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ARC2015-1871

The Compatibility of Ion Plating Plasma Assisted Technologies for Preservation Antique Ceramics

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This paper should be cited as follows:

Perrotta, V., Aversa, R., Misiano, C. and Apicella, A. (2016). **"The Compatibility of Ion Plating Plasma Assisted Technologies for Preservation Antique Ceramics",** Athens: ATINER'S Conference Paper Series, No: **ARC2015-1871.**

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Tel: + 30 210 3634210 Fax: + 30 210 3634209 Email: info@atiner.gr URL: www.atiner.gr
URL Conference Papers Series: www.atiner.gr/papers.htm
Printed in Athens, Greece by the Athens Institute for Education and Research.
ISSN: 2241-2891
20/04/2016

The Compatibility of Ion Plating Plasma Assisted Technologies for Preservation Antique Ceramics

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Abstract

The aim of this study is the application of ION PLATING PLASMA ASSISTED technology in the field of Cultural Heritage, applied to a case study, the tiles of the Certosa di San Lorenzo (UNESCO Heritage). Specifically, we want to investigate the possibility to apply not-invasive and reversible coatings for the conservation of historical ceramic tiles with strong chromatic valence, which is an important part of our artistic and cultural identity. We have identified two types of ceramic support for the tests: a fragment of tile of the XIX sec. and a new product, both of which with strong chromatic valence surfaces. Preliminary tests with two plasma treatments were carried out on contemporaneous tile samples in order to find the optimal processing conditions. The first TiO_2 thin film deposition test was carried out by ION PLATING PLASMA ASSISTED source by magnetron sputtering with atmosphere Argon treatment has generated color change on the surface of the coating. A second test was carried out by ION PLATING PLASMA ASSISTED from the heat source with evaporation of SiO_2 (silicon dioxide R.I. 1.46), having as a result a thin-film, transparent, achromatic. The treated samples were verify with the spectrophotometric profile and compared with a slide UNC. The protective nanometer treatment SiO_2 (silicon dioxide) achieved by ION PLATING PLASMA ASSISTED does not modify the chromatism and creates no reflection, strengthening representing a valid innovative method of preservation and conservation of ancient ceramic tiles.

Keywords: Ceramic artefacts, Historical coatings, Ion plating plasma assisted, Vacuum materials

Introduction

Preserving Cultural Heritage is a general concern and the use of non-invasive techniques to protect and conserve ancient materials is an important challenge [4]. The actual problem of the ancient ceramic tiles called "petenate-spetenate" derives from abrasion due to the mechanical tourist passage and also the effect of surface waxing (given the restoration process), therefore compromising the porous structure. The methodology used has imposed a number of considerations which are: to address the choices and the ability to find new solutions, balancing the modern needs, with respect for the important traces of our past, proposing new technologies for the deposition of protective coatings that prevent the degradation of old tiles. Ion Plating Plasma Assisted certainly constitutes an important frontier for the deposition of thin films. Today, thin films are widely used in every field and constitute a strong potential for usability. They can be used under conditions of low temperature and relatively fast in the lay, so they are perfect for industrial applications. In this work, we have addressed the main characteristics of inorganic thin films as TiO_2 and SiO_2 to protect ancient ceramic artefacts. They have used three ceramic supports for testing: a tile of the nineteenth century and two tiles of contemporary manufacturing, all characterized by a strong color effect. This particular type of tile is called "spetenata/petenata" and is characterized by a porous surface in earthenware and with a glazed colored part. Titania and Silica should be achromatic and transparent layers and able to guarantee a high barrier to O_2 and H_2O (g). Thus, three conditions can be satisfied: acromaticity, transparency and protection against atmospheric agents.

Element for a Process under Vacuum Materials and Equipment

The basic elements of a process of vacuum deposition are the source or the sources for the vaporization of the material, the mechanism of the passage from the source of the vaporized material to the substrate in a gaseous environment at low pressure (and eventually through the use of a plasma) and the condensation of the material itself on the underlayer. In some cases the material to be deposited is made to react with a gas introduced into the vacuum chamber, in order to form in a process of deposition of the reactive type, a different film from the vaporized material such as, for example, an oxide or a nitride starting from a source of metal vapor. The reasons that led to the development of the techniques of vacuum deposition may be currently sought in the need to obtain materials with surfaces that have particular physical and chemical properties.

Materials and Equipment

Three ceramic supports have been utilized for the tests: a tile of the XIX century and two contemporaneous production tiles, all characterized by strong chromatic valence and by mixed porous ("cotta") and glazed surfaces. This particular type of tile is named "spetenata" and it is characterized by having a mixed surface with glossy glazed and porous structures.

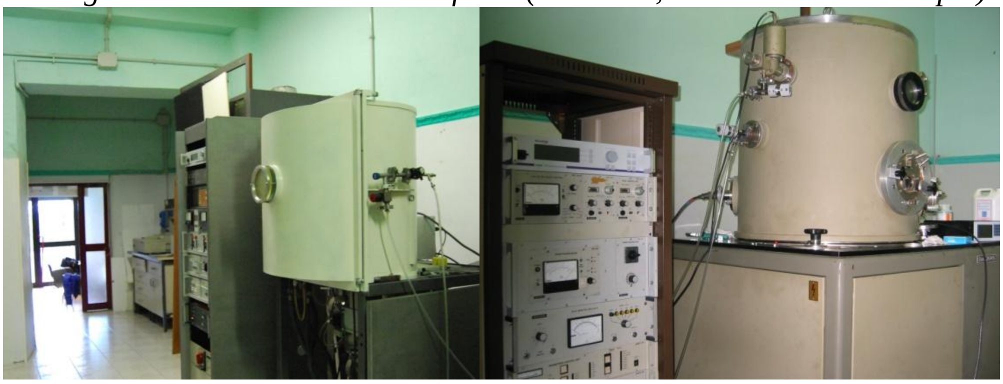
Testing Procedures and Equipment

Four fragments were cut from the same tile and two of them were spray coated with traditional acrylic resins characterized by low and high strengths, respectively. One fragment was not treated while the forth one was plasma coated. The traditional organic resin coated tile fragments were compared with the as received and plasma treated ones.

Ion Plating Plasma Assisted (IPPA)

The two devices used for Ion Plating Plasma Assisted deposition with magnetron sputtering source and thermal source were composed by a high-vacuum chamber containing sources of Ti for sputtering source and SiO for thermal source attached to a magnetron sputter source, powered by a direct current (DC), which is shown in Figure 1. In the chamber, along with the samples, a gas mixture of argon and oxygen was introduced. The percentage of ionized depositing material Titanium or SiO particles emitted by the DC powered magnetron sputtering or thermal sources, are accelerated by a negative bias produced by a radiofrequency electric field (RF) applied to the substrate holder. Such radiofrequency produces plasma in the process atmosphere, which produce a ionization of the depositing materials and produces an ion bombardment of Argon and Oxygen on the growing film [2-3].

Figure 1. *Experimental Set-up for Ion Plating Plasma Assisted IPPA (Also Including the Thermal) Source for Transparent SiO_2 Thin Film Protective Coating on Porous-Glazed Tile Surfaces (Chamber, Source and Tile Sample)*

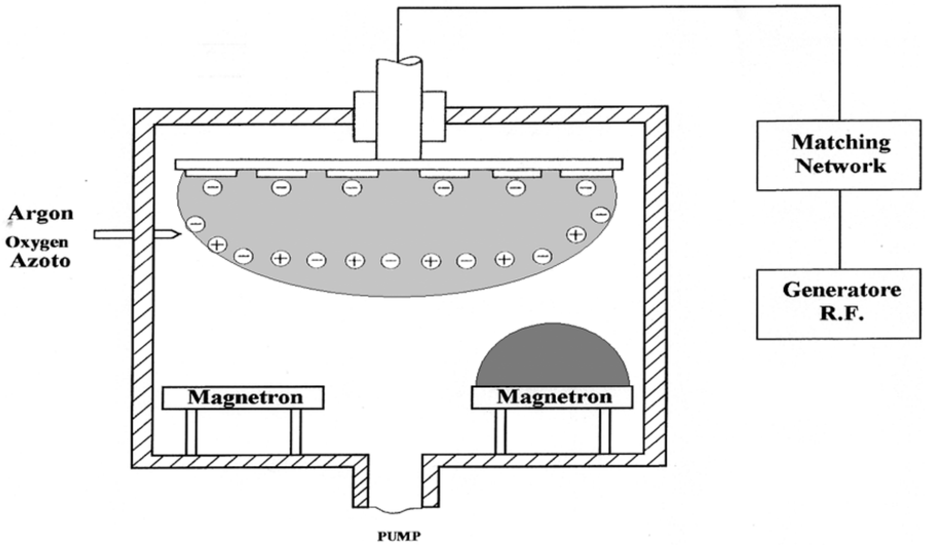


The Schematics of the IPPA Process are presented at Figure 2 and the most important characteristics of the IPPA are the following:

- The energy of the particles emitted by the Magnetron Sputtering source is in the range of a few eV but a certain percentage of these (valuable around 10%) is emitted in ionised form;
- The emitted particles crossing the second plasma, generated by the bias

in front of the substrates, are partially ionized, too;
- The ionized particles arriving on the substrates are accelerated by the bias up to an energy of eVbias;
- The growing film is continuously bombarded by the ions of the plasma (Argon + added reactive gas).

Figure 2. *Schematics of the IPPA Process [1]*



Equipment

The samples were produced and partially characterized (adhesion tape test, abrasion) in the laboratory of Romana Film Sottili in Anzio (RM, Italy). The deposition processes were carried out on Balzers BA 710 plant modified to a configuration IPPA (IPPARCUS shown in Figure 3).

Figure 3. *System BALZER BA 710 (IPPARCUS)*

The plant shown in the figure has a cylindrical deposition chamber of 710mm diameter. The pumping system consists of a mechanical rotary pump with a dual-stage of 100 m3/h and by a diffusion pump from 5,000 lit/sec. Between the diffusion pump and the vacuum chamber a trap is inserted for vapors of "Chevron baffle" type which are water-cooled to minimize the back diffusion of oil vapor. The deposition source is a MAGNETRON SPUTTERING DC with a target size of 5 "x10". The DC generator is a Balzers maximum 5 KW. For door-underlayers a "Cathode Sputtering" configuration type has been chosen for a polarizable radiofrequency screen mass frame. Its structure allows it to look out on the existing Magnetron Sputtering source of 5"x10" dimensions. The radiofrequency was generated by a generator of RF (13.56 MHz) to which the corresponding automatic matching network is coupled. For the creation of the plasma, an RF generator of 1000W was chosen from the Dressler company (shown in Figure 4), with a built-in meter of the direct power, the reflected power and especially by the self-polarization voltage.

Figure 4. *Generator of RF*

To measure the thickness and the deposition speed, a microbalance quartz was used oscillating at high sensitivity, capable of measuring thickness and with a deposition rate which has an accuracy of 0.1 A ° / sec (shown in Figure 5).

Figure 5. *Measuring the Thickness of a Quartz Oscillation*

Results

The first test was carried out by ION PLATING PLASMA ASSISTED (Figure 6), source by magnetron sputtering with atmosphere. Argon P.R.I. $7.0X10^{-5}$ mbar, PRAr $3.0X10^{-3}$ mbar, PRO_2 closed PRN_2 closed; The treatment has generated discoloration on the surface of the coating. Result: thin protective film, transparent, not-achromatic.

Figure 6. *TiO2 Thin Film Coating by Magnetron Sputtering IPPA on Porous-Glazed Tile Fragment. Achromatic Transparent Protective thin film*

Deposition SiO_2 (silicon dioxide RI 1.46) by ION PLATING THERMAL PLASMA ASSISTED from source, (Figure 7, Table 1), (reactive bank), with evaporation of SiO_2. Result: thin protective film, transparent, achromatic, abrasion resistant.

Figure 7. *SiO_2 Thin Film Coating by Magnetron Sputtering IPPA on Porous-Glazed tile Fragment. Achromatic Transparent Protective Thin Film*

Profile Spectrophotometric
Specimen: SiO_2

Table 1. *Spectrophotometric Profile Compared with an Uncoated, Glassslide. The SiO_2 Treatment on Glass Substrate is Antireflective. It can be Expected that the Coating does not Alter he Chromatic Effect of the Sample Treated Tiles*

L onda nm	UNC	SiO_2	% SiO_2/UNC
350	12.2	12.6	103.27
400	35.9	36.1	100.27
450	32.9	33.4	101.56
500	53.3	53.9	101.12
550	98.2	98.6	100.41
600	123.3	123.3	100
650	120	119.7	99.75
700	116.6	116.4	99.82
750	113.4	113.4	100
800	109.6	110	100.36
850	108.2	108.9	100.64
900	109.4	110.2	100.73
950	45.3	45.8	101.10
999	21.8	22.1	101.38

Figure 8. *Low Strength Acrylic Based Resin Spay Coating (Left) and Untreated Tile Surface (Right)*

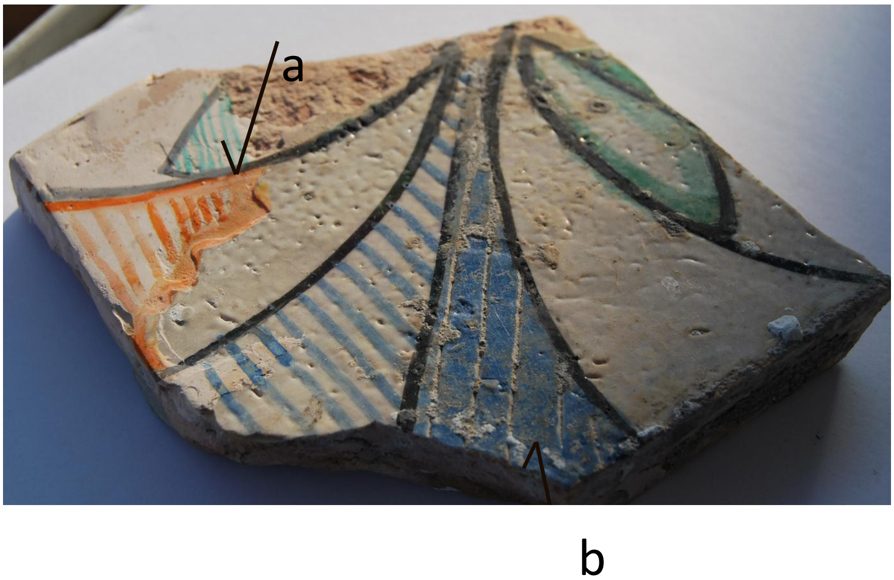
1. Deposition SiO_2 (Figure 8), (silicon dioxide) by ION PLATING PLASMA ASSISTED
2. Deposition resin spray acrylic-based high-strength

Result 1: thin protective film, transparent, achromatic

Result 2: Surface significant alteration condition of things

IPPA Coating on Ancient Historical Tile (Figure 9)

Figure 9. *Effects of the New IPPA Coating on a XIX Century Historical Tile Before (Upper Part of the Image) and After the Thin Film Deposition (Lower Part of the Image): Colours Have not Been Altered in the Repaired Portion (a) and on the Original Glazed Surfaces (b). The Porous "Cotto" Surface Aspect Has Not Been Modified (c).*



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Conclusions

As cited above, the assessments results of the characteristics of treatments developed with the use of the technique IPPA (also including the thermal), show that this deposition technique has produced excellent results for the protection of porous glass surfaces of ancient tiles.

The best result is obtained by TiO_2 thin film coating by magnetron puttering IPPA

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