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TESTING OF STAINLESS STEEL BY DOUBLE YOKE DC MAGNETOMETER

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In the present work the microstructure and the strain induced phase transformation of Lean Duplex Stainless steel were studied. Sheet samples were cold rolled, the number of samples was nine; the applied deformation was up to 80%. The first magnetization curves and the saturation magnetization loops were measured by a double yoke DC magnetometer. The double yoke magnetometer is based on the former Stablein-Steinitz type magnet tester. The idea of the measuring set up was reconstructed and modernized at the Department of Materials Science and Engineering of BUTE. The obtained laboratory measuring device is an accurate closed-circuit DC magnetometer which widely can be used for testing bulk magnetic samples. The measuring set-up was found to be especially suitable for testing structural steel and hard magnetic samples. This equipment was successfully applied for studying the ferritic-martensitic phase transformation of lean duplex stainless steel due to cold rolling deformation.

Keywords: double yoke DC magnetometer, phase transformation, strain induced martensite, lean duplex stainless steel, magnetic characterization

1 INTRODUCTION

Stainless steels are composed of iron, chromium and carbon, and by other elements such as nickel, molybdenum, silicon and titanium *etc*. Each alloying element has particular influence on the properties of the alloy. Their excellent corrosion resistance is due to a complex oxide film on their surface. This film protects the alloy from corrosion, and it can recreate if it damaged.

Duplex stainless steels (DSS) have double phase structure of austenite (γ) and ferrite (δ). These stainless steels offer an excellent combination of properties like high mechanical strength, excellent resistance to corrosion in aggressive environments and good weldability. Unfortunately the DSSs are susceptible to secondary phase precipitation like chi- and sigma-phases. The appearance of these phases is typical at about 500°C and they strongly decrease toughness and can cause embitterment problems. Additionally, in the last years the large cost fluctuation of certain expensive elements like Ni and Mo lead to the development of low cost lean duplex stainless steels (Lean DSS). Lean DSSs contain decreased amount of nickel to lower the price and to reduce problems such as the precipitation of dangerous intermetallic phases that strongly promote localized corrosion and brittle fracture behaviour [1]. The nickel must be replaced in lean DSSs by nitrogen and manganese alloying to stabilize the austenitic phase, and to maintain the austenite/ferrite balanced microstructure. The FCC microstructure of most austenitic stainless steels is not thermodynamically stable around the room temperature. Therefore, stress or plastic deformation may induce a diffusionless martensitic phase transformation, by which the metastable austenite is transformed to the thermodynamically more stable martensite [2, 3]. The metastable austenite can transform into body-centred cubic (BCC) α'-martensite directly (γ→α') or via a hexagonal close-packed (HCP) ε-martensite phase (γ→ε→α') [4]. The initial austenite and ε-martensite phases are paramagnetic while the α'-martensite is a ferromagnetic phase. Therefore this martensitic phase transformation can be studied by measuring the magnetic properties of lean DSS samples.

2 THE DOUBLE YOKE DC MAGNETOMETER

The coercivity of the tested cold rolled lean DSS samples were in the range of 500-3500 A/m which is too high for saturating them by an AC magnetometer. For the high field measurements a special DC magnetometer was developed and applied. This magnetometer is based on the setup of the old Stablein-Steinitz magnet tester. The original version of the Stablein-Steinitz tester was developed in the middle '30-s for measuring bulk-shaped medium hard magnetic samples [5]. Nowadays this measuring arrangement is practically sunk into oblivion because of the outworn design of the original set-up. While keeping the basic principle of the measurement, we successfully combined the advantages of the original set-up with the opportunity given by modern electronics and sensors. The result is a double yoke DC magnetometer device that can be used widespread for research, industrial and educational purposes. It is suitable for testing structural steel and hard magnetic samples as well. The set-up consists of two facing U-shaped bulk soft-iron yokes with four field coils placed on the arms of the yokes. The yokes can be distanced – while keeping the geometrical symmetry – to achieve two adjustable and identical air gaps. Therefore there are two equal air gaps in the yoke: the measuring air gap for the sample and the reference are gap. The measured sample touches the surfaces of the yoke without air gap. Consequently the system behaves as a closed magnetic circuit for the sample. The coils are excited so, that a magnetic symmetry appears (after compensation, if necessary), thus the magnetic flux in both air gaps will be equal. The set-up contains a so called bridge-branch in the

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middle of the arms (Fig. 1), which is flux less in case of geometrical and magnetic symmetry. Upsetting the symmetry by any means the air gaps obtain different fluxes, and the difference of those can only be conducted via the bridge branch. By creating a negligible air gap in the bridge-branch containing a field sensor the flux of the bridge is measurable.

The double yoke setup behaves as a symmetrical magnetic bridge. Without sample the A and B points of the yoke (Fig. 1) are equipotent in the magnetic circuit. Therefore there is no magnetic flux through the bridge-branch. If sample is taken into the measuring air gap it upsets the symmetry of the yoke and there will be a magnetic flux thought the bridge-branch. The flux of the bridge-branch can be calculated by a simple concentrated parameter model of the magnetic circuit. After the proper simplifications it can be derived.

$$\mu_0 M_{Sample} = B_{Bridge} \frac{C_1\left(1+\frac{C_2}{l}\right)}{A} \quad (1)$$

This demonstrates that the magnetic polarization of the measured sample is linearly proportional with the magnetic induction detected within the bridge-branch.

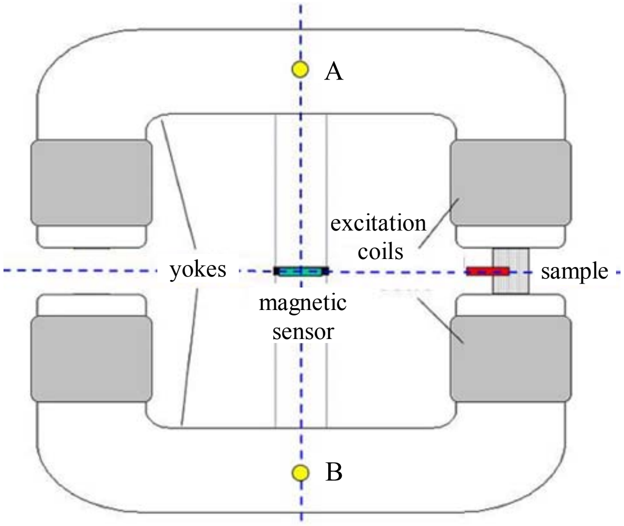

Fig. 1. The schematic of the DC magnetometer set up

Where: M_{Sample} (A/m) is the magnetization of the tested sample, B_{Bridge} (Vs/m^2) is the magnetic induction within the bridge-branch, l (m) and A (m^2) are the length and the cross section of the sample respectively. The C_1 (m^2) and C_2 (m) values are the calibration constants of the device.

The double yoke measuring set-up was developed and constructed at the Department of Materials Science and Engineering of BUTE in the year of 2010. The magnetometer is completely computer controlled, the measurement and the excitation are performed by a National Instruments data acquisition card (DAQ) and a PC. The magnetic fields in the bridge and the measuring air gap are both measured by up to date Hall-sensors. Low noise and small sized Hall-sensors (thickness is 0.5 mm) were applied in the system. They allow us to use thin air gap in the bridge-branch that makes the measurement of its flux precise. The sensor which measures the magnetic field strength (H) of the sample is taken close to the surface of the sample. The system utilizes the well known electromagnetic principle: the normal component of magnetic induction (B) and the tangential component of magnetic field strength (H) do not change when they cross the border between two substances having different permeabilities.

The developed electronics contains a stabilized current source for driving the Hall-elements with the lowest possible noise; it also contains a filtered instrumental amplifier, so the total voltage range of the data acquisition card can be utilized, thus the quantation error of the digitalization can be minimized. The excitation is driven by a computer-controlled power supply, also adapted to the DAQ. The data acquisition, processing and the control of the set-up were done by a LabView VI.

The double yoke magnetometer can apply cyclic demagnetization before measuring the sample. It can measure symmetrical and all types of asymmetrical hysteresis loops as well as the first- and normal magnetization curves. One of the unique features of the system is that it can be applied for measuring the anhysteretic magnetization cure as well.

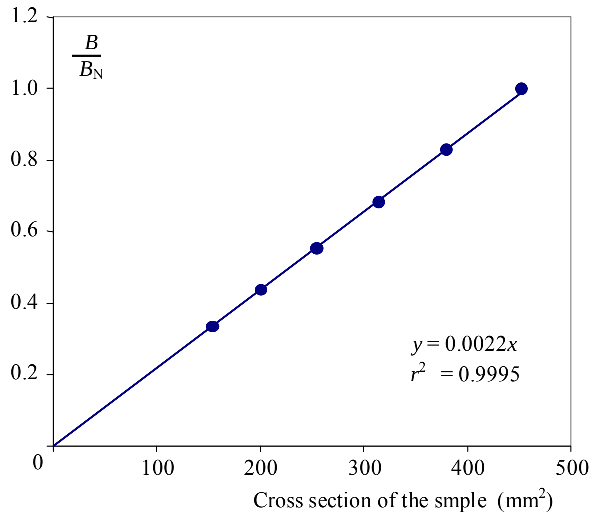

Fig. 2. Measured calibration curve of the applied measuring yoke.

The double yoke magnetometer was calibrated by cylindrical Armco steel and nickel samples. The calibration samples were made of Armco steel can be seen in Fig.2. It was determined measuring the saturation polarization of six cylindrical samples, with the length of 20mm but different diameters (14, 16, 18, 20, 22, 24 mm). According to the plotted results (Fig.2) it can be concluded that the measured polarization values are in good agreement with the theory (1). The magnetic induction in the bridge-branch is practically linearly proportional to the cross section of the samples.

The main advantages of the developed double yoke tester are the followings:

- The system forms a closed magnetic circuit with the sample therefore there is no demagnetization effect.
- Polarization of the sample can be measured directly without integration. It can avoid all the problems which normally can arise from the integration of the magnetic flux (drift etc.).
- The anhysteretic magnetization curve of the samples can be measured.
- Medium and high coercivity samples (H_c < 500 A/cm) including structural steel and Alnico magnets can be tested.
- Ideal for small ratio of length to transverse dimensions (dumpy bulk) samples.
- Stable, completely PC controlled

The main characteristic properties of the constructed double yoke tester:

- Air gap: 0 - 50 mm
- Yokes: bulk low carbon steel
- Pole diameter: 80 mm
- H_{max}: 14.000 A/cm (10 mm)
- Frequency: only DC
- Accuracy (M, H): ±1%

3 EXPERIMENTAL

The tested material was a lean duplex stainless steel received as hot rolled plates of 8 mm in thickness. Its chemical composition measured by EDS shown in Table 1. The metallographic volume fractions of austenite and delta-ferrite phases were 20.4% and 79.6% respectively.

Table 1. The chemical composition of the tested steel.

Cr	Mn	Ni	Si	Mo	Cu	Fe
22.12	3.50	1.25	0.81	0.19	0.38	Rest

The samples were cold rolled at room temperature. The plates were cold rolled in one direction, through many constant passes, to gradually reduce its thickness by compression. Seven percentages between 10% and 80% of thickness reduction were obtained.

Samples were specially prepared for measuring them by the double yoke magnetometer. The elongated stripe was cut into pieces and laminated bulk samples were constructed containing 3-8 layers. The size of block samples was 20×15 mm their thickness was in between 15–20 mm.

Our previous X-ray diffraction investigations detected fully ferromagnetic structure in case of the largest deformation. Therefore it can be concluded that due to 80% of thickness reduction all the paramagnetic austenite phase were transformed completely into ferromagnetic α′- martensite.

Vickers hardness (HV) tests were also performed using a Buehler MMT-3 digital micro hardness tester. All measurements were carried out using a load of 500 N.

4 RESULTS AND DISCUSSIONS

The first magnetization curves and saturation hysteresis loops were measured by our double-yoke magnetometer. The maximum applied external field was about 2250 A/cm which was absolutely sufficient for the magnetic saturation of the samples (Fig. 3). As it can be seen in Fig.3 the saturation polarization values are gradually increased due to plastic deformation. Its initial value was 0.752 T which rose to 0.94 T as a result of the martensitic phase transformation.

It is well known that the saturation polarization is linearly proportional with the amount of ferromagnetic phase in alloys [6]. It allowed us to quantify the amount of ferromagnetic α′-martensite in cold rolled samples by direct comparison with saturation polarization obtained by magnetic tests.

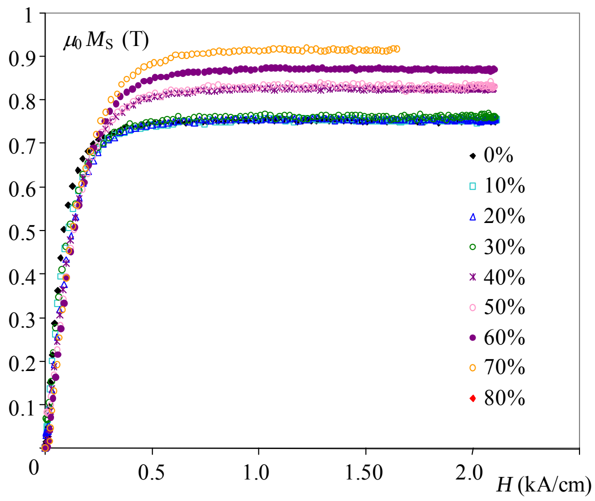

Fig. 3. The measured first magnetization curves.

The amount of martensite was calculated from

$$\mu_0 M_S^{Sample} = R^{\delta} \mu_0 M_S^{\delta} + R^{martensite} \mu_0 M_S^{martensite} \qquad (2)$$

Where: M_S^{Sample}, M_S^{δ}, $M_S^{martensite}$ are the saturation magnetization values of the tested sample, delta-ferrite and martensite phases respectively. R^{δ} and $R^{martensite}$ are the relative ratios of delta ferrite and martensite phases. The determined saturation polarization values of delta-ferrite and martensite phases are 0.95 T and 0.92 T respectively.

The saturation polarization values and the quantified amount of strain induced martensite phase can be seen in Fig. 4. The saturation polarization seems to be almost the same at lower thickness reduction (up to 30%). Further increase in cold deformation has the consequence of stronger and gradual increase in magnetic saturation field, reaching the highest value of 0.94 T for the highest thickness reduction imposed (80%). The amount of α´-martensite at each thickness reduction is reported in Fig. 4 as well.

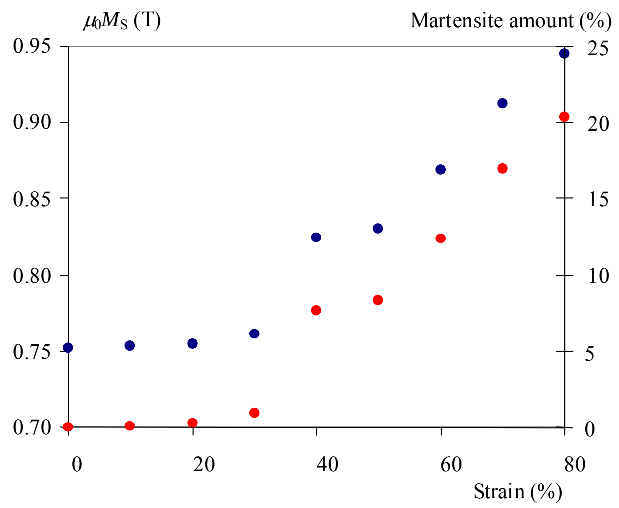

Fig. 4. The saturation polarization values and the quantified amount of martensite *vs* strain.

The results of Vickers hardness and the amount of strain induced martensite phase are plotted together in Fig. 5. It seems that at low strain rates the Vickers hardness increases rapidly up to 20% of thickness reduction. For further deformations martensitic transformation takes place, causing a slight increase of the hardness. Above 20% thickness reduction the hardness practically linearly increases with the amount of strain induced martensite phase. It can be concluded that above 20% deformation only the appearance of martensite phase causes the strain hardening. In a low deformation range (under 20%) the hardening process is supposed to be associated with the increase of dislocation density. It is remarkable that this microstructural background of hardening process of lean duplex stainless steel is different from the AISI 304 austenitic stainless steel reported earlier [4]. In contrast to the demonstrated behavior of lean duplex stainless steel in AISI 304 steel the strain induced martensite phase has no significant role in the hardening process.

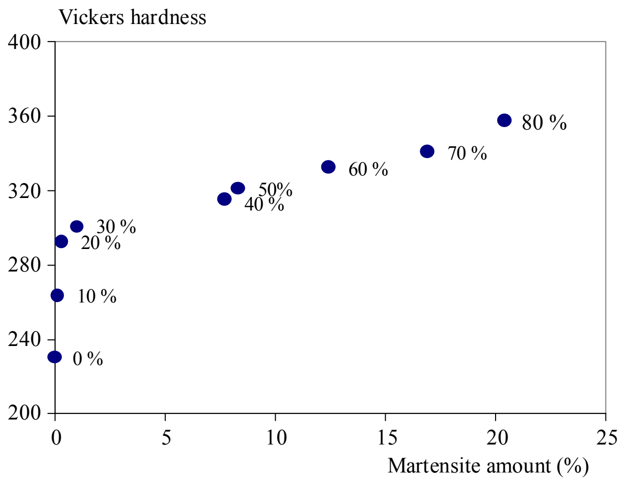

Fig. 5. The Vickers hardness *vs* amount of strain induced Martensite. Indicated are the thickness reduction values in %.

CONCLUSION

In this work a new double yoke DC magnetometer set up was constructed on the basis of the old Stablein-Steinitz magnet tester. The idea was reconstructed and modernized at the Department of Materials Science and Engineering of BUTE. The magnetometer is completely PC controlled and equipped with modern magnetic field sensors, sensing and driving electronics. The obtained laboratory measuring device is an accurate closed-circuit DC magnetometer which widely can be used for testing bulk magnetic samples. The measuring set-up is especially suitable for testing structural steel and medium hard magnetic samples.

This double yoke magnetometer was successfully applied for testing of cold rolled lean duplex stainless steel samples. The achievements in metallographic point are the following:

- The austenite-α´ martensite phase transformation starts at about 20% thickness reduction and became complete at 80% thickness reduction.
- The amount of martensite seems to be not affected by cold rolling up to 20% of thickness reduction, while further increase in cold deformation lead to a linear increase of the amount martensite phase up to 80% thickness reduction.
- The saturation polarization values of delta-ferrite and martensite phases were determined. Their values are 0.95 T and 0.92 T respectively.
- It was demonstrated that above 20% thickness reduction practically only the appearance of strain induced martensite phase is responsible for strain hardening.

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