EFFECT OF ISOTHERMAL HEAT TREATMENTS PARAMETERS ON NANOSTRUCTURED BAINITE TRANSFORMATION

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Summary: Nanostructured bainitic steels have a microstructure consisting of nanosize baintic ferrite plates interwoven with thin films of carbon enriched retained austenite. Such microstructure can be obtained in specially designed steels inhibiting the precipitation of cementite during isothermal transformation at low temperatures, between $150 - 350^{\circ}$ C, by the addition of a high Si content (1.5 - 2 wt. %). In this work, the evolution of bainitic transformation in two high silicon steels was studied between 300 and 350°C, by the complementary use of High Resolution Dilatometry, X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM). The influence of different heat treatments parameters, i.e. isothermal temperature and time, on the phase fraction and their chemical compositions, as well as austenite thermal stability has been studied as a function of the transformation degree.

Keywords: nanobainite, isothermal treatment, phase transformations, thermal stability.

1. INTRODUCTION.

Nanostructured bainite refers to a microstructure composed of nanocrystals of bainitic ferrite interwoven with thin films of retained austenite, the microstructure is essentially carbide free due to the judicious use of Si [1, 2]. Such microstructure is specially obtained in steels by isothermal transformed at very low temperatures, resulting in excellent combination of strength and ductility [3, 4] with no need of severe plastic deformation or complex thermomechanical treatments [5]. The nucleation of bainite takes place under paraequilibrium conditions, i.e. only C diffuses, and its growth is displacive and diffusionless in nature. Displacive growth of bainitic ferrite implies the deformation of the parent austenite leading to a shape change in the transformed region, with large shear and small dilatation normal to the plane [6]. Immediately after the growth of a plate of bainitic ferrite, the excess of C is rejected to the parent austenite, increasing its thermal stability and decreasing the martensite starttemperature (Ms). From the point of view of mechanical properties, it is desirable that the C enriched austenite remains stable at room temperatures after the isothermal treatment (Ms $< 25^{\circ}$ C), since formation of martensite might impair toughness of such microstructures. What is more, in order to get a better balance between the strength and ductility, it has been proven that from the two present austenite morphologies, blocks, between the sheaves, and thin films, between the plates of bainitic ferrite, the thin films must be favored due to their higher mechanical stability and better mechanical response. In any case, further developments on these type of alloys goes through the understanding of its evolution during the whole transformation process. Therefore, this work is focused on the study of the bainitic transformation

evolution and the detailed study of the different phases obtained at room temperature, between 300 and 350°C, both temperatures between the bainite-start temperature (Bs) and Ms [7], of two high silicon steels.

2. EXPERIMENTAL PROCEDURE.

Chemical composition of steels used in this study is shown in Table 1. The amount of Si was enough to inhibit the cementite precipitation, while C, Mn and Cr ensure low bainitic transformation temperatures.

Table 1. Chemical Composition of the alloys (wt. %)

	С	Si	Mn	Cr	Mo	Си
1C2Si	1.00	2.45	0.75	0.98	0.02	0.19
04C3Si	0.43	3.05	0.71	0.97	0.21	0.14

Cylindrical specimens of 4 mm in diameter and 10 mm in length were machined to be used in a High-Resolution Dilatometer Bahr DIL805, where all the heat treatments were performed. The materials were austenitized at two different temperatures: 950 °C during 5 min for 1C2Si and 990 °C during 4 min for 04C3Si. Then, samples were cooled down to the selected isothermal temperatures, i.e. 300 °C and 350 °C. The cooling rate was high enough to avoid the formation of ferrite and pearlite prior to bainite transformation. Finally, specimens were cooled down to room temperature at 10 °C/s. A comprehensive diagram of the heat treatment is shown in Figure 1. Different times for the isothermal treatment were considered in order to evaluate the evolution of the transformation and its effect on the final microstructure.

For microstructural characterization, Field Emission Gun Scanning Electron Microscopy, FEG-SEM Hitachi S48000, and X-Ray Diffractometer, Bruker AXS D8, were used. FEG-SEM operated at an accelerating voltage of 7 kV. The preparation of samples for SEM was standard grinding with SiC paper from 600 to 1200 grit, and final polish with diamond paste from 3 to 1 µm. The microstructure was revealed by chemical etching with 2% Nital. Bruker AXS D8 diffractometer is equipped with a Co X-ray tube, Goebel mirror optics and a LynxEye Linear Position Sensitive Detector for ultra-fast XRD measurements. In this study, the version 4.2 of Rietveld analysis software TOPAS (Bruker AXS) was used to quantify the volume fraction of different phases, the lattice parameters and the carbon content in solid solution. The lattice parameter was calculated by Dyson-Holmes equation [8]. Specimens for XRD were prepared in the same way as for SEM. Subsequently, Vickers hardness was performed according to ASTM E92. Hardness values (HV10) corresponds to an average of at least 3 measurements.



Figure 1. Heat treatments performed by dilatometry. T_{γ} and t_{γ} stands for austenitization temperature and time, respectively.

3. RESULTS AND DISCUSSION.

The dilatometry results of 1C2Si and 04C3Si steels, corresponding to complete transformation at 300 and 350°C, are shown in the Figure 2, where it is possible to distinguish three regions:

- *Incubation period* where the transformation has not started and the microstructure is still fully austenitic. Relative change in length is cero.
- *Transformation period* when the nucleation and growth of bainite occur. Rapid increase in the relative change in length.
- *Steady state,* where the transformation has finished and no further transformation occurs. Relative change in length is constant with time.

In the presented cases in Figure 2, it is clear that the steady state has been reached, and therefore the transformation is assumed to be finished. At this stage the microstructure is only composed by two phases, bainitic ferrite and retained austenite [4, 9]. For the same steel, the amount of bainitic ferrite that is transformed increases as the isothermal transformation temperature decreases, which is in agreement with the incomplete reaction phenomena ruling this transformation, T_0 line concept [10, 11]. Also, and in

accordance with the same theory, the amount of bainite obtained in the lower carbon content alloy (04C3Si) is higher than that of the 1C2Si at the same isothermal transformation temperature.

As expected and observed in Figure 2, the relative change in length is directly related to the amount of bainitic ferrite that it is formed, so, higher fraction of bainite leads also to bigger longitudinal expansions in dilatometry. The explanation resides in the fact that these expansions are associated to the difference in the specific volume between austenite, face-centered cubic and (FCC), bainitic ferrite. body-centered tetragonal/body-centered cubic (BCT/BCC), due to the change of crystal structure and the variation of volume due to the carbon enrichment of retained austenite [12]. It is also worth mentioning that the transformation is faster the higher the transformation temperature is, characterized by a decrease in the incubation time and the time needed to reach a steady state.



Figure 2. Relative change in length obtained by Dilatometry for a) 04C3Si and b) 1C2Si

In order to study the evolution of the bainitic transformation, three intermediate isothermal times were defined and new interrupted dilatometric tests were performed. Details of the degree of transformation corresponding to the mentioned interrupted tests are shown in Table 2.

In this work, special care was taken in analyzing the possibility of martensitic transformation on cooling to room temperature after isothermal treatment, as a way to stablish the thermal stability of austenite. In a similar manner, the microstructure was described in terms of phase fractions, their C contents determined by XRD and overall hardness as a function of the transformation degree.

The SEM micrographs sequence in Figure 3 clearly show a gradual increase of bainitic ferrite (α_b) as the transformation progresses. In addition, the two mentioned morphologies of retained austenite were observed regardless of temperature and chemical composition: block (γ_b) and thin films (γ_f). Thin films can be found between the bainitic ferrite plates while blocky austenite can be observed between the sheaves of bainite (aggregates of plates of ferrite separated by untransformed austenite).



Figure 3. Micrographs obtained by SEM as a function of the transformation degree for 1C2Si at 350°C

When the degree of transformation is low, and not suffice enrichment of C happens in austenite, its corresponding Ms is higher than room temperature, and the final microstructure is composed of bainitic ferrite, retained austenite and martensite (α '). As the

transformation progresses, further enrichment of C in austenite takes place, decreasing its corresponding Ms and therefore the amount of martensite formed. There is a "degree of transformation" that allows for the C enriched austenite to have a Ms below room temperature, and the final microstructure is then solely composed of bainitic ferrite and retained austenite. This is also supported by results in Table 2 that gathers the Ms measured by dilatometry under all conditions. As already mentioned, an increase of the isothermal temperature leads a decrease of the amount of bainitic ferrite formed, which implies that the percentage of retained austenite is higher, but less enriched in carbon, therefore the martensite formation is more favorable [13]. Obviously, if the bulk carbon content is higher, although less bainite is formed, austenite needs of less carbon enrichment to be stable at room temperature, and this is clear in the Ms listed in Table 2. On the contrary, for the lower carbon steels, 04C3Si, the level of C enrichment might not be enough even at 100% of transformation, as shown for the case of 350°C, and martensite will be always present in the microstructure.

Table 2. Experimental Ms as a function of the transformation degree. RT stands for room temperature.

	1C2	Si	04C3Si		
TISO	Transf	Ms	Transf	Ms	
(°C)	(%)	(°C)	(%)	(°C)	
	40	73	43	254	
200	63	53	51	213	
300	75	< RT	67	140	
	100	< RT	100	< RT	
	36	110	19	276	
250	46	98	60	242	
350	85	52	72	205	
	100	< RT	100	< RT	

The performed XRD analysis clearly supports the mentioned behavior, see Figure 4. It has to be noted that martensite by XRD was only distinguishable in the 1C2Si alloy due to its high tetragonality as compared to that of bainitic ferrite. Due to its lower carbon content, and lower martensite tetragonality, in the case of the 04C3Si it was impossible to distinguish bainitic ferrite from martensite and therefore there is a certain region of the plots where the results of both phases overlap, those corresponding to the degrees of transformation where an Ms was detected in Table 2. In general it is clear that the C content of bainitic ferrite is fairly stable through the whole transformation, as already reported [14], being around 0.2 wt.%. Austenite is enriched in C as the transformation proceeds, and eventually is stabilized at room temperature.

As expected, at low transformation degrees, the martensite formation is more favorable and the volume fraction decreased until becomes zero as see in Figure 4. The martensite carbon content remained constant close to 0.9 wt. % when it was present in the microstructure, while that the bainite carbon content was 0.2 wt. % for both conditions [10].



Figure 4. Evolution of carbon content and volume fraction of different phases obtained by XRD and Vickers Hardness as the bainite transformation progresses.

In addition, as expected the austenite and martensite carbon content experimentally obtained were not the same at the end of heat treatment due to the different C enrichment of austenite before cooling to room temperature. The blocky austenite has lower carbon content than films, hence the blocks transformation to martensite will be more favorable, while the films trapped between subunits of bainitic ferrite will remain retained and carbon enriched [13]. All these changes are also reflected in the evolution of the HV values, see Figure 4. At low transformation degrees, high hardness values were due to the presence of a high amount of martensite. When the transformation progresses, a decrease of hardness was observed because the volume fraction of martensite decreases in favor of bainitic ferrite. In the most advanced states of transformation, when the martensite transformation doesn't occur. hardness will be mainly controlled by the bainitic ferrite volume fraction.

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