown (LIBS), Raman;
- Object physical structure and defects localization:
 - multispectral imaging;
 - interferometry, speckle interferometry;
 - holography, holographic interferometry;
 - laser vibrometry;
 - thermography;
 - laser tomography (OCT);
- Object morphology:
 - 3D laser scanning;
 - triangular scanning;
 - surface profilometry (mechanical, laser);
 - laser scatterometry;
 - laser tomography (OCT).

Particularly popular analytical methods have become spectroscopic techniques (laser and non-coherent), mainly due to their sensitivity, flexibility and analytical methodology [9]. Spectroscopy delivers information, which is directly or indirectly connected with chemical nature of investigated materials. Wide application in the diagnostics of historical object found classical Fourier infrared spectrometry (FTIR) or its DRIFT variety with the utilization of diffuse reflection of radiation. Range of FTIR spectroscopy applications include:

- identification of molecular compounds created at the artwork surface,
- studies of composition of painting layers,
- identification of fibers material, chemical composition and soiling of paper and parchment,
- investigations of epoxy resins.

Optical measurement methods (scatterometry, shadowgraphy, microscopy, reflectometry) are commonly supplementing sets of diagnostic methods. Increases interest in application of multispectral imaging for evaluation the results of laser cleaning, identification and mapping of painting materials and visualization of top surface layers.

Diagnostic techniques that utilize X-ray radiation and methods of nuclear physics and chemistry are also supporting conservation of artworks. The most popular is scanning electron microscopy (SEM), frequently with radiation energy dispersion (EDS or EDX). Chemical and crystallographic surface modifications, composition and volume structure of pigments and other materials are studied with the use of X-ray diffraction (XRD) and fluorescence (XRF). Additional basic materials research is sometimes realized using complex systems of mass spectrometry and atomic force microscopy.

4. LASER BASED DIAGNOSTIC METHODS

4.1 Raman spectroscopy

Raman spectroscopy is a light scattering technique, and can be thought of in its simplest form as a process where a photon of light interacts with a sample to produce scattered radiation of different wavelengths (Figure 2a). Molecular Raman spectroscopy is particularly well-suited technique for the determination of the artistic materials in a non-destructive way. By focusing a low power laser on a sample the intensity of the inelastically scattered light is plotted against the Raman

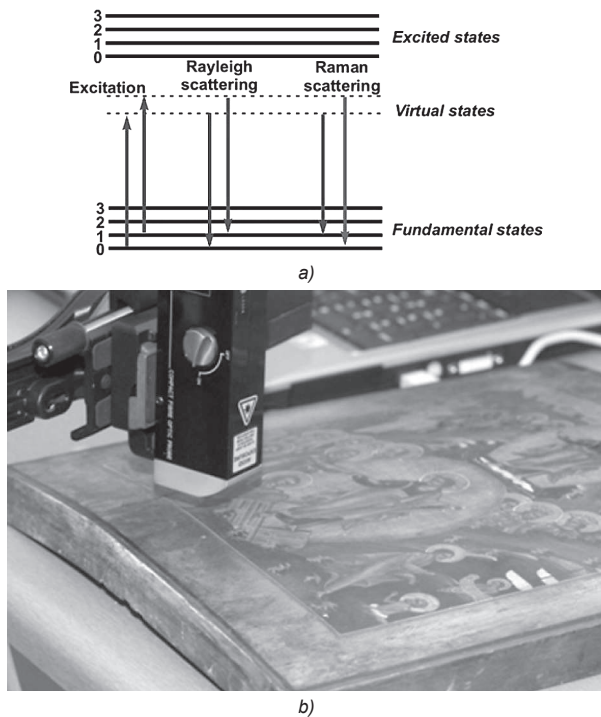


Fig. 2. a) Schematic illustration of energy levels involved in Raman phenomenon; b) R-3000 Raman system head during tests of medieval icon

wavenumber, which is proportional to the difference in energy between the laser and the scattered light.

Identification material tests and preliminary studies of deposits were conducted using two small Raman spectrometers, namely R-2001 from Ocean Optics, Inc., and R-3000 from Raman Systems, Inc. (Figure 2b). Raman spectra of superficial layers before and after laser cleaning were registered using a dispersive Nicolet Almega Raman spectrometer, equipped with two laser illumination sources (532 nm and 780 nm), Olympus BX research-grade confocal microscope (magnification 50 \times , long FD) and high precision motorized x-y stage. Nominal laser power of 25 mW (532 nm) and 35 mW (780 nm) was usually reduced (to around 30%) due to the small diameter of the focused laser spot ($\sim 2 \mu\text{m}$) and sensitivity of the samples. Exposition time was 15-60 s in each of the two averaged scans of the sampled areas. Species identification followed application of the Thermo Scientific OMNIC Spectra software.

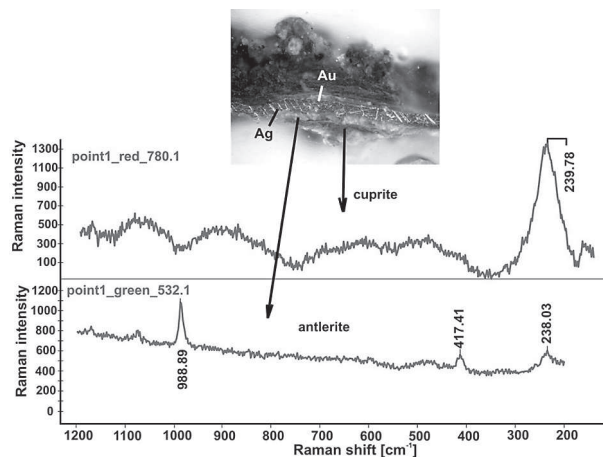


Fig. 3. Raman spectra of corrosion layers near the putto gilding. Small photograph shows the section of gilding. Identified metals and compounds are indicated by lines and arrows

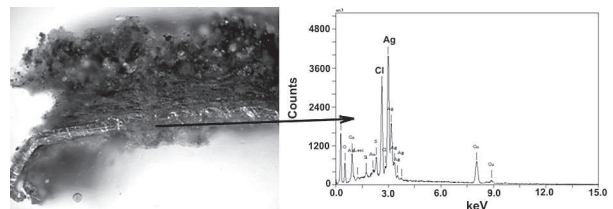


Fig. 4. a) Section of putto gilding damage with developed area of grey corrosion near Ag layer; b) SEM EDS spectrum of corrosion in the point indicated by black arrow and identified as cerargyrite (AgCl)

Application of Raman spectrometry allowed for the identification of several corrosion compounds on metal artworks and specimens. Two examples are shown in Figures 3-5.

Analysis of gilding sections taken from putto with laurel (Figure 1a) is presented in Figures 3 and 4. The pair of gilded bronze putti are dated to the end of the 17th century and attributed by annotations to the Rome studio of the Dutch sculptor Disgenue. Their function as decorative elements of the Wilanów Palace in Warsaw (garden façade) has caused serious soil, local corrosion of the alloy under the gilding as well as numerous mechanical damages. Raman spectra of green salts, registered at section shown on a small photograph in Figure 3 have been identified as antlerite $\text{Cu}_3\text{SO}_4(\text{OH})_4$ (Figure 3: upper plot), and can be found below and above the metallic layers. Cuprite Cu_2O has been identified as the red compound indicated below green layer of antlerite. Grey salts, shown in Figure 4 (left photograph) which were not identified in Raman spectra, are probably silver compounds, as indicated by supplementary SEM EDS analysis (right side spectrum in Figure 4).

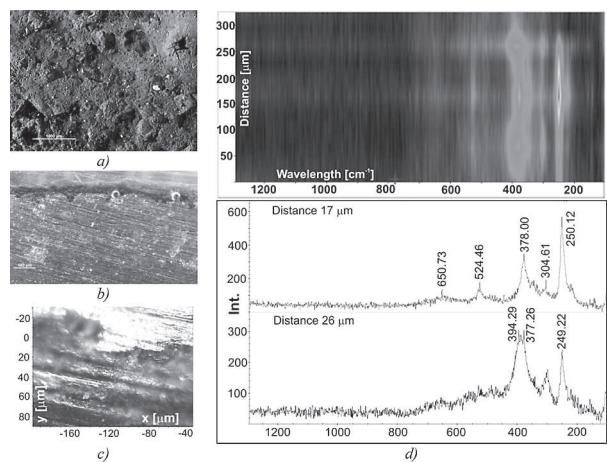


Fig. 5. Raman analysis of corrosion products on carbon steel specimen after annual outdoor exposition in the Railway Museum in Warsaw: a) photograph of surface; b) section of corrosion layers; c) microphotograph from Raman microscope indicating measurement line; d) Raman spectra image (upper photo) and graph below

The second example – analysis of a carbon steel sample after annual outdoor corrosion test in the Railway Museum in Warsaw is presented in Figure 5. Two main compounds were identified: lepidocrocite (red line 378 cm^{-1} and 250 cm^{-1}) and goethite (blue line, 378 cm^{-1} and 298 cm^{-1}).

The laser-deposit interaction in the long laser pulse range resulted in melting, evaporation and sputtering of the surface layer.

Interesting results have been obtained during tests of the influence of laser pulse duration on the quality of post-processing surface of artwork [10]. It is common knowledge that the interaction of long laser pulses (dozens of microseconds) with a solid in a free running regime of Nd:YAG laser

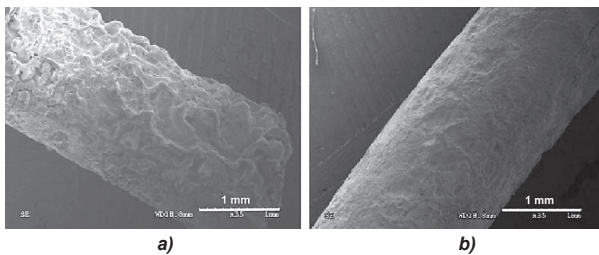


Fig. 6. SEM photographs of fragments of archaeological bow from the collection of the Wilanów Palace Museum in Warsaw after laser cleaning: a) laser pulse duration 150 ms; b) laser pulse duration 120 ns

causes the generation of intense heat on the worked surface. However, the melting and mixing of layers is so efficient that the Raman identification of the resulting surface composition is not possible due to the overlapping of many Raman peaks. An example of such strong influence of laser induced surface heating and melting of archaeological bronze bow is shown in Figure 6a. At the other side, interaction of short, nanosecond pulses in the Q-switched generation regime of Nd:YAG laser resulted in selective, step by step removal of corrosion layers, without damage of the underlying substrate (Figure 6b). The final results of Raman analysis of bow are following:

Corroded surface before cleaning consists of cuprite Cu_2O , antlerite $\text{Cu}_3(\text{SO}_4)(\text{OH})_4$, malachite $\text{Cu}_2\text{CO}_3(\text{OH})_2$ and posnjakite $\text{Cu}_4(\text{SO}_4)(\text{OH})_6\text{H}_2\text{O}$;

Surface after cleaning with long microsecond laser pulses contain a mixture of different copper compounds with metallic copper and tin (from the original bow substrate)

Surface after cleaning with short nanosecond laser pulses shows preserved original substrate and patina, consisting of not touched cuprite layer and thin layer of antlerite.

4.2. Laser interferometry

Laser interferometry is a well-established, highly sensitive technique for non-destructive testing and analysis. It can be useful in the field of historical object conservation in reconstruction of 3D structure of the object under observation and its deformation under stress, layer-to-layer detachments, delaminations and surface cracks [11]. As it is shown in Figures 5-6, sensitive laser interferometry and its variant – Doppler VISAR can be also utilized for “on-line” analyses of shock waves generated during laser cleaning of quite thick objects. As the amplitudes of registered shock wave pulse decrease with the decay of soiling layer, it can serve as an indicator of laser cleaning level. At the other side, substantial signal increase may warn against possible hidden defect in the structure of fragile object and stop the laser. This technique may be useful particularly during the “on-line” diagnostics of cleaning of thin and fragile objects.

In our first experiments, samples of stone materials (granite, marble and sandstone) were specially prepared for interferometric studying of generation of shock waves during interaction of high intensity pulse laser radiation [12]. Recently, a new approach has been introduced, based on optical fiber Doppler velocity interferometer (VISAR) [13], developed by Martin Froeschner and Associates, USA. The example of measurement scheme and overall view of the system are presented in Figure 5.

Optical backscattered signals can be registered from the front or back side of the object under treatment. Acquired information includes surface movement amplitude and velocity data in the time domain scale. Figure 6 shows examples of

oscillograms registered for 4 mm thick marble samples irradiated with Nd:YAG Q-switched laser (1064 nm).

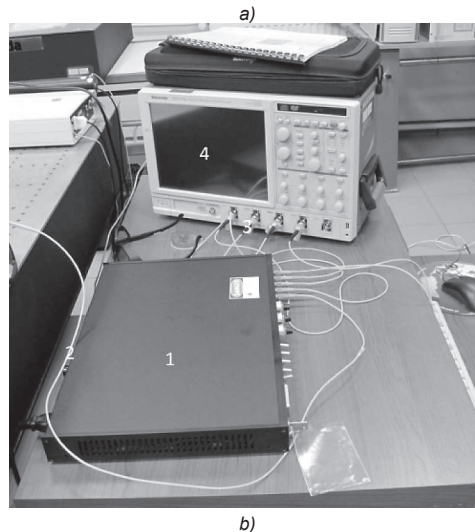
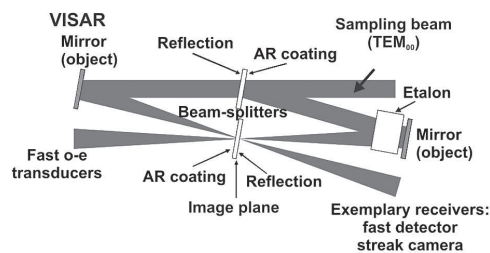


Fig. 7. a) Optical scheme of Doppler velocity interferometer (VISAR); b) Photograph of model FDVI Mark IV-3000 system: 1 – main interferometer body, 2 – signal optical fiber, 3 – digital scope

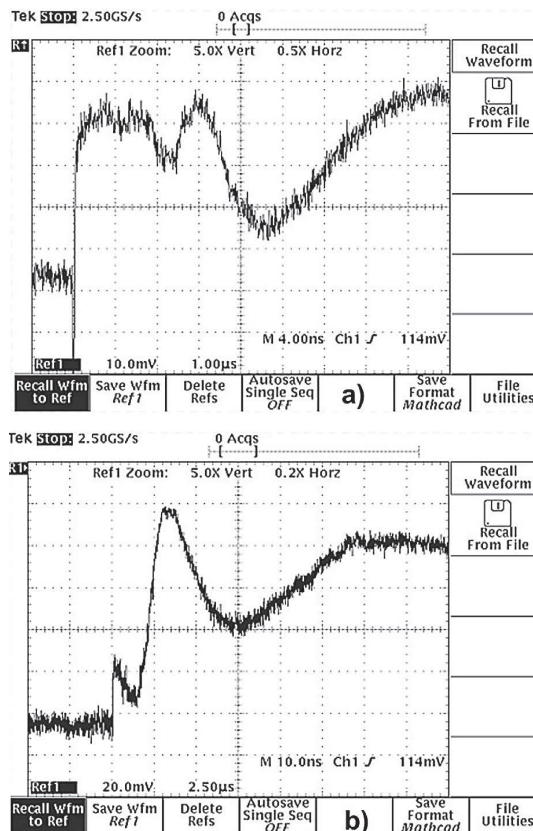


Fig. 8. Oscillograms of shock-wave generated in stone samples by intense pulse laser irradiation: a) fluence 2.5 J/cm²; b) fluence 4.5 J/cm². Note change of vertical scale in b)

5. MEASUREMENT OF LIGHT DIFFUSION REFLECTION COEFFICIENT AND COLOURIMETRY

Physical parameter which allows description of inhomogeneous encrustation optical characteristics is average reflection coefficient of backscattered white light (or laser light). Its modified version – spectrometric measurement of amplitude of diffusely reflected white light in the function of wavelength represents synonymous and objective colourimetry. Apart from easy discrimination between clean and contaminated surface of artwork, the results of spectrometric measurement of reflection coefficient are useful in matching laser radiation wavelength to the highest absorption coefficient of layers to be removed.

Our sophisticated measurement system, developed for precise measurements of spectral reflectivity to determine laser cleaning level and optimise processing wavelength of radiation was described elsewhere [14]. Figure 9b shows the results of measurements of light amplitude scattered from an ivory artwork (photograph in Figure 9a).

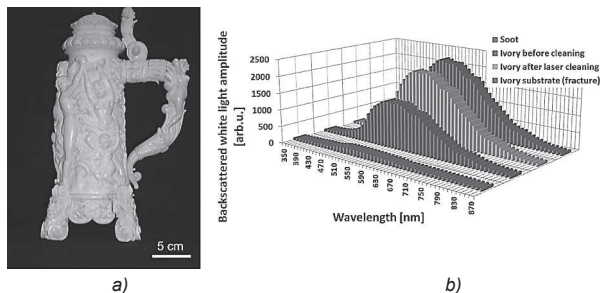


Fig. 9. a) Ivory mug (original view before cleaning); b) Spectral intensity of diffusely reflected white light [14]

Spectral reflectance data can also express colors ranking by means of tone (hue), clarity (brightness) and saturation (chromaticity). Determination of their scale creates possibility of objective digital and convenient color measurement. Different data representation are calculated, from which the colour space $L^*a^*b^*$, determined also as CIELab, is now one of the best known and wide used in almost all domains for object colour measurements. Also the well known empirical test of laser cleaning efficiency relies on comparison of object color in dependence on laser fluence [15]. L^* describes the brightness, a^* the red-green color and b^* the yellow-blue color in CIELab colour space. Additional attribute of colourimetric measurement is documentary notation, which determines reference point and will allow to return to same hue after few dozens of years during next renovation procedures.

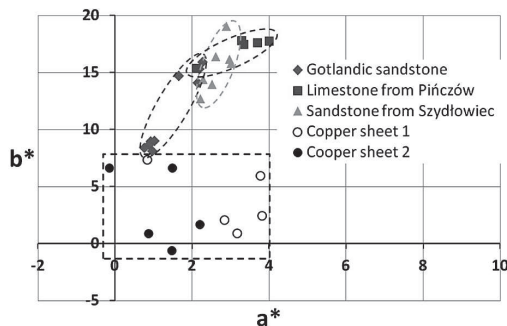


Fig. 10. Set of colourimetric experimental data $b^* = f(a^*)$ for laser cleaning of different materials

Figure 10 contains the image of colorimetric data obtained with the Konica Minolta CM2600d spectrophotometer in the four experiments: laser cleaning tests of Gotlandic sandstone sample, in two places of throne wall of the King's Batory Chapel in Wawel Cathedral, Cracow [15], and for two regimes of laser cleaning of old copper sheets, using 6,2 ns (cooper sheet 1) and 8.4 ns (cooper sheet 2) laser pulses. Charts show two important analytical conclusions. The first indicates arrangement of stone measurement points for stones along a characteristic line (hue angle = $\arctan(b^*/a^*)$, which is similar for sandstones (Gotlandic, from Mysłenice) and quite different for limestone from Pińczów. In case of cleaning of copper sheets, large variations of colourimetric coordinates a^* and b^* (points in rectangular dashed area) are connected with transition of laser cleaning beam between individual layers of deposit and corrosion – from black crust to brochantite (increase of green component a^* and blue in b^*), and from brochantite to cuprite (decrease of a^* and b^*) [16]. It can be easily concluded that colourimetric analysis of laser cleaning process should be limited to the homogeneous superficial layers on artworks and monuments.

6. CONCLUSIONS

Discussion presented in this paper is limited to the selected diagnostic methods. Authors extensively investigated also LIBS [17], acoustic effect of laser-matter interaction [18], and Optical Coherence Tomography (OCT) [19], but analysis of all other methods exceeds the available volume of the present paper. Summarizing, the paper presents our modest attempt to describe and discuss analytical techniques applied to mainly to characterize laser cleaning – comfortable and effective technique for the process of encrustation removal. Raman microscopy is the ideal technique for the investigation of materials used on works of art because it is reliable, sensitive, specific, nondestructive and can be applied *in situ*, therefore avoiding any sampling and consequently any damage to the object under examination. Interferometric systems (VISAR) seem to be present and future advanced technique particularly useful in the basic research in the field of laser – art matter interaction. Colorimetric representation of reflectance data in the CIE- $L^*a^*b^*$ color space is widely utilized to study several conservation problems, including physical-chemical modifications of the surface induced by cleaning (laser irradiation), effectiveness of cleaning procedure, artwork ageing process as well as serves as a documentation of artwork surface digital color representation for future restoration works.

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Abstract

The estimation and analysis of damages (present condition), object conservation (cleaning process), and the protection of an object against further degradation are the main tasks of conservator. One of the physical methods that is becoming more and more popular for unwanted deposit removal is the laser cleaning method. Laser tool is non-contact, selective, local, controlled, self-limiting, gives immediate feedback and preserves even the gentlest of relief – the trace of a paintbrush. Paper presents application of different, selected physical methods to characterize condition of works of art as well as laser cleaning process itself. It includes, tested in our laboratories, optical surface measurements (e.g. colourimetry, interferometry), thermography, and acoustic measurements for “on-line” evaluation of cleaning progress. Results of laser Raman spectrometry analyses will illustrate identification of objects superficial layers.

Streszczenie

Ocena i analiza uszkodzeń (stanu aktualnego) oraz obiektu poddanego konserwacji (np. w procesie czyszczenia) i zabezpieczenie obiektu przed dalszą degradacją stanowią główne zadania konserwatora. Jedną z metod fizycznych, którą staje się coraz powszechniej w usuwaniu niepożądanych nawarstwień, jest metoda z wykorzystaniem promieniowania laserowego (metoda laserowa). Laser, jako narzędzie konserwatorskie jest: bezkontaktowe, selektywne, lokalnie kontrolowane, samo się ograniczające, dające natychmiastowe sprzężenie zwrotne i zachowujące nawet najdelikatniejszy relief – zachowujące ślady pędzla artysty malarza. W artykule przedstawia się zastosowanie różnych, wybranych metod fizycznych, aby scharakteryzować aktualny stan dzieła sztuki, jak również sam proces czyszczenia laserowego. Artykuł zawiera opis przeprowadzonych testów w laboratoriach, pomiary optyczne powierzchni (np. kolorymetryczne, interferometryczne), termowizyjne i akustyczne, w celu oceny procesu czyszczenia laserowego „on line”. Przedstawiono ponadto przykłady analizy warstw wierzchnich z wykorzystaniem spektrometru Ramana.