TEM STUDY OF DISLOCATIONS IN DUPLEX STAINLESS STEEL

ŠTIJUDA DISLOKACIJ V DUPLEKSNEM NERJAVNEM JEKLU S PRESEVNO ELEKTRONSKO MIKROSKOPIO

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Vzorci dupletskega nerjavnega jekla (DSS, tip zlitine 258) so bili izotermno starjeni (starani) pri temperaturah 300 °C in 350 °C v trajanju 10000 h in 30000 h. Med toplotnim staranjem so se v materialu pojavile spinodalne razrede ter zmanjšali raztezanje in zarezno `ilavost. Zato je bila izvedena `utrditev s presevnim elektronskim mikroskopom (TEM) in morfologijo in gostoto dislokacij.

Keywords: duplex stainless steel, ferrite, austenite, spinodal decomposition, transmission electron microscopy, dislocations

1 INTRODUCTION

Many metallic alloys, including duplex stainless steels (DSSs), consist of austenite (γ-Fe) and ferrite (α-Fe). The two phases have different crystal lattices, austenite having a face-centred cubic lattice (fcc) and many slip-plane systems that prevent cleavage, and ferrite having a body-centred cubic lattice (bcc) that is more easily deformable than austenite, with a propensity for cleavage. The susceptibility to cleavage is greater at lower temperatures, increasing with an addition of certain alloying elements such as chromium, silicon, phosphorus, etc., and it is higher for a coarse-grained microstructure.

Nowadays, DSSs are used in a wide range of industries, especially in the oil and gas, petrochemical, paper and nuclear industries, because of their excellent combination of mechanical properties and corrosion resistance. Austenitic Fe-NiCrMo alloys have high toughness and good resistance to oxidation and scaling.² An addition of Ni, Cr and Mo to iron alloys suppresses the polymorphic transformation. In the alloys containing high amounts of Cr, the solid solution of Cr and Ni or Co in α-Fe is not stable³ and it decomposes with a spinodal process up to 750 °C.³⁴ The ferritic-austenitic DSS possesses a specific microstructure, where austenite and ferrite may create internal stresses during the deformation due to their elastic and plastic differences.⁵

The localization of the plastic strain is a typical feature of the fatigue of metallic materials. One of the basic classes of crystal defects is the linear defect, consisting of dislocations, where the rows of atoms are in irregular positions. There are two general types of dislocation: the edge (the Burger vector is perpendicular to the dislocation line) and the screw (the Burger vector is parallel to the dislocation line) dislocations. In 1934 Taylor and Polanyi confirmed dislocations as the carriers of plastic deformation.⁶,⁷ Dislocations have the strain fields arising from the distortions at their cores – the strain drops radially with the distance from the dislocation core. Edge dislocations introduce compressive, tensile and shear lattice strains. Screw dislocations introduce the shear strain only. According to Roters, Raabe and Gottstein⁸, there are three dislocation classes: the density of mobile dislocations travelling through a cell structure (ρm), the immobile dislocation density inside the cells (ρi), and the density of immobile dislocations in the cell walls (ρw).

The aim of our research was to investigate ferrite and austenite grains using transmission electron microscopy...
TEM or high-resolution TEM (HRTEM) with energy-dispersive X-ray spectrometry (EDXS) and to observe dislocations (the kind of dislocations and their locations in the specimens) in austenitized (non-aged) and annealed (aged at 300 °C and 350 °C for 10000 h and 30000 h) DSS, the 258-alloy type.

2 EXPERIMENTAL WORK

The specimens of DSS (the 258-alloy type) were austenitized at 1100 °C for 6 h, water quenched and then isothermally annealed (aged) at 300 °C and 350 °C for 10000 h and 30000 h. Thin foils of specimens were prepared using argon ion slicing with a JEOL EM-09100IS Ion Slicer9,10 or electrochemical thinning with the final application of jet electropolishing.

The preparation of thin foils by means of argon ion slicing using an ion slicer is a novel method that enables a quick preparation of high-quality specimens for TEM. The specimens with a thicknesses of around 500 μm were cut out from the bulk material, having a rectangular shape of (0.5–1.0) mm × 2.8 mm (a bulk cross-section preparation), then thinned to less than 100 μm with the grinding paper SiC 800 (the grain size of around 22 μm), and further thinned with an argon ion beam. The thinning process started at the pressure of 10–5 Pa or 10–4 Pa and alternated between the front and backside of the rotating specimens. The beam was tilted between 1.0° to 2.5°. The accelerating voltage between 4.0 kV and 6 kV, the argon-gas-flow rate between 7.1 and 7.5 (arbitrary units) and the side change interval of 30 s or 60 s were chosen. After a large thin area of the specimens up to (300–500) μm × 700 μm was obtained, a small hole was made in the thinnest region of the specimens. Polishing was done at the tilt angle of 0.5°, at the accelerating voltage of 2 kV and the side change interval of 15 s or 40 s for 7 min, 10 min or 15 min. The total time of thinning was up to 9 h and 40 min.

The other method of thin-foil preparation consisted of diamond-saw cutting of about slices 0.3 mm, mechanical grinding, electrochemical thinning, punching out diameter 3 mm thin disks and final jet electropolishing with a Struers Tenupol-5 device.

After the thin-foil preparations, the non-aged and aged specimens were examined with TEM (JEOL JEM-2100) at the electron accelerating voltage of 200 kV using conventional TEM (CTEM), high-resolution TEM (HRTEM), electron diffraction (ED), and an energy-dispersive X-ray spectrometer (EDXS, JED-2300T).

The results of the inductively coupled plasma atomic emission spectroscopy (ICP-AES) bulk average chemical composition in mass fractions (w/%) of the starting (non-aged) material of DSS, the 258-alloy type, are shown in Table 1. The base metal is iron.

3 RESULTS AND DISCUSSION

Thin foils for the investigations of non-aged and aged DSS (the 258-alloy type) were studied by means of TEM in order to define ferrite and austenite phases and to observe dislocations.

Dislocations in the non-aged DSS are shown in Figure 1. An HRTEM image and EDXS spectra of ferrite and austenite in the same material are shown in Figures 2 and 3, respectively. The insets are fast Fourier transforms (FFTs). The disorientation that is slightly less evident in Figure 2 (the α-phase) may be an indication of the size of the spinodal domain. This is yet to be investigated. The elemental composition of the non-aged DSS determined with the TEM/EDS analysis is shown in Table 2. Ferrite has a higher content of Cr than austenite and austenite has a higher content of Ni than ferrite. The CTEM bright-field images of dislocations in the aged (annealed) DSS are shown in Figure 4.

An example of the interactions of electrons and elastic stresses at the grain boundary between ferrite and austenite in one of the aged DSS specimens (350 °C for 30000 h) is shown in Figure 5. The elemental composition of the small areas of the specimen determined with the TEM/EDS analysis is shown in Table 3. Compared to the non-aged specimen (Table 2), the aged specimen

<table>
<thead>
<tr>
<th>C</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Mn</th>
<th>Si</th>
<th>Cu</th>
<th>Co</th>
<th>Nb</th>
<th>P</th>
<th>S</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.033</td>
<td>22.28</td>
<td>9.92</td>
<td>2.34</td>
<td>0.91</td>
<td>0.86</td>
<td>0.26</td>
<td>0.06</td>
<td>0.051</td>
<td>0.031</td>
<td>0.003</td>
<td>0.051</td>
</tr>
</tbody>
</table>

Figure 1: CTEM bright-field image of dislocations in the non-aged DSS, 258-alloy type
Slika 1: Posnetek CTEM v svetlem polju prikazuje dislokacije v nestaranem DSS, tip zlitine 258
has a slightly different composition: its ferrite has a slightly higher content of Mn, Ni, Mo and Si, and a lower content of Cr and Cu, while the austenite has a slightly higher content of Mn and Cu, and a lower content of Ni, Cr, Mo and Si.

Spinodal decomposition occurs during thermal ageing of this type of material. It is characterized by the formation of a nanocellular microstructure of ferrite domains with the austenite regions enriched with Cr and the other alphagene alloying elements as well as the regions enriched with Co, Ni and the other gammagene alloying elements. Because of the difference in the local chemical composition, the lattice parameters are modified and their accommodation creates elastic stresses that increase the hardness and change the mechanical properties (the tensile strength increases, while the ductility and toughness decrease). The change in the mechanical properties may also be related to the changes in the material’s internal structure (stacking faults, the morphology and density of dislocations). It has been established that the density of dislocations may have increased during the ageing at 300 °C and that these dislocations appeared in different configurations with many of them being substantially mobile (numerous side trails and traces of dislocations escaping to the foil surface) (Figure 4), while many other dislocations became immobile.
after the ageing at 350 °C (Figure 4) when the transformation of the matrix occurred (Figure 6), leading to a virtually equilibrium state according to the effect of the ageing temperature on the change in the Charpy notch sharpness. Spinodal decomposition is faster at a higher temperature because of a greater diffusion rate. Due to spinodal decomposition, elastic stresses are generated at the austenite/ferrite phase boundary. Dislocations may be slowed down in their movements or become immobile because of the grain (phase) boundary or obstacles. However, until now, we have not been able to detect such

**Table 3:** Elemental composition of ferrite and austenite in the aged (annealed at 350 °C for 30000 h) DSS, 258-alloy type, determined with the TEM/EDXS analysis

<table>
<thead>
<tr>
<th>Element</th>
<th>Energy (keV)</th>
<th>ferrite Mass (%)</th>
<th>Error (%)</th>
<th>austenite Mass (%)</th>
<th>Error (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>1.739</td>
<td>0.55</td>
<td>0.31</td>
<td>0.46</td>
<td>0.25</td>
</tr>
<tr>
<td>Cr</td>
<td>5.411</td>
<td>27.13</td>
<td>0.01</td>
<td>20.31</td>
<td>0.01</td>
</tr>
<tr>
<td>Mn</td>
<td>5.894</td>
<td>2.28</td>
<td>0.12</td>
<td>1.40</td>
<td>0.14</td>
</tr>
<tr>
<td>Fe</td>
<td>6.398</td>
<td>59.85</td>
<td>0.00</td>
<td>63.77</td>
<td>0.00</td>
</tr>
<tr>
<td>Ni</td>
<td>7.471</td>
<td>6.27</td>
<td>0.05</td>
<td>11.67</td>
<td>0.02</td>
</tr>
<tr>
<td>Cu</td>
<td>8.040</td>
<td>0.63</td>
<td>0.64</td>
<td>0.56</td>
<td>0.48</td>
</tr>
<tr>
<td>Mo</td>
<td>2.293</td>
<td>3.29</td>
<td>0.20</td>
<td>1.82</td>
<td>0.24</td>
</tr>
<tr>
<td>Total</td>
<td>100.00</td>
<td>100.00</td>
<td></td>
<td></td>
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</tr>
</tbody>
</table>
obstacles (inclusions) with TEM/EDXS. This should also be studied, in detail, in the future, possibly with a better TEM resolution. Further investigations (TEM/EDXS) will enable a better understanding of the characteristics of spinodal decomposition and the change in the compositions of both domains. They will allow us to explain the real rearrangement of the alloying elements and the formation of the eventual new phases during spinodal decomposition. As a result, we will be able to calculate the extent of the internal stresses.

4 CONCLUSIONS

A small difference caused by the change in the ageing temperature was observed. The density of dislocations in the specimens of the non-aged and aged duplex stainless steel (DSS, the 258-alloy type) increased after the ageing at 300 °C and these dislocations appeared in different configurations with many of them being substantially mobile. After the ageing at 350 °C the dislocation configurations changed and many dislocations became immobile (or at least slowed down in their movements) when the transformation of the matrix occurred.

Acknowledgments

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5 REFERENCES