NDT&E International 44 (2011) 490–494

Contents lists available at ScienceDirect

NDT&E International

journal homepage: www.elsevier.com/locate/ndteint

# Detection of spring steel surface decarburization by magnetic hysteresis measurements

O. Perevertov [a,*], O. Stupakov [a], I. Tomáš [a], B. Skrbek [b]

[a] Institute of Physics of the AS CR, v.v.i., Na Slovance 2, 18221 Prague, Czech Republic
[b] Technical University of Liberec, Studentská 2, 46117 Liberec, Czech Republic

ARTICLE INFO

*Article history:*
Received 23 December 2010
Received in revised form
6 April 2011
Accepted 10 April 2011
Available online 19 May 2011

*Keywords:*
Decarburization
Magnetic hysteresis
Spring steel

ABSTRACT

Surface decarburization of steel was *non-destructively* and *quantitatively* investigated by magnetic hysteresis. Flat samples were prepared from spring steel 54SiCr6 and decarburized layers of different thickness were produced on their surfaces by annealing at 800 °C in air for 1, 4, 8 and 20 h. Three types of treatment were applied on different samples in order to remove the simultaneously appearing surface oxides. The decarburized layers were examined magnetically by hysteresis loops measurements and the results were related to their optically determined depths. The magnetic measurements showed high sensitivity with respect to free ferrite and mixed ferrite/pearlite layers detection even without removal of the oxide layer. The free ferrite layers were easily and quantitatively detected by minor loops measurements with low field amplitude.



## 1. Introduction

Steel processed at elevated temperatures suffers frequently by two unwanted phenomena, namely by oxidation and decarburization [1–5] of their surfaces. Decarburization can significantly change mechanical properties of the final product since hardness, fatigue resistance; strength and wear properties depend on carbon content. For springs it is particularly critical because fatigue life is one of their most important features [2,6,7]. Oxides are usually cleared away from the surfaces chemically or mechanically by processes, which can also modify surface properties of the product. A quantitative, non-invasive way of detection of the surface decarburization, which would be also sensitive towards the oxide-removing processes is sought for.

Steel industry is interested in replacing time consuming and expensive destructive mechanical tests by non-destructive methods. The international standard ISO 3887:2003 has specified three methods for measuring the depth of decarburization of non-alloy and low-alloy steels: (1) a micrographic method, which measures the microstructure variation due to the loss of carbon, such as the increase in ferrite fraction in a ferrite/pearlite mixed microstructure; (2) determination of the gradient of micro-indentation hardness on a cross-section; (3) direct measurement of the carbon content by chemical or spectrographic analysis [6,8]. All these methods are destructive, time consuming and cannot be applied during the production process.

In this work we investigate applicability of magnetic non-destructive testing of decarburized steel by methods based on magnetic hysteresis measurements.

## 2. Experimental

### 2.1. Samples

For investigation we have chosen high silicon 54SiCr6 spring steel. Its chemical composition is shown in Table 1. The samples were cut from an as-delivered 3 mm sheet into 3 mm × 30 mm × 110 mm plates and then annealed in air at 800 °C for 1, 4, 8 and 20 h in order to obtain different decarburization depths. Reference samples were prepared by annealing at the same temperature in vacuum for 2 h. After the annealing an oxide layer appeared on top of the samples and from some of them this layer was removed either by chemical etching (acid-pickling) or by sand-blasting. At the end there were three series of samples with differently treated surfaces: (1) no surface treatment, (2) acid-pickled and (3) sand-blasted. As there were samples with five different decarburization depths and always two samples identically treated, each of the three series contained 10 specimens.

### 2.2. Magnetic measurements

The setup for magnetic measurements is described in [9]. The pick-up coil was wound on the sample. The field was measured by an array of Hall sensors situated at 1, 2, 3 and 4 mm above the

* Corresponding author. Tel.: +420 266052617; fax: +420 286890527.
*E-mail address:* perever@fzu.cz (O. Perevertov).

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doi:10.1016/j.ndteint.2011.04.010

**Table 1**
Chemical composition of the 54SiCr6 steel.

| Element | C | Mn | Si | P | S | Cr | Ni | Cu | Al |
|---|---|---|---|---|---|---|---|---|---|
| wt% | 0.540 | 0.680 | 1.470 | 0.017 | 0.009 | 0.610 | 0.050 | 0.070 | 0.032 |

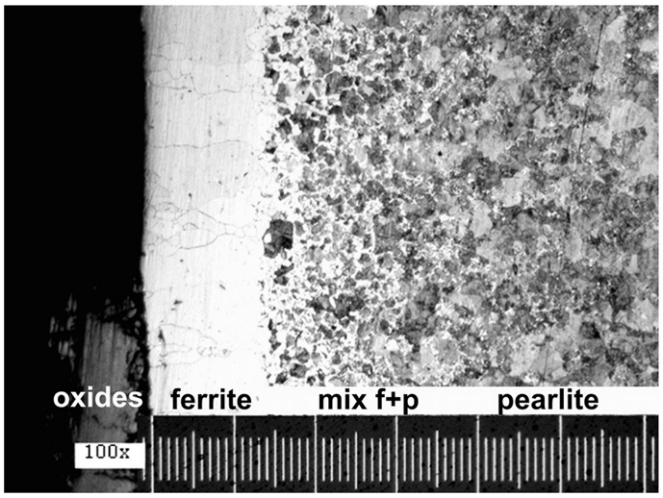


**Fig. 1.** Metallography of cross-section of steel 54SiCr6 after 4 h exposition to air at 800 °C. The smallest division of the scale is 10 μm.

sample and the surface field was found by extrapolation [10]. The Hall sensor array was magnetically shielded by two plates in order to suppress any unwanted stray field gradient near the surface. The only difference as compared with [9] is that in the present work there was a second magnetizing yoke below the sample so that it was a double-yoke setup. A triangular waveform of the magnetizing current was used. The measurements frequency was 0.03 Hz, and the maximum field was 13 kA/m. Digital filtering on-the-fly was used to get rid of the noise in the pick-up coil and in the field signal. The frequency of sampling was 120 kHz. Every 2400 points were averaged into a single value so that the resulting frequency of reading was 50 Hz.

## 3. Results

### 3.1. Metallography

The effect of annealing in air was investigated metallographically. Thickness of the decarburized layers and Vickers hardness HV0.1 were measured on a cut cross-section of each sample as shown in Fig. 1. The measured depths and values of HV0.1 at the core and at the surface of the samples are given in Table 2.

In Fig. 1 the layers of (1) oxides, (2) ferrite, (3) mixture of ferrite/pearlite and (4) pearlite can be recognized as one goes from the sample surface into the bulk.

Also carbon concentration as a function of the distance from the surface was measured by wave dispersive spectroscopy, see [11].

### 3.2. Magnetic results

Results of hysteresis measurements on pickled samples and samples with no surface treatment are shown in Fig. 2a. As compared with the usual sigmoid shape of the hysteresis loop of the sample with zero annealing in air (i.e. with the reference sample annealed in vacuum only), the loops become more and more bulged with annealing time in air, and the coercive field decreases. Results for both series were very close to each other. Also a similar loop measured on an as-delivered sample was very close to that annealed in vacuum (0 h) and it is not plotted in the figure.

**Table 2**
Vickers hardness and depths of the surface layers after annealing at 800 °C.

| Annealed in | | Vacuum | Air | Air | Air | Air |
|---|---|---|---|---|---|---|
| Annealing time (h) | | 2 | 1 | 4 | 8 | 20 |
| HV0.1 | Surface | 250 | 171 | 145 | 160 | 131 |
| | Core | 260 | 268 | 288 | 305 | 270 |
| Thickness of the layers (μm) | Oxides | 0 | 30 | 140 | 165 | 260 |
| | Ferrite | 0 | 110 | 170 | 200 | 350 |
| | Ferrite/pearlite mix | 0 | 175 | 360 | 510 | 650 |

In Fig. 2b hysteresis loops measured on the sand-blasted samples are shown. Here only the sample annealed for 20 h has a substantially distorted loop. Shorter annealing periods make the loops narrower but the typical incurved shape does not show.

The bulged shape means that the loop describes two magnetization processes, in our case that the sample consists of two magnetically different phases. This fact is much better seen on differential permeability curves shown in Fig. 3. The coercive field for all three series of samples is plotted in Fig. 4.

## 4. Discussion

From the results above it is seen that appearance of ferrite and mixed ferrite/pearlite layers has a substantial influence on the magnetization process. The ferrite layer makes the hysteresis loop bulged and it corresponds to the thin permeability peak around 100 A/m. The mixed layer leads to a large decrease in the total coercive force – from 1150 down to 500 A/m. It is evident that the sand-blasting procedure made the ferrite layer magnetically harder, by introducing large plastic deformation into the surface. That is why hysteresis loops measured on 1, 4 and 8 h sand-blasted samples are not bulged. Indeed, large surface residual stresses around 300 MPa were measured on the sand-blasted surfaces by X-ray diffraction stress measurements (see [11]).

It is logical to suggest that there is a range of the magnetizing fields within which most of the magnetic flux change is given by magnetization of the soft ferrite layer and where participation of the core material is very limited. By measurements of minor loops with a proper amplitude we should measure mostly flux of the ferrite only. Obviously, amplitude of such a minor loop should be higher than the field position of the first permeability peak in Fig. 3 and at the same time lower than the position of the second peak. In Fig. 5 minor hysteresis loops measured on samples with no surface treatment with the field amplitude 300 A/m are shown. By plotting maximum magnetic induction, $B_{300}$, of those loops as a function of the ferrite layer depth one gets a good linear dependence both for the pickled and the not-treated samples (see Fig. 6). For sand-blasted samples there is very little change for the 1, 4 and 8 h samples and only the 20 h sample is close to that of the other two series. The proper amplitude of such a minor loop can also be approximately estimated from values of the magnetic induction at a given field on virgin curves, which are measured after demagnetization, see Fig. 7.