MECHANICAL AND THERMAL INDUCED PHASE TRANSFORMATIONS IN SUPERDUPLEX STAINLESS STEEL

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ABSTRACT

The aim of this work is to study the microstructural changes in SAF 2507 type superduplex stainless steel due to heat treatments and mechanical fatigue process. Specimens were heat treated in the 400-1360 °C temperature range for 1 and 5 hours respectively. An other series of specimens were periodically loaded by using a fatigue testing machine. The microstuctural changes were investigated by using a complex micromagnetic measuring system which includes a Barkhausen noise measuring and a magnetic harmonic analysing system. The RMS value of the Barkhausen noise, the distortion factor (k), and magnetic coercivity values were measured. The microstuctural changes were investigated by metallography using scanning electron microscope.

KEYWORDS

duplex stainless steel, heat treatment, fatigue process, Barkhausen noise, harmonic analysis, micromagnetic measurement

INTRODUCTION AND BACKGROUND

Development of duplex stainless steels significantly broadened the application possibilities of stainless steels. The corrosion resistance of duplex stainless steels much better compared to the traditional austenitic especially in chloride ion containing surroundings. Moreover, the large yield stress of duplex steels offer significant advantages in structural applications. The so called superduplex stainless steels have the superior corrosion resistance and mechanical properties as well. There is intensive research activity on weldability because the welding and the thermal load naturally, have strong effect on the microstructure [1, 2]. The goal of these research works is to clarify the connections of precipitation processes in δ-ferrite + austenite (γ) structures which happen due to thermal effects [3]. The segregation and precipitation processes are normally divided into two large thermal ranges, these are the 300-600 °C and 600-1000 °C. In the 300-500 °C interval the Ni- and Si-rich G-phase and the Cr- and Mo-rich α′-phases come into being. At about 600 °C the appearance of Mo-rich Laves-phase is typical. These phase transformations significantly modify the properties like in case of the embrittlement at 475 °C [4].

At higher temperatures the appearance of Cr-nitrides, Cr- and Mo-rich σ-phase, chi-phase (χ) and secondary austenite (γ_2) is typical which can be observed by optical microscopic and SEM investigation as well. Usually, the mechanism of these processes the eutectic decomposition of δ-ferrite ($\delta \rightarrow \sigma + \gamma_2$). The γ_2-phase can appear due to Widmännstatten-type or martensitic transformation as well. Naturally, these phase transformations strongly effect the amount of δ-ferrite phase. According to the TTT diagrams the shortest incubation time of the segregation processes is at around 450 °C and 900 °C respectively.

In the present work SAF 2507 type superduplex stainless steel was studied using magnetic Barkhausen-noise measurement, harmonic analysis (HA) and metallographic techniques. The work has two separated parts.

In the first part the effect of heat treatments was studied on the amount and microstructure of δ-ferrite. The usage of magnetic measurement techniques are specially useful at this field because, as it was mentioned before, the segregation and precipitation processes mainly result submicroscopic phases which can be detectable by magnetic techniques.

In the second part the effect of fatigue load was investigated on the magnetic properties and the microstructure.

MATERIAL

SAF 2507 type superduplex steel was tested. The experimental material was in its original cold worked state, containing austenite and about 40 % δ-ferrite. According to the micro-hardness measurement results there is no significant difference between the hardness of ferrite and austenite phases. Table 1. contains the data of the nominal composition of the bulk material and the composition (measured by EDS-analyser) of phases.

Table 1. The chemical composition of the investigated steel and its phases . (In mass %.)

	Fe	Cr	Ni	Mo	Si	Mn	Cu	C	N
SAF 2507 nominal	Balance	25	7	4	0.8	1.2	0.5	0.02	0.3
Austenite	63.27	23.20	8.62	2.73	0.51	1.15	0.53		
Ferrite	62.34	26.12	5.59	4.21	0.44	0.7	0.6		

MICROMAGNETIC MEASUREMENTS

The detailed description of the magnetic measuring arrangement used for the Barkhausen-noise measurement and for the harmonic analysis can be find in our earlier work [5]. The flat surfaced specimens were measured by a mobile measuring head which contains a magnetising and a detector coil. The magnetising coil was supplied by sinusoidal current with a frequency of 10 Hz and 20 Hz respectively. The applied magnetic field strength exceeded the irreversible domain wall displacement range of the hystersis loop. The signal of the detector coil was amplified. The amplification was 200 in case of Barkhausen measurement and it was 10 for harmonic analysis. The amplified signal was digitised and analysed by an Eckelman-Krenz TRB 4000 type signal analysing unit. The RMS value of the Barkhausen-noise was used to characterize the structural changes. A PC software was used to calculate the Fourier-components of the signal. The odd harmonics of the signal were used to characterize the magnetic properties of the ferromagnetic phase induced by microstructural changes.

The amplitudes of the first, third, fifth and seventh harmonics were measured. In both cases the sampling frequency of the signal processing unit was 100 kHz.

The Barkhausen-noise is influenced by the ferromagnetic ratio of the alloy and the mobility of magnetic domain walls. The inclusions, precipitates as obstacles can delay or hinder the movement of domain walls. In contrary, the distortion factor (k) is mainly influenced by the amount of the ferromagnetic phase. The k-factor is calculated by the following equation:

$$k = \sqrt{\frac{A_3^2 + A_5^2 + A_7^2}{A_1^2}}$$

Where: A_1 is the amplitude of the first harmonics,
A_3 is the amplitude of the third harmonics,
A_5 is the amplitude of the fifth harmonics,
A_7 is the amplitude of the seventh harmonics.

EFFECT OF HEAT TREATMENTS ON MICROSTUCTURE

Isothermal heat treatments were done in the temperature range of 400-1360 °C. The specimens in the first series were kept at constant temperature for 1 hour, in the second series for 5 hours. The heat treatment was followed by the fast cooling in water (quenching).
The specimens were investigated by metallography, Barkhausen-noise measurements and harmonic analysis.

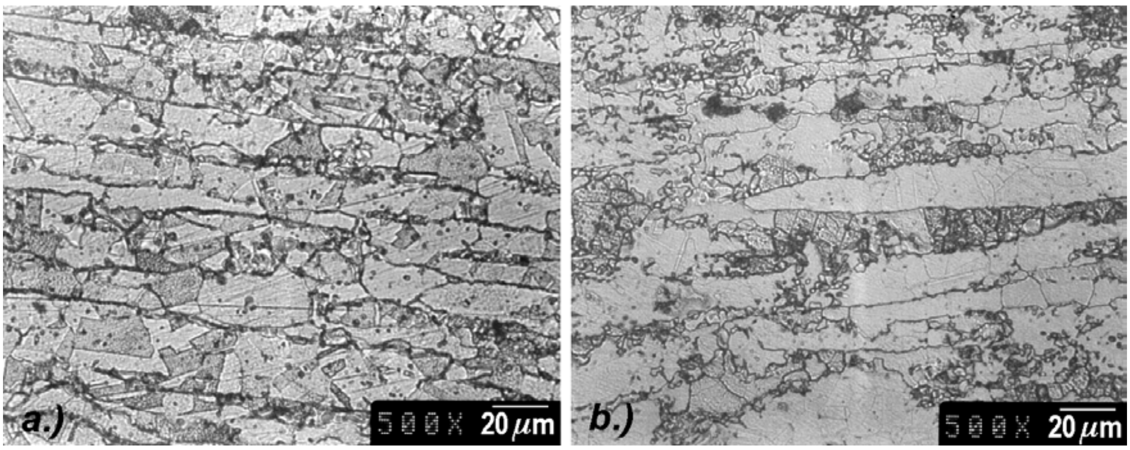

Fig 1. Microstructure of the rolled sample after 500 °C (a.) and 700 °C (b.) heat treatments

In the specimens which were heat treated at 500 °C there was no significant change in the ratio of ferromagnetic ferrite and paramagnetic austenite phases. At 500 °C decrease of ferrite grain size started which was continued with increasing temperature as it can be seen in Fig 1.a and 1.b. This microstructural change of ferrite is enhanced by the intercrystalline and intracrystalline inclusions as well, which results the decrease of amount of the ferromagnetic ferrite phase. It is visible in Fig 1.b that the eutectoid decomposition of ferrite is started which results the appearance of σ-phase.

This Fe-Cr-Mo intermetallic compound can be investigated after etching by Groessbeck-agent. The amount of δ-ferrite was measured after etching in 40% KOH solution.

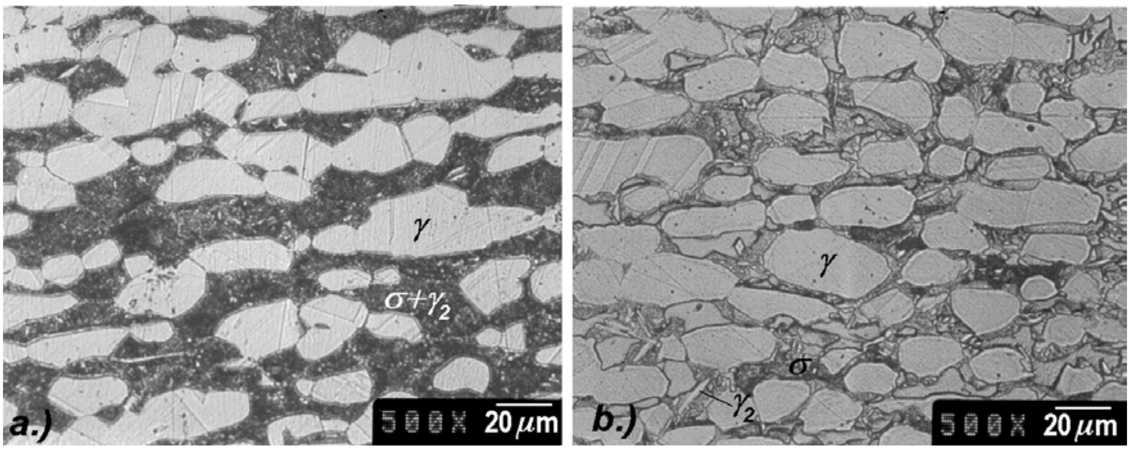

Fig 2. Microstructure after 800 °C (a.) and 900 °C (b.) heat treatments.

It clearly seems that the decomposition of δ-ferrite is over in Fig 2.a and 2.b. The structure of the σ-γ_2 eutectoid (which appeared at 800 °C) is fine, the morphology of secondary austenite is basically eutectoid type (Fig 2.b). At 900 °C another form of the secondary austenite came into being, it is the Widmannstätten-type needle like structure (Fig 2.b) [6, 7].

The segregation processes significantly modified the concentration distribution of alloying elements. The local concentrations of the specimen which was heat treated at 900 °C can be seen in Table 2. It demonstrates that the appearance of σ-phase causes the concentration increase of Mo and Cr and results Ni-depletion. In contrary, the appearance of secondary austenite causes enrichment of Ni and Cu.

Table 2. The chemical composition of different phases formed in 900 °C (In mass %.)

	Fe	Cr	Ni	Mo	Si	Mn	Cu
Secondary austenite, γ_2	65,13	21,72	8,37	2,22	0,51	0,96	1,11
σ-phase	56,46	29,49	4,60	7,62	0,59	0,84	0,40

Fig 3, 4, and 5 show the results of magnetic measurements. Up to the 400 °C temperature the applied methods could not detect any structural change. At 400 °C the BN level started to decrease meanwhile the k-factor and the coercivity remained at their original level up to about 600 °C where the k-factor started to decrease and the coercivity started to increase. It is believed, that the decrease of BN in the range of 400-600 °C is only due to the domain wall delaying effect of the Ni- and Si-rich submicroscopic phases which were precipitated inside the ferrite grains. The decomposition of δ-ferrite starts at about 600 °C.

The low level of BN in between 800-900 °C is caused by two effects. The eutectoid decomposition of δ-ferrite is the fastest at the range of 800-900 °C and this process causes the decrease of the amount of ferromagnetic δ-ferrite.

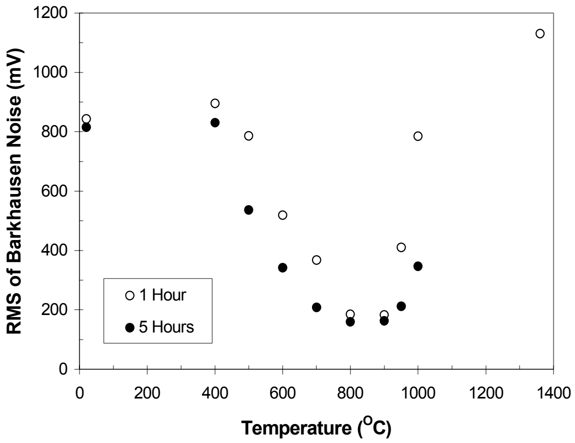

Fig 3. RMS of Barkhausen noise in function of heat treatment temperature.

Moreover, large number of inclusions appear inside the remaining δ-ferrite grains. These inclusions delay the domain wall movement therefore, decreasing the RMS value of Barkhausen noise. These inclusions are supposed to be Laves-phases. Above 900 °C Barkhausen noise values are increased rapidly due to the increasing ferrite content. Because, the incubation time of the ferrite decomposition over 900 °C requires more time than the time of the applied heat treatments. The curve which belongs to the 5 hours heat treatment is under the curve of 1 hour heat treatment in the whole temperature range (Fig. 3). It is because in case of longer heat treatment (5 hours) larger amount of δ-ferrite transformed to non-ferromagnetic phases (γ_2 and σ phases). The distortion factor (k) is strongly affected by the amount of ferromagnetic phase. As can be seen in Fig. 4 the k-factor starts to decrease at 600 °C where the δ-ferrite decomposition begins. The magnetic coercivity of the specimens were measured by ballistic method. It starts to increase at 600 °C and has maximum at the 800°C temperature. The high coercivity confirms the existence of inclusions within the remaining ferrite phase. It seems that the longer heat treatment, the larger amount of inclusions (Fig 5).

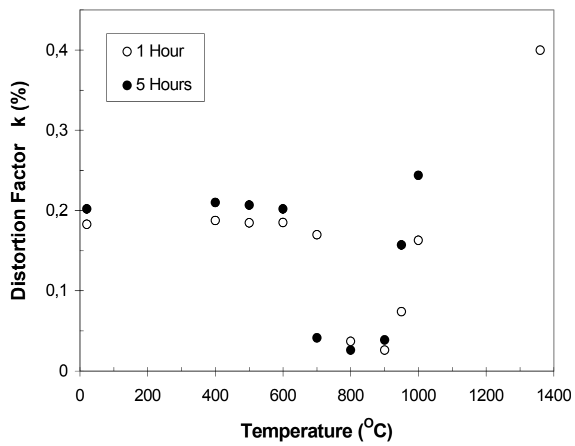

Fig 4. Distortion factor (k) in function of heat treatment temperature.

These results are in good agreement with the before discussed metallographic results. The Barkhausen measurement together with the harmonic analysis and the coercivity measurement can give us complex microstructural information. The BN level is sensitive to the amount of the ferromagnetic phase and the domain wall hindering inclusions as well. The HA and coercivity measurement can give additional information about the amount and nature of inclusions. They can be used to distinguish the two different types of microstructural changes. Therefore, the effect of changing amount of ferromagnetic phase and the effect of inclusions on magnetic properties can be separated.

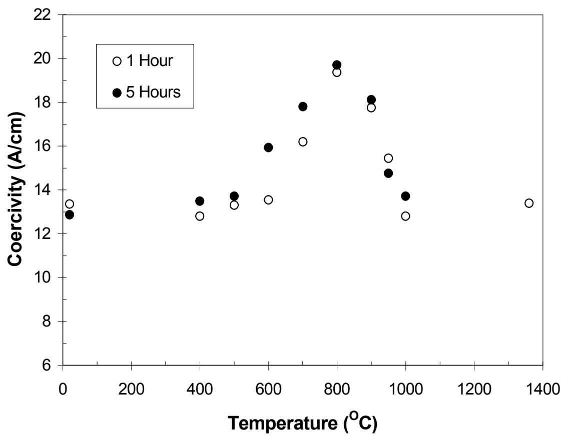

Fig 5. Coercivity (H_c) in function of heat treatment temperature.

EFFECT OF CYCLIC LOAD ON THE MAGNETIC PROPERTIES AND MICROSTRUCTURE

FATIGUE SPECIMENS

The fatigue specimens were machined from an SAF 2507 type superduplex stainless steel rod. The material was tested in its original, strongly cold worked condition.

For the experiments standard fatigue specimens were used which have a 15 mm long cylindrical part in the middle with the diameter of 7 mm. The specimens were fatigue tested by using an MTS 810 hydraulic testing machine.

The mechanical fatigue experiments were done in two-two series of experiments. In the first series of experiment the strain amplitude was constant 0.53 % and the number of cycles changed from 667 to 6091 in eight steps. The fatigue limit was at 6091 cycles, where the specimen was completely deteriorated and broken. Large number of cracks were found in the completely deteriorated specimen by optical microscopic metallography. In the second series of experiment the number of cycles was constant 4000 and the strain amplitude had the values of 0.16, 0.24, 0.3, 0.36, 0.41, 0.46 %.

MICROMAGNETIC MEASURING ARRANGEMENT FOR FATIGUE SPECIMENS

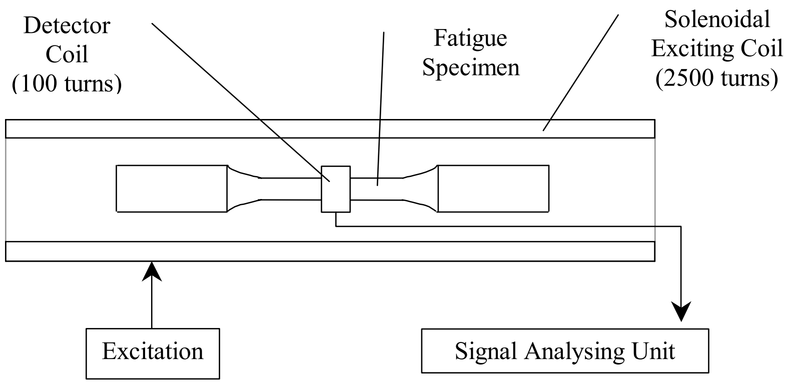

Fig.6 Sketch of the measuring arrangement which was used for measuring the micromagnetic properties of cylindrical fatigue specimens.

The measuring arrangement used for the Barkhausen-noise measurement and for the harmonic analysis is similar. Following the mechanical cycling detector coil with 100 turns was reeled onto the cylindrical middle part of the fatigue specimens and they were taken into the middle of the 15 cm long solenoid exciting coil (Fig. 6). The exciting coil was supplied by sinusoidal 20 Hz current produced by signal generator and a power amplifier. The magnetic field strength in the measuring coil was 350 A/m..
In one of our earlier papers it was shown that the RMS of the noise linearly proportional with the amount of the ferromagnetic phase [8] which is the α'-martensite in the present case.

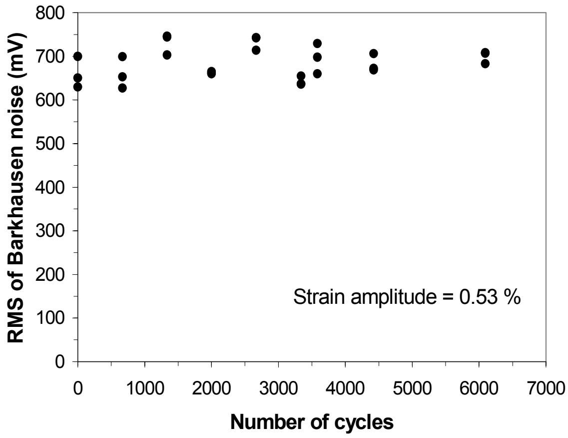

Fig 7. RMS of Barkhausen noise in function of number of cycles.

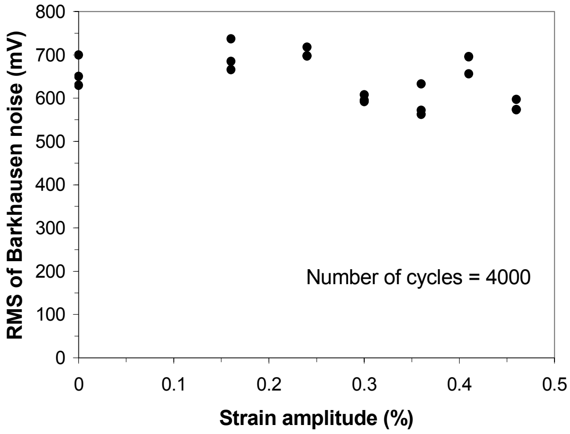

Fig 8. RMS of Barkhausen noise in function of the strain amplitude.

As it can be seen in Figure 7 ,8, 9 and 10 the RMS of the Barkhausen noise and the distortion factor are practically independent of the number of fatigue cycles and strain amplitude values. The magnetic hysteresis loops were constructed from the amplitudes and phases of the upper harmonics. The calculated coercivity values are independent of the fatigue load. It means that the material in its original condition has practically the same magnetic properties like in its completely fatigue deteriorated state.

This magnetic behaviour holds a lot of structural information of the investigated duplex steel. The applied magnetic measurements can measure only the ferromagnetic phases of the alloy. The two possible ferromagnetic phases in this system are the δ-ferrite and the α′- or strain induced martensite. The amount of ferrite is not affected but the amount of α′-martensite could increase due to fatigue load. The stable value of the Barkhausen noise RMS expresses that the amount of α′-martensite was not increased. It can be explained by the high stability of austenite due to its high N, Mo and Cr content [9].

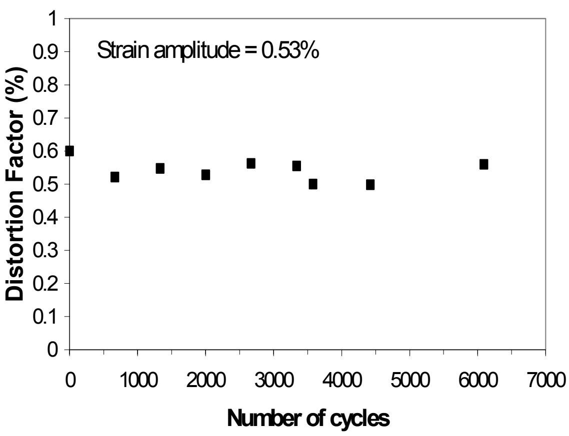

Fig 9. Distortion factor (k) in function of number of cycles.

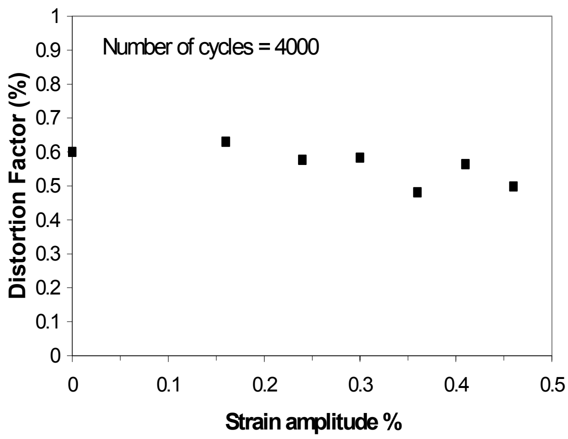

Fig 10. Distortion factor (k) in function of strain amplitude.

The periodical deformation caused by fatigue load must increase the dislocation density of the affected grains. The constant value of the harmonic amplitudes and the calculated coercivity values express that the dislocation density of δ-ferrite grains did not increased significantly. It was proved by microhardness measurement as well. The Vickers hardness (measured by 20 grams of load) of the ferrite grains in the original and the completely deteriorated specimens were the same (they were in the range of 310-320). It is well known that the plastic deformation of austenite is easier than the ferrite. Therefore, it can be concluded that during periodical loading only the softer austenite grains deform, in contrast to the δ-ferrite grains which are practically not deformed.

CONCLUSIONS

The microstructural changes of superduplex stainless steel were investigated by Barkhausen-noise measurement, harmonic analysis, coercivity measurement, optical- and scanning electron microscopy.

It was found that the Barkhausen-noise and the distortion factor (k) can be used for monitoring the change of the amount of ferrite and the inclusions within ferrite phase. The common usage of Barkhausen measurement and harmonic analysis has special advantage because they can supplement each-other and can help to distinguish between different microstructural changes.

The thermal induced decomposition process of δ-ferrite (600-900 °C) and the precipitation processes within ferrite grains (400-600 °C) are clearly separated by using Barkhausen and harmonic analysis techniques. Our ferrite decomposition results achieved by micromagnetic measurements are in good agreement with published results of Wang et al. [10] measured by using Ferritgehaltmesser 1.054 ferrite measuring equipment.

It was demonstrated that the austenite of the investigated duplex stainless steel does not transform to α′-martensite. The δ-ferrite grains are not deformed during periodical loading.
Therefore, the applied micromagnetic investigation methods can help us understand better the microstuctural changes of duplex stainless steels due to periodical loading.

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