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Autor: Voûte, Alexander

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The Plasma Equipment at the Swiss National Museum – Observations and Improvements

by ALEXANDER VOÛTE

Abstract

The Plasma equipment at the Swiss National Museum is described. In the beginning the plasma-machine had to be used also to dry the fresh samples. Specially the vacuum-pump was highly stressed. A separate vacuum-drying oven allowed a much better use of the equipment, with no need for supervising the drying process. The safety circuits were also improved. As it was very difficult to assess the contribution to the success of the treatment of the single parameters, several additional measuring devices were installed. The measured values are monitored to be able to determine their influence on the treatment or on the other measurements. Specially the measurement of temperature within the extremely strong highfrequency field inside the treating vessel found a satisfactory solution.

Zusammenfassung

Das Gerät für die Plasma-Konservierung am Schweizerischen Landesmuseum wird beschrieben. Wie sich anfangs bald herausstellte, wurde die Anlage, insbesondere die Vakuumpumpe, durch den notwendigen Trocknungsvorgang der Proben stark beansprucht. Dies konnte durch ein getrenntes Vakuumtrocknungsgerät vermieden werden. Gleichzeitig musste der Trocknungsvorgang nicht mehr ständig überwacht werden. Die Sicherheitsvorrichtungen wurden ebenfalls deutlich verbessert. Die erzielbaren Resultate sind stark von den Behandlungsparametern abhängig. Um die einzelnen Einflüsse besser abschätzen zu können, wurden einige zusätzliche Messinstrumente montiert. Der Verlauf von sämtlichen Messwerten wird registriert, um die Wirkung der Parameter auf die Proben und die gegenseitige Beeinflussung zu erfassen. Insbesondere die Temperaturmessung in dem extrem starken Hochfrequenzfeld im Behandlungsgefäss konnte befriedigend gelöst werden.

Plasma equipment (Fig. 1)

After extensive trials with plasma reduction at the University of Zürich by Stan Vepřek with the assistance of Jörg Elmer from the Swiss National Museum, a decision was made to order plasma equipment for the museum made by Vacotec in La Chaux de Fonds[1–4]. This equipment was delivered in 1990.

The two bottles in the Figure on the left symbolise the gas bottles of hydrogen, argon nitrogen and methane. Not shown is a safety valve in the lead from the hydrogen bottle. The gases enter the plasma vessel after passing flowmeters and regulating valves. A Baratron gauge measures

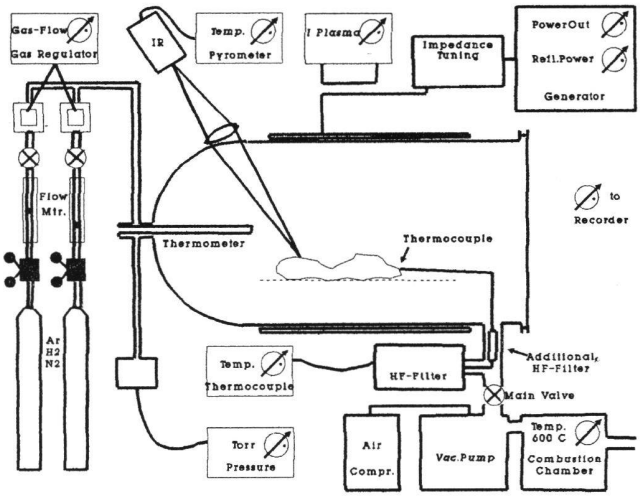

Fig. 1 Plasma Layout.

the gas pressure at the same inlet, and a thermometer, to read the temperature in the vessel, is introduced into a glass tube. This temperature is referred to as Gas Temperature. The diameter of the glass plasma vessel is 400 mm and it has a useable length of 85 cm between the electrodes. The vessel may contain up to three glass supports with objects. The generator can deliver 4 kW (Kilowatt) power at 27 MHz (Megahertz) and it is connected to the electrodes through an adapter for impedance tuning. Two instruments read output- and reflected power. After passing through the vessel, the used gas reaches the pumping outlet which is situated just behind the door.

From the vacuum pump, after compressed air is added, the gas-mixture enters the combustion chamber where all dangerous components will be burnt. Since this equipment was first delivered to us we have gained a lot of experience in its use and in improvements for conservation treatments.

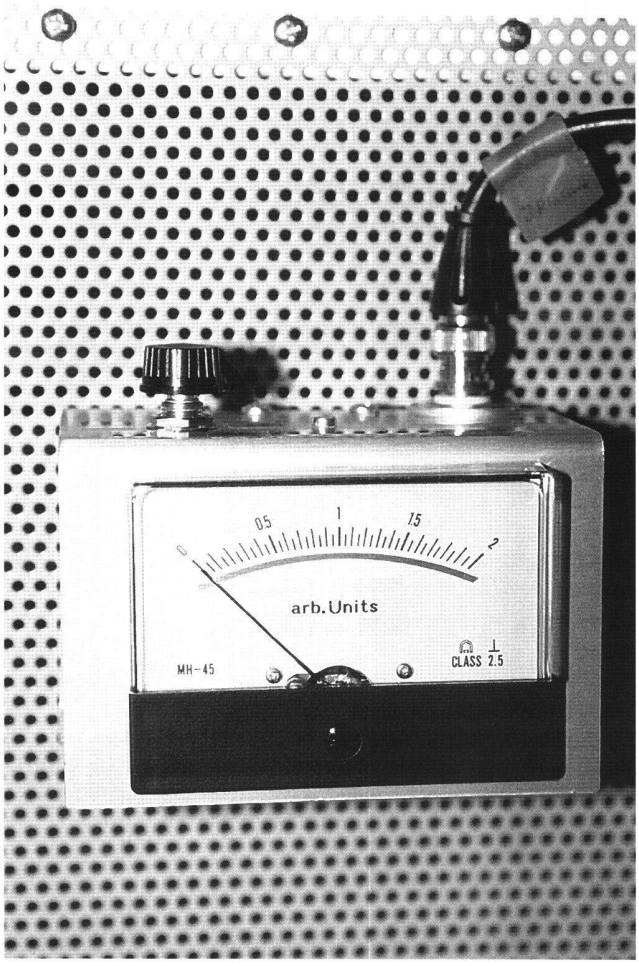

Fig. 2 Plasma current instrument.

At first all objects were dried in the plasma vessel itself. But eventually a vacuum drying oven was installed instead for this purpose so that the plasma apparatus could be used solely for the active treatment. This oven that was installed has similar dimensions to the plasma vessel, so it allows for the same glass supports to be used, enabling an easy transfer of the newly dried objects from the oven to the plasma vessel. Since 1995, safety measures have also been improved. It was in that year that the plasma apparatus was moved to its present location.

The safety measures with which it was equipped did not correspond to the most recent standards, and we were forced to make several changes on the Faraday cage. This, in turn, affected the machine's overall performance. Though we were not very pleased with these consequences, we were lead to a better understanding of the plasma apparatus from a technical point of view. As operational safety measures were addressed, several changes to the circuits were made. Oven temperature can now be checked; it must be high enough to enable the safety valve for hydrogen to work. The generator has been connected to a safety circuit. Opening the door of the Faraday cage, for example, will immediately switch the generator to standby so that health hazards may be avoided. Until recently, working with the plasma equipment for conservation treatment has been carried out more or less on a basis of trial and error. Although the results achieved with plasma as a treatment for archeological metalwork are in many cases remarkably good, we know that a lot more must be understood about the process[5–8].

High quality work depends upon a precise knowledge of all the mechanisms involved and how they affect the objects being treated. An intricate system of monitoring is a good way to obtain the information we need. The following values are of interest: gas composition, gas flow, gas pressure, output power of the generator, reflected power, plasma current, gas temperature, temperature inside the object being treated, surface temperature of the object. For monitoring treatment parameters, electrical values must be given. Suitable instruments for reading the pressure, output power and reflected power were in use from the beginning. A thermometer without electrical output measured the gas temperature. All other measuring devices were not yet present.To monitor gas flow and composition we were able to locate a suitable instrument made by Brooks which regulates the gasflow of up to four gases to a high degree of accuracy. Gasflows may also be adjusted in fixed relations to one another. Together with the given capacity of the vacuum pump, very stable values for gas composition and pressure in the treating vessel can be obtained. Exact impedance matching and tuning to the frequency produced by the generator is essential for obtaining a stable plasma. Such tuning is dependent upon the size and number of objects being treated in the vessel, as well as on the gas properties being used. So far, tuning has been done by adjusting the "Module" and "Phase" knobs on the adapter to minimum reflected energy. After altering the Faraday cage we have noticed that the "Phase" knob had only a minor influence and that the cooling of the generator was not anymore sufficient to remove surplus heat under all conditions. As a result, the machine has sometimes shut off in mid-process. The indication here was that there was resonance in another part of the high frequency circuit, rather than in the electrode circuit.

Plasma current instrument (Fig. 2)

In trying to find a better way to adjust these settings we constructed a small instrument. See also Fig. 1. It consists of a short wire loop near the lead from the adapter to the upper electrode where it picks up a signal that is proportional to the current flow in the gas discharge. Adjusting the tuning to maximum plasma discharge current resulted in a moderate amount of reflected energy and in a significant decrease of dissipated heat in the generator. Because of this new plasma current indicator, both "Module" and "Phase" adjustments now have more of an influence on the fine tuning of the adapter, and adjusting has become much easier. A similar plasma current instrument on the new machine in Roztoky near Prag has been of great help in figuring correct adjustments within a short time. The reflected energy is strongly dependent upon gas composition. A discharge in argon, for example, results in a much higher amount of reflected energy than as with hydrogen. Measuring temperatures in a strong high frequency field causes several problems. A mercury thermometer introduced within a glass tube that protrudes into the plasma vessel indicates the temperature. Temperature is measured by pulling this thermometer out and making a fast reading before it starts dropping. Replacing this thermometer with an alcohol thermometer that has a red column has allowed readings through the vessel glass, but it reads only temperatures lower than 200 centigrade. Monitoring the temperature with these two types of thermometers is, in the end, not feasible. An attempt to replace them with a commercial thermocouple instrument nearly lead to a disaster that could have resulted in the sudden destruction of the instrument.

Even more problems have arisen with attempts to measure the temperature of treated objects directly. We have been measuring the surface temperature of these objects with a pyrometer which calculates the temperature from the amount of infrared radiation emitted by the surface of an object. We use a Vanzetti thermal monitor. Pyrometers have good precision for high temperatures but for the region that interests us most – clearly below 300°C – performance is only moderate. These instruments cannot be calibrated below 120 centigrade. Another difficulty with pyrometers is that the reading is dependent upon the emissivity which is given by the structure, material and color of the measured surface. During plasma treatment this surface is likely to alter, and calibration will be accordingly uncertain. A simple experiment has been made to ascertain that the plasma discharge itself does not produce any infrared radiation which would disturb measurements with the pyrometer. We observed that no sudden change of the measured temperature so far occurred when the plasma discharge is switched off. Despite the mentioned problems we have decided to continue to use the instrument. In order to be able to make measurements on small objects, an additional lens is used to focus on a spot about 6 to 8 mm in diameter. For consistent measurements it is necessary to use thermocouples provided with suitable high frequency filters.

High frequency filter (Fig. 3)

Wolf-Dieter Schmidt-Ott from the University of Göttingen was so kind as to build such a filter for us, with the thermocouple entering the vessel through the pumping connection. At low power it functioned well. At a higher energy however a strong discharge was pulled along the wire down the pumping tube and the reading on the instrument became erratic. Additional filtering with two wirewound resistors near the exit of the vessel stopped this effect, although a weak discharge along the thermocouple wire is still a possibility under unfavourable circumstances. We are sure that it is only a question of improvement in the positioning or the splitting up of this additional filter to be completely successful. The thermometer to measure the gas temperature will also be replaced by such a filtered thermocouple instrument. Filters, small enough to fit into the glass tube, have to be constructed.

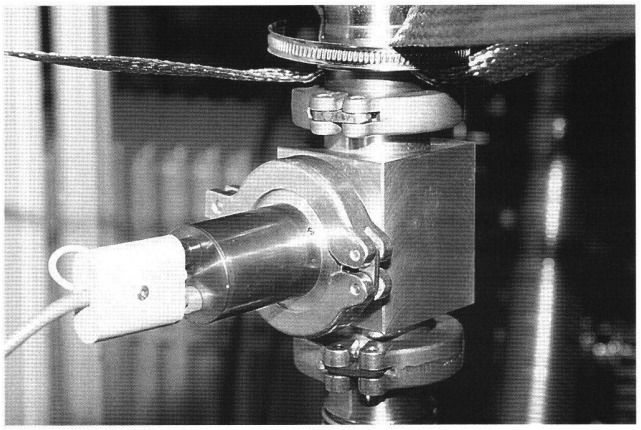

Fig. 3 Arrangement of the high frequency filter.

First results with the new measuring possibilities showed that the temperature inside an object rises rather slow. It takes almost one hour to reach its final temperature. The gas thermometer goes up faster in the beginning and then slows down. The gas itself reaches its final temperature very quickly, whereas the gas thermometer and the object have significant thermal lags caused by slow heat transfer from the gas. It seems that the gas temperature is mainly dependent on gas pressure and the influence of output power is only marginal. At a given primary gas composition, the reflected energy can be used as a not quantitative indicator for the amount of gas or vapour released from the objects undergoing treatment.

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