LOAD PARTITIONING IN A DUPLEX STAINLESS STEEL WITH SURFACE STRENGTH GRADIENT AND RESIDUAL STRESSES

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ABSTRACT

The load partitioning between surface and bulk was studied by means of in-situ synchrotron energy dispersive diffraction in an SAF 2507 superduplex stainless steel having a strain hardened surface layer with compressive residual stresses. Two loading cycles with a peak tensile strain of 0.5% and 0.87%, respectively, were successively applied and the evolution of stresses in the respective constituent phases (austenite and ferrite) were measured in a 0.026 mm thick surface layer and at 0.75 mm depth, from which the macrostress development was derived, respectively, for the two locations. It was found that the surface and the bulk followed different loading paths and that such inhomogeneous load sharing was related to variation in strength and residual stresses over the load-carrying cross-section. During macroscopic elastic loading, the load partitioning, characterized by increasing tensile stress in the bulk and decreasing compressive stress in the surface, depended mainly on the initial residual stress distribution. Upon yielding of the bulk, a rapid load transfer from the bulk to the hard surface layer started and continued until the surface also reached its elastic limit. At the 0.87% peak strain, the plastic incompatibility over the cross-section resulted in a much higher stress in the surface (1070 MPa) than in the bulk (680 MPa), which also led to a large tensile residual stress in the surface after unloading. In the paper, the observed inhomogeneous microscopic load partitioning was also presented.

INTRODUCTION

Many manufacturing processes impose, intentionally or unintentionally, changes to the surface layer of engineering components, such that a strength gradient is induced and residual stresses are created. It is well known that such changes play an important role for the mechanical behavior of the components. In particular, it is well established that fatigue properties can be enhanced by a hardened surface layer with compressive residual stresses but reduced by a surface layer in tension. Residual stresses add to stresses applied during service, through which the local load partitioning is altered. However, the actual stress distribution is a result of complex interactions between the applied and residual stresses and depends on a number of factors, including stress and strength distribution, external loading, microstructure and defects. As component failure is closely related to the local stress distribution, such investigations on inhomogeneous load sharing will not only help understand the true mechanisms for the development of material damage under the action of mechanical load but also provide valuable experimental input for simulation and operation of engineering structures.

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Using finite element modeling, residual stresses and local strength changes can be directly incorporated to study their influence or to predict the component behavior [1-3]. On the other hand, experimental studies of interactions between residual stress and applied load, either independent or in association with simulations, are mostly indirect; namely, such analyses are based on stresses measured before and after loading, from which the interactions are inferred. Experimental work that analyses the actual stress distribution across the load carrying cross section under external loadings and studies the influence of load type, strength gradient and residual stresses on the induced load partitioning is limited. Both neutrons and high energy X-rays are capable of analysing subsurface stress non-destructively in crystalline materials. With the help of a compact loading frame mounted on a diffractometer, diffraction measurements can be performed to analyse stress distributions in the cross section under in-situ mechanical loading. Such setups were successfully applied to study microscopic loading sharing in composites and polycrystalline materials [4,5] and stress distributions around crack tips [6]. In the current work, we have used in-situ synchrotron diffraction to study load partitioning between the surface and bulk for a duplex steel of type SAF 2507 having a strengthened surface with compressive residual stresses and the influence of low cycle fatigue loading. The results from in-situ tensile experiment are presented in this paper. The stress obtained from a diffraction measurement in a duplex stainless steel is related to the so-called phase specific stress, which is the sum of macrostress and the average microstress in the measured volume. While the macrostress is, by definition, the same for both phases, the microstress, originating from phase incompatibility such as thermal mismatch or different mechanical properties, is balanced between the phases. Therefore, based on the synchrotron measurements, the load partitioning on both macroscale and microscale could be analyzed.

EXPERIMENTAL DETAILS

Materials and characterization
A duplex stainless steel of grade SAF 2507 having a microstructure of austenite (γ) and ferrite (α) was used in this investigation. The chemical composition of the steel is (in weight %): 24.58 Cr, 6.64 Ni, 3.73 Mo, 0.85 Mn, 0.28 N, 0.017 C and Fe (balance). Plates with a thickness of about 6.4 mm were provided in solution treated and water quenched condition. Both surfaces of the plate were mechanically treated, a process that introduced strain hardening and compressive stresses to a layer of about 0.2 mm. The 0.2% proof stress and tensile strength were found from tensile testing to be 688 MPa and 867 MPa, respectively. Microstructure characterization by electron back scatter diffraction (EBSD) mapping in the mid-thickness of the plate revealed 55 vol. % γ and 45 vol. % α as well as a strong texture (Fig. 1). The main texture components are \{001\}<100>, \{011\}<211> and \{112\}<111> in γ but \{001\}<110> and \{011\}<100> in α.

The residual stress field in the plate was characterized by X-ray diffraction analysis on a 2 cm wide strip cut from the plate. To remove the oxide scales, a 0.015 mm layer was electrolytically polished away from the original surface in an ethanol solution of perchloric acid. Using the Cr-kα radiation, phase specific stresses in the rolling direction were measured on the γ-220 and α-211 reflections, respectively, using the \sin^2\psi method [7]. Assuming isotropic properties, the diffraction elastic constants applied are calculated using the Kröner-model (Table 1). In order to obtain the depth profile of stress, electrolytic polishing was used for successive layer removal. Stress relaxation in the removed layer was corrected according to the method proposed in [8].
Fig. 1. Electron back scatter diffraction (EBSD) image of (a) a longitudinal, (b) a transverse cross-section, with the lighter phase being $\gamma$ and darker phase $\alpha$. RD, TD and ND are the rolling, transverse and normal directions, respectively. c and d are serial sections of orientation distribution function (Bunge) for $\gamma$ and $\alpha$, respectively, derived from EBSD mapping.

Table 1 Calculated diffraction elastic constants (DEC) $\frac{1}{2}S_2$ (x10$^{-6}$ MPa$^{-1}$)

<table>
<thead>
<tr>
<th></th>
<th>$\alpha$-110</th>
<th>$\alpha$-200</th>
<th>$\alpha$-211</th>
<th>$\alpha$-220</th>
<th>$\alpha$-321</th>
<th>$\gamma$-111</th>
<th>$\gamma$-200</th>
<th>$\gamma$-220</th>
<th>$\gamma$-311</th>
<th>$\gamma$-222</th>
<th>$\gamma$-420</th>
</tr>
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<tbody>
<tr>
<td></td>
<td>5.8</td>
<td>7.7</td>
<td>5.8</td>
<td>5.8</td>
<td>5.8</td>
<td>5.14</td>
<td>8.86</td>
<td>6.07</td>
<td>7.11</td>
<td>5.14</td>
<td>7.07</td>
</tr>
</tbody>
</table>

As mentioned before, a diffraction measurement yields phase specific stress $\sigma^i$ (i is $\alpha$ or $\gamma$). Using the following equation, the macrostress acting on both phases can be derived according to

$$\sigma_{\text{Macro}} = \sigma^\alpha V^\alpha + \sigma^\gamma V^\gamma$$

(1)

where $V^i$ is the volume of phase i. The microstress balanced between the phases, $\sigma^i_{\text{micro}}$, can be calculated according to:

$$\sigma^i_{\text{micro}} = \sigma^i - \sigma_{\text{macro}}$$

(2)

The stress components are indexed as follows. $\sigma_{11}$ is the component in load direction, $\sigma_{22}$ transverse to load direction and $\sigma_{33}$ is the component acting in the direction normal to the sheet plane. For duplex stainless steels, since the stress normal to the specimen surface $\sigma^i_{33}$, may not be assumed zero, a stress component ($\sigma^i_{11} - \sigma^i_{33}$) is instead obtained from the $\sin^2\psi$ analysis using high energy synchrotron radiation. In the current work, $\sigma^i_{33}$, if it exists in the specimen, is mainly microstresses originating from thermal or mechanical mismatch between $\gamma$ and $\alpha$ during manufacturing. As they are balanced between the phases, the macrostress derived using Eq. (1) with $\sigma^i$ being replaced by the stress component ($\sigma^i_{11} - \sigma^i_{33}$) is essentially the axial stress, $\sigma_{11}$ [9].
The results of the X-ray diffraction measurements are given in Fig. 2. The depth profile of the diffraction peak width of austenite and ferrite reveals a hardened surface layer of about 0.23 mm in which compressive residual stress also prevails. Microstresses, evident by the different phase specific stresses in austenite and ferrite, are found in the hardened zone as well as in the bulk. The measured macrostress profile is characterized by a surface compressive stress of 700 MPa and a peak tensile stress of 120 MPa at 0.27 mm depth. The tensile stress seems to stabilise to about 85 MPa from 0.6 mm depth.

In-situ synchrotron experiment
The in-situ synchrotron experiment was carried out at the high energy synchrotron beamline EDDI-beamline@Bessy in Berlin using energy dispersive diffraction (EDD). The Bragg angle $\theta$ was fixed at 4.5°, and the energy spectrum used for stress analysis ranged between 36 and 110 keV. A general description of the EDD method and set-ups of the diffractometer at Bessy can be found in [10] and [11], respectively. The tensile specimen used was prepared along the rolling direction of the plate (Fig. 3). Like the X-ray specimen, a surface layer of about 0.015 mm thick and 35 mm long was electrolytically polished away from the gauge section which is 5 mm wide. Via a compact test rig, two successive tensile loading-unloading cycles were applied. While the applied loads were monitored by strain gauges attached on both side surfaces of the specimen, the evolution of phase specific stresses was measured using synchrotron diffraction. The peak strain in the first cycle was 0.5%, which is also the maximum strain used in a study of low cycle fatigue test to be reported elsewhere. The second load cycle to 0.87% strain was applied to investigate the load partitioning when the surface layer also yields. Creep, possibly accompanied by slight stress relaxation, was observed when the applied strain reached 0.4% and became more significant under larger loads. Therefore, the synchrotron stress measurements always started after the creeping had reached a stable stage.

Based on Fig.2, two locations were chosen for the in-situ synchrotron experiment. One is the very surface layer and the other 0.75 mm depth, the behavior of which can be considered to be representative for the unhardened bulk. Using the conventional reflection mode, stress information in a surface layer of about $\tau_{\text{mean}} = 0.026$ mm was obtained according to the $\sin^2 \psi$-
method of stress analysis. The information depth $\tau$ is given for the diffracted intensity being attenuated to 1/e of the primary intensity by natural absorption by the test material. The information for the individual hkl-planes considered within the analysis is given by $\tau = (\tau_{\text{max}} + \tau_{\text{min}})/2$. The mean information depth $\tau_{\text{mean}}$ characterizes the average value for all hkl-planes of the individual phases. Stress measurements at 0.75 mm depth were carried out in transmission mode (Fig. 3). In contrast to the measurements in reflection geometry, where the nominal gauge volume defined by the slits in the primary and in the secondary beam path, respectively, is located with its center of mass at the very surface, the center of mass of the gauge volume in transmission mode is located in a certain distance to the surface. The respective depth defines the information depths of the stress measurements. In the reflection measurements, the $\psi$-angle was stepped in 4° from $\psi=0°$ to $\psi=+80°$ and afterwards in 1° to $\psi=+89°$, while in the transmission measurements 20 angles, spreading evenly between $\sin^2(\psi=0°)$ to $\sin^2(\psi=+89.9°)$, were used. The diffraction elastic constants used for stress calculations are listed in Table 1.

Fig. 3 Schematic illustration of the diffraction geometry for transmission measurements. The gauge width of the specimen is 5 mm. The origin of the specimen coordinate system x-y-z is located 0.75 mm below the specimen surface. As the incident beam is along the y axis and the $\psi$-angle is defined in the x-z plane, the actual direction of measurement is moving in a plan that is inclined by 4.5° to the x-z plane.

RESULTS AND DISCUSSION

Because of the strong texture, a non-linear a (lattice constant)-$\sin^2 \psi$ distribution is observed for certain hkl-planes, which is particularly strong for measurements of the ferritic phase. By combining the lattice constants obtained from several hkl-planes, a more linear (mean) a-$\sin^2 \psi$ curve is obtained. For surface stress analysis in reflection geometry, the first four $\alpha$-reflections and the first five $\gamma$-reflections given in Table 1 were used for the calculation of the phase specific stresses. The theoretical mean penetration depth is 25 $\mu$m for $\alpha$ and 26 $\mu$m for $\gamma$. For measurements in transmission mode, the $\alpha$-110 interference lines showing only poor intensities are replaced by $\alpha$-321 in stress analysis. For the same reason, the phase specific stress of the austenite is calculated based on measurements on $\gamma$-220, $\gamma$-311, $\gamma$-222 and $\gamma$-420.
Macroscopic load sharing
The synchrotron macrostress-strain curve for both cycles is shown in Fig. 4. Uncertainties of the synchrotron stress data, i.e. the standard deviation from fitting a linear curve to the experimental $a\sin^2 \psi$ distribution, are given as error bars in the stress plots. They are in general rather small. As indicated in Fig. 4, the surface layer and bulk follow completely different loading paths. In the surface, the applied load is compensated for by the compressive residual stress. The non-linear stress variation with applied strain indicates a load transfer between the surface and other regions of the cross-section of the specimen. In the bulk, 0.75 mm below the surface, where a small tensile residual macrostress exists, the macrostress increases at first almost linearly with applied strain. With increasing applied strain, the region with tensile stress expands gradually into the surface regions where compressive residual stresses have been balanced out by the applied stress. Over 0.2% applied strain, the bulk yields and deviates from the linear loading behavior. A rapid load transfer appears, such that the macrostress increases rapidly in the surface layer that is still elastic but slowly in the bulk. At the peak strain of the cycle, a somewhat larger macrostress is found in the surface than in the bulk. Unloading to zero stress leaves a 0.2% permanent strain, and the surface is in tension. It is also clear that the occurrence of plastic deformation in the bulk relaxes the compressive stresses of the surface layer and the plastic incompatibility between different regions of the cross section introduces tensile stresses in the hardened surface layer.

![Graph showing macrostress in surface layer (0.026 mm) and bulk (at 0.75 mm depth) as a function of applied strain measured by strain gauges. c1 and c2 are cycle 1 and cycle 2, respectively.](image)

For the second loading cycle, as the bulk is now strain hardened, an almost linear behavior appears for both the surface and bulk before the peak strain of the first cycle, 0.5%, is reached (Fig. 4). Because of its initial tensile residual stress the surface layer is subjected to a higher stress than the bulk. However, there is no macroscopic load transfer occurring in the specimen and both loading curves go parallel. When the applied strain exceeds 0.5%, plastic flow starts again in the bulk, while the surface, having a higher yield strength, remains elastic. The load partitioning changes such that the tensile stress in the bulk increases very little with applied load while loading in the surface still follows a linear behavior. Over 0.62% strain, yielding also occurs in the surface layer. The surface macrostress seems to increase at a larger rate than the bulk macrostress, which could be attributed to a different strain hardening rate between the two regions. However, given that the loading step is rather large, the difference could also be an
artificial effect caused by yielding of the surface at a higher load than 0.62% at which the synchrotron measurement was made. At the peak strain of the second cycle, the surface stress (1070 MPa) is much higher than the bulk stress (680 MPa). This difference in macrostress, which is caused by plastic incompatibility, remains as tensile residual stress in the surface after unloading that leaves a plastic strain of 0.54%.

Load sharing between the phases
The measured phase specific stresses ($\sigma_{11}^i - \sigma_{33}^i$) are shown in Fig. 5 for the bulk and surface layer. Both cycles are plotted in the same figures, however, for a clear illustration of the microstress evolution with applied load, two data points, namely unloading at 0.2% in cycle 1 and loading at 0.5% strain in cycle 2 are excluded. The exact contribution of the residual stress component, $\sigma_{33}^i$, is not known. Nonetheless, as the phase interactions that cause microstresses are most significant in the loading direction, the observed evolution of phase specific stresses can be considered reflecting mainly the influence of the applied load and phase incompatibility induced microstresses on the axial stress, $\sigma_{11}^i$. Obviously, the microscopic load partitioning is also inhomogeneous and dependent of the initial residual stresses and the phase property ratio, as often observed for such materials [5,9,12]. In the bulk (Fig. 5.a), $\gamma$ having a high tensile residual stress yields first, at 0.2% strain, which leads to load transfer from $\gamma$ to $\alpha$. At the 0.3% when plastic deformation starts in the ferrite, the ferrite shows a higher tensile stress than the austenite and further loading hardly affects the microstresses balanced between the phases. In the surface layer, the phase microstresses vanish rapidly during initial loading to 0.2% strain but increase again over 0.38% strain (Fig. 5.b). Yielding may have occurred first in the ferrite, resulting in a load transfer to the austenite. In the current experiment, the diffraction peaks do not show a significant peak broadening for either the surface or the bulk measurements.

![Fig. 5 The evolution of phase specific stresses ($\sigma_{11}^i - \sigma_{33}^i$) in the bulk (a, 0.75 mm depth) and in the surface layer (b, 0.026 mm) with applied strain as measured with strain gauges.](image)

**SUMMARY**

The partitioning of applied tensile load in a flat specimen of SAF 2507 with a strain hardened surface layer and high surface compressive residual macrostresses was investigated. In-situ synchrotron measurements in the near surface region at a mean information depth of about 0.026 mm and in the bulk material at 0.75 mm depth clearly illustrate an inhomogeneous load.
distribution which is related to the different strength and residual stress state and varies with applied load. The main findings are summarized below.

1. The surface layer and bulk follow different loading paths. During macroscopic elastic loading, the bulk shows increased tensile stress with applied strain, while the applied stress is compensated for by the compressive residual stress in the surface layer.
2. By the occurrence of bulk yielding a significant load transfer from the bulk to the hardened surface layer starts. The surface stress increases rapidly and eventually exceeds that of the bulk. Such a load transfer is reduced or may stop when the surface layer also undergoes plastic deformation.
3. The difference in strength leads to an inhomogeneous applied stress distribution when the bulk has become plasticized. At 0.54% plastic strain, the surface stress reaches 1070 MPa and the bulk stress 680 MPa. This macrostress difference induced by macroscopic plastic incompatibility remains as a tensile residual stress in the surface after unloading.
4. Inhomogeneous load distribution on a microscale is also observed in the bulk and maybe in the surface.
5. In-situ diffraction stress analysis using high energy synchrotron radiation offers a potent tool to extract information about the load partitioning between different regions as well as between constituent phases within the material.

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