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Heat-Treatment of 16MnCr Steel in a Linear Non-Isotherm Plasma Reactor

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Abstract: Over the last few years a Direct Current Plasma Nitriding (DCPN) equipment has been built at the Sapientia-University. This equipment was built on the basis of an older linear plasma reactor. Over the years the vacuum-system and vacuum-measurement, gas feeding system was upgraded and a brand new modern voltage supply was developed to generate and sustain the plasma discharge. Using the upgraded DCPN equipment we treated 16MnCr steel pieces at 530 degrees Celsius, for one, two, three, four and 6 hours long, respectively. We present optical- and electron microscopy investigation and the Vickers-hardness study of the prepared treated pieces. The results of the DCPN experiments reported in this paper serve as comparison basis for the following ASPN (Active Screen Plasma Nitriding) experiments, which will be performed at the same sample temperature and time-span conditions.

Keywords: plasma, nitriding, heat treatment, coating.

1. Introduction

Plasma nitriding is one of the most efficient, environment and health-friendly coating and hardening techniques.

Plasma nitriding is widely used in case of industrial applications and it is also object of many developments and plasma physics studies [1-6]. There are two different plasma nitriding methods, the DCPN (Direct Current Plasma Nitriding) and the ASPN (Active Screen Plasma Nitriding) method. In case of the DCPN method the nitrided pieces represent the cathode of the plasma discharge, thus the pieces are heated directly by the discharge. In case of the ASPN method there is a separate metallic screen which plays the role of the cathode, the screen is heated by the discharge while the nitrided pieces are

heated by heat transfer. Starting from the basis of a linear plasma reactor, we developed a modern DCPN equipment which is suitable for heat treatment of different steels in nitrogen-hydrogen plasma. Steels are treated at 530 degrees Celsius, for about 1-6 hours, at working pressure 2 torr, depending on the industrial requirements. The equipment is also suitable for dedicated local plasma diagnostics research, involving different types of electrostatic (Langmuir) probes such as cylindrical-, double- and emitting probes. In the present paper the construction of the equipment is presented, along with detailed description of the constructing modules, first nitriding experiments involving *16MnCr* steel pieces, optical- and electron microscopy investigations and Vickers hardness study of the adequately prepared samples.

2. General presentation of the Direct Current Plasma Nitriding (DCPN) equipment

The present DCPN equipment is based on an older DCPN machine called NITRION-10 [7]. We discarded the vacuum-system, electrical-system and gas feeding lines, and retained the discharge chamber which is the anode of the discharge (grounded) and the cathode which holds the pieces to be treated too (the cathode in *Fig.1* is a solid metallic cylinder mounted coaxially with the chamber axis).

The experimental equipment is presented in *Fig.1*. The upper flange and the body of the vacuum-chamber is water cooled. The upper flange is provided with gas-inlet hole, electrical feed-through and the cathode of the plasma discharge is also mounted in the top flange. There is a motion feed-through on the side of the chamber which is used to introduce a Langmuir-probe for plasma diagnostics research. Optionally, a heat shielding metallic cylinder can be coaxially positioned inside the plasma chamber.

We adopted a reasonable vacuum line involving an Alcatel 2105 type fore-vacuum pump and an absolute pressure gauge for pressure measurement. We can reach pressure in the range of 1-0,02 torr, which is by far sufficiently low for plasma nitriding. The working pressure for plasma nitriding is about 3,5 torr which is obtained mixing and introducing gaseous nitrogen and hydrogen.

It is very important to ensure proper and safe gas feeding system for the heat treatment. For this purpose hydrogen and nitrogen is needed, which are provided by a combined nitrogen-hydrogen generator. We provided the nitrogen and the hydrogen outlets of the generator with precision valves to set the proper gas flows. After the valves, the hoses are introduced into a mixing chamber which is connected to the plasma chamber.

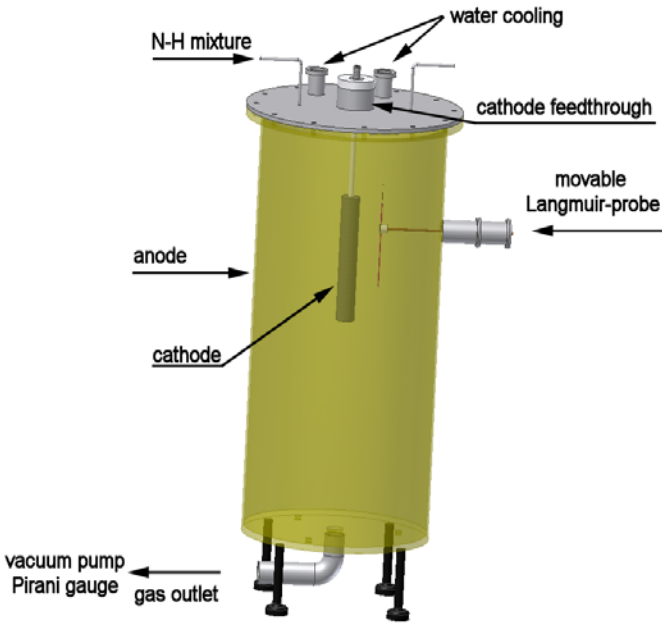


Figure 1: DCPN equipment.

Due to the low pressure obtained by pumping the plasma and mixing chambers, nitrogen and hydrogen can be mixed as ideal gases in the mixing chamber, and can be introduced as perfect mixture suitable for plasma nitriding.

3. Development of a Regulated Direct Current plasma discharge Power Supply (RDC-PS)

A new DC power supply was developed in order to obtain accurate output voltage and current, with 1% precision compared to the setvalues. The new digitally controlled power supply has several benefits, namely programmable arc management, fast protections, and communication interface with higher level control system. The main characteristics of the power supply are described in *Table 1*.

Table 1: Specifications of the DC power supply.

AC input:	230Vac, 10A
DC Output:	0-1000VDC, 0-2A controllable.
Control:	Fast, DSP based voltage and current control. PLC for process control and external communication. Interface to PC via PLC.
Mode of operation:	Voltage control with current limitation or vice versa. Programmable restart in case of arc discharge (arc management).
Protections:	Fast overcurrent and di/dt protection. Overvoltage protection. Cooling water flow monitoring.
Human-Machine Interf.	Siemens OP3.

According to *Fig.2* the DC power supply consists of an input rectifier and a filter circuit, an inverter unit feeding the voltage transformer followed by the rectifier and the output filter. Due to the single-phase mains input voltage, the DC-link has an important role on smoothing the rectified voltage, but the filter has to block also the high frequency noise coming from the inverter.

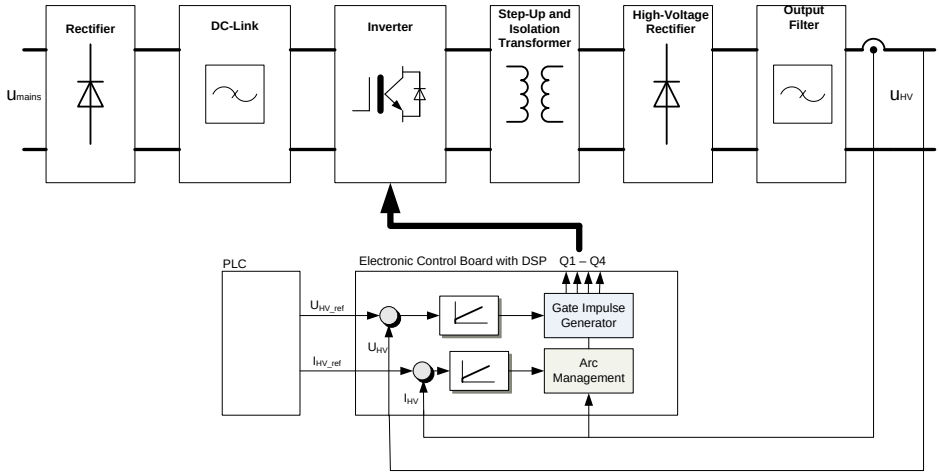


Figure 2: Schematic diagram of the DC power supply.

The inverter and the high-voltage rectifier have been realized with the state of the art semiconductors. In the inverter, two pairs of 40 Amps IGBTs are used, working at 100 kHz. The high-voltage rectifier has been built with Silicon Carbide (SiC) diodes, thus the switching losses can be neglected. With this

technology the cooling demand of the converter is reduced and the size of the reactive elements, mainly the output transformer, is reduced. The output filter has the role of reduction of the HV voltage ripple, but the energy stored in the capacitor has to be considered when the load is a vacuum chamber with plasma discharge. In case of electric arc, the energy of the capacitor (Cp) will be discharged on the surface of the treated part by a DC arc, having a strong erosion effect on the surface. The higher the energy stored in the capacitor, the higher the evaporated metal quantity. In our case a good compromise was to use a capacitor of 1 μF.

The control of the power supply is based on an inverter control board containing all necessary signal conditioning circuits, a dsPIC30F6010 digital signal controller for all the measurements and control functions, and finally a Lattice type CPLD for gate impulse generation and fast error handling. The control board communicates with a VIPA PLC, where the setvalues are entered and the mode of operation is selected. The user can select between two modes of operation, namely voltage control with current limitation, or vice versa. Beside of these, the arc management can be enabled or disabled from the PLC. In case of enabled arc management, the restart time is programmable. In case if the arc management is disabled, when overcurrent occurs on the output, the power supply stops, reporting overcurrent error. Simulations and practical measurements regarding the power supply operation are presented in [8].

4. Graphic User Interface - Control from PC

The nitriding process is controlled from a process computer, where a LabWindows CVI based software is running.

The process control software has been developed to offer an easy interface for data visualization and logging, controlling of the different process parameters. The most important control problem is the temperature control. Based on a simplified model of the system, a PI controller with anti-windup has been implemented to control the temperature of the treated part. The actual temperature can be seen on the main screen of the GUI and the temperature can be logged for further data processing. The main variable from the DC power supply are displayed (voltage, current, power), the power supply can be controlled through the PC in manual mode or closed loop temperature control mode.

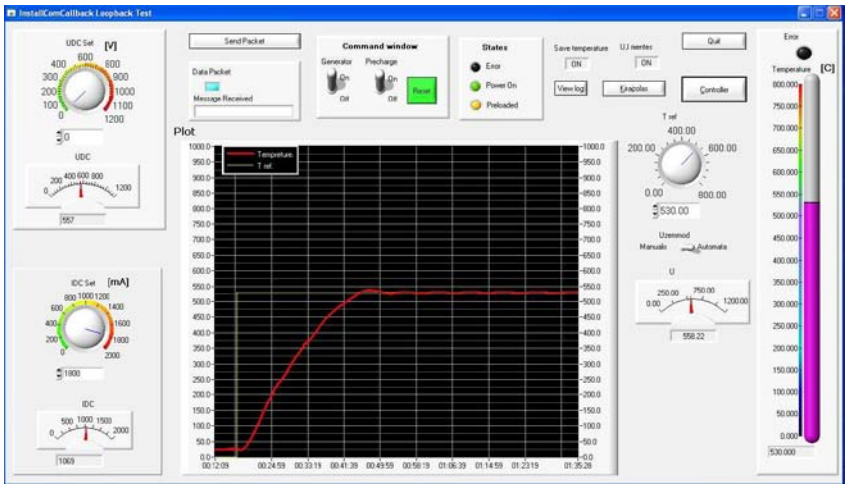


Figure 3: The graphic user interface (GUI) of the process control software.

The startup of the process is an important technological task, namely the temperature of the part has to be increased to the nitriding temperature with a well-defined ramp. The ramp can be set from the GUI of the process control software.

5. Piece holder system – material selection of the treated pieces

For test experiments, we selected a material whose properties are well-known with respect to plasma-nitriding heat treatment. This material is a 16MnCr steel. We planned to perform a series of heat treatments using the same material, same plasma conditions, and same geometry varying only the time-span of the heat treatment. The cathode arrangement used for heat treatment is presented in *Fig. 3*.

Two identical pieces are shown, which are positioned symmetrically with respect to the plasma chamber axis. This is important to have the same plasma conditions in the vicinity of each piece; hence the pieces reach the same temperature during heat treatment. We measure the temperature of the pieces using a K-type 1,5 mm diameter thermocouple, positioned inside a ceramic insulator tube to ensure galvanic isolation and introduced in Piece “B” through a hole. Piece “A” is changeable; we manufactured 4 identical pieces for this experiment.

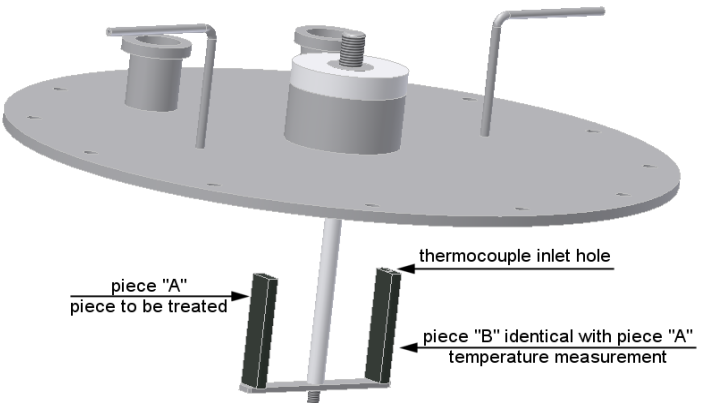
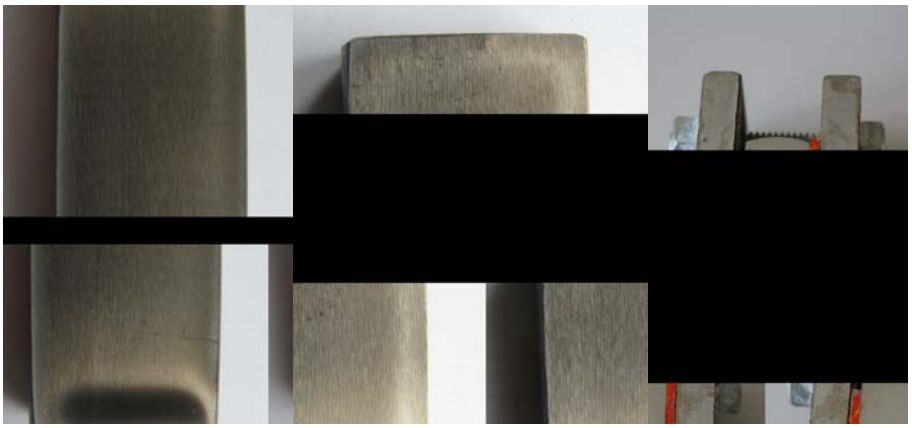


Figure 3: Experimental arrangement. Piece "A" is the piece to be treated; Piece "B" is an identical piece with Piece "A" located in symmetrical position with respect to the chamber axis.

6. Sample preparation for different investigations

It is very important to perform different investigations of the treated pieces, such as optical microscopy, electron microscopy and hardness measurements. For this reason the pieces must be properly prepared.



a. b. c.

Figure 4: Heat treated steel piece
a. deposition caused by cathode evaporation
b. edge-effect due to DCPN heat treatment
c. sample preparation for optical and electron microscopy.

At first all treated pieces must be cut perpendicularly to the plane side presented in *Fig. 3*. After cutting the treated pieces the samples were locked between two copper plates. After that the surfaces of the samples must be grinded, and polished in many steps involving different water resistant *SiC* abrasive papers with different grades of granulation, e.g. 100, 250, 500, 1000 until the surface become buff. After polishing, all surfaces were etched using 3% nital solution (solution of alcohol and nitric acid).

7. Conclusions

7.1. Visual observations

Visual observations of the samples provide important information. One can see, that the heat treatment of the pieces is not uniform. The bottom region of the piece in *Fig.4a* is covered by deposition of the evaporated cathode material. This is inevitable, because in DCPN treatment, the piece to be treated is the cathode of the discharge, so it must be in galvanic contact with some piece holder.

One other thing can be seen in *Fig.4b* namely the edge-effect. Also due to the fact that the treated piece is the cathode of the discharge, the appearance of the pieces is not uniform. The electric field distribution in the surroundings of the treated pieces is influenced by the shape of the piece. Of course the electric field is inhomogeneous; it becomes high at the edges of the pieces, leading to higher charge carrier transport to these regions.

7.2. Optical and electron microscopy

The prepared samples were observed using an optical microscope, than photographed. Such photo involving 60×10 optical zoom is presented in *Fig.5*.

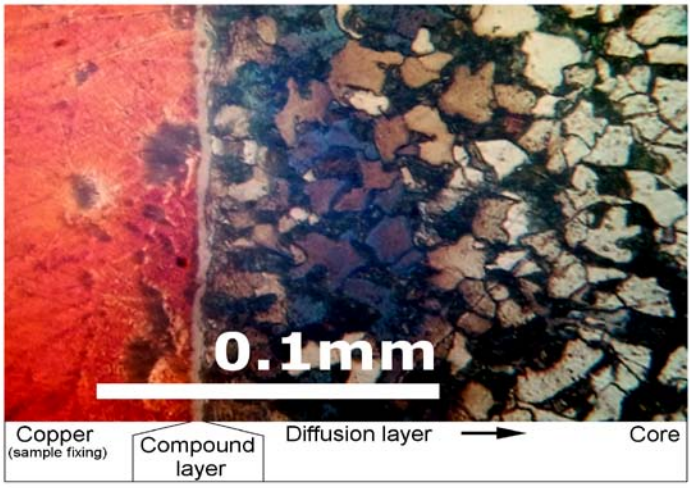


Figure 5: Heat treatment temperature 530°C, time-span 1 hour, magnification of the optical microscope 60x10.

Fig.6 presents a further magnified scanning electron microscope picture of the same sample.

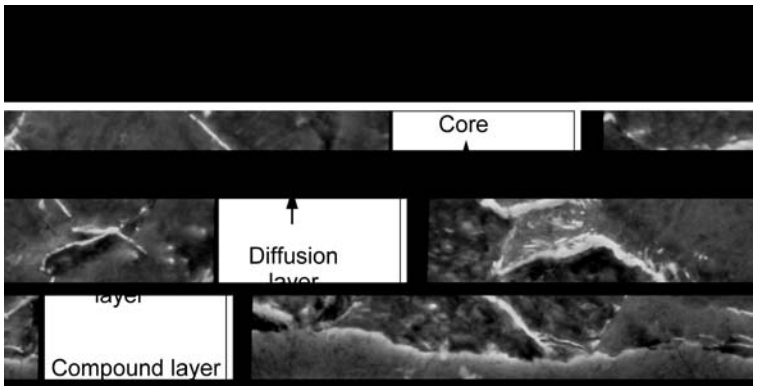


Figure 6: SEM picture using secondary electrons; 2-3 μm thick compound layer-magnification of the electron microscope ×5000.

We prepared all our samples as described in Section 5 and investigated them according Section 6. We measured in each case the thickness of the diffusion layer and obtained the curve presented in *Fig. 8*. One can conclude, that the diffusion layer thickness (d_{diff}) is proportional to $\sqrt{t}$.

7.3. Micro-hardness profile measurement (Vickers-hardness)

Another method to measure the thickness of the diffusion layer is to determine the hardness-profile of the cross section of the treated pieces. We applied the Wickers-hardness test using standard 25 gram weight pushed perpendicular to the sectioned surface. The first point to be tested was located 10mm away from the edge of the piece and continued with 25mm steps along a line perpendicular to the edge of the piece toward the inner regions. Usually the distance measured form the edge of the piece to the inflexion point of the micro-hardness profile is used as layer thickness. In case of the steel used in our experiments this point is at about 400MHv, hence we consider as layer thickness the distance measured from the edge of the piece where the hardness of the piece drops to the 400MHV value.

Fig. 7. represents the micro-hardness profile of the piece treated for one hour at 530ºC temperature. Similar micro hardness profiles are shown in [9].

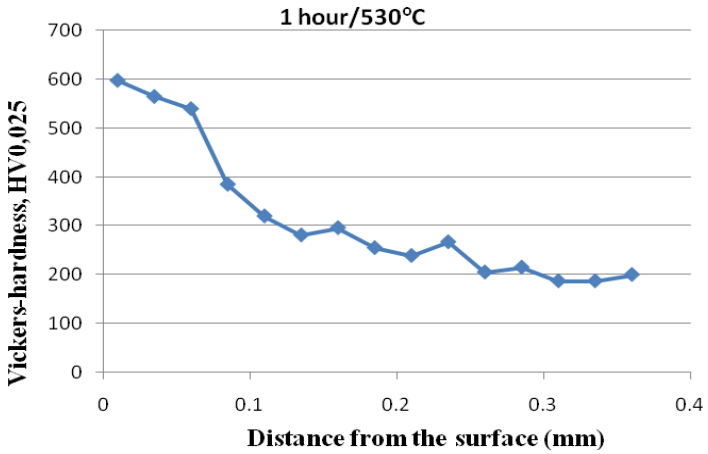


Figure 7: Micro-hardness profile of the piece treated for one hour at 530ºC.

The layer thickness values determined using micro-harness test are 10% higher than the ones determined using optical microscopy (*Fig. 8.*). The micro-hardness test is much more reliable than the optical measurements, because the optical ones are influenced by the sample preparation and the time-span of the etching. Evidently the micro-hardness measurements are also loaded by errors caused by inhomogeneous structure of the sample.

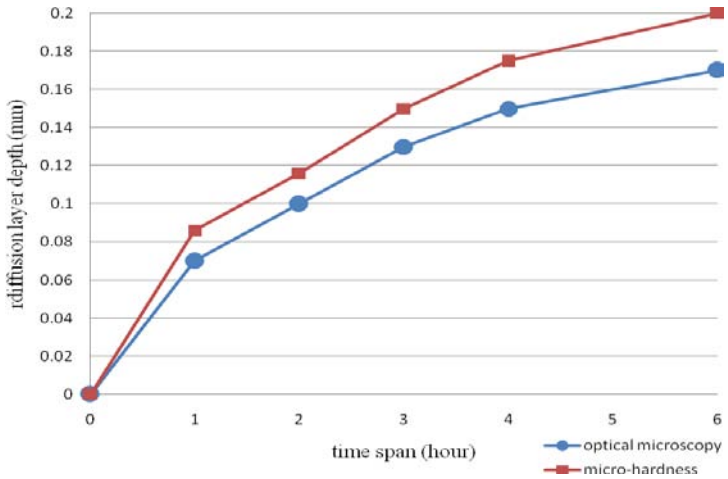


Figure 8: Diffusion layer depth.

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