

THE IN-DEPTH STRESS DISTRIBUTION FOR 1H13 SPECIMEN AFTER CUTTING

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Abstract

Measuring the in-depth stress state is of vital importance for materials scientists. Strain gauges methods are capable of yielding information only about the surface stress state. Diffraction methods using synchrotron or neutron radiation, which allow totally non-destructive stress measurements inside the material, are not widely available. In this context, the best widely available method combines the X-ray diffraction stress measurements and gradual removal of the outer layer by means of electropolishing. Here, this method was applied to the specimen made of 1H13 stainless steel cut with under water on a corundum cut-off wheel. The idea was to investigate how deeply an additional stress state resulting from cutting was introduced and whether the technique of combining of X-ray diffractometry and electropolishing can be used widely for determining the stress state inside the specimen.

Keywords: In-Depth stress distribution, Electropolishing, Diffraction methods, 1H13 stainless steel.

INTRODUCTION

Nowadays a number of different techniques of performing stress measurements are being used; including strain gauges application, hole drilling methods, ultrasonic and magnetic methods, and diffraction methods employing different types of radiation. However, the only methods that enable non-destructive and semi-destructive in-depth stress measurements are diffraction methods. The really non-destructive are these diffraction methods which employ synchrotron and neutron radiation. The main disadvantage of diffraction methods is that access to radiation sources is not common: the neutron radiation being produced by nuclear reactors and the synchrotron radiation obtained through a synchrotron. The compromise can be employing the X-ray diffraction method: X-ray diffractometers are widely accessible and do not need any special infrastructure. The X-ray diffractometry combined with electropolishing and successive incremental material removal can give information on the variation of residual stress relative to depth; it can be described as a semi-destructive method (in contrast with hole-drilling and other diffraction methods). Although the material is successfully removed, the measurement can be performed many times at each successive stage of the electropolishing procedure. The hole-drilling method can be classified as a destructive method (the measurement on one specimen can be performed only once during the drilling of a hole) while diffraction methods employing more penetrating types of radiation: synchrotron or neutron radiation are 100% non-destructive.

The purpose of measurements made was to investigate the effect of both cutting a 1H13 specimen on the corundum cut-off wheel and the depth of stress changes induced by it.

THEORETICAL BACKGROUND AND THE METHODOLOGY OF THE EXPERIMENT

Electropolishing idea and theory

When there is no access to other diffraction measurements methods, the X-ray diffractometry provides an optimal way to measure in-depth distribution of stresses. This method involves successive removing of the by electropolishing and stresses analyzing at every stage of the polishing procedure.

The idea of electropolishing consists in subjecting the surface of the material to a specially mixture of acids saturated with the metals' salts and a current flow. This procedure causes the material's surface to be removed ion by ion. For the local electropolishing procedure required by X-ray diffraction measurements, electrolyte is pumped only into a specified area of the sample (the sample is not submerged with electrolyte like for example metallographic samples are). Voltage is applied to a specimen for a very straight, defined period of time depending on the required layer removal. Essential for an amount of the removed material is Faraday's first law stating that the mass of the metal removed Δm (i.e. the mass of metal ions) is proportional to the current ΔI^+ during time Δt , and is given by:

$$\Delta m = k \Delta I^+ \Delta t \quad (1)$$

where:

k – the electrochemical equivalent of the sample material (equal to the mass of ions carrying the charge of 1 coulomb). Its value can be calculated based on Faraday's second law (for the reaction of $M \rightarrow M^{z+} + ze^-$):

$$k = \frac{A}{zF} \quad (2)$$

where:

A – the atomic weight of the metal M ,

F – Faraday's constant equal to 96500C.

The key electropolishing variables and parameters are: the selection of the appropriate electrolyte, appropriate for given material; electropolishing procedure parameters which are: current, voltage and time. Before electropolishing the voltage – current characteristic, which indicates the voltage range for specified material – electrolyte, should be obtained. A good practice prior to electropolishing is to perform some trials before starting measurement cycle to check the relation between adopted parameters (voltage, current and time) and electropolishing depth.

Corrections of in-depth X-ray diffraction stress measurements data

Although, compared to other methods of the outer layer removal (e.g. mechanical polishing or EDM), electrolytic polishing affects the stress state in the least, some corrections with reference to stress redistribution and relaxation should be taken into account, especially when the examined specimen is relatively thin.

A rudimentary example is a flat specimen. Sikarskie [3] proposed a generalized solution:

$$\sigma(z_1) = \sigma_m(z_1) + 2 \int_{z_1}^H \frac{\sigma_m(z)}{z} dz - 6z_1 \int_{z_1}^H \frac{\sigma_m(z)}{z^2} dz \quad (3)$$

where:

H – an original plate thickness,
 z_1 – plate thickness after layer removal,
 Δz_1 – removed layer thickness,
 σ_m – measured stress,
 σ_z – real stress.

Integrands of the above equation can be expanded in a Taylor series. The first simplification is when taking into account only the first term of the Taylor series. It can be made if electropolishing is relatively shallow (a few percent of the specimen thickness):

$$\sigma_z(z_1) = \sigma_m(z_1) + \left(-4\sigma_m(H) \left(\frac{\Delta z_1}{H} \right) \right) \quad (4)$$

The above corrections were taken into consideration in this work.

The measurement methodology: material, specimen preparation, electropolishing and X-ray diffraction parameters

The crucial aspect of depth profiling measurements is the accuracy of the removed material amount. This can be achieved by using a calibrated travelling microscope, digital gauge or calibrated micrometer. In this work the latter method was applied. It is important to ensure that the electropolished surface is flat so number of electropolished layer depth is recommended. When the electropolished surface is uneven it is recommended that the mean value of the measured layer's thickness should be used. The next very important decision prior to performing the experiment is choice of the depth increment used to measure the profile. It should be appropriate to expected stress values and to grasp the changes. Chosen depth increments can be incorrect so the stress values for different depths shouldn't be connected – it can stress states cannot be correct falsify the results.

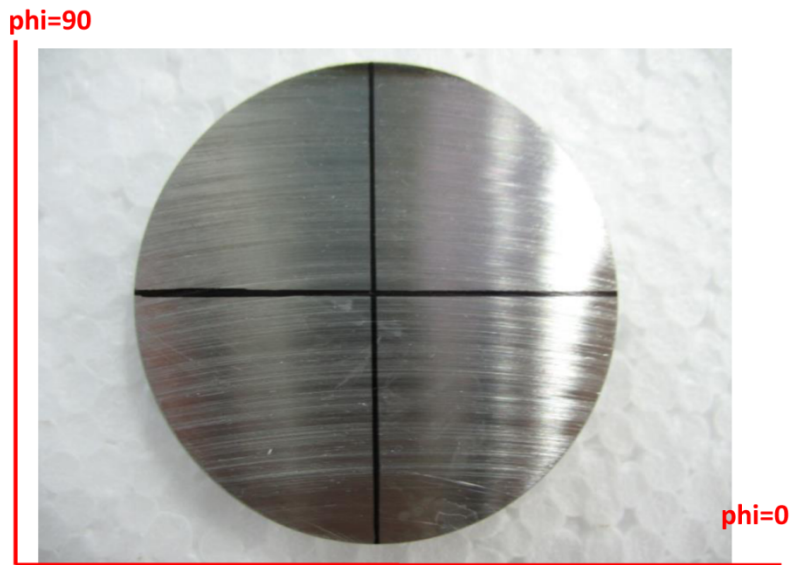


Fig. 1. The specimen surface with the measurement directions and the measurement point location (lines cross-section).

The material investigated was 1H13 chromium stainless steel with about 0,08% – 0,15% content of carbon and addition of sulphur, manganium, nickel, silicon and potassium. The specimen was cut under water on aluminum oxide cut-off wheel to prepare the surface for the experiment (Fig. 1.).

The electrolyte used was K1 electrolyte distributed by the ATM GmbH whose composition is perchloric acid, ethanol and Propandiol-1,2. The most appropriate current intensity for this type of material and electrolyte was 0,6A; this setting allowed to remove a layer of about $10\mu\text{m}$ in 30s. The effect of electropolishing on some chosen steps is presented on Figure 2.

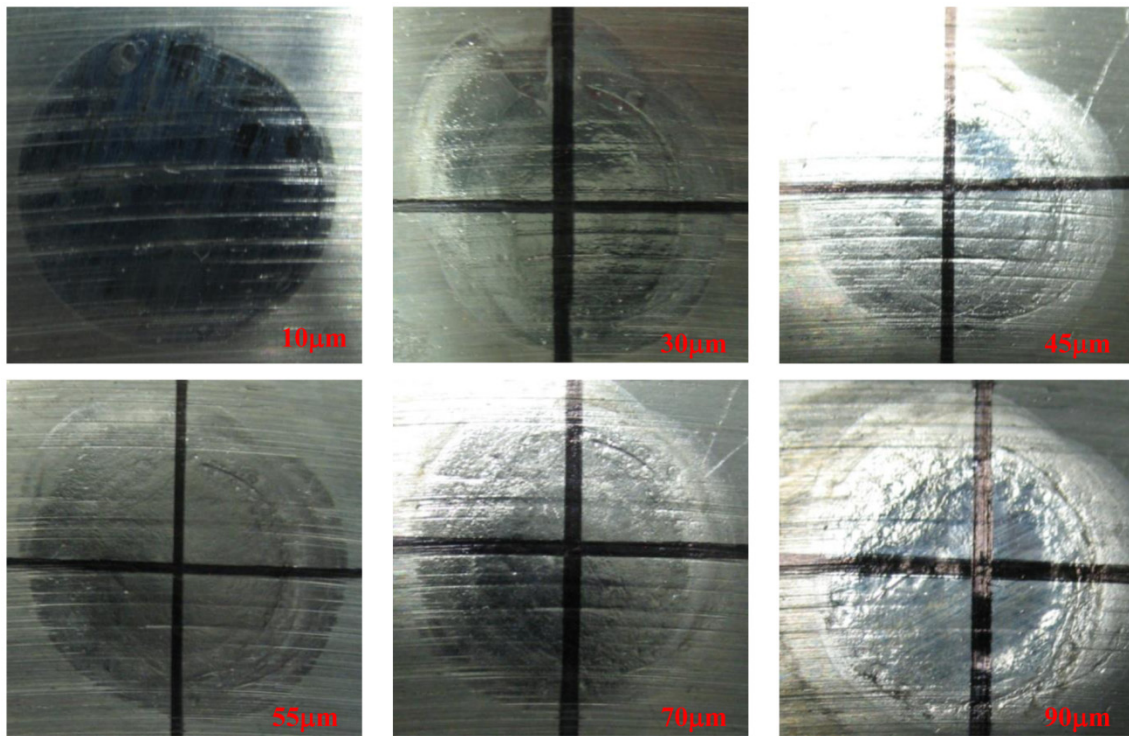


Fig. 2. Specimen surface after some succeeding electropolishing steps.

The X-ray stress measurement was performed with Xstress3000 equipment following each step of electropolishing. The radiation applied was K_{α} of chromium X-ray tube ($\lambda K_{\alpha} = 0,22897\text{\AA}$). The diffraction peak was obtained after reflection in (211) lattice planes of the ferrite phase (Braggs' angle $2\theta = 156,4^{\circ}$). Material data taken for calculation were: Poisson's ratio equal to 0,3 and Young modulus – 211GPa . The measurements at every successive stage were performed for 3 directions as marked in Figure 1. The time of single exposure was 5s and the measured area was 3mm in diameter and was located in the center of the specimen.

RESULTS ANALYSIS

The results of the experiment are presented in Figures 3 and 4. Figure 3 shows the in-depth corrected stress distribution in three directions ($\phi = 0^{\circ}$; $\phi = 45^{\circ}$; $\phi = 90^{\circ}$, for the direction definition see Fig. 1.) including the difference between measured values and values corrected with equation (4). It can be observed that the effect of cutting (about $100 - 300\text{MPa}$ depending on the measurement direction) disappeared at the depth of about $50\mu\text{m}$. Additionally, the information on the reliability of the measured values was obtained: Figures 3 d-f show that the deeper electropolishing process is the bigger difference between the measured and real values of stress is. In this case the difference for a depth of $90\mu\text{m}$ is about $6 - 18\text{MPa}$. It is negligible compared to the stress measurement uncertainty (even $30 - 40\text{MPa}$).

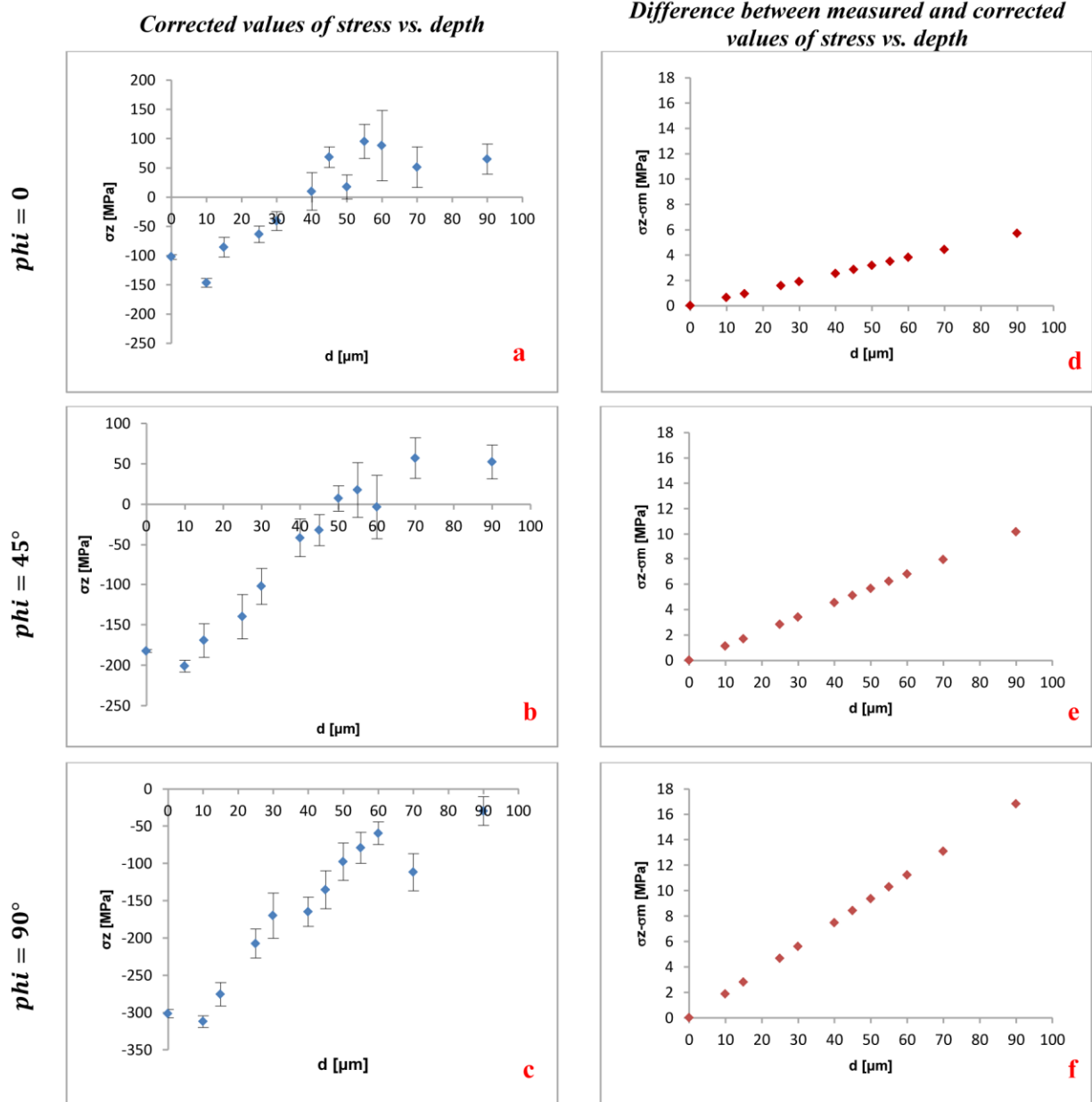


Fig. 3. The corrected values of stress (left column) and the difference between corrected and measured stress values (right column) versus depth into the specimen in three directions with respect to the specimen (a, d: $\phi = 0$; b, e: $\phi = 45^\circ$; c, f: $\phi = 90^\circ$).

Analysing the data shown in Figures 4 has led to the conclusion that the outer layer of the specimen was deformed plastically by the cutting process: the full width at half maximum (FWHM) values are double for the outer layer than for the inner part of specimen. Furthermore, the cutting process had no influence on the peaks intensities i.e. it can be assumed that it doesn't introduce any characteristic texture of the material.

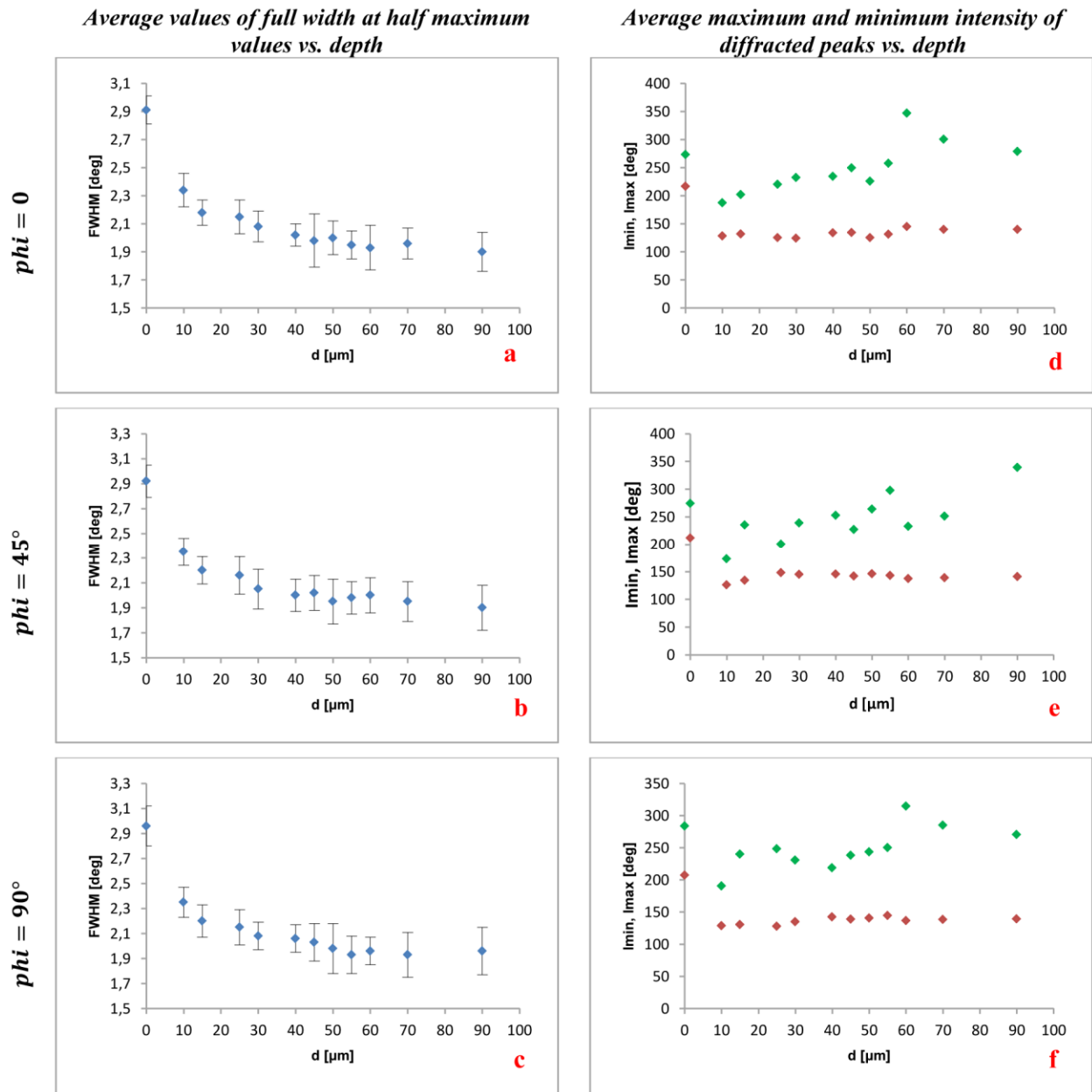


Fig. 4. The average values of FWHM of diffracted peaks (left column) and average minimum and maximum peaks intensity values (right column) versus electropolished depth (a, d: $\phi = 0^\circ$; b, e: $\phi = 45^\circ$; c, f: $\phi = 90^\circ$).

CONCLUSIONS

The X-ray diffraction stress measurement coupled with electropolishing is an advantageous tool for measuring in-depth stress distribution without affecting the original stress state. It can be applied in every laboratory equipped with an X-ray diffractometer and a portable electropolisher. The measurements obtained by this method are reliable and enable finding out the absolute stresses values inside the specimen with the accuracy of few micrometers.

Additionally, the X-ray diffraction method makes it possible to describe the plastic deformation of the material and to describe its texture. The only condition for ensuring reliable in-depth results is determining the optimal current intensity for the investigated material – electrolyte doublet – and to choosing the best time in order to not miss stress values changes.

The above results for 1H13 stainless steel after cutting showed that this process had a significant impact on the stress state and the plastic deformation of the outer layer (about $50\mu\text{m}$) of the specimen. The theoretical corrections of the stress values are not significant in relation to the uncertainty resulting from the nature of the diffraction measurement so they can be ignored.

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