AFM measurements on ferrous materials

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The Atomic Force Microscope (AFM) has been successfully used for the characterisation of the surface topography of a very wide range of materials. The AFM is an interesting technique to study the surface of materials and specifically to study surface effects in steel, e.g. after polishing and/or after etching. This technique allows not only to study the topography of the samples, but it is also a useful tool to obtain structural details of surfaces and for the identification of different phases. In this paper, some applications are shown concerning the study of the microstructure of multiphase-steels.

Key words: atomic force microscope, steel metallography, multiphase alloys

Uso de la Microscopía de Fuerza Atómica (AFM) en materiales ferrosos

El Microscopio de Fuerza Atómica (AFM) es utilizado en la caracterización topográfica de una gran variedad de materiales. El AFM es una interesante técnica para el estudio de la superficie de los aceros, ya sea con ataque químico o no. Esta técnica es también una útil herramienta para obtener detalles estructurales en las superficies y en la identificación de fases. En este trabajo algunas de estas aplicaciones son mostradas en el estudio de la microestructura de aceros multifásicos.

Palabras claves: Microscopio de Fuerza Atómica, metalografía de aceros, aleaciones multifásicas

1. INTRODUCTION

Microstructural observations in multiphase steel are usually made with optical microscopy, scanning and transmission electron microscopy, X-ray diffraction and color etching methods. The use of AFM for the study of multiphase steels can give good results concerning phase identification and better understanding of the distribution of phases through the application of the different techniques that it offers:

- Identification through the analysis of the topography of an observed region
- Identification using Lateral Force Microscopy (LFM)

The topographic study can be carried out using indistinctly Contact mode or Non contact mode. The result will be an image that corresponds to the topography of the sample due to the deflection of the cantilever. In our experiments we used the Contact mode regimen: an image is obtained corresponding to the topography of the surface due to direct contact between the tip and the sample. The repulsive forces between the tip and the surface cause to bend the cantilever. A laser beam is used to measure the cantilever deflection as the tip is scanned over the sample, or the sample is scanned under the tip. The cantilever deflections allow a computer to generate a map of the surface topography.

Furthermore, working in the Contact mode simultaneous images can be obtained in LFM to establish comparisons between the images. The images in LFM show changes in the physical properties of the surface analysed. In multiphase regions, the tip experiences changes in friction when it crosses

some areas causing bigger or smaller torsion of the cantilever. This will be recorded in the image through changes of colour contrasts and it will be possible to carry out qualitative comparisons between the different areas (a quantification of the results obtained can also be carried out). The images in LFM can be obtained indistinctly on polished or attacked samples, because the image is a map of the physical properties of the surface. When the image is taken of a polished sample the interpretation is easier because there is no signal due to topography.

2. EXPERIMENTAL PROCEDURE

Two samples of multiphase steels were selected, sample A (0.11%C, 1.53%Mn, 1.26%Si) and sample B (0.19%C, 1.57%Mn, 1.46%Si). In both samples intercritical annealing was done at 800 °C to obtain a two-phase structure of ferrite and austenite. Sample A was quenched in water immediately. After this treatment, all the austenite should have transformed to martensite, but frequently, a small fraction of the austenite does not transform. The final microstructure was composed by the phases martensite and ferrite.

In sample B, the intercritical annealing was followed by a salt bath quench, where the specimen was held isothermally for 2 minutes at 400 °C in order to transform the austenite into bainite. The final microstructure contained ferrite, bainite and austenite. Finally the sample was cooled down in air.

The attack by the LePera method (tint etching) allows the identification of the phases in optical microscopy according to

Figure 1. Light micrographs of steel A (a) and B (b) after tint etching by the LePera technique (1)

the coloration taken during the attack. Figure 1a shows the microstructure of the steel A where the ferrite takes a brown colour and the yellow colour corresponds to the martensite areas. In the sample B (figure 1b) three phases are identified, polygonal ferrite of green colour, brown bainite and yellow austenite. However it is impossible to distinguish between the austenite and the martensite.

2.1 Study of sample A with AFM

Figure 2 shows the topographic image in three dimensions, the higher areas have been identified as martensite and the lower areas as ferrite. In specimens attacked with Nital, the ferrite is attacked more deeply than other phases, so the ferrite regions will always be lower in relation to the regions of other phases. Also the typical polygonal shape of the ferrite allows a clear identification.

In the zones identified as martensite it is possible to distinguish clearly the presence of two regions with different morphology (figure 3). One zone (I) seems to be more resistant to the attack with Nital: the topographic level is higher and a higher degree of roughness is observed. The other zone (II) is included inside the first zone with lower roughness and a lower height. This way, we defined two kinds of martensite, called MI and MII.

The topographic study confirmed the presence of three regions in the sample, which have differences in height (figure 4). The height histogram shows the distribution in three peaks

Figure 2 Topographic image of martensite and ferrite regions. Field of view $18\mu\text{m}$

Figure 3. AFM images on regions of martensite. The heterogeneity of martensite can be easily distinguished. (b) Detail of the martensite regions.

Figure 4 Topographic study showing the presence of three regions with different height. (Detail of figure 3)

Figure 5 Topographic (a) and LFM images (b and c). Field of view $20\mu\text{m}$

Figure 6 Possible austenite zones in the martensite

corresponding to ferrite (lowest level) and two types of martensite (MI and MII) and a calculation confirms the previous affirmation: the heights have been normalised, ferrite was located in level 0 and martensite I in 100, being calculated MII according to this scale. The MII level was always found between 60 and 75.

Figure 5 shows the topographic image (5a) and the images simultaneously obtained in LFM mode (5b and 5c). The contrast changes in the analysed area with LFM reveal the presence of changes in properties: a bright pattern in the trace image becomes a dark pattern in the retrace image and vice versa, which coincides with the martensite and ferrite areas. For example, the area identified as ferrite in the image 5b appears as a step and in the other scan direction (figure 5c) it appears like a cavity. The combination of the topographical and LFM images permits to distinguish clearly between ferrite and martensite, nevertheless, there are no differences inside the martensitic regions.

Figure 6 shows the lath structure of the martensite and areas where there possibly is some quantity of retained austenite between the laths, while the structure of the area MII corresponds to another type of martensite.

Figure 7 Lath of bainite surroundings by austenite. Field of view $3\mu\text{m}$

2.2. Study of sample B with AFM

The sample B should present three phases, ferrite, retained austenite and bainite, being possible also some martensite. It is well known that austenite and bainite can be identified according to the kinetics of bainite formation during the ageing stage. Bainite transformation proceeds by repetitive events leading to the formation of many sheaves of bainite across the prior austenite grains. The kinetics of bainite transformation is therefore controlled by the nucleation rate of individual sub-units which nucleate at austenite grain boundaries and by the growth mechanism segregating the excess of carbon into the surrounding austenite, which becomes consequently enriched in carbon. The displacive transformation of a new sub-unit in this zone can thus be prevented since nucleation is energetically favoured in low carbon austenite. This inhomogeneous carbon distribution therefore explains the sheaf morphology commonly observed for bainite (2,3).

According to this explanation retained austenite has been mainly identified in the central areas and bainite in the border areas of the former austenite grains. Measurement which XRD show that the quantity of retained austenite in this sample is 9.5%. Making use of the topographical analysis in AFM it is possible to demonstrate that there are indeed three types of phases in the sample, bainite corresponding to the higher areas while the height of the austenite is intermediate between the bainite and the ferrite. Nevertheless, in this case we were not able to quantify typical height levels for each phase, as we could do in sample A. Further investigation will help us to identify clearly the height levels. In figure 7 two laths of bainite are observed, surrounded by austenite.

3. CONCLUSIONS

The topographic study in sample A showed that the higher areas in the sample correspond to the martensitic phase. The lower areas correspond to ferrite.

In the sample A the martensite is not homogeneous, the images show and the topographic study indicates the existence of two types of martensite. It is possible to explain this through the inhomogeneous manganese distribution in the prior austenitic grain. During the formation and growth of the austenite grain, a gradient of manganese concentration will exist from the center to the grain boundary. Several models explain the diffusion of this element during this stage, transport of manganese either through the ferrite or along the grain boundary leads to the formation of a high-manganese rim around the austenite grain. As a result, this rim could be the reason of the martensite differences.

Through the topographical analysis in sample B it is possible to demonstrate that three types of regions exist in the sample, differed by the heights. Bainite corresponds to the higher areas, the austenite in the intermediate areas between the bainite and the ferrite. Nevertheless it is not always possible to affirm categorically that the identification of the phases is the correct.

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