Microscopical Characterisation of Martensite Formation in a Metastable Austenitic Stainless Steel

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Abstract : Stainless steels have widespread applications due to their good corrosion resistance, but for certain types of large naval constructions, other requirements are imposed such as high strength and toughness, and modified magnetic characteristics. 302 type metastable austenitic stainless steel after solution treatment possesses a fully austenitic microstructure. However, its tensile strength in the solution treated condition, is low. Consequently, cold rolling following solution treatment is applied and results in the strain induced transformation to α' -martensite within the austenitic matrix and enhancement of its tensile strength. Following cold rolling a mixed martensitic-retained austenitic microstructure is obtained. In addition, when ferromagnetic α' -martensite is introduced to an otherwise fully paramagnetic matrix, the magnetic response of the steel changes. The magnetic hysteresis loop for the cold rolled 302 type stainless steel was recorded, under a static field, together with the hysteresis loop obtained for an 817M40 type steel (reference material). It is observed that the 302 type stainless steel shows much lower maximum induction and remanence values, without any increase in the coercivity. TEM examination revealed in greater detail the fine elements of the microstructure of the material. Hexagonal ϵ -martensite forms initially and then at a later stage of deformation α' -martensite forms. This behaviour is typical of 302 type stainless steel deformed below its M_d temperature. In addition to conventional electron microscopical investigation, magnetic domain imaging has been performed using the low magnification mode of the TEM. Magnetic domains then appear as lines of dark and light contrasted areas, when the sample is imaged in the out of focus condition. These domain boundaries are present only in the α' -martensite regions and are normally oriented at an angle to the long axis of the martensite lath. It has been identified that significant reductions in the maximum magnetic induction and remanence values can be achieved when a 302 type stainless steel is substituted for the conventional types of low alloy medium carbon steel. These changes can be attributed to the paramagnetic austenite fraction and the magnetic domain structure existing within the α' -martensite areas (ferromagnetic phase). There was no magnetic domain structure within the paramagnetic ϵ -martensite region.

1. INTRODUCTION

Stainless steels have widespread applications due to their good corrosion resistance, however for certain types of naval constructions other requirements are imposed on the candidate material, namely a high yield strength, high impact toughness coupled with suitable magnetic characteristics.

Several types of stainless steels were examined during this ongoing research project and the results were compared [1-5] to a low alloy medium carbon steel (817M40 type) that was the reference material. The aims are to provide a steel with a high yield strength (~ 700 MPa), high impact values (80J at -84°C), improved corrosion resistance in a marine environment and possessing suitable magnetic characteristics (low permeability at earth's field, as well as low remanence and coercivity).

Austenitic stainless steels are obvious candidate material for such a study, since they have improved corrosion resistance, compared with low alloy medium carbon steels, and improved magnetic characteristics (austenitic steels are paramagnetic unlike ferritic ones that exhibit a strong ferromagnetic response). However, they possess in the solution treated condition, yield strength values way below those required.

In this paper, results are presented on the magnetic behaviour of this steel, on the SEM and TEM characterisation, and on the magnetic domain imaging of a metastable austenitic stainless steel.

2. EXPERIMENTAL

A 302 type metastable austenitic stainless steel (its composition given in table 1, together with its Pitting Resistance Equivalent-PREN) was used in order to provide a material capable of hardening after relatively low cold deformations.

Table 1: Composition of steels in wt. %

	C	Cr	Mo	Ni	N	PREN
302 type	0.05	18.34	0.29	8.20	0.039	19.92
817M40	0.44	1.4	0.35	1.6	-	2.5

The 302 type stainless steel was solution treated at 1120°C followed by water quenching. In this condition the material is composed mainly of austenite (with minor amounts of residual δ -ferrite) and therefore exhibits paramagnetic behaviour. However, its yield strength is ~220 MPa, significantly lower than that required, therefore cold deformations at different levels (ranging from 13-21%) and at different temperatures were applied to this steel in order to increase its yield strength. The samples were subjected to only small amounts of cold deformation in each pass, after which they were rapidly transferred to a cooling bath in order to avoid adiabatic heating during cold rolling.

Samples of the 817M40 reference steel were austenitised at 850°C then oil quenched, followed by tempering at 675°C and air cooling. Specimens for metallographic study (either light or electron microscopy), after the heat treatment, were prepared using standardised techniques of grinding and polishing, etching and jet polishing [6].

Conventional transmission electron microscopy dark field imaging techniques, micro diffraction techniques and EDS microanalysis was performed using either a JEOL 200 CX analytical microscope or a Philips EM 430ST High Voltage Microscope. The high voltage TEM microscope was used also for the magnetic domain imaging of the 302 samples. The microscope was operated in the Low magnification mode with the objective lens weakly excited (in effect switched off), so that in the vicinity of the sample the magnetic field created by the objective lense is negligibly small. Magnetic domains then appear as lines of alternate light and dark contrast bands when the sample is imaged in the underfocussed or overfocussed condition.

The use of a standard solenoid was employed to create the hysteresis loops under a static magnetic field, and in addition a standard magnetometer (Fischer, Ferritoscope) was employed in order to measure the percentage of ferromagnetic phases in the samples.

3. RESULTS

3.1 Magnetic characterisation

Upon cold deformation and depending on the temperature and the amount of cold deformation applied, austenite partly transforms to initially ϵ -martensite (a hexagonal-paramagnetic phase) and then with increased amounts of cold deformation formation of α' -martensite occurs (a tetragonal ferromagnetic phase), in accordance with previously published work [7-9]. However, since significant amounts of cold deformation (ranging from 16.5% at -35°C to 25% at 20°C) had to be applied, on 302 steel samples, to achieve the required yield strength values most of the specimens contained significant proportions of α' -martensite. In table 2 the different amounts of cold rolling reduction applied at different temperatures are given together with their corresponding percentages of ferromagnetic α' -martensite, as measured using the Ferritoscope.

Table 2: Percentages of ferromagnetic phase after cold rolling.

Cold rolling temperature	Cold rolling reduction (%)	Ferromagnetic α' -phase (%)
-35°C	11	17.5
	16.5	24.5
-20°C	13	14
	16	24.2
	18.5	27.5
0°C	14	14
	20	16
20°C	17	4.1
	21	6.8
	25	13.5

In addition the magnetic hysteresis loops of these partly transformed samples were measured and results compared to the reference material. An example of a hysteresis loop produced for a 302 sample cold rolled at -35°C at 16.5 % cold rolling reduction is given in Figure 1, together with the hysteresis loop of the reference material (817M40 tempered at 675°C).

Figure 1: Hysteresis loops produced under a static field for a quenched and tempered 817M40 sample (A) and a 302 sample cold reduced by 16.5% at -35°C (B).

It is evident that the 302 sample, which exhibits a similar yield strength value to that of the 817M40 sample, shows significantly lower saturation magnetisation (points A and B), as well as lower remanence values (points C and D). However, its coercivity is higher than that exhibited by the reference material (points E and F). Similar magnetic characterisation results were obtained by the remaining cold rolled 302 samples with the only variation noted on the saturation magnetisation, depending on the amount of α' -martensite (ferromagnetic constituent) present in the 302 microstructures. The higher the amount of α' -martensite the higher the saturation magnetisation.

3.2 SEM and TEM characterisation

SEM microscopical investigation was carried out on samples of the cold rolled 302 type stainless steel. On the level of the SEM examination, only the general details of the microstructure were revealed as shown in Figure 2, where it can be seen that within the generally austenitic microstructure, slip bands, and variable amounts of martensite are present.

Figure 2: SEM micrographs of a 302 type metastable austenitic stainless steel after solution treatment followed by a) 25% cold rolling reduction at 20°C and b) 16.5 % cold rolling reduction at -35°C .

TEM microscopical examination of a sample of 302 steel after 25% cold rolling reduction at room temperature, revealed the fine microstructure in greater detail. The presence of ϵ -martensite and α' -martensite plates within the austenitic matrix is clearly shown in Fig.3.

Figure 3: Bright field TEM micrograph of a 302 type metastable austenitic stainless steel solution treated and given a 25% cold rolling reduction at 20°C.

Similar results were obtained also for samples cold rolled at different temperatures, as can be seen in Figure 4, where a pair of dark and bright field micrographs is provided.

Figure 4 : A pair of dark and bright field TEM micrographs of ϵ -martensite and α' -martensite within the austenitic matrix, produced in a 302 type metastable austenitic stainless steel solution treated followed by 16.5% cold rolling reduction at -35°C. Notice the presence of α' -martensite plates illuminated in the dark field micrograph.

In the dark field micrograph the coarse areas of tetragonal α' -martensite are illuminated, whereas the fine laths of ϵ -martensite clearly belonging to a different crystallographic orientation are visible only in the bright field mode. However, in Figure 5, which shows a pair of bright field and dark field TEM micrographs of a different area of the same sample presented in Figure 4, it can be clearly seen, especially in the dark field micrograph that ϵ -martensite needles are illuminated within the residual austenite matrix.

Figure 5 : A pair of dark and bright field TEM micrographs of ϵ -martensite within the austenitic matrix, produced in a 302 type metastable austenitic stainless steel solution treated followed by 16.5% cold rolling reduction at -35°C . Notice the presence of ϵ -martensite needles illuminated in the dark field micrograph.

Figure 6: Low magnification bright field TEM micrograph of the magnetic domain boundaries observed within the martensitic lath region of an α' -martensite packet area. The sample was solution treated followed by 18.5% cold rolling reduction at -20°C .

3.3 Magnetic Domain Imaging

Magnetic domain imaging was also performed so as to illustrate clearly the presence of magnetic domain boundaries present only within the α' -martensite (ferromagnetic constituent) regions of the complex microstructures. In Figure 6 a low magnification bright field TEM micrograph of the magnetic domains observed within α' -martensite regions of a sample is presented. It can be seen that magnetic domain boundaries are only present within the α' -martensite plates, whereas residual paramagnetic austenite and ϵ -martensite regions are free of domain boundaries.

4. DISCUSSION

It has been demonstrated that upon cold deformation, metastable 302 type stainless steel initially transforms to ϵ -martensite followed by the subsequent formation of α' -martensite.

During cold rolling, strengthening of the 302 steel is achieved through work hardening of the austenitic matrix, as well as with the formation of α' and ϵ -martensite. However, significant amounts of cold deformation need to be applied depending on the temperature of the cold rolling experiment, so as to achieve the required strength values. In addition, the introduction of ferromagnetic α' -martensite in a paramagnetic austenitic matrix modifies the magnetic characteristics of the steel, namely the coercivity, remanence and saturation magnetisation. However, the remanence and saturation magnetisation values at all cold rolling conditions were significantly lower than those measured on the reference medium carbon low alloy steel. In fact remanence values of ~ 0.1 T were measured on the 302 type steel, following cold deformation, as compared to the 1.4 T measured on the reference material, and saturation magnetisation values of ~ 0.2 T were measured on the 302 steel as compared to the ~ 1.9 T measured on the reference material.

Finally, the coercivity value measured on the 302 stainless steel following cold deformation was higher than that measured on the reference material (~ 0.4 kA/m for the 302 steel versus ~ 0.15 kA/m for the 817M40 steel).

Magnetic domain imaging revealed, as expected, the presence of magnetic domain boundaries present only within the α' -martensite regions of the 302 stainless steel but absent from the paramagnetic residual austenite and ϵ -martensite regions.

Therefore, taking into account the significant superior corrosion resistance of the 302 type stainless steel in a marine environment, compared to that of the reference material, the high yield strength values, and the favourable saturation magnetisation and remanence results, it is concluded that an alternative material for use has been identified if the higher coercivity values exhibited by the 302 stainless steel can be tolerated.

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