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On the mechanism of nondestructive evaluation of cementite content in steels using a combination of magnetic Barkhausen noise and magnetic force microscopy techniques

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ABSTRACT

The influence of carbon content in the form of globular cementite precipitates in unalloyed steels was macroscopically characterized by means of magnetic hysteresis loop and Barkhausen noise techniques. The choice of the frequency of the applied field has a strong influence on the Barkhausen noise profiles. At sufficiently high frequency (0.5 Hz) there are two peaks, one at lower field, the amplitude of which corresponds to the amount of ferrite and one at higher field, the amplitude of which corresponds to the amount of the cementite phase, respectively. Magnetic force microscopy and electron backscattered diffraction techniques were used to determine the magnetic and crystallographic microstructures of the steels. Cementite has its own domain structure and stray fields which influence the magnetization process of the steel by its own magnetic contribution. When an external magnetic field is applied, the magnetization process in ferrite occurs mainly at lower fields through the 180° and 90° domain walls. A higher field is required for the observation of 180° domain wall movements in cementite.

1. Introduction

In most steels, among all the phases or constituents that can be obtained by choosing the chemical composition and thermo-mechanical treatments, two are frequently encountered: ferrite and cementite. Ferrite shows high ductility and low strength values and therefore low mechanical and also – very often – magnetic hardness in terms of coercivity. Cementite on the other hand shows high mechanical and magnetic hardness and is much more brittle. Thus, the relative volume fraction of the ferrite and cementite phases gives rise to the final mechanical and magnetic properties of the steel. The knowledge of the amount of cementite in steels is thus crucial.

Hysteresis loop and magnetic Barkhausen noise (MBN) evaluations are widely used magnetic measurement techniques for the microstructural characterization of ferromagnetic materials and determination of residual stress states. In physics, regular hysteresis measurements providing reproducible and reliable results can only be performed by using special hystrometer measurement devices asking for specially shaped test specimens like spheres and cylinders with well-designed geometry combined with encircling coils to measure the magnetic induction. As a consequence the technique cannot be applied to real components, as for instance vessel shells or pipes or high speed running steel sheets in a cold rolling mill [1], and is therefore destructive. All techniques based on magnetic circuit approaches [2] suffer from influence of lift-off on absolute value, ensuing value fluctuations, and shearing of the hysteresis curve [3]. Only in case of transformer steel sheets industrial consensus standards exist (i.e. Epstein frame measurements [4]) based on destructive batch tests. In contrast to these facts MBN-analysis can be performed with sensors positioned locally on top of the surface of a component, which means that MBN is non-destructive. However, the use of the magnetic Barkhausen noise techniques is “not yet” regulated by a standard. This makes the comparison of results published by different authors extremely difficult, especially as the side influences of different experimental conditions such as magnetization set-up, signal pick-up, band width, etc. are often insufficiently described. First attempts into standardization are initialized by the German Engineering Society (VDE Guidelines) [5].

During the magnetization process the domain structure of a ferromagnetic material is altered which involves different movements of the domain walls. There are exclusively 90°- and 180°-domain walls in case of iron materials. In the case considered here, the domain wall movement takes place in a microstructure consisting of a ferrite matrix with cementite precipitates. In basic physics the precipitates are either assumed acting as nonmagnetic foreign bodies [6,7] in the ferromagnetic matrix, or they are considered to

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interact with the domain walls of the matrix via their residual stress fields [8]. Domain walls tend to cling to nonmagnetic inclusions in order to minimize the magnetostatic and the wall energy. It was considered in the literature in a first approach that cementite behaves as a nonmagnetic inclusion in a ferrite matrix [9] or even as a nonmagnetic phase at all [10]. However, cementite is a ferromagnetic phase [11,12], and therefore – depending on its size, shape, crystalline orientation and amount of defects - it has its own domain structure and stray fields. The stray fields of inclusions, which are a source of magnetostatic energy, may also interact with the domain walls within the matrix. In order to reduce the magnetostatic energy, supplementary domains (closure domains) are built close to the inclusions which in turn interact with the domain walls in the matrix [13,14]. The other source of interaction of the cementite precipitates with the domain walls in the ferrite phase are – as mentioned above – the residual stresses which are built-up due to different thermal expansion coefficients of the two phases during the solidification process of the material, and lattice defects, e.g. dislocations, which are created on the interface between ferrite and cementite.

Several studies have been done on the individual influence of different microstructural parameters, e.g. second phase particles, on the generation of the magnetic Barkhausen noise. For example, the observed MBN activity profile in a microstructure containing cementite in a ferrite matrix showed either a single [15] or a double peak [8,16]. The description of a double peak for a Barkhausen noise profile has also encountered divergences in the literature. The weaker field peak was attributed to the pinning of 180° walls in the matrix by secondary particles and the stronger field peak was explained by annihilation of 90° domain walls at grain boundaries [17]. Contrarily, Moorthy et al. [16] stated that the weaker field peak is caused by irreversible domain walls in the ferrite and the stronger field peak by irreversible movement of domain walls overcoming second phase particles. By measuring the MBN signal as a function of temperature of a compact cementite and unalloyed white cast iron samples, Altpeter [18] demonstrated that the cementite actively produces its own MBN signal. With increasing temperature the ferromagnetic coordination decreases, and consequently the MBN signal intensity decreases. The Curie temperature of cementite (~210 °C) is lower than the Curie temperature of ferrite (~770 °C). Altpeter observed a Barkhausen noise amplitude of the compact cementite specimen, which decreased with increasing temperature and disappeared at the Curie temperature of cementite [18]. Furthermore, the MBN amplitude of white cast iron showed qualitatively the same behavior, i.e. it decreased strongly towards the Curie temperature of cementite and remained at a low almost constant level above this value. In addition, the MBN decrease of white cast iron was stronger with increasing amount of cementite.

In this work, we investigate the opportunity of assessing the relative proportion and contributions of the cementite and ferrite phases in unalloyed steels by optimizing the measured hysteresis loop and Barkhausen noise parameters. The evolution of the magnetic microstructure is directly correlated to the macroscopic measurement quantities by means of a superposed magnetic field applied to a Magnetic Force Microscope (MFM).

2. Materials and methods

Three different materials were examined in this study, a high purity iron (99.99%) and two unalloyed steels, Fe–0.8%C and Fe–1.5%C, containing globular cementite (Fe_3C) embedded in a ferrite matrix. The samples were provided as-cast and machined in a cylindrical shape of 8 mm diameter and 50 mm length. In order to remove all processing-induced residual stresses the samples were vacuum annealed at 600 °C for 4 h. The resulting microstructure has an average grain size of 80 μm for all samples. The size of the cementite precipitates ranges from a few hundred nanometers to about 10 μm in diameter.

The hysteresis loop and Barkhausen noise measurements were performed inside an electromagnet with a computer-controlled bipolar power supply (Fig. 1). The magnetic tangential field strength H was measured by a Hall probe. The cylindrical samples were magnetized along their axial direction up to a maximum magnetic field strength of 11,000 A/m at different excitation frequencies of 0.05 Hz, 0.1 Hz and 0.5 Hz, respectively. The change in magnetic flux density B and the magnetic Barkhausen noise amplitude M were measured by a pick-up coil with 300 turns (wire diameter: 0.1 mm, resonance frequency: 710 kHz) surrounding the sample (Fig. 1). The envelope of the noise signals (analyzed frequency range f_A=200 Hz–50 kHz) and the magnetic flux density were recorded as a function of the tangential field strength.

Small specimens ($3\times3\times1$ mm^3) were cut by spark erosion from the annealed cylindrical samples for the atomic force microscopy (AFM) and magnetic force microscopy investigations. The specimens were mechanically polished using standard procedures and slightly etched using Nital (95% ethanol+5% nitric acid). The AFM and MFM techniques were used to image the topography and the magnetic microstructure of the samples, respectively. The measurements were performed in tapping-lift mode using a commercial AFM/MFM instrument (Nanoscope III® multimode, Bruker AXS Inc. (formerly Digital Instruments/Veeco), Madison, WI, USA). The sensor tips were CoCr-coated with a coercivity of ~32,000 A/m (MESP, Bruker AXS Inc., Madison, WI, USA). The topography images show the local height of the sample surface displayed in gray scales. The magnetic images are taken by vibrating the AFM sensor at its resonance frequency at a predefined lift height above the sample surface. The gradient of the magnetic interaction forces cause a phase shift in the cantilever vibration which is displayed in gray scales. A lift-height of 60–100 nm was chosen for all measurements reported here. In order to investigate the evolution of the magnetic microstructure and the resulting domain configuration, an external electromagnet was combined with the MFM as shown in Fig. 2. The pole shoes of the electromagnet were adjusted such that the sample inside the AFM was magnetized parallel to its surface.

3. Results and discussion

3.1. Bulk magnetic Properties

The unalloyed steels investigated here consist of a relatively hard ferromagnetic phase (cementite) embedded in a soft ferromagnetic phase (ferrite). The different microstructural states lead to characteristic changes of the hysteresis loops and Barkhausen noise profiles as shown in Fig. 3.

Magnetic hysteresis curves of the three samples for three different frequencies f (0.05 Hz, 0.1 Hz, and 0.5 Hz, respectively) of the applied external field are shown as dotted lines in Fig. 3. The coercivity H_c and loss per cycle W increase with frequency and carbon content, while the relative permeability μ_r and the saturation magnetization B_S at 10,000 A/m decrease with increasing frequency f of the applied magnetic field. This means that the material reacts magnetically harder with increasing frequency of the external magnetic field. In addition, the magnetic hardness increases as the carbon content increases. A summary of the measured parameters is given in Table 1.

With increasing amount of carbon in form of globular cementite precipitates in the ferrite matrix the pinning of the domain walls in the ferrite matrix is enhanced due to the presence of the