ORIGINAL PAPER

METALLURGICAL STUDY OF MANGANESE STEEL

F. SABIR[1], O. BEN LENDA[1], S. SAISSI[1], Y. TAMRAOUI[2], F. MIRINIOUI[2], B. MANOUN[2], A. IBENLFASSI[3], K. OUZAOUIT[4], R. BOULIF[5], E. SAAD[1,6]

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Abstract. *This study shows the effects of heat treatment on the manganese steel which microstructure consists essentially of 18.447% of manganese, 0.745% of chromium, and 1.169% of carbon. The techniques used are: X-ray diffraction, optical microscope, scanning electron microscope.After heat treatment at 600 °C and 1050 °C for 30 minutes, metallurgical analysis will connect the microstructural changes occurring during the temperature maintenance to the mechanical behavior of the material. The characterization of the microstructures and the surface morphology of the alloy before and after the heat treatment determine that this treatment process favors the dispersion of carbides in the austenitic matrix and the appearance of precipitates which causes structural hardening.*

Keywords: *X-ray diffraction, optical microscope, electronic scanning microscope, microstructural changes, structural hardening.*

1. INTRODUCTION

Many researchers have begun to focus on the replacement of conventional steels Fe-Cr-Ni by Fe-Cr-Mn, because Mn substitutes for Ni with decreasing the Cr content. Indeed, Cr was found to be most effective for improving high-temperature oxidation resistance and embrittlement of the environment [1, 2]. Carbon promotes the hardening of the alloy by precipitation and manganese improves the mechanical properties at elevated temperatures [3-6]. Manganese steels have many advantages, including low cost, corrosion resistance [7-18]. Thus, they are suited to use in shipbuilding, and the land of the automobile industry.

The mechanical properties of manganese steels at casting raw state are low, but these can be considerably increased by the completion of heat treatment, and also have to study and examine the effects of content C, Mn and Cr. The characterization of the microstructures and the surface morphology of the alloy before and after heat treatment were studied by optical microscopy, scanning electron microscope, X-ray diffraction (XRD) and thus the hardness measurements indicate the evolution of the mechanical properties of our steel.

[1] University Hassan 1[st], Laboratory of Physicochemistry of Materials and Processes (MPCP), Settat, Morocco. E-mail: saadelmadani73@gmail.com.
[2] University Hassan 1[st], Laboratory of Materials Science, Media and Modeling, 25000 Khouribga, Morocco.
[3] University Hassan 1[st], Department of Environmental Sciences and Development, Settat, Morocco.
[4] Managem Group, REMINEX Research Center, Marrakech, Morocco.
[5] OCP Group, Laboratory of Research and Development, 24000 El Jadida, Morocco.
[6] University Hassan 1[st], Higher Institute of Health Sciences, Settat, Morocco.

2. MATERIALS AND METHODS

The alloy that will be the subject of the metallurgical study is manganese steel. Its composition is shown in Table 1. It essentially consists of iron with a ratio of chromium and manganese. Our alloy is prepared as follows: the elements are melted in an electric arc furnace provided with carbon electrodes at a temperature almost equal to 1500 °C [19]. The crude steel, before being poured into molds, it is refined to adjust the chemical composition by adding elements (i.e. Manganese, Chromium, Silicon, Copper, Nickel, Phosphorus, Vanadium, Molybdenum, sulfur, titanium) to form requested alloy.

Table 1. Chemical composition [%] of steel based on iron.

C	Si	Mn	P	S	Cr	Mo	Ni	Cu	V	Fe
1.169	0.668	18.447	0.001	0.0065	0.745	0.034	0.184	0.302	0.0017	78.368

Hardness

The treated sample was cut into smaller samples; sections are obtained by sawing and mechanical abrasion. All sections are heat-treated at various temperatures for 30 minutes at 600 °C and 1050 °C in the furnace, and then quenched with water.

The objectif to place the hardness test at the center of this study comes from the fact that it is a fast and reliable mean to follow the kinetics of aging. This Hardness tests is performed by Rokwell method using a durometer testweel under a load of 60 kgf. This protocol makes possible monitoring the overall behavior of the material.

Metallographic Examination

Manganese steels after heat treatment has undergone a mechanical polishing with a series of abrasives (from 120 to 1200), then etching using nitric alcoholic solution, this reagent is composed of 2 ml to 5 ml of acid nitric in 100 ml of pure ethanol. Observation of the structure of the steel was observed by optical microscope and scanning electron microscopy of type Philips XL30, the microscope is coupled to a type detector EDAX (Energy Dispersive X-Rays Spectrometer) which permits quantitative analysis for local composition.

X-Ray Diffraction

The X-ray diffraction of the spectra of the samples during aging and over aging are obtained at room temperature using a diffractometer D2 PHASER Bragg-Brentano geometry, type of radiation Cu Ka = 1.5406 Å (40 kV, 40 mA) and the 2θ scanning range is 15° to 100 ° with a step of 0.02 (2θ).

X-Ray Fluorescence

Quantification and qualification of all elements of the alloy was given by mobile Spectrometry XRF the mark of Bruker.

3. RESULTS AND DISCUSSION

Manganese steel based on iron in the initial state

The hardness measurements of austenitic steel with manganese at the receiving state before heat treatment give an average of 47 HRF. The micrographic study of manganese steel in the rough cast shows that the sample structure is austenitic with presence of carbides of manganese and chromium which precipitated at the grain boundaries by altering the strength and ductility (Figs. 1-2). From Table 2, we see that the matrix and the precipitates are composed essentially of manganese and chromium carbides.

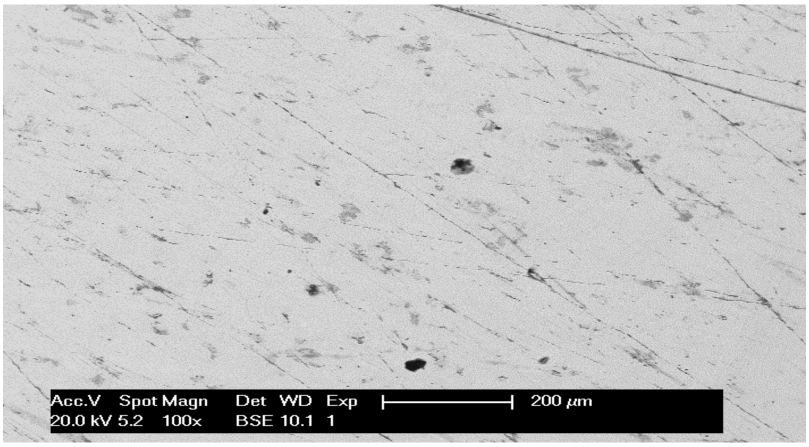

Figure 1. Scanning Electron Microscopy - the structure of manganese steel for the raw cast shows heterogeneous precipitates of chromium and manganese dispersed in the austenitic matrix.

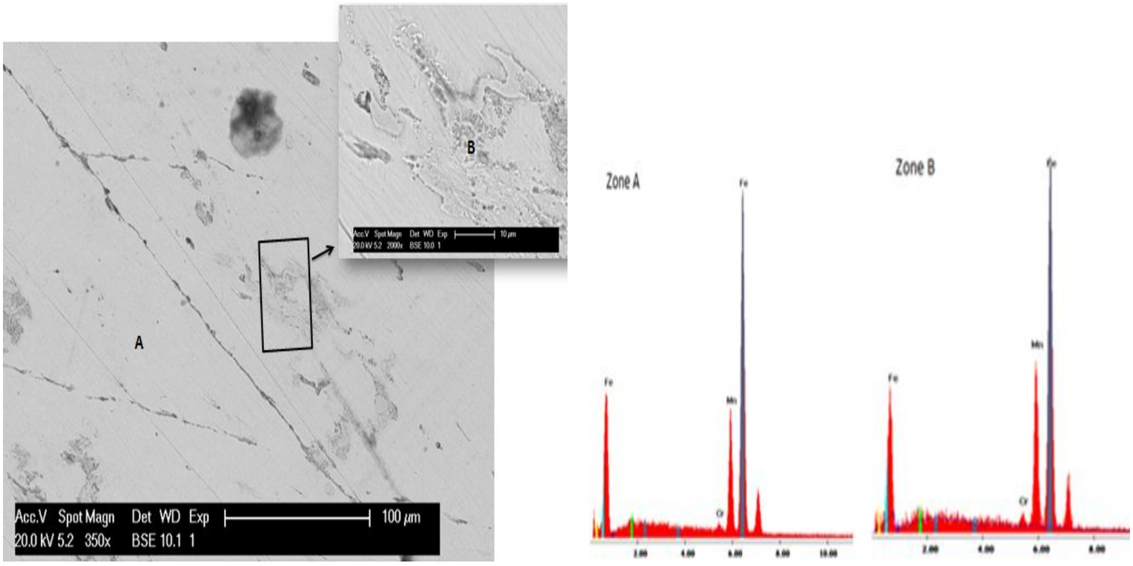

Figure 2. The SEM micrograph and EDAX of As-cast manganese steel.

Table 2. Chemical Composition of the target points.

ZONE/ Element	Fe		Mn		Cr	
	Wt%	At%	Wt%	At%	Wt%	At%
A	76.69	76.34	22.15	22.42	1.16	1.24
B	71.10	70.69	27.27	27.56	1.64	1.75

Study of heat treatment effect on the hardening of manganese steel

After heat treatment at 600 °C, we have followed the evolution hardness of manganese steel treated on time. We see a linear increase in hardness with time in order to achieve a hardness value of 49 HRF after 20 minutes of aging at room temperature. The hardness stays constant before decreasing to a 47 HRF value after 3 hours. For maintain long time the hardness remains constant [20].

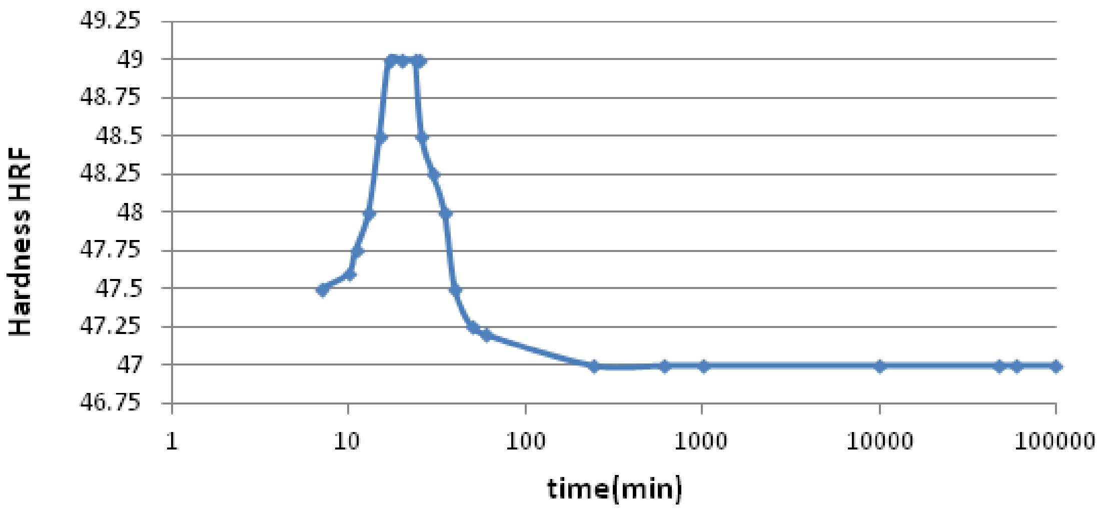

Figure 3. Evolution hardness of the manganese steel treated at temperature 600 °C as a function of time.

The micrographic study of the heat treated alloy at 600 °C shows a non-uniform dispersion of the second phase in the microstructure which can be observed in Fig. 4.The figure shows also the heterogeneity of the distribution of second phase particles of the steel treated at 600 °C.

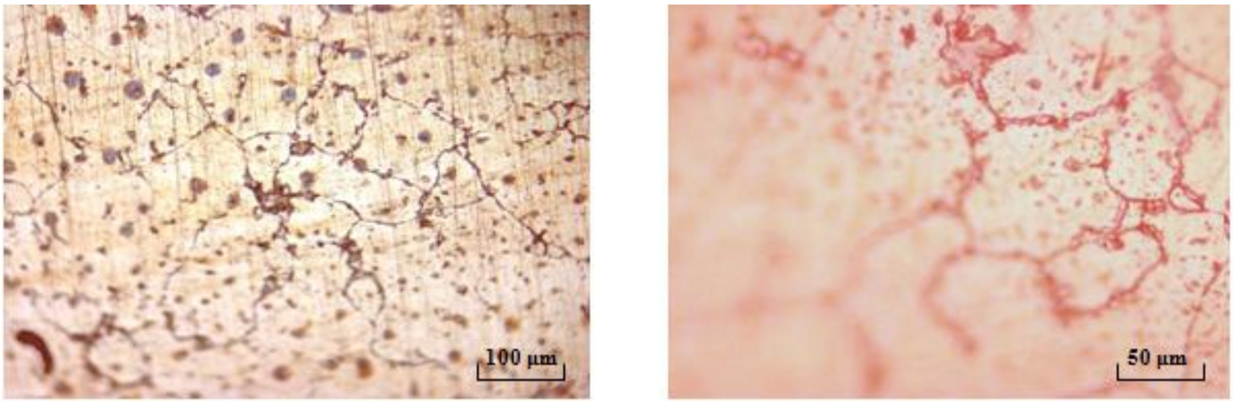

Figure 4. Visualization of the manganese steel structure after 20 days of aging treated at 600 °C, quenched with water, attacked by the nitric alcoholic solution.

Figs.5-6 show the result of SEM and EDAX. The structure of the steel after the treatment at 600 ° C is composed by a matrix essentially rich in Fe and Mn (Fig. 5), and the precipitates rich in Mn and Cr (Fig. 6). In fact the observation of the structure by scanning electron microscope shows that the structure is heterogeneous, this observation is similar to the optical microstructure obtained in Fig. 4.

Fig. 7 shows the evolution hardness of manganese steel as function of time. For prolonged maintain the hardness remains constant and have a value of 47.5 HRF to 1050 °C. The increase in the hardness may be due to a relatively uniform distribution of the carbide phase in the austenite phase which explains the increase hardening structure increase treated at 1050 °C compared to the As-cast state of manganese steel [21].

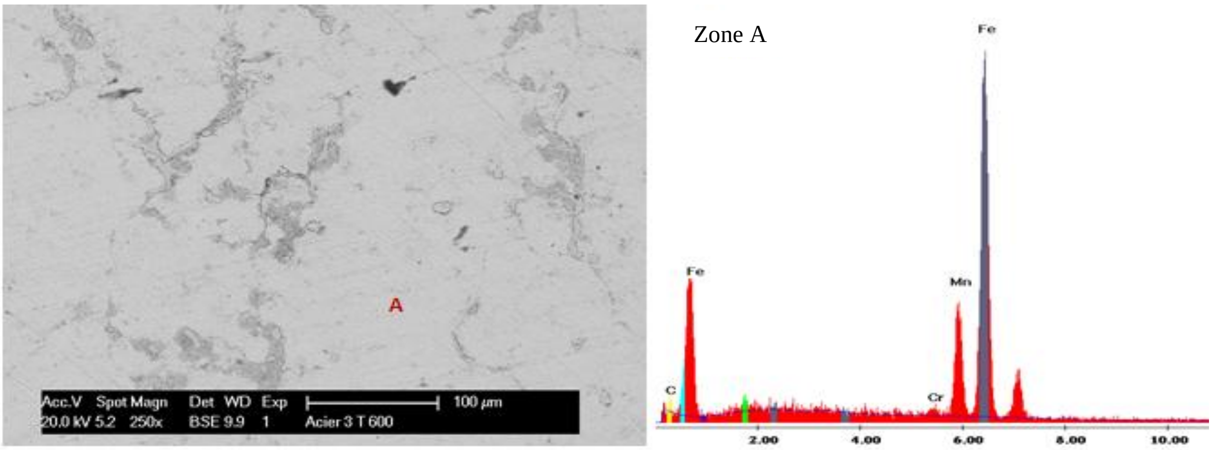

Figure 5. Visualization manganese steel by a scanning electron microscope, treated at 600 °C.

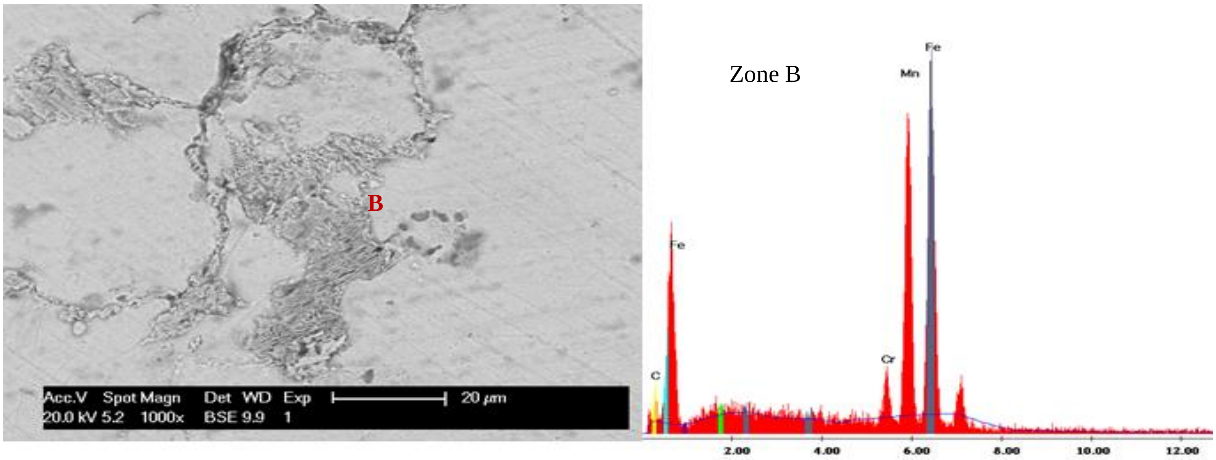

Figure 6. Identification of phases with different chemical compositions by EDAX on manganese Steel treated at 600 °C.

Table 3. Chemical Composition of the target points.

ZONE/ Element	Fe		Mn		Cr	
	Wt%	At%	Wt%	At%	Wt%	At%
A	78	78	21	21	1	1
B	55	55	41	41	4	4

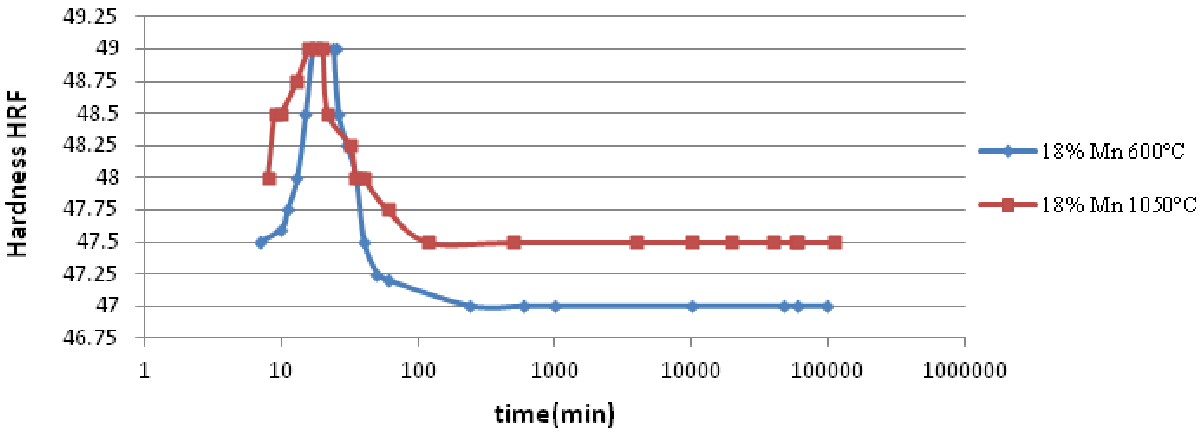

Figure 7. Evolution of hardness of manganese steel treated at 600 °C and 1050 °C depending on time.

Table 4 shows the chemical composition of manganese steel obtained using mobile X-ray fluorescence spectrometry; we see that there is no heat treatment influence on the chemical composition of treated alloy.

Table 4. Chemical composition of manganese steel before and after heat treatment.

Steel with 18% Mn before heat treatment		**Steel with 18% Mn after heat treatment at 1050 ° C**	
Element	**%**	**Element**	**%**
Cr	0.7 ± 0.02	**Cr**	0.69 ± 0.02
Mn	17.46 ± 0.08	**Mn**	17.28 ± 0.08
Fe	81.45 ± 0.15	**Fe**	81.58 ± 0.16
Ni	0.13 ± 0.02	**Ni**	0.16 ± 0.02
Cu	0.22 ± 0.01	**Cu**	0.27 ± 0.01
Mo	0.03	**Mo**	0.03

Visualization of manganese steel structure treated at 1050 °C after one month of aging showed a significant effect of the heat treatment on the structural behavior of the sample. For maintain prolonged in time, the grain boundaries are characterized by strong segregation of chromium carbides (Fig. 8).

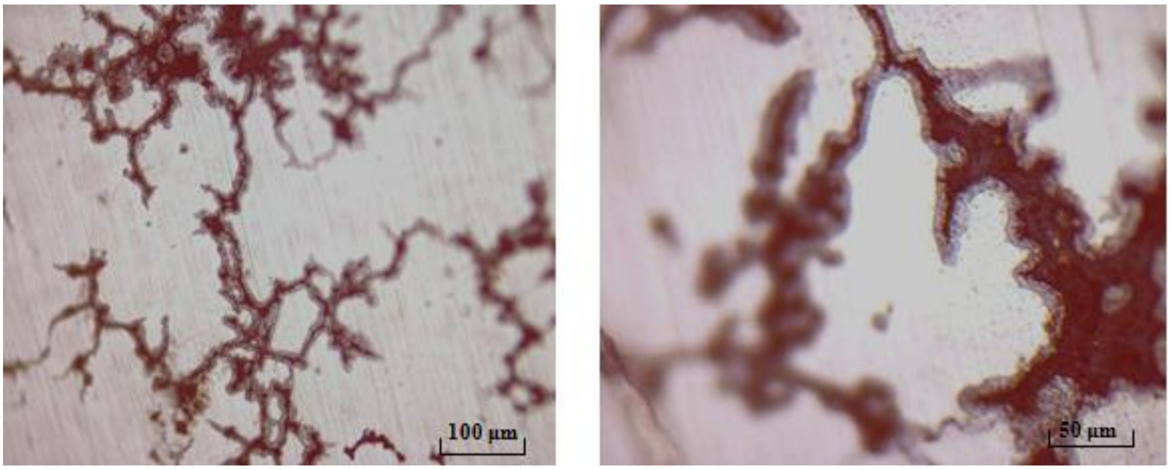

Figure 8. Optical micrograph manganese steel treated at 1050 °C.

The SEM micrograph of manganese steel heat treated at 1050 ° C is shown in Fig. 9, the second phase particles, is uniformly dispersed with the austenitic matrix; this treatment helps to dissolve the carbides in the matrix [19].

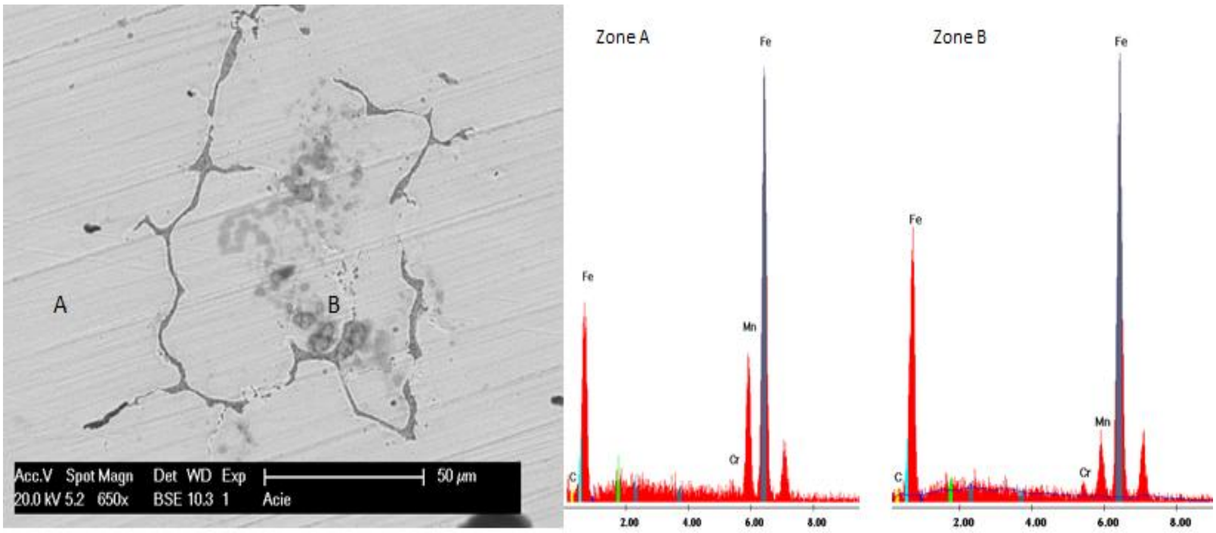

Figure 9. Identification of phases with different chemical compositions by EDAX for manganese Steel treated at 1050 °C.

Table 5. Chemical Compositions of target points of manganese steel after treatment at 1050 °C.

ZONE/ Element	Fe		Mn		Cr		O	
	Wt%	At%	Wt%	At%	Wt%	At%	Wt%	At%
A	82.10	81.82	16.94	17.16	0.95	1.02	0.00	0.00
B	89.00	89.00	10.00	10.00	1.00	1.00	0.00	0.00

Fig. 10 shows the X-ray diffraction analysis of manganese steel before heat treatment and also treated at 600 °C and 1050 °C. We observe the displacement of the peak position to the right, this change of position is due to changes of the phases that characterize each treatment (Table 5). In the result of X-ray diffraction, the carbides are uniformly dispersed with the austenitic matrix; the operation of the heterogeneity of second phase carbide segregation was dissolved at 1050 °C with the appearance of new precipitated phases.

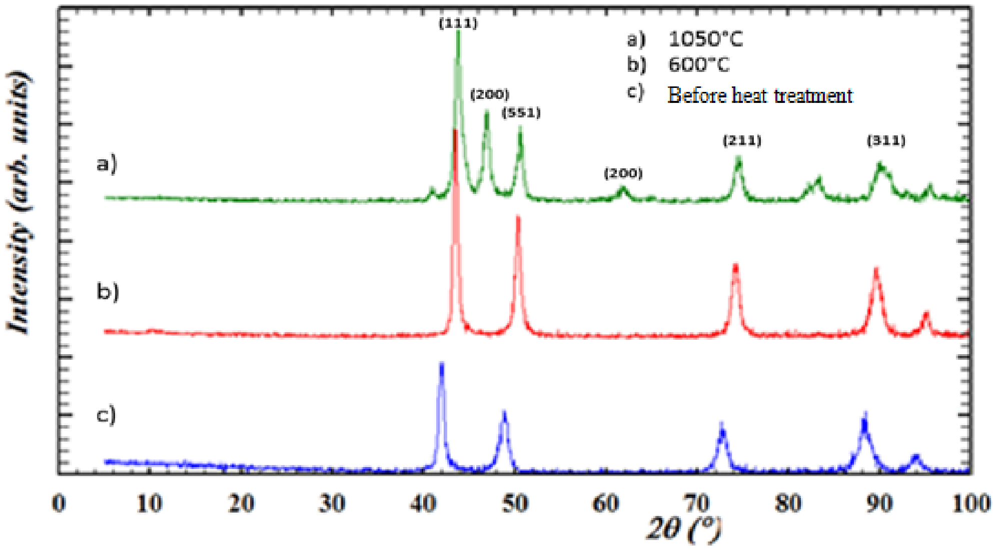

Figure 10: Evolution of the X-ray diffraction patterns for manganese steel as a function of temperature 600 °C, 1050 °C and cast state.

Table 6. Characteristics of diffraction pattern at 1050 °C, and at 600 °C before heat treatment.

	Steel before heat treatment		**Steel after heat treatment**		
hkl	2Θ	I_{hkl}	2Θ	I_{hkl} at 1050°C	I_{hkl} at 600°C
002	-	-	40.573	11.952	-
111	42	57.63	43.228	100	100
420	-	-	46.548	46.181	-
200	48.80	30.19	49.867	29.208	62
551	-	-	61.152	9.689	-
220	72.57	23.07	73.765	25.813	40
211	-	-	82.129	20.156	-
311	88	29.17	90.228	26.096	37
222	93.68	10.78	94.742	9.406	12

Table 7 shows the crystal parameters a, b, c, and the angles α, β, ɤ of the crystal structures. Stitch parameters have been refined from the experimental positions of diffraction lines using the program PARAM [22, 23], we find that the crystal system changes from cubic to orthorhombic after heat treatment (Table 7). Thus, as and as the treating temperature increases, the cell volume decreases, passes from 46.9 $A^{\circ 3}$ to 45$A^{\circ 3}$.

Table 7. Parameter values and volume determined and refined using the software "PARAM".

	Refinement By PARAM		
	Steel before heat treatment	**Heat treatment at 600°C**	**Heat treatment at 1050°C**
crystal system	cubic	orthorhombic	orthorhombic
Lattice parameter(A°)	a=3.606±0.01923	a= 3.649±0.011	a= 3.586±0.039
		b= 3.588±0.016	b= 3.589±0.064
		c= 3.656±0.019	c= 3.458±0.0689
Volume of the stitch ($A^{\circ 3}$)	V= 46.856±0.750	V= 47.808±0.356	V= 44.5±1.318

4. CONCLUSIONS

A metallurgical study connects the microstructural changes during the heat treatment with the mechanical behavior of the material. The temperature and time are parameters influencing the kinetics of microstructural transformations. For this reason, two aging temperatures were selected; 600 °C and 1050 °C for industrial and scientific interests.

Characterization manganese steel at raw casting states showed that they have a structure consisting of austenite, manganese and chromium carbides which precipitate, in great quantities, in the austenitic grain boundaries. For that, the steel has followed an annealing treatment at a temperature of 1050 °C; this treatment favors the dispersion of carbides in the austenite matrix.

The scanning electron microscope is coupled to sensor type EDAX allowing for local quantitative analysis of the composition. The EDAX analyzes carried out on the steel show that for lower temperatures, the precipitates are very rich in carbon and the matrix consists of Iron, Manganese and Chromium.

X-ray diffraction spectra obtained after the treatment revealed that the structure is orthorhombic. These spectra also show the presence of several phases that characterize the structure of the steel.

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