Journal of ELECTRICAL ENGINEERING, VOL 66. NO 7/s, 2015, 134-137

STUDY OF FERRITE DECOMPOSITION IN DUPLEX STAINLESS STEEL

Bálint Bögre * — István Mészáros **

In this work the eutectoidal decomposition of ferrite in 2507 type super duplex stainless steel was investigated with magnetic measurements. The main task was how the previous cold rolling of the duplex plate can influence the heat assisted phase decomposition of ferrite phase. The obtained results can be applied in welding technology. It could give information about the critical temperature at the dangerous sigma phase can appear in cold rolled samples. The rolled and heat treated samples were tested by hardness tester, AC (alternating current) magnetometer and DC (direct current) coercimeter.

Keywords: Duplex stainless steel, eutectoidal decomposition, magnetization curves

1 INTRODUCTION

In this work the thermal induced eutectic decomposition process of ferrite ($\delta \rightarrow \sigma + \gamma_2$) was studied. The special goal of this project was studying the effect of previously applied cold working (rolling) on the rate of eutectic decomposition.

Thirty-five samples of 2507 type super duplex stainless steel (SDSS) were cold rolled at different rates, analyzed in this state and also after the ageing for 30 minutes at 700, 750, 800 and 850°C temperatures. After heat treatment the hardness of the samples was measured by a Vickers hardness tester. The amount of ferromagnetic phase (δ-ferrite) was determined by magnetic measurements. The hysteresis and normal magnetization curves were measured by an AC magnetometer. The exact value of coercivity was determined by a Foerster-type DC magnetometer.

2 THEORY

Duplex stainless steel (DSS) is a particular category of stainless steels characterized by a double-phase microstructure with about equal proportions of austenite and ferrite. The combination of properties, including high strength and excellent resistance to corrosion and chloride stress corrosion cracking made DSS very attractive for many applications. The mixed ferrite/austenite microstructure leads to different advantages if compared with the unique structure of the austenitic and ferritic stainless steels. In fact, DSS show higher toughness than most ferritic grades, improved SCC (stress corrosion cracking) resistance than most austenitic grades, and higher strength than most grades of either type. [1, 2]

Unfortunately, there are several disadvantages due to the metastable structure of duplex stainless steels. Undesirable secondary phases can form during improper heat treatments in the critical temperature range 300-1000°C [3]. The precipitation is mainly associated with the ferritic phase, due to the larger amounts of Cr and Mo and the lower solubility and faster diffusion of N and C within the BCC (body-cantered cubic) lattice than austenitic phase. The decomposition of ferrite leads to the formation of many different secondary phases, as σ-phase, χ-phase, carbides of M_7C_3 and $M_{23}C_6$, nitrides Cr_2N and CrN, secondary austenite, R-phase, π-phase. The appearances of these chemical compound phases are dangerous because it can cause decrease the ductility dramatically. The most important phase transformation in duplex stainless steels is the eutectoidal decomposition of δ-ferrite [4]. The kinetics of the eutectoidal decomposition of the δ-ferrite is known. The chromium and molybdenum rich sigma phase grains appear along grain boundaries of δ-ferrite and austenite. Therefore the neighbouring δ-ferrite grains become poor from these components. So the stability of the δ-ferrite decreases and it transforms to secondary austenite simultaneously with the appearance of sigma phase [5, 6].

3 EXPERIMENTS

At the beginning of the experimental work 35 samples were made from the base DSS plate which had a size 10x15x100 mm.

A pre-experiment was prepared to determine the maximum rate of the possible rolling. The sample was rolled as long as it began to curve. Therefore the maximum deformation was chosen to be 60 %, so the 10 mm thick of the sample was reduced to 4 mm by cold rolling. Differently cold rolled sample series were prepared with 60 %, 50%, 40%, 30%, 20%, 10% deformation and the undeformed, reference sample series (0 %). 5-5 samples were rolled in the case of every deformation extent. After that 1-1 samples were heat treated for 30 minutes at 700°C, 750°C, 800°C and 850°C from every deformation extent, and one sample series stayed without heat treatment.

* PhD Student, Department Budapest University of Technology and Economics, Department of Materials Science and Engineering, H-1111 Bertalan L. u. 7. Budapest, Hungary; bbalint1113@gmail.com ** Associate Professor, Budapest University of Technology and Economics, Department of Materials Science and Engineering, H-1111 Bertalan L. u. 7. Budapest, Hungary; meszaros@eik.bme.hu

ISSN 1335-3632

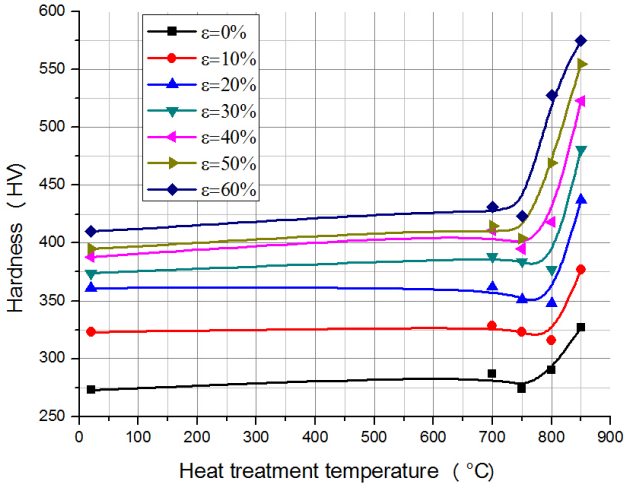

Fig. 1. Hardness values as a function of the heat treatment temperature and rolling extent

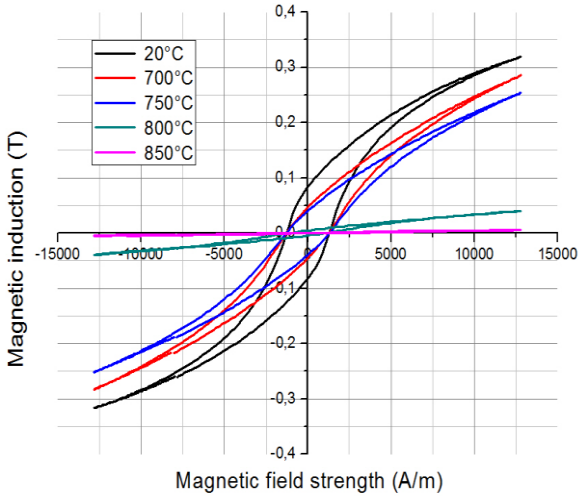

Fig. 2. Magnetic hysteresis curves of those samples which were rolled at extent of 60 %

After heat treatment the hardness of the samples was determined by a Vickers hardness tester. Figure 1 shows the hardness values of the samples as a function of the heat treatment temperature and the rolling extent. It can be seen that the hardness increases slightly in the case of the undeformed (ε = 0 %) samples as a function of the heat treatment temperature. The difference of the hardness values is about 40 HV between the not heat treated and at the 850°C heat treated samples. The growth of hardness is more intensive in case of the rolled samples (for example at ε = 50 % or ε = 60 %). When the extent of the deformation was 60 % the increase of the hardness was more than 140 HV, compared to the not heat treated samples with the 850°C heat treated samples.

It can be explained by the eutectoidal decomposition of the δ-ferrite. In this process the δ-ferrite phase partially transformed into secondary austenite and sigma phase due to the heat treatment and the appearance of the brittle sigma phase causes the growth of the hardness. It can be seen that the increase of the hardness is significantly higher at 800°C and 850°C by those samples which were deformed at 60 % compared to the undeformed ones (ε = 0 %). It is considered that the reason of this phenomenon is the deformation stored energy which is an additional driving force of the phase transformation.

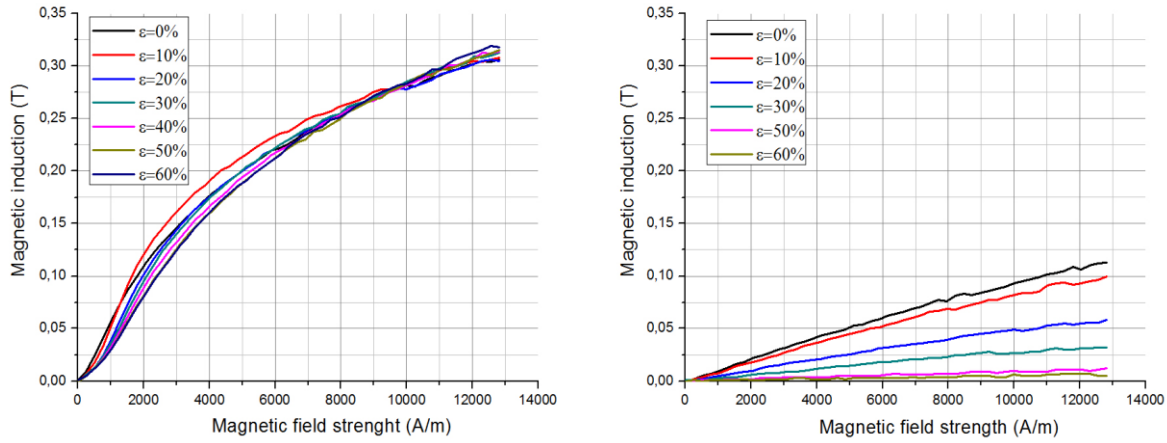

Fig. 3. Normal magnetisation curves of samples: (left) - not heat treated, and (right) - heat treated at 850 °C

Thus, more measure of the rolling causes a higher hardness rising because of the growing amount of sigma phase in the case of the same heat treatment temperature. It can be concluded that the cold rolling before heat treatment really speeded up the decomposition rate of the δ-ferrite.

An AC single sheet tester type magnetic analyzer was applied for measuring the series of symmetrical minor magnetic hysteresis loops. The applied measuring yoke contains two symmetrical U-shaped laminated FeSi iron cores to close the magnetic circuit. The excitation current

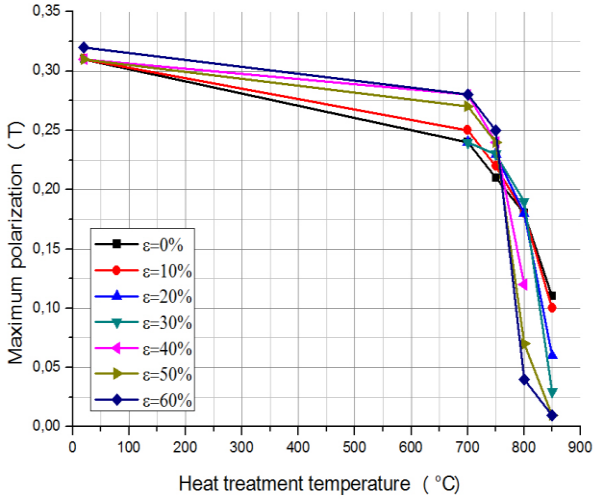

Fig. 4. Maximal polarization as a function of the heat treatment temperature

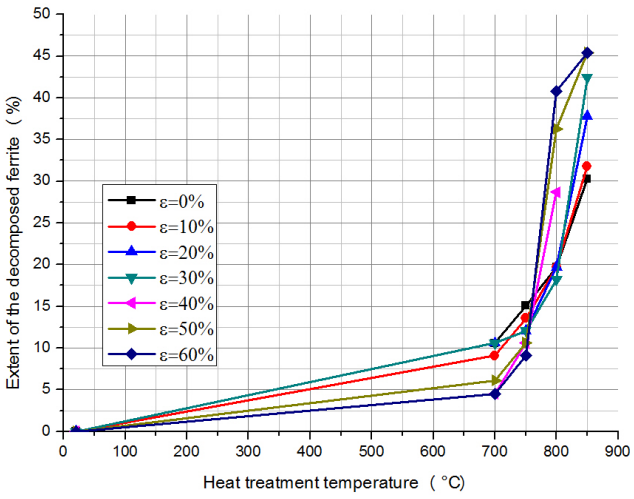

Fig. 5. Extent of the decomposed δ-ferrite as a function of the heat treatment temperature

was sinusoidal produced by a digital function generator and a power amplifier, used in voltage regulated, current generator mode. The driving coil and the pick-up coil were around the middle part of the specimen. The magnetic tester was controlled by a computer in which a 16 bit input-output data acquisition card accomplished the measurements. The applied maximum excitation field strength was 12800 A/m. In case of each samples 200 minor hysteresis loops were measured. Each minor hysteresis loops were recorded by measuring 500 points of them. The measurements were carried out by using sinusoidal excitation at a frequency of 5 Hz.

Magnetization curves were constructed from the peak points of the symmetrical minor loops. The set of the measured hysteresis and normal magnetization curves can be seen in Fig. 2 and Fig. 3. The aim of the AC magnetometer experiment was to determine the maximum polarization of the samples. There is a well known direct ratio between the ferrite content of the samples and the measured maximum polarization [7].

Ferrite content of the original SDSS was known from its data sheet. Maximum polarization of the original, undeformed and not heat treated sample was determined by AC magnetometer measurement. This maximum polarization is proportional with the original δ-ferrite content. Therefore it was possible to deduce the δ-ferrite content of the heat treated and rolled specimens too.

Figure 2 shows the higher the heat treatment temperature is, the smaller the value of the maximum polarization is. Figure 3(a) shows that the normal magnetisation curves covered each other by those samples which were not heat treated but were rolled. The decomposition of the δ-ferrite and the increase of the extent of the sigma phase didnot occur without heat treatment. On the other hand the previous rolling did not change the values of the maximum polarization. So the appearance of strain induced martensite phase can not be detected. The austenite of the studied SDSS is thermodynamically stable.

Figure 3(b) shows that the maximum polarization decreased when the temperature of the heat treatment was 850°C. The bigger the extent of the previous rolling was, the smaller the rise of the curves was. So the amount of the ferromagnetic phase reduced progressively in the deformed samples. It can be told those samples which were rolled in 50 % and 60 % were practically paramagnetic. The AC magnetometer measurement verified too, the bigger the measure of the previous deformation was, the more the δ-ferrite decomposed during the heat treatment.

Figure 4 shows the maximal polarization as a function of the heat treatment temperature in the case of the different rolling extents. It can be seen that the maximum polarization of the undeformed samples decreased progressively from 0.31 T to 0.11 T during the heat treatment. But the maximum polarization reduced from 0.31 T to 0.01 T in case of the 60 % rolled samples. This curve fell down suddenly between 750°C and 800°C. So the decomposition of δ-ferrite occurred at lower temperature by the strongly deformed samples. However same extent decomposition of the δ-ferrite happened at higher temperature by the small extent rolled samples.

The amount of the decomposed δ-ferrite as a function of the heat treatment temperature in the case of different rolling extents can be seen in Fig. 5. The ferrite content of the samples was determined from the maximum polarization as it was mentioned previously. It can be seen that the intensive decomposition of the δ-ferrite began above 750°C. At 800°C the extent of the decomposed δ-ferrite was nearly equal by the less deformed samples (0, 10, 20, 30 %). So the rate of the decomposed δ-ferrite was independent from the extent of the rolling. But in the case of more deformation the higher the measure of the rolling was the more the decomposed δ-ferrite was. The tendency

is similar at 850°C and the amount of the decomposed ferrite was the most significant on this temperature.

The exact value of coercive field was measured by a DC coercimeter. The coercive field strength (H_c) is determined by a Foerster-type DC coercimeter. It contains a solenoid coil in an open magnetization circuit. The measured sample is magnetized inside the coil to the saturation. The polarization of the sample is measured by two Fluxset sensors which are exactly in the middle outside of the coil. A reverse field is subsequently built up until the polarization becomes zero. In this case the reverse field strength is equal with the coercivity of the sample. The highest magnetization field strength in the applied DC coercimeter was 1000 A/cm which is definitely enough for the complete saturation of the sample.

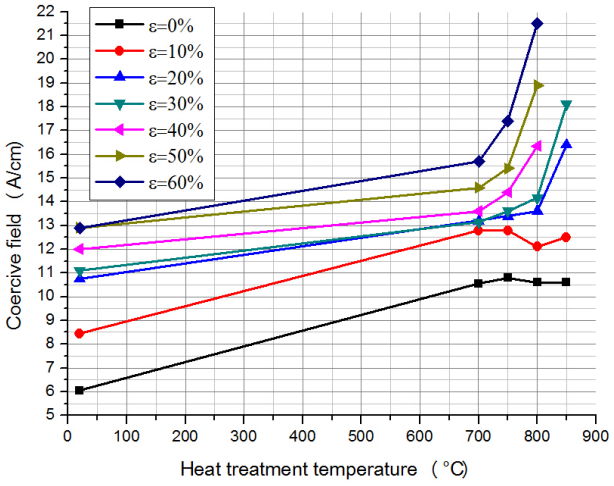

Fig. 6. Coercive field as a function of the heat treatment temperature

Figure 6 shows the coercive field as a function of the heat treatment temperature. It can be seen that the value of the coercive field increases less in case of the undeformed samples than in case of the strongly rolled samples.

There are two parts at the diagram. It is considered that the reason of the increasing coercive field under 750°C is the strain hardening. Above 750°C the coercive field increases due to the appearance of the sigma phase and the sigma phase grains prohibits the movement of the domain wall movement. If the previously applied cold rolling is higher, larger amount of sigma phase will appear in the material so the coercivity increases significantly.

SUMMARY AND CONCLUSIONS

In this work the eutectic decomposition process of ferrite was studied in 2507 type super duplex stainless steel.

It was found that if more rolling is used before the heat treatment, the amount of δ –ferrite will decrease more during the heat treatment process.

The harness of the samples increased more intensively if the extent of the previous rolling was higher because of the appearance of more sigma phase nuclei.

The results showed that the higher the extent of the previous rolling was, the lower the value of the maximum polarization was. The amount of the ferromagnetic phase (δ-ferrite) decreased because of the increasing previous deformation. The decomposition of the δ –ferrite started at lower temperature in the case of strongly rolled samples.

It was found if the extent of the previous cold rolling is higher the value of the coercive field will increase.

The experiment results could have an important effect on the welding processes. If a strongly rolled duplex plate is welded, the sigma phase already appears at lower temperature. It could be especially important in the heat effect zone, where the appearance of the brittle sigma phase could reduce the toughness of the material.

REFERENCES

[1] BÖDÖK, K.: Az ötvözetlen, gyengén és erősen ötvözött szerkezeti acélok korrózióállósága, különös tekintettel azok hegeszthetőségére, CorWeld, (1997), 225-254.

[2] THIELE, Á. – HOŠEK, J.: Mechanical Properties of Medieval Bloomery Iron Materials - Comparative Tensile and Charpy-tests on Bloomery Iron Samples and S235JRG2, Period. Polytech. Mech. Eng. (59)1 (2015), 35-38.

[3] BERECZ, T. – SZABÓ, P. J.: Crystallographic relations during decomposition of the ferritic phase by isothermal ageing in duplex stainless steel, Journal of Applied Crystallography 46 (2013), 135-141.

[4] BERECZ, T. – SZABÓ, P. J.: Study of the Isothermal Phase Transformations in Duplex Stainless Steels by EBSD Method, Materials Science Forum Vols. 473-474 (2005), 177-182.

[5] GUNN, R. N. (editor): Duplex Stainless Steels, Abington Publishing Co. (1997).

[6] CHARLES, R. N.: Duplex Stainless Steels, Steel Research International Vol.79 (2008), 455-465.

[7] FIORILLO, F.: Measurement and Characterization of Magnetic Materials, Elsevier (2004).

Received 30 November 2015