

THERMAL AND PHASE TRANSFORMATIONS ANALYSIS IN A PREMOMET® STEEL

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Thermal analysis in a PREMOMET® steel has been performed by differential scanning calorimetry (DSC) and high-resolution dilatometry. The phase transformation temperatures (A_{c1} , A_{c3} , M_s and M_f) of this steel were obtained by the two methods at different heating rates showing good agreement between both techniques. The enthalpy of α - γ transformation for this steel was measured using the thermograms acquired by DSC and microstructure was analyzed by scanning electron microscope (SEM). The results showed that this steel retained a martensitic structure for all conditions.

Keywords: martensitic steel, phase transformation, DSC, dilatometry, microstructure.

INTRODUCTION

PREMOMET® is a high strength martensitic steel, it was produced using combination of vacuum induction melting (VIM) and vacuum arc remelting (VAR) and followed by casting as ingots. This steel offers an attractive combination of high strength and toughness in the quench and tempered condition [1]. A fundamental investigation on the high temperature thermodynamic stability in martensitic steels are very important due to that the microstructural conditions at these temperatures should provide the required set of mechanical properties and corrosion resistance [2]. Phase transformations are one of the factors that most influence in the mechanical properties of steels, especially the γ - α transformation [3], while the martensite formation depends on the austenite chemical composition and defects in the structure [4, 5].

Different thermal analysis methods can be applied in order to analyze the phase transformation temperatures.

One of them is the dilatometry, which provides information on the consequences of transformations by the study of changes in the sample length, related to phase change, the lattice structure become altered and produces a change in specific volume [6, 7].

DSC is a very powerful method for the non-isothermal study of solid-state phase transformations in material science [8, 9].

On the other hand phase transformation temperatures of PREMOMET® steel has not been investigated extensively. In this work were measured A_{c1} , A_{c3} tem-

Table 1 **Chemical composition of PREMOMET® steel / %wt**

C	Si	Ni	Co	Mn	Cr	Mo	V	Fe
0,4	1,5	3,8	0,5	0,9	1,25	0,5	0,3	Bal.

peratures and the martensitic transformation using thermal analysis techniques like dilatometry and differential scanning calorimetry (DSC). These results were correlated with microstructural analysis and microhardness tests.

EXPERIMENTAL PROCEDURE

Samples of PREMOMET® steel were obtained from a billet in form of a slide of 6 inches of diameter, the material was sectioned from an ingot and was solubilized at 1 040 °C, the chemical composition of this steel is shown in Table 1.

For dilatometric studies a set of 4 mm diameter and 10 mm length samples were cut and machined, then were analyzed using a L78 R.I.T.A. high-resolution dilatometer. During the tests, specimens were protected from oxidation using helium gas; the heating rates were 60 and 600 °C/min, for this tests the samples were heated up to 850 °C for 2 min to reach austenite region in this steel, the cooling rate was 18 °C/min for all tests.

Calorimetry tests were performed in a Differential Scanning Calorimeter NETZSCH model DSC 404C Pegasus, using platinum pans for sample and reference with an argon atmosphere in order to protect the samples from the oxidation. The studies were carried out using small discs, 6mm diameter and 13 - 16 mg. These samples were prepared by mechanical thinning and finally electropolishing in order to eliminate the microdeformation caused by the previous step. Samples were

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heated until 850 °C for 2 minutes using two heating rate, 10 and 25 °C/min and were cooled at 18 °C/min.

Samples were sectioned transversally, mechanically polished and etched with Vilella's reagent for metallographic inspection after dilatometric tests, after that microhardness tests were performed in each sample, using a Shimadzu microindenter with a load of 980,7 mN for 15 sec.

RESULTS AND DISCUSSION

Figure 1 shows the micrograph of PREMOMET® steel as received condition, it can be observe that the material has a martensitic structure, and the hardness was measured of 640 HV, this values corresponds with a martensitic microstructure typical for this type of steels [1].

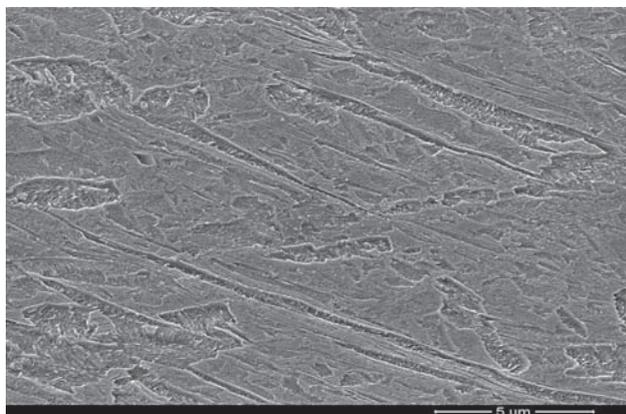


Figure 1 Micrograph of PREMOMET® steel as initial condition.

Figure 2 shows a DSC thermogram, the heating and cooling rates were 10 °C/min and 18 °C/min respectively, two endothermic peaks can be observed and were related to Ac_1 transformation temperature, which corresponds to austenite temperature onset, this transformation starts at 690 °C and Ac_3 temperature of 820 °C was measured. During the cooling down, it can be observed that the adiffusional formation of martensite resulted in a decomposition of austenite [10] started at 415 °C, this value was considered as M_s temperature while M_f was measured at 364 °C with an enthalpy formation of 170 J/g. These temperatures were estimated from the analysis of the energy curves using the change in the slope for each transition and complemented with the calculation of the first derivate of the curve, the same analysis was carried out on each curve at different heating rates.

Figure 3 shows the thermogram with heating condition of 25 °C/min and cooling condition of 18 °C/min, where the Ac_1 temperature was measured at 692 °C and Ac_3 at 827 °C. The martensitic transformation was observed in the cooling down at 407 °C in the starts and the M_f was registered at 363 °C with finally enthalpy formation of 180 J/g. In DSC results Ac_1 and Ac_3 temperatures were similar due to the heating rates are similar, it can be observed the presence of two peaks in the

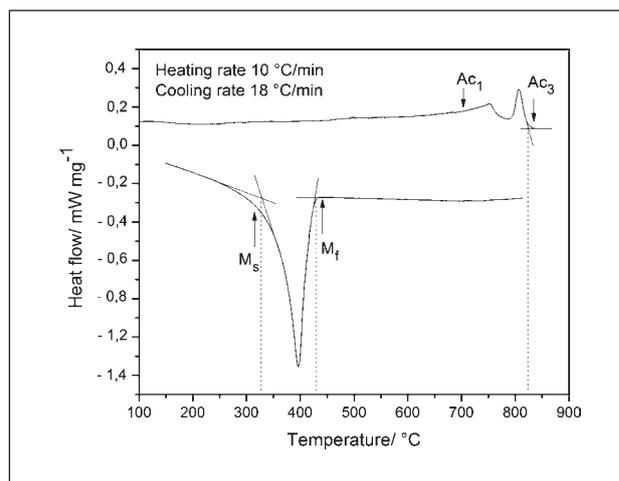


Figure 2 DSC thermogram using a heating rate of 10 °C/min showing the transformation temperatures using intersection lines method.

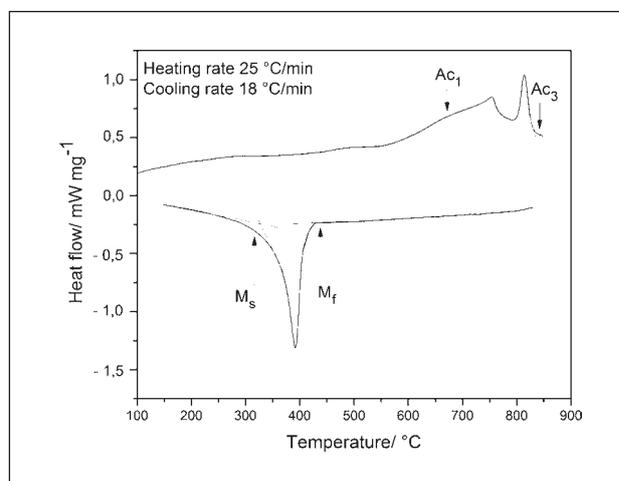


Figure 3 DSC thermogram using a heating rate of 25 °C/min showing the transformation temperatures using intersection lines method.

curves in the region of the α - γ transformation. This is associated with a change in the mechanism of phase transformation: the formation of austenite starts by the shear mechanism and then the transformation continues as diffusive [11,12].

Figure 4 and 5 show the length changes of PREMOMET® steel during heating and cooling rates. In the heating cycle it can be observed the α - γ transformation, which correspond to the Ac_1 - Ac_3 temperatures, while martensitic transformation was measured during cooling at 18 °C/min from the austenitic region. All transformation temperatures were determined with the changes in the curve slope and calculating the first derivate function.

At 60 °C/min the Ac_1 temperature was measured at 698 °C and Ac_3 temperature at 757 °C, during cooling can be observed an expansion, which represents the start and end of martensitic transformation, this were estimated at 243 °C and finished at 172 °C as shown in Figure 4.

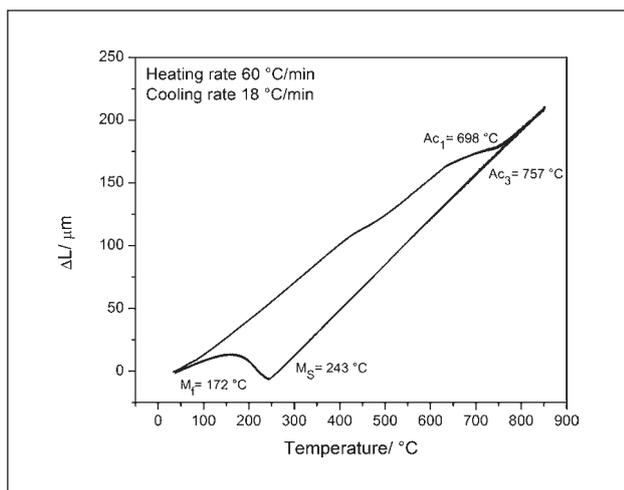


Figure 4 Experimental dilatometric curve of PREMOMET® steel heated at 60 °C/min.

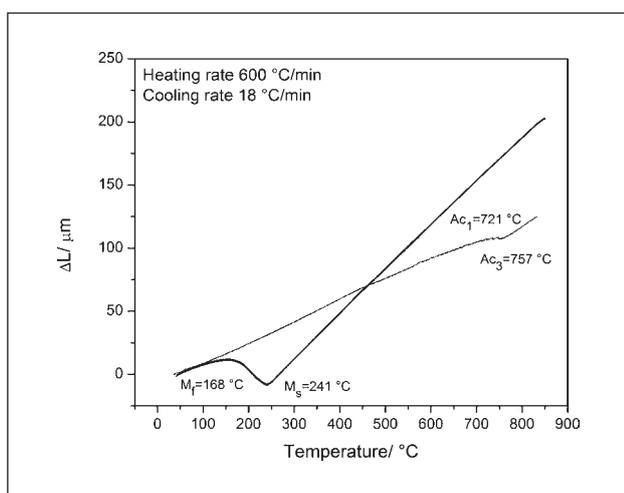


Figure 5 Experimental dilatometric curve of PREMOMET® steel heated at 600 °C/min.

Figure 5 shows the dilatometric curve for the steel heated at 600 °C/min; though this heating rate the Ac_1 and Ac_3 temperatures were registered at 721 °C and 757 °C respectively and the M_s and M_f , which corresponds to the martensitic transformation, were determined at 241 °C and 168 °C.

The Ac_1 and Ac_3 temperatures measured are similar in two techniques. Nevertheless in the martensitic transformation can be observed a data differences, this can be attributed to the martensitic transformation occurred at high velocities and the cooling is not controlled in the same way in both techniques employed. Additionally in both dilatometric curves, it was observed a change in the slope during the heating in the range of 450 °C. In this region of temperatures is possible the loss of tetragonality in the martensitic structure and the transition carbides.

Figure 6 shows the microstructure after dilatometric test, this is very similar in both heating rates and it only presents a martensitic structure without evidence of precipitates at least in SEM. The hardness likewise does not show a significant change, which was measure at

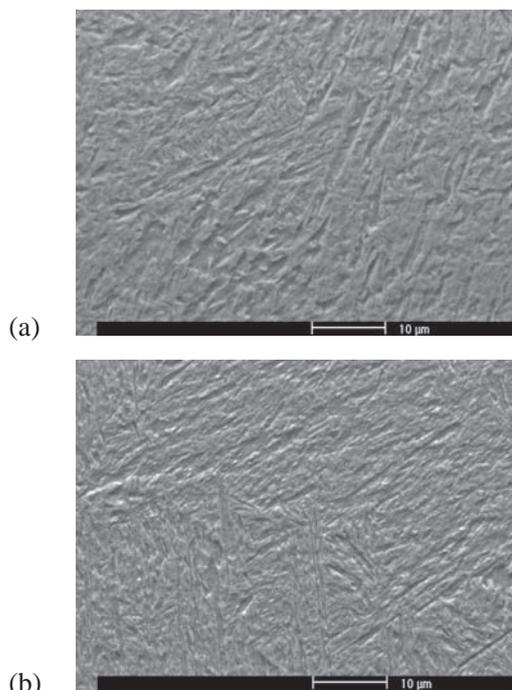


Figure 6 Microstructure after dilatometric test;... (a) 60 °C/min, (b) 600 °C/min.

622 HV for a heating rate of 60 °C/min and 632 HV for 600 °C/min.

CONCLUSIONS

A comprehensive characterization of phase transformations in a PREMOMET® steel has been performed. The Ac_1 and Ac_3 temperatures in the PREMOMET® steel are similar in each technique used in this investigation. M_s and M_f temperatures were measured in both techniques: in DSC the martensite formation appears as an exothermic peak and in the dilatometric curves there is an evident expansion caused by the change of crystalline structure, on other hand all the conditions analyzed in the steel shows a martensitic microstructure, this steel does not evidenced show precipitates in the microstructure and finally the hardness was similar after dilatometric tests

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Note: The responsible person for English language is the translator from University Autonomous of Nuevo Leon, Mexico