EAN '97

EXPERIMENTAL RESEARCHES CONCERNING THE DEFLECTIONS PRODUCED BY RESIDUAL STRESSES DURING PLASMA NITRIDING

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Abstract

Within the paper some experimental results are introduced concerning the evolution of bending deformations, occurring in some flat, prismatic specimens and measured, by means of an *in situ* technique, during the plasma nitriding process. The nitriding steel under investigation is 38 MoCrAl 09 and the applied temperatures are: 480 °C, 500 °C, 520 °C and 550 °C. The measurements have been performed on an original experimental installation, specially - built for this purpose.

1. Introduction

In order to consider, starting even from the design stage, the complex implications of residual stresses caused by manufacturing processes, both their initial distribution and subsequent evolution must be known. Special attention has been paid to those processes which represent the last operations within the technologic flux, because they are the very one which have caused the stress state inherited by the part at the beginning of its "life".

Maximum exploitation of the possibilities offered by both the material and technology involves knowing the evolution of residual stresses during the different stages of the process, in order to find out the potential influences of technological parameters on residual stresses distribution.

In this context, certain research staffs have been lately formed, in several countries, concerned in the modeling of the generating mechanism of residual stresses during the plasma nitriding process. The information offered by literature are still insufficient, the continuation of the researches on residual stresses formation being required both at the end and during the process, especially in the case of plasma nitriding.

After analysing the references it follows that only the X - ray diffraction method has been employed, until now for monitoring both the occurrence and evolution of residual stresses during the nitriding process. The researchers U. Kreft, F. Hoffmann, T. Hirsch and P. Mayr from Stiftung Institut für Werkstoffiechnik, Bremen, Germany, are the first who carried out, by applying the above method at the beginning of this decade, measurements of the residual stresses from the compound layer, during the gas nitriding process [4], [5].

In reference [3], from the present Conference, we have introduced a new method for experimental *in situ* research of the residual stresses generating phenomenon, during either the plasma or gas nitriding process.

The method is based on the measurement of bending deflections which occur in a flat prismatic specimen, asymmetrically - nitrided during a complete cycle of thermochemical treatment.

In the following, some experimental results are detailed, regarding the evolution of these deflections during plasma nitriding, at different temperatures, applied to a specimen made from 38 MoCrAl 09 steel.

2. Experimental

Material

For the research, the 38 MoCrAl 09 steel, STAS 791 - 80, has been chosen as representative among Romanian nitriding steels. The chemical composition of the alloy under study was determined by spectral analysis with a Quantovac - type apparatus, being listed in Table 1.

| 38 MoCrAl 09 | | С | Si | Mn | s | P | Cr | Мо | AJ | Residual elements | | |
|--------------|------|------|------|------|-------|-------|------|------|------|-------------------|------|------|
| | | | | | | | | | | Ni | Cu | Ti |
| STAS | min. | 0.35 | 0.17 | 0.30 | - | - | 1.35 | 0.15 | 0.70 | - | - | - |
| 791-80 | max. | 0.42 | 0.37 | 0.60 | 0.033 | 0.035 | 1.65 | 0.30 | 1.10 | 0.30 | 0.30 | 0.02 |
| Specimen | | 0.37 | 0.36 | 0.51 | 0.019 | 0.021 | 1.58 | 0.15 | 0.98 | 0.26 | 0.29 | 0.00 |

Table 1 Chemical composition of material, wt-%

In order to determine mechanical characteristics, tensile test have been performed at the following temperatures: 20 °C, 500 °C and 550 °C.

The following parameters have been determined: proof stress, Rp_{02} , tensile strength, Rm, percentage elongation, A.

Tensile tests were performed according to the standards SR EN 10002-1:1994 and SR EN 10002-5:1995. The tests results are given in Table 2.

| Temperature, [°C] | R р₀₂ [MPa] | Rm [MPa] | A [%] |
|----------------------|----------------------------------|--------------------|----------|
| 20 | 795 | 1080 | 12 |
| 500 | 625 | 740 | 20 |
| 550 | 480 | 530 | 25.8 |

Table 2 Mechanical characteristics

Specimens

The specimen have a flat prismatic geometry (Figure 1).

The surface protection against nitriding is done by galvanic copper coating, the thickness of the protection layer being of approximately $20 \,\mu m$.



Figure 1. The specimen geometry

Experimental installation

The experimental set [2], specially designed and built for these tests, contains besides all the elements of an usual plasma nitriding unit, a displacement transducer and a special specimen fixing device.

The electrical power required for the testing procedure is about 0.8 kW.

The nitriding chamber (Figure 2) is made of a heat-resistant glass container (10), fixed with two fasteners on a steel plate (3) representing the anode. Pressurization is ensured by rubber seals. The chamber volume is about 1.25 dm^3 .

The steel wall of the chamber is fixed with screws, on two pads, in a vertical position.

In the vertical symmetry plane of the chamber, are located:

- the supporting device (8) of the specimen (11) which represents the cathode and includes the thermocouple;

- the transducer (7) for specimen temperature measuring (in infrared rays) without contact;

- the inductive displacement transducer (6), [1].

On each side of this plane, there are two orifices:

- a passage orifice, for the vacuum coupling pipe on which a thermal PIRANI-type gauge is fastened which measures the pressure in the vacuumed chamber;

- an inlet orifice for working-gas supplying.

The flow adjustment of technologic gases is made through a tap provided with a micrometric gauge-screw, attached on the chamber's steel wall, right in front of the admission orifice.

On the steel wall of the chamber the socket (9), is positioned; that makes all the necessary electrical connections.

In order to dissipate the thermal radiated energy, chamber wall has a groove which achieves a circuit for the water cooling according to a "U" - shaped circuit,

around all devices and transducers placed in its central area. Water admission and exhaustion is made through the leakage pipes (1).

The flat prismatic specimen, protected against nitriding on all its surfaces except one is considered to be a rigidly fixed bar in the supporting device, at one end, and free at the other one.

At the free end, at a 100 mm distance from the supporting device, the specimen is in contact with the transducer free gauge. The gauge allows to measure the displacements that occur during the plasma nitriding as a result of specimen bending.



Figure 2. The plasma nitriding chamber

For a given geometry of the specimen, the displacement magnitude "transduce" the generation and development of residual stresses during the entire period of the nitriding process.

3. Results

Corresponding to four plasma nitriding regimes, applied to specimens with the form and dimensions given in Figure 1 and made from 38 MoCrAl 09 steel, the displacement values have been recorded at the free end of each specimen, during the entire process.

Plasma nitriding parameters - working medium: nitrogen and hydrogen mixture, obtained from ammonia dissociation at 875 °C;

- gas pressure: 3 -4 torrs;

the voltage between anode and cathode can variate continuously from 300 V to 1000 V, so that for a certain gas working pressure, the needed specimen temperature could be obtained;
nitriding temperatures were: 480 °C, 500 °C, 520 °C and 550 °C.

Each specimen was maintained at the above nitriding temperatures until its displacement, measured at a 100 mm distance from the fixed end, reached 3 mm.

In Figure 3, the evolution of these displacements is illustrated during plasma nitriding, until the moment before cooling, corresponding to each of the above four nitriding temperatures.



Figure 3. Deflections evolution of the specimens made from 38 MoCrAl 09 steel, during plasma nitriding (displacements measured at a distance of 100 mm from the embedded end)

After vacuum cooling, the final deflection has been measured at room temperature, inside the nitriding chamber. The results are shown in Table 3.

Table 3. Final values of the deflections, after cooling the plasma nitrided specimens

| | Nitriding temperature (°C) | | | |
|--------------------|----------------------------|-----|-----|-----|
| Total deflections | 480 | 500 | 520 | 550 |
| after cooling (mm) | 3.97 | 4.1 | 4.2 | 4.5 |

4. Discussion and conclusions

By analysing the curves shown in Figure 3, it is noticeable that the deformation rate of the specimens largely increases with increasing nitriding temperature. Thus, for instance, in the case of the 480 °C maintaining temperature, the 3 mm deflection of the specimen was

measured after 174 minutes (time elapsed from the moment the 350 °C temperature was reached) while in the case of the 550 °C nitriding temperature the above deflection was recorded after only 34 minutes.

The above results have been explained as follows:

- on are hand, with increasing deformation rate, nitriding kinetics is intensified which induces an intensification of the kinetics of both the growth of nitrided layer thickness and the level of residual stresses;

- on the other hand, due to the high nitriding temperatures, a marked creep is observed in the basic material. This fact was experimentally - ascertained by means of creep tests performed on the same material subjected to tension. Thus, for instance, in the case of a constant stress, $\sigma = 200$ MPa, an average plastic deformation $\epsilon_p = 0.22 \cdot 10^{-2}$ has been obtained at 500 °C after 700 minutes, while at 550 °C the plastic deformation accumulated in the same period of time (700 minutes) was considerably larger, $\epsilon_p = 3.07 \cdot 10^{-2}$.

Thus a thermal relaxation of residual stress occurs, as a consequence of the difference between the specific volumes of the phases produced by the treatment and the basic material. In the case of asymmetrical nitriding, the creep phenomenon also causes a considerable increase of deformations.

- during the cooling stage, the deformations increase of asymmetrically nitrided specimen is mainly caused by the deformations incompatibility, as a consequence of the difference between the values of thermal expansion coefficients corresponding to the resulted phases and the basic material, respectively. During the first part of this stage, when temperature is still high enough, the creep phenomenon is present.

The obtained results will be used for ascertaining different mathematical models which describe the residual stresses generating mechanism, during the nitriding process, according to the method introduced by us in reference [3].

The above-described experimental technique constitutes an important means for *in situ* investigation of the complex phenomenon of stress generating, both in the case of gas and plasma nitriding.

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