THE INFLUENCE OF MECHANICAL SURFACE TREATMENT TO HYDROGEN EMBRITTLEMENT OF A HIGH-STRENGTH STEEL.

L'INFLUENCE DE L'ETAT DE SURFACE USINE SUR LA FRAGILISATION PAR L'HYDROGÈNE D'UN ACIER MARTENSTITIQUE PENDANT LE TRAITEMENT ELECTROLYTIQUE.

SCHMIDT-THOMAS KH.G. - WOLLRAB P.M.  
HOFFMEISTER B.  
IMM, TECHNISCHE UNIVERSITÄT LIEBHERR-AEROTECHNIK  
MUNICH  
LINDENBERG  
W. GERMANY

RESUME Trois surfaces différentes peuvent être produites mécaniquement par meulage, sablage et grenaillage et ont été chargés électrolytiquement en hydrogène. Les propriétés topographiques et les tensions résiduelles aux surfaces ont été étudiés à l'aide du microscope à balayage et de diffraction des Rayons-X.

SUMMARY Three different surfaces are produced mechanically by grinding, sandblasting and shot-peening and are electrolytically charged with hydrogen. These surfaces are investigated by SEM for topographic properties. The state of residual stress at the surfaces is studied by means of X-Ray-Diffraction.

II. INTRODUCTION

The invested material is a low-alloy steel. A suitable heat-treatment can bring it up to a tensile strength of 190 hbar. After annealing the steel it is necessary to remove the oxide. Especially the producers of aircraft-parts - e.g., the landing gear - use mechanical methods, before galvanizing those parts to protect them against corrosion and wear. The application of chemical methods, as for example pickling or etching is not possible for materials with the require for highest properties. Besides removing the oxid by mechanical surface-treatment there is great importance in inducing residual stress and in producing certain roughnesses.

In our experiments we produced mechanically three different surfaces as they are in utilization for landing gears of aircrafts. These surfaces have been investigated by SEM for topographic properties. The state of residual stress has been studied by means of X-ray-diffraction.

Electrolytically charging with hydrogen under defined conditions has been chosen to simulate the hydrogen absorption during electrolytical plating with Cadmium and Chromium. Tensile testing after the charging with hydrogen has shown, that the charging-time and the mechanical behaviour of the specimens can be assigned to each other. By means of SEM we could find out a relationship between the extent of hydrogen embrittled microstructure and the facts mentioned before.

II. EXPERIMENTAL

II.1. The high-strength steel with the french indication 35 NCD 16 has the percentage:

- 4.7 Ni
- 1.9 Cr
- 0.43 Mo
- 0.35 C
- 0.27 Si
- 0.20 Mn

After the austempering at 875°C and after the annealing at 200°C the tensile strength at 190 hbar and the yield strength at 155 hbar is attainable. Of course, before heat-treatment the specimens got their shape. We used tension specimens, see FIG. 6.
MECHANICAL SURFACE TREATMENT

II.2. To produce mechanically three different surfaces we used the

    dores:

    **GRINDING**
    Grinding disk 70 JOT
    Diameter of disk 400 mm
    Speed of grinding disk 1400 R/mn
    Speed of specimen 250 R/mn

    **SAND BLASTING**
    Substance Korindan that is composed of oxids of the elements
    Ti, Al, Si, Zr
    Particle-size 10 to 30 µm (90 to 150 mash)
    Air-pressure 4 to 4.5 bar
    Blasting-time 20 s

    **SHOT PEENING**
    Ball-diameter 0.5 to 0.8 mm
    Ball-hardness HV 0.5
    Drop-speed 1.2 m/s
    Speed of specimen 18 R/mn

    II.3. SEM-Investigation for topographic properties
    See FIG. 1, 2 and 3.

![SEM-image of a ground surface](FIG_1.png)
Magnification 200
FIG. 2.: SEM-image of a sand-blasted surface
Magnification 200

FIG. 3.: SEM-image of a shot-peened surface
Magnification 200
II.3. Residual Stress Measurements

The determination of the state of residual stress at the ground, blasted and shot-peened surfaces was performed by means of the SIN²-γ-Met. This method is based on Bragg's Equation \( n \lambda = 2d \sin \theta \). The device is a S-ω-Goniometer \( 2 \). The origin of this method is the assumption a layer close to the surface being inhomogeneously plastically deformed by mechanical treatment regions of the microstructure are more or less intensively deformed with the consequence of required elastic strain that is measurable by X-ray-diffraction. The existence of elastic strain the coherence of the microstructure would possible.

We only recorded macroscopic residual stress (Eigenspannungen 1.; we know that recording microscopic residual stress (Eigenspannungen 2.Art \([3,4]\] will be necessary in the future, to have more profound view of plastic deformation within areas like grains.

The SIN²-γ-Method considers the shift of the Bragg-angle \( \gamma \) due to plastic deformation in correlation to angles \( \gamma \). \( \gamma \) is the angle between the beam and the surface to be investigated, see FIG. 4.

![FIG. 4.: Compiling results by means of the SIN²-γ-Method](image)

The test values at \( \gamma \)-angles of 0°, 25°, 35°, 42° and for laterations at additional \( \gamma \)-angles of 12.5°, 30°, 38.5° should be situated under ditions onto a straight line. The gradient of this line permits to calculate value and the plus or minus sign of the residual stress.

The position of the peak-maximum was determined by:

- points of intersection of the tangent lines
- calculating the center of the envelope curves.
II.4. Electrolytically charging with hydrogen

Tensile specimens (see FIG. 6) have been charged with hydrogen in \( \text{In-H}_2\text{SO}_4 \) at a temperature of 55°C. This temperature is conform to the medium temperature required for certain Chromium-plating processes. The current density of 3 mA/mm² was generated by a direct-current constanter. A net of Platinum of cylindric shape and of a diameter of 25 mm was the anode (see FIG. 7). Times of 6s, 30s, 60s, 90s and 120s have been chosen for charging.

FIG. 6.: Tensile specimen DIN 50 125, \( d_o = 6 \text{mm} \), \( L_o = 30 \text{mm} \), \( d_1 = 8 \text{mm} \)

FIG. 7.: Charging-device
1 glass-container, 2 stirrer, 3 heater, 4 thermometer
5 glass-electrode, 6 anode, 7 specimen, 8 reference-electrode

Immediately after charging the specimens have been cleaned to begin the tensile testing as soon as possible. The time between charging with hydrogen and tensile testing was about 3 min.

II.5. Tensile Testing

Tensile testing has been executed at a deformation-speed of 0.35 mm/min at room-temperature. A relatively low speed like this one is required for visualizing embrittled microstructure. We could confirm this fact in our own experiments. The mechanical properties were recorded by the stress-strain diagram.

Most of the specimens have been loaded as high as their ultimate strength. All the planes of failure have then been prepared for SEM. Besides general views to make obvious the macro-fracture appearance we tried to find some typical feature by application of higher magnification.
III. RESULTS

Measuring the state of residual stress renders the following values:

- Ground surface: +6.0 hbar (residual tensile and compressive stress)
- Sand-blasting surface: -17.4 hbar (residual compressive stress)
- Shot-peening surface: -35.5 hbar (residual compressive stress)

To explain the results of tensile testing we use FIG. 8 and FIG. 9

The relationship of the tensile strength to the period of the charging-time is obvious. More remarkable in our opinion is the linearity of relation to be conclusive for the ground and shot-peened surfaces. Clearly to be seen is the favorable influence of the sand-blasting process to avoid hydrogen embrittlement. Issuing from a percentage of the decrease of tensile strength we can say that there is a variation of about 20% during the first 60s depending upon the surfaces. For longer periods of charging with hydrogen there is a range of about 15% to 35%.
Plotting $\log \delta$ vs. charging-time, we found out a rather close linearity for the ground, sand-blast and shot-peened surfaces. Only specimens without any defined surface-treatment cannot be approximated to linearity.

Onto the fracture planes of the tensile specimens a general view gives characteristic features of the macro-fracture of the investigated high-strength steel (FIG. 10, 11).

The typical 'cap and cone'-fracture (specimens without hydrogen) and the typical brittle fracture are examples of the interaction of hydrogen in steel. The image of the brittle fracture appears almost without any deformation and without any necking and the plane of fracture is orientated vertically to the direction of tensile stress. The embrittled microstructure is conspicuous located into an area that is limited by two concentric circles. The depth of penetration of embrittled microstructure is about 700 $\mu$m.

FIG. 12,13,14 show fracture planes of specimens with ground surfaces. One has been charged with hydrogen for a period of 30 s, one for 60 s, and another for 120 s.
FIG. 10: Macro-fracture image of a tensile specimen that has not been charged with hydrogen by SEM, magnification 20

FIG. 11: Macro-fracture image of a tensile specimen that has been charged with hydrogen, charging time 120s, by SEM, magnification 20
MECHANICAL SURFACE TREATMENT

FIG. 12.: Fracture image of a ground tensile specimen, hydrogen-embrittled microstructure in the external zone, charging-time 30s, by SEM, magnification 150

FIG. 13.: Fracture image of a ground tensile specimen, hydrogen-embrittled microstructure in the external zone, charging-time 60s, by SEM, magnification 150

FIG. 14.: Fracture image of a ground tensile specimen, hydrogen-embrittled microstructure in the external zone, charging-time 120s, by SEM, magnification 150
MECHANICAL SURFACE TREATMENT

Charging-time 30 s: depth of penetration about 240 μm
Charging-time 60 s: depth of penetration about 380 μm
Charging-time 120 s: depth of penetration about 480 μm

For these and all the other investigated specimens there was no difficulty in distinguishing intercrystalline from transcristalline appearance of fracture.

IV. GENERAL CONCLUSIONS AND REMARKS

Our procedures of surface-treatment correspond to those of procedures of high-strength construction units for airplanes. Besides the structural properties we paid attention to the state of residual stress. A compact surface is as well of interest as a surface full of fissures regarding the amount of hydrogen absorption. The advantage of a shot-peening surface is on the one hand side the low notch effect in relation to the ground or sand-blasting surface. On the other hand side the favourable influence of the needle effect especially onto the sand-blasting surface has to be regarded. The needle effect promotes hydrogen recombination before atomic hydrogen being able to migrate into the metal lattice [6,7].

The grinding-treatment was purposely manufactured at two nearly vertical directions to prevent as far as possible any influence of texture [8]. The grinding-procedure needs special care of temperature effects. Grinding without local increase of temperature seems to be impossible even under attentive conditions. The temperature-induced degradation of residual stress makes the scattering of results nearly zero explicable. The possibility of hydrogen absorption even by grinding has not been taken into account. The influence of the cooling agent [11] might be the subject matter of further investigations.

The surfaces onto which the highest values of residual compressive stress is measurable are owing to shot-peening. Shot-peening causes cold work-hardening within the plastic range of the tensile strength. This verification can be taken for granted [10]. A profile of residual stress is recordable by etching in steps of about 10 μm, see FIG. 15. The interaction dislocations-atomic hydrogen in steel [14,15,16,17,18] shall not be discussed, though an increase of the density of dislocations is to be premised. Another point of view is the question, whether there is any influence of the parameters of shot-peening to the intensity of residual stress or whether there happens primarily shifting of the position of maximum residual stress relative to the distance from the surface. This opinion [19] includes nearly constant values of residual stress and was made firm by our own experiments.

Before starting our investigations we expected shot-peening to be the most efficient way to reduce hydrogen-embrittlement during electrolytical plating. So we were surprised finding out sand-blasting the favourable surface-treatment of high-strength low-alloy steel. Nevertheless our results are in good conformity with some other authors [20,21]. Regarding the first 60 s of electrolylical plating we shall be able to establish a compact layer of Chromium or Cadmium onto the surface to act against hydrogen diffusion during the whole period of plating. On this field of research there exist some investigations not all leading to the same recommendation [12,21].

The classification of fracture areas is a very significant facility for failure analysis [19]. Also for basic research microfractography has become greater importance. In our investigations we were able to find out a relation between the charging-time and the depth of penetration of the embrittled microstructure.
FIG. 15: Residual stress vs. distance from the surface, Plus: tensile stress
Minus: compressive stress

REFERENCES

Ing. Techn. 45, (1973), 760.
[9] Syren B. - Wohlfahrt H. - Macherauch E., Röntgenographische Eigenspannungsbestim-
mungen an Ck 45, HTM 31, (1976), 90 - 94.
[13] Franz H.E., Einsatz der röntgenographischen Spannungsmessmethode in der Luftfahrt-
industrie, Metall 12, (1975), 1215 - 1221.
[20] Schmitt-Thomas Kh.G. - Klingele H., Entstehung und Erscheinungsform mikrofraktogra-
phischer Bruchmerkmale, Der Maschinenschaden 42,(1969), 183-193