Microstructural and magnetic investigations of duplex steel

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Abstract. In this work, the eutectoid decomposition of δ-ferrite was investigated in 2507 type super duplex stainless steel. Samples were cold rolled in different extents and then heat treated at different temperatures. The ferrite contents of the specimens were determined by magnetic measurements and electron backscatter diffraction. The microstructure was examined by optical microscope and electron backscatter diffraction. The results showed that the cold rolling before the heat treatment significantly increases the rate of eutectoid decomposition and decreases the starting temperature of the ferrite decomposition. The measured ferrite contents with various methods showed diverse results because of the different principles of the measuring methods.

1. Introduction

DSS combines the advantages of the austenitic and ferritic steels thereby the microstructure of this corrosion resistant steel contains about 50-50% austenite and δ-ferrite. This coupling effects that the strength of the steel significantly increases and the corrosion resistance raises mainly in chloride ion containing environments. The yield strength of duplex stainless steel (DSS) can be two or three times higher than the yield strength of the traditional austenite steel.

The carbon content is limited strongly to avoid the formation of harmful precipitations. The aim of the chromium alloying is to increase the corrosion resistance, the nickel makes the steel tougher. Nitrogen can raise the strength and revises the resistance against the pitting corrosion. DSS is usually alloyed by molybdenum, tungsten and copper too [1, 2].

The austenite ensures good ductility, toughness and weldability until the δ-ferrite increases the resistance against the pitting, crevice and stress corrosion. Corrosion resistance of DSS can be determined by the pitting resistance equivalent number (PREN) which depends on the chemical composition. In case of super-duplex stainless steel (SDSS) the PREN index exceeds 40 [1, 2].

The upper application temperature of DSS is about 280-325°C. If the temperature is higher than this value, precipitations (intermetallic phases) can appear which cause the embrittlement and the reduction of the corrosion resistance of the material. The damageability of steels was simulated earlier [3]. The most important phase transformation in DSS is the eutectoid decomposition. During this process, the δ-ferrite transforms into σ phase and secondary austenite ($\delta \rightarrow \sigma + \gamma_2$). The σ-phase can emerge between 600-1000°C and the chance of its appearance increases if the amount of the alloying elements is higher. It shows up mainly in SDSS. The χ-phase can turn up at 700-850°C. The $M_{23}C_6$ and M_7C_3 are high chromium content complex carbides. The Cr_2N has a hexagonal lattice and it settles down at the grain boundary of the δ-ferrite. The R-phase and π-phase appear between 550-650°C and these can cause significant embrittlement [4-8].

There are several publications which deal with the eutectoid decomposition and the formation of the σ-phase in SDSS but there is just limited information about how these transformations influenced by previous plastic deformation. The aim of this study was to investigate the changing of the process of the eutectoid decomposition in 2507 type SDSS if the samples were plastic deformed in different extents before the heat treatment.

2. Sample preparation

Samples were prepared from the basic material which sizes were 10×15×100mm. The specimens were cold rolled in 6 different extents in longitudinal direction at room temperature. Values of the rolling reductions were 10, 20, 30, 40, 50, 60% and there was a sample series without deformation. 5-5 samples were rolled in case of every deformation rates.

After the cold rolling the specimens were heat treated during 30 minutes at 700°C, 750°C, 800°C, 850°C and there was a sample series without heat treatment. After rolling every specimen were milled at same geometry which was 3,4×10×100mm.

Table 1. shows the chemical composition of the 2507 type SDSS.

Table 1. Chemical composition of the 2507 type SDSS (%)

| C	Mn	P	S	Si	Cu	Ni	Cr	Mo	Nb	Ti	N
0,021	0,822	0,023	0,0004	0,313	0,178	6,592	24,792	3,705	0,008	0,005	0,264

3. Experiments

The ferrite content of the samples was firstly determined by an alternating current (AC) magnetometer as it was reported in our previous publication [9].

In the present paper the results of the Stablein-Steinitz direct current (DC) magnetometer, optical microscopic and electron backscatter diffraction (EBSD) measurements were summarized.
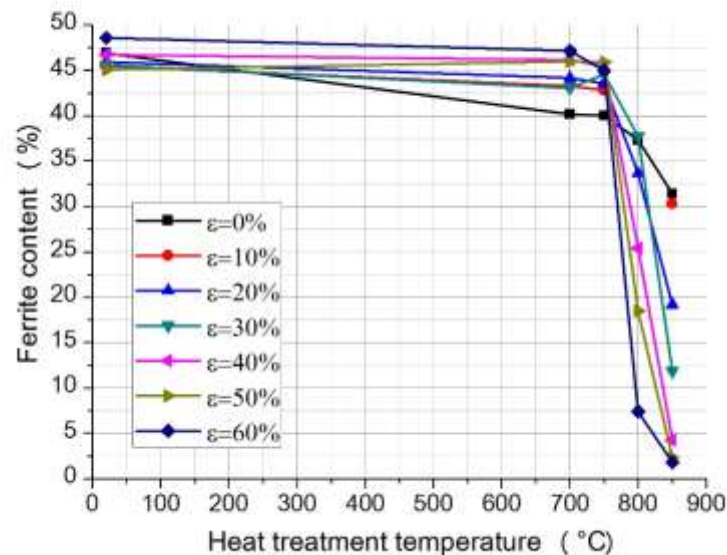
3.1. DC magnetometer measurement

The samples were measured by the DC magnetometer which is an especially suitable method to determine the ferrite content of samples. The Stablein-Steinitz DC magnetometer requires bulk samples so the longish, rolled specimens which were measured by the AC magnetometer were cut into smaller pieces and fixed to cubic shape. Fig. 1. shows the sketch of the applied Stablein-Steinitz DC magnetometer. This set-up contains a symmetrical yoke which stands two U-shaped parts and a cross bridge. There are four excitation coils on the main arms of the yoke. The set-up consists two Hall sensors, a reference and a measuring air gap too. If there is no sample in the measuring air gap the arrangement is symmetrical therefore there is no flux through the cross bridge. If the sample is put in the measuring air gap the symmetry ends and some part of the flux closes across the cross bridge. The Hall senor in the cross bridge measures this magnetic field which is proportional with the magnetization of the sample.
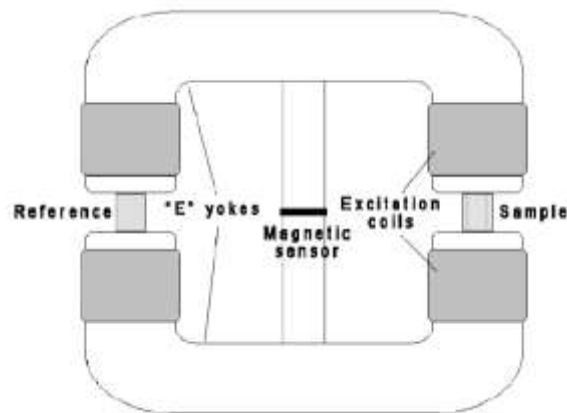
Figure 1. Sketch of the Stablein-Steinitz DC magnetometer

The set-up recorded the saturation magnetic hysteresis loop of the samples. The value of the saturation polarization which can be determined from the hysteresis loop are known to be proportional to the ferrite content of the sample [10]. Fig. 2. represents the calculated ferrite contents in function of the heat treatment temperature and the deformation rate. It can be seen that the ferrite content reduction is more moderate by the undeformed and less deformed samples. The ferrite content suddenly drops at 750°C by the strongly rolled samples. This means that the previous cold rolling before the heat treatment significantly influences the eutectoid decomposition. The stronger the deformation rate is the more active the eutectoid decomposition is. Much more paramagnetic σphase and secondary austenite begin to appear in effect of the heavy deformation which reduces the ferrite content appreciably. On the other hand, the previous cold rolling seems to decrease the starting temperature of the eutectoid decomposition as well.


Figure 2. Results of the DC magnetometer measurement

The ferrite contents measured by the DC magnetometer were compared with the results of the AC magnetometer measurement. The details and results of the AC magnetometer measurement had already been published in a previous paper [9]. It was found that the AC magnetometer measured lower ferrite values than the DC magnetometer. This difference can be explained by the significantly

different excitation levels. The DC magnetometer is able to excite the whole volume of the samples to their magnetic saturation in contrast to the AC magnetometer.

3.2. Metallographic investigation with optical microscope

All samples were investigated by metallography using optical microscope. The specimens were fixed in resin, grinded, polished and prepared by Beraha etch. Fig. 3. shows the microscope pictures in case of different deformation levels and different heat treatment temperatures. The magnification of the microscope was 1000×. The etching time of the samples were different for achieving the best image quality. It caused the diverse colour of the same phases on the different samples. The dark area (brown and green) demonstrates the ferrite (δ), the bright area is the austenite (γ). σ-phase, ferrite and austenite are indicated on the Fig. 3.

The pictures demonstrate obviously that the previous cold rolling can significantly influence the eutectoid decomposition. The heat-treated samples on 850°C show most expressively this phenomenon. The stronger the extent of the previous cold rolling is the more δ-ferrite transforms mainly into σ-phase and secondary austenite. It can be seen the decomposition of δ-ferrite appears slightly in case of the ε=20% deformed sample. However, the decomposition of the δ-ferrite is almost complete at the sample which belongs to the highest deformation level (ε=60% and heat treated at T=850°C) in good agreement with the DC magnetometer results.
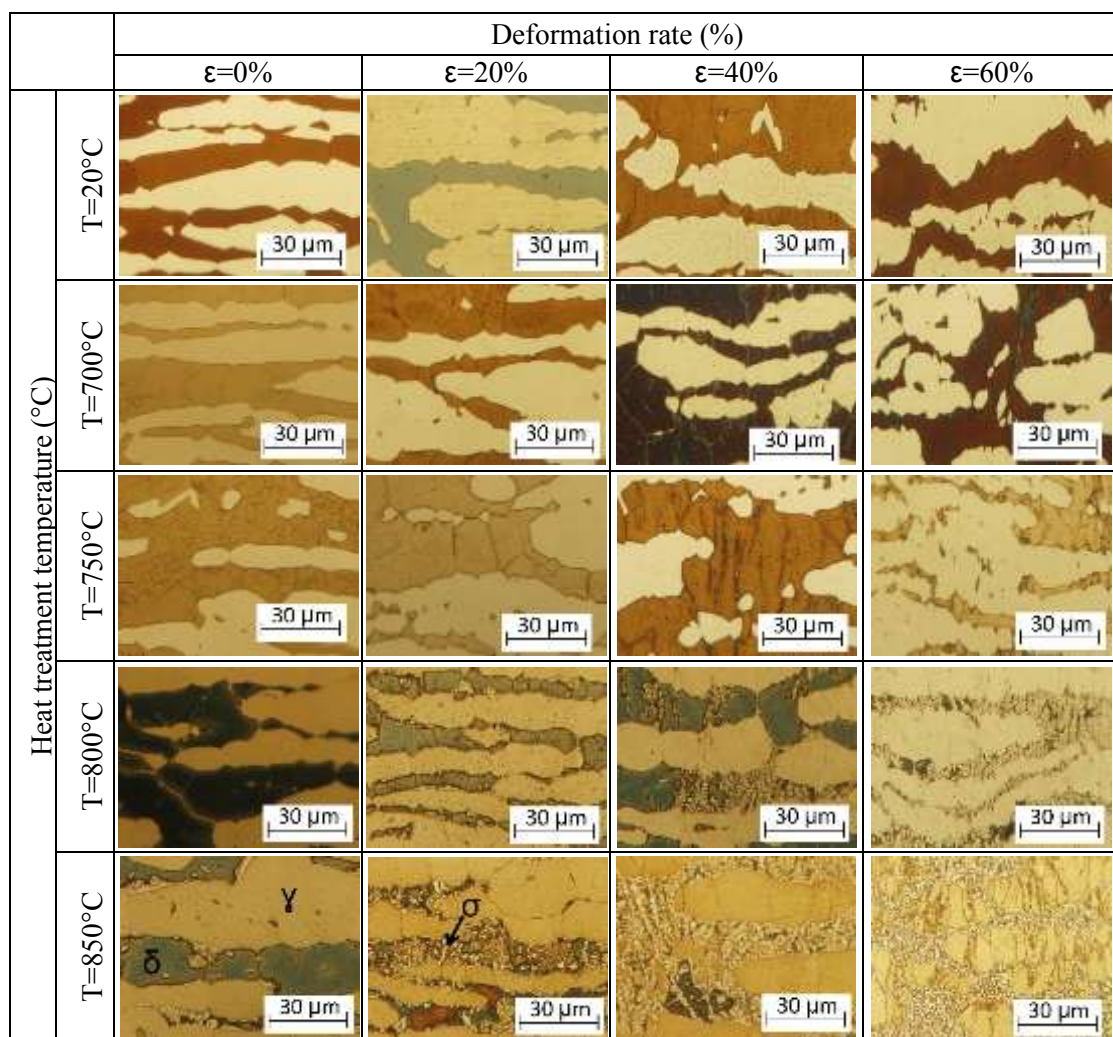
Figure 3. Microstructural images of 2507 type SDSS (magnification of the microscope was 1000×)
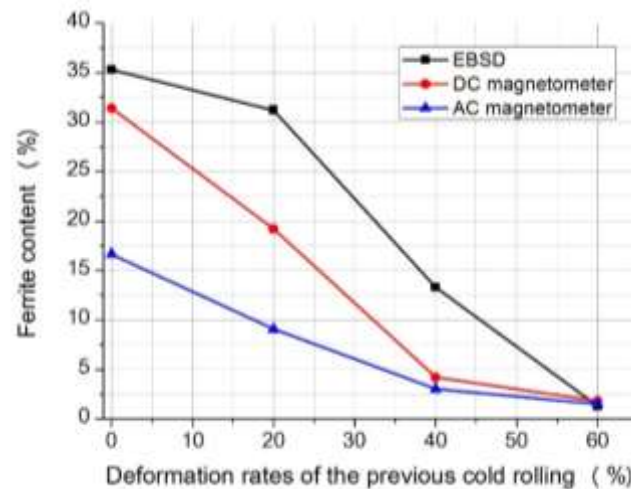
3.3. EBSD measurement

Samples were investigated by electron beam scattering diffraction technique (EBSD) as well. Fig. 4. represents the EBSD phase maps of four samples. All the four samples were heat treated at 850°C and the deformation rates are the following from left to right in the top line: ε=0%, ε=20% and in the bottom line: ε=40% and ε=60%. The red, green and yellow fields indicate the δ-ferrite, austenite and σ phase respectively.

The EBSD phase maps show similar results as the optical microscope pictures. If the deformation rate is stronger the amount of the σ-phase increases, because the previous cold rolling before the heat treatment decreases the starting temperature of the eutectoid decomposition.

The EBSD phase map is suitable also to determine the phase ratio. The EBSD showed different ferrite contents than the DC and the AC magnetometers. Fig. 5. compares the ferrite values of the EBSD, DC and AC magnetometer measurements. It can be seen that the difference is remarkable, the ferrite contents are far higher at the EBSD than at the DC and AC magnetometer measurements. The lowest ferrite values were showed by the AC magnetometer. The reason of this difference can be that the EBSD is a surface measurement which uses a two-dimension picture to calculate the ferrite values in contrast to the DC and AC magnetometers which are volumetric measurements. [11]
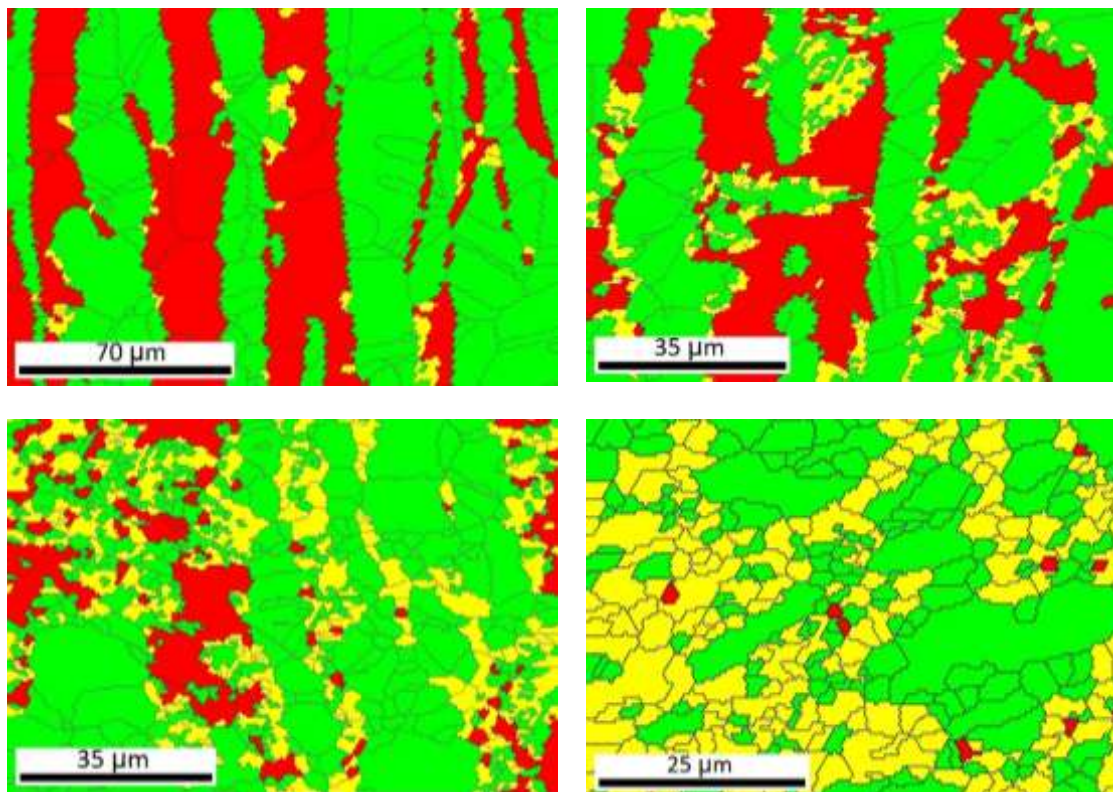
Figure 4. EBSD phase maps of cold rolled (upper line left side ε=0%, upper line right side ε=20%, lower line left side ε=40% and lower line right side ε=60%) and heat treated samples at 850°C

Figure 5. The difference of the ferrite values between the EBSD,
DC and AC magnetometer measurement

4. Summary

The main goal of this study was that to investigate the eutectoid decomposition of ferrite phase in the 2507 type SDSS. The samples were plastic deformed in different extents before the heat treatment. The extents of the cold rolling were 0, 10, 20, 30, 40, 50 and 60% which was followed by an isothermal heat treatment at the temperatures of 20, 700, 750, 800 and 850°C.

Three methods were used to study the structural changes due to the decomposition of ferrite phase. Magnetic ferrite content measurements, metallography and EBSD were applied. The results of all these measurements demonstrated expressively the decomposition of the δ-ferrite into σphase and secondary austenite. The obtained results are in good agreement in the tendency of the process. The followings were proved by all the three methods;

- the previous cold rolling before the heat treatment significantly increases the rate of eutectoid decomposition,
- the previous cold rolling decreases the starting temperature of the eutectoid decomposition.

Naturally, some differences were also observed among the three applied measurements. The DC magnetometer detected higher ferrite values than the AC magnetometer measurement because only the DC magnetometer can excite the samples into magnetic saturation.

The ferrite contents determined by EBSD and DC magnetometer are different. The EBSD seems to be detected higher ferrite amount than the DC magnetometer. This difference is caused by the different principles of these measuring methods. The EBSD is a surface (2D) while the DC magnetometer is volumetric (3D) measurement. The EBSD derives the phase ratio from the tested surface area in contrast to the DC magnetic measurement which measures the total volume of the sample.

Finally, it must be stressed that magnetic measurements, the metallography and the EBSD supplied useful and coherent information about the details of the eutectoid decomposition of the ferrite in the studied duplex stainless steel.

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