MATERIALS CHARACTERIZATION BY ELECTROMAGNETIC NDE FOCUSING ON THE PETROCHEMICAL INDUSTRY

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ABSTRACT

Electromagnetic nondestructive evaluation (NDE) is widely used for detection and sizing of defects in industrial equipments. Since this technique measures the electric and magnetic properties of materials, it can also be used for their characterization. This is particularly true in the case of material degradation during “in service” operations where the microstructure may experience some changes which can be correlated to its electromagnetic properties. This paper reviews the operation principles and some applications of electromagnetic NDE currently used to characterize materials degradation in the petrochemical industry.

Keywords: Materials Characterization, Electromagnetic Testing, Petrochemical Industry

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magnetic permeability, and a gradient of magnetic properties is formed through the thickness of the tube wall. The method was applied on test specimens representing serpentines in mint condition and on samples taken from furnaces where they had been subjected to distinct conditions over different operational times. The degree of carburization of these tubes was successfully determined by measuring the volume fraction of chromium carbides and following their through-wall distribution. An NDE technique that determines the distribution of the volume fraction of the chromium carbides through the wall is described in the present paper. The objective is to characterize the structural evolution in the tubes during carburization correlating it to their ferromagnetic content.

**DUPLEX STAINLESS STEELS**

**Microstructure**

DSS samples with varying amounts of γ austenite and δ ferrite can be obtained by heat treatment at different temperatures and times. It is also possible to induce the precipitation of different volumetric fractions of σ phase. Figure 1 is an example of some of these microstructures. It shows the scanning electron microscopy (SEM) microstructure images of an as received DSS sample with similar amounts of δ and γ phases. After heat treatment the γ/δ ratio changes (figures 2b and 2c) and some σ phase precipitation can be observed in the DSS (figure 2d).

**Eddy current measurements**

The eddy current (EC) probe used for the measurements has a cylindrical design, with height of 30 mm, a core with diameter of 10 mm, and a bobbin with 1000 turns, external diameter of 19 mm and height of 4.5 mm [2]. On both situations the bobbin was operated with an external perturbation (Sine) of 4 Vpp and frequency of 4 kHz. A gain = 32dB, probe drive = 4 V, Y-spread = 10 dB and phase = 0 deg were used with the OmniScan equipment in order to obtain those results. The lift-off signals for the eddy current measurements in the impedance plane obtained are shown in figure 2. It is clear that this technique can successfully be applied to differentiate the samples tested in this study, which received different heat treatments and thus exhibited different volumetric fraction of σ phase (figure 2a).

The presence of σ phase causes a change of the electromagnetic properties of DSS. As already mentioned, ferrite is

![Fig. 1: SEM micrographs of a microstructure of DSS samples. In these images, the σ, δ and γ phases are clearly indicated by arrows: (a) Solubilized sample with δ/γ = 50/50, (b) Sample with δ/γ = 30/70, (c) Sample with δ/γ = 20/80 and (d) Sample with σ phase precipitation.](image)

![Fig. 2: Lift-off signals in the impedance plane signals obtained for the samples through the EC technique with frequency of 4 kHz. (a)Samples with different amounts of σ phase and γ/δ ratio of 50/50. (b) Samples without σ phase but with varying amounts of γ/δ ratio.](image)
ferromagnetic while \( \gamma \) austenite and \( \sigma \) phases are paramagnetic. Thus an increase of \( \sigma \) phase percentage and the resulting decrease of \( \delta \) phase volumetric fraction render the material behavior more paramagnetic. This characteristic makes the EC technique an interesting non destructive tool for the detection of material degradation caused by an increase of \( \sigma \) phase percentage. But it could be argued whether the difference depicted for the samples through the EC technique, figure 2a, could be due to a decrease in the \( \delta \) ferrite which necessarily follows \( \sigma \) phase precipitation. The answer can be obtained from figure 2b. The lift-off signals of samples free from \( \sigma \) phase but with different amounts of \( \gamma/\delta \) ratio are exactly the same.

SERPENTINE TUBE CARBURIZATION IN PYROLYSIS FURNACES

Microstructure

The temperatures required for the cracking process in a pyrolysis furnace give rise to surface temperatures higher than 1000°C in some regions of the serpentines. Such conditions allied to the nature of the fluid, facilitate the diffusion of carbon in the tubes through the internal surface, causing the detrimental effect known as carburization. Carbon precipitates in the austenitic matrix as chromium carbides, as depicted in the optical micrographs, figure 3. As expected, the chromium carbide volumetric fraction increases with the exposure time at high temperature. In each cross-section, there is also an increase in the chromium carbide volumetric fraction from the outer wall to the inner wall. Figure 4 shows the variation of the carbide volumetric fraction along the wall thickness for several carburized HP samples extracted from furnaces in service over different operation times [3-4].

Magnetic Measurements

The technique used measures the density of the magnetic flux generated from a small magnet placed near the external surface of the specimen. A magneto resistive sensor measures the magnetic field. The value of the magnetic field in air is subtracted from the value measured in the specimen, obtaining the response of the field. The magnetic flux density values of each sample are displayed in figure 4. The magnetic flux

![Fig. 3](image-url)  
Samples removed from a deactivated pyrolysis serpentine furnaces after being in operation for 25,600 hours. Optical micrographs from samples ranging from the internal surface of the tube (a) to the center (b) and finally close to the external wall. Chromium carbides are delineated by the etching.

![Fig. 4](image-url)  
Carbide volumetric fraction along depth, measured from the external wall. Exposure time of each sample: S0, as received, S1, 6,800h, S2 25,600h and S3 more than 90,000 hours.

![Fig. 5](image-url)  
Area under the chromium carbide volume fraction curves (arbitrary units) versus the measured magnetic flux density.
density of samples in whose carbide volumetric fractions are depicted in figure 4, ranges from 20μT for the as received sample to a maximum of 413μT for the samples having operated for more than 90,000h. It is possible to establish a linear relationship between the magnetic flux density and the areas under chromium carbide volumetric fraction curves as shown in figure 5. This relationship is expected as the area under these curves is proportional to the total number of carbides along each sample wall thickness [3-4].

From these results, it can be stated [3-4] that HP steel, when not carburized, is paramagnetic and has an austenitic microstructure, but as the degree of carburization increases it gradually becomes ferromagnetic, due to the loss of chromium from the matrix through chromium-carbide precipitation. Carburization increases the magnetic permeability, creating a gradient of magnetic properties through the thickness of the tube wall.

CONCLUSIONS

Electromagnetic nondestructive evaluation has proved to be an efficient tool for materials characterization. This technique can be successfully applied to materials with a microstructure feature that can be correlated to an electromagnetic or electric property. However, for this kind of evaluation it is necessary that just one microstructure feature is responsible for the observed material property change.

A limitation of this technique is the necessity of a preliminary calibration which in most cases implies in having standard test pieces that are not always available. This limitation can be overcome through modeling and simulation tools.

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REFERENCES