

**MICRO AND MACRO HARDNESS CORRELATION
FOR HIGH SPEED STEEL HSS WITH MC AND
M₂C CARBIDE PRECIPITATION, USED TO
PRODUCE LAMINATION ROLLS**

**R. SERVIN-CASTAÑEDA¹, I. CALDERON-RAMOS¹, F. EQUIHUA-
GUILLEN¹, R. D. MORALES², R. D. MERCADO SOLÍS³
and VEGA-LEBRUN CARLOS ARTURO⁴**

¹Facultad de Ingeniería Mecánica y Eléctrica Unidad Norte
Universidad Autónoma de Coahuila
Av. Barranquilla S/N, Colonia Guadalupe, C.P. 25750
Monclova, Coahuila
México
e-mail: romualdoservinc@prodigy.net.mx

²Instituto Politécnico Nacional- E. S. I. Q. I. E.
Ed. 7 UPALM, Col. Zacatenco, C.P. 07738
México D.F.

³Universidad Autonoma de Nuevo Leon-UANL-FIME
Avenida Universidad S/N, Cd. Universitaria
San Nicolas de Los Garza
Nuevo Leon
C.P. 66451
Mexico

⁴Universidad Popular Autonoma del Estado de Puebla Ed. de Postgrado
17 Sur 901, Puebla, Pue
C.P. 72410
Mexico

Keywords and phrases: carbides, high speed steel (HSS), hardness, lamination, rolls.

Received March 30, 2017

Abstract

In the present study, the effect of the percentage of carbides MC and M_2C is analyzed on the hardness of HSS steel used to manufacture lamination rolls; heat treatment was given to one piece of the material, with high Cr, Mo and V contents. This was subsequently cut and prepared, according to ASTM the standards to measure microhardness from the carbides, the percentages of each one of them and the macrohardness of the material. Applying different techniques of characterization the evolution of the hardness in the microstructure became evidenced; what we found was that the highest hardnesses is obtained when the amount of carbides M_2C is approximately 50% of the amount of MC carbides (ratio of about 2% MC per 1% M_2C). It is also possible to verify that there is a hardness drop of 3.14% of the surface with respect to the center, this is due to a 6.27% reduction in the %MC and % M_2C , which means that there is a hardness drop of 1% approximately with respect to the reduction of carbides in 2%.

1. Introduction

Hardness is a fundamental property of a material, it is related to plastic and elastic properties, hardness numbers have no intrinsic meaning, so therefore values are only used as a comparison among materials. The selection of a hardness test is generally determined by the desired accuracy, the reason why is divided into microhardness for minor impressions of very small parts with very thin sections and macrohardness of robust sections (Avner [1]).

The materials have different mechanical properties, which are related to the external forces that are exerted on them, the hardness is one of the most important variables in the prediction of the behaviour of materials that are exposed to mechanical contact. In recent years, the technological development of materials resistant to wear has increased considerably, and in regard to steels, one of the best options for this type of materials are high speed steels, known as HSS, by its initials in English High Speed Steel. This have the peculiarity of precipitating very hard carbides of very small size that measure their hardness in micro scale and the matrix of this material that forms the greater part of the

alloy that is measured in macro scale. Many types of carbides are present in this type of alloys, each of them have different effects on its properties and among them, the morphology and quantity of carbides are important factors that concern them (Colnet et al. [2]; Hwang et al. [3]; Chang et al. [4]).

The hardness in the HSS steel is characterized by the microstructure that is obtained with the heat treatment and is influenced by hard particles deposited in the matrix called carbides. These carbides are very hard substances present in the steel structure in the form of inclusions. Through the addition of alloying elements such as chromium, molybdenum, vanadium, tungsten, and titanium etc. The iron carbide (Fe_3C) is transformed into other carbides of higher hardness. The degree of hardness depends on the nature of each of the carbides present in the steel; HSS steels are some of the materials that considerably increase their hardness due to the presence of MC , M_2C , and M_7C_3 carbides. Studies on HSS materials used for the steel forming process through rolling, establishing that alloying elements such as coal, tungsten, molybdenum and vanadium generate about 25% MC , M_2C , and M_7C_3 carbides, significantly influencing the microstructure, hardness and wear resistance of HSS steels (Dae Jin Ha [5]; Kim et al. [6, 7]; De Colnet [8]). However, it has also been shown that the high percentage of carbides increases considerably the coefficient of friction at the contact of the materials, mainly the vanadium content, so therefore is recommended to reduce as much as possible this alloy element (Park [9]).

Most of the studies carried out for the development of HSS steels agree that carbides are much harder than the matrix of the material, therefore the plastic properties of the matrix are made of special interest, where toughness, hardness, and strength to wear depends mainly on its morphology, size distribution and volumetric fraction of carbides (Blaha et al. [10]; Boccalini Jr. and Sinatora [11]; Rodenburg and Rainforth [12]).

There are six types of indentator hardness tests, which are used to determine the microhardness and macrohardness of metallic materials. The indentator macrohardness tests are Brinell, Meyer and Rockwell, and the microhardness tests are Berkovich, Knoop and Vickers (Johnson [13]).

The hardness had a very important role, in which Wojciech Sitek developed a mathematical model to calculate the hardness of HSS steels used to manufacture cutting tools, this model has a margin of error of 0.7 HRC and uses as a base the elements of alloy and the temperatures applied in the heat treatment of steel (Sitek [14]).

High hardness values are desired in HSS steels and are subjected to mechanical contact and wear, such as those used to produce lamination rolls, it is required to know the nature of the carbides and matrix that constitute mechanical properties such as hardness; to evaluate the microstructural configuration in terms of carbide and matrix density; so, it is interesting to study the correlation between the microhardness of the carbides, the percentages of the same and the macro hardness of the matrix.

2. Materials and Experimental Procedure

2.1. Material used

The material used for the study is a cylindrical bar of 50.8mm in diameter by 150mm in length, heat treated with tempering and then annealing to relieve stresses. The tempering was performed at 1100°C, heating the test piece at a rate of 10°C/min until reaching the tempering temperature, remaining there for a time of 40 minutes and then letting it cool rapidly to the air with a speed of 35°C/min, in the Figure 1, you will be able to see the graph showing the process that was made.

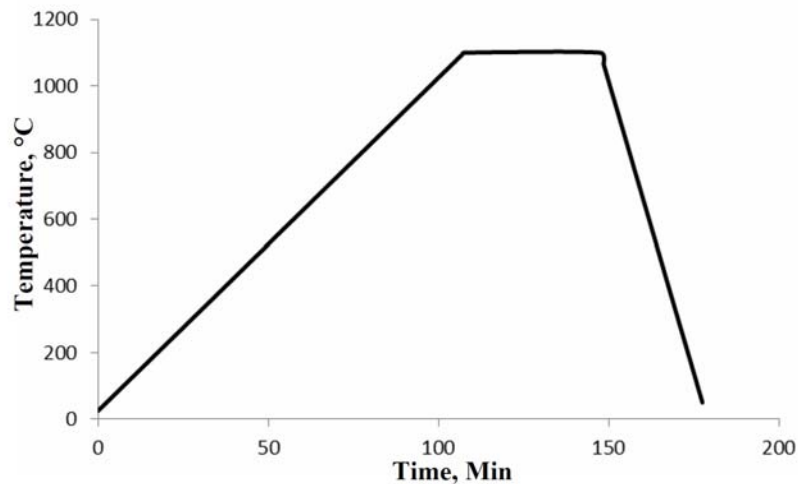


Figure 1. Heat-cooling curve applied in the quenching process.

The chemical composition of the alloying elements corresponds to steel with 1.6% C, and is combined with the main carbide formers such as chromium, molybdenum, and vanadium, which according to ASTM standard correspond to an HSS steel; the exact values and the rest of the alloying elements can be seen in Table 1.

Table 1. Main alloying elements present in the analyzed HSS material

Alloying Element	C	Mn	P	S	Si	Cu	Cr	Ni	Mo
%	1.673	0.406	0.023	0.009	0.762	0.070	2.904	0.949	2.979
Alloying Element	Al	V	Cb	Ti	Sn	Al sol	Ca	B	
%	1.014	4.276	0.019	0.004	0.009	0.003	0.0016	0.0001	

2.2. Samples description

In order to analyze the largest possible area of the heat treated material and trying to have a more complete and homogeneous sampling, the length of the cylindrical bar was divided into three equal parts of 50mm, and three test areas of analysis of 20mm of width by 20mm of

depth, equidistant in its circumference every 120°; in Figure 2, we can see the distribution and dimensional description of the cells that have been analyzed above.

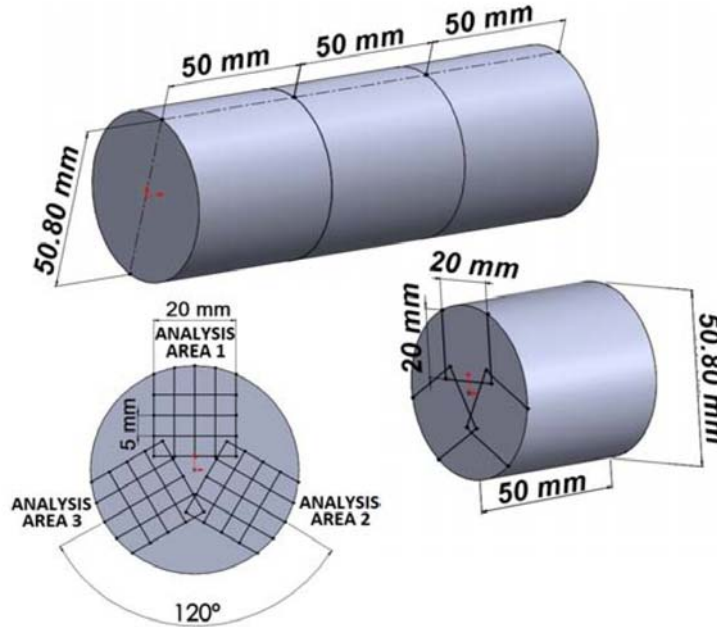


Figure 2. Schematic representation of the samples extracted from the analyzed material.

2.3. Preparation technique

All the tests performed in the study according to ASTM (1991) standards were carried out, for example, the preparation of the metallographic samples, the ASTM E3 [15] was applied, for the micro and macro hardness tests, the standards ASTM E92 [16] and ASTM E18 [17], respectively, for Vickers and Rockwell hardnesses have been applied; in addition, in order to facilitate the identification of the areas analyzed in the sample, a subdivision of the dimensions of 5mm of width by 5mm of height was made, obtaining a square cell of approximately

25mm^2 , the identification of the cell is considered taking a line and one column for each of the samples; for example, in Figure 3(a), it is possible to observe in dark colour the cell 1C3, which corresponds to the analysis area 1 column C with the intersection of row 3; in Figure 3(b), cell 2B4, corresponding to the analysis area 2 column B of row 4, is observed, and in Figure 3(c), cell 3A2 is observed, corresponding to the analysis area 3 column A of row 2; following the same pattern to identify the 16 cells that correspond to each analyzed area. The analysis cells close to the surface of the piece are all those that are located in row 1, for example, A1, B1, C1, and D1 of the three areas analyzed, its analysis interval would be 0 to 5mm depth in the part, for row 2 the analysis interval is 5 to 10mm depth, row 3 is 10 to 15mm depth and finally row 4, is 15 to 20mm depth.

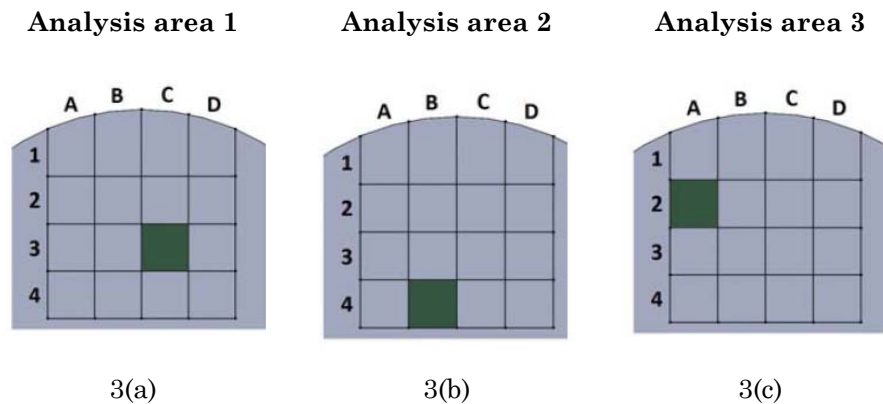


Figure 3. Graphical representation and location of cells 1C3, 2B4 and 3A2.

3. Results and Discussion

3.1. Microstructural composition and carbide density

The solidification sequence of the speed steels comprises two transformations; (1) formation of the primary austenite, and (2) eutectic decomposition of the interdendritic residual liquid. In the case of the eutectic decomposition the interdendritic residual liquid, the formation of

different eutectics is made, depending on the alloying elements; in this case we can analysis and could confirm this hypothesis since we had three eutectics which are $\gamma + MC$, $\gamma + M_2C$, and $\gamma + M_7C_3$. Figure 4(a) shows the microstructure of martensitic matrix with the precipitation of MC and M_2C secondary carbides, and in Figure 4(b) the image of total carbide density is shown, these images are repetitive for each one of the analyzed cells. The average values in the tables and graphs shown below are examined later in the report.

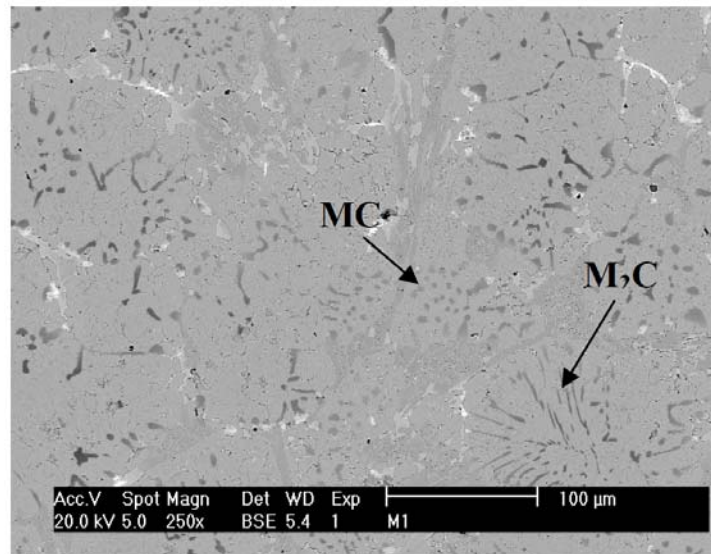


Figure 4(a). HSS microstructure with secondary precipitates of MC and M_2C carbides.

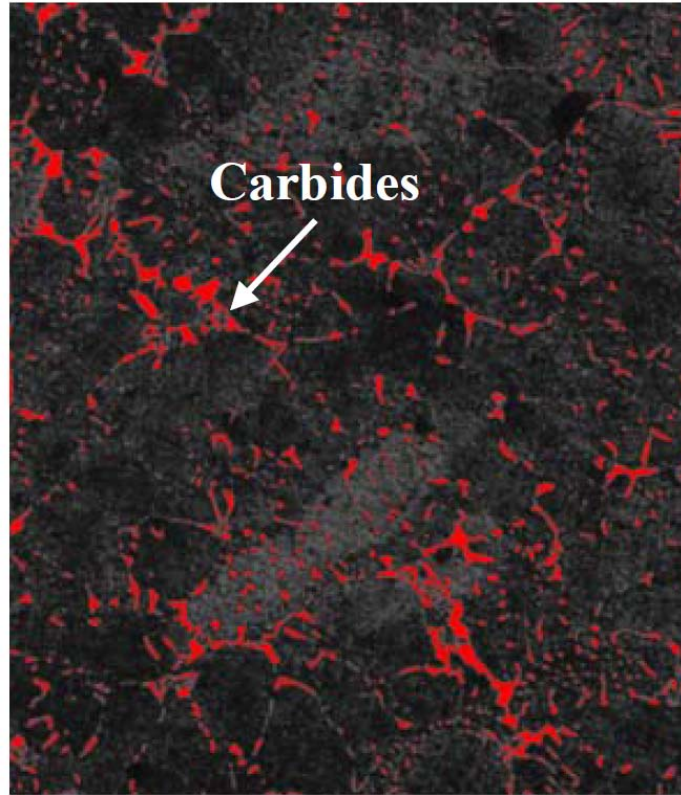


Figure 4(b). Total density of MC and M_2C carbides.

3.2. The percentage effect in carbides MC and M_2C in the hardness frame

The main characteristics of HSS steels are the high hardness that are obtained, which makes them high resistant to mechanical contact and wear, this is mainly due to the hardness of the carbides present; the microhardness obtained for the matrix and the carbides is very repetitive, Table 2 shows the averages of each of these values.

Table 2. Microhardness averages of matrix and precipitated carbides

Carbide type	MC	M ₂ C	M ₇ C ₃	Matrix
	2700	2100	1500	550

As shown in Table 2, the microhardness of carbides are very high, however due to the low density and morphology of the current carbides, the hardness descend because the martensitic matrix form the greater part of the area and its value prevails; in order to have an idea of the relation of the percentages of carbides with their respective microhardness and the influence they have in the macrohardness of HSS material, we analyzed the values obtained in the study, which are shown in Table 3.

Table 3. Relation of the hardness in function with the percentages of MC and M₂C carbides

Analysis area 1				
Cell	A	B	C	D
1	%MC 8.21	%MC 8.18	%MC 7.75	%MC 8.35
	%M ₂ C 4.18	%M ₂ C 4.55	%M ₂ C 5.04	%M ₂ C 5.08
	HRC 59.25	HRC 59.05	HRC 59.08	HRC 59.35
2	%MC 7.75	%MC 8.05	%MC 7.99	%MC 8.00
	%M ₂ C 5.04	%M ₂ C 4.35	%M ₂ C 4.55	%M ₂ C 4.55
	HRC 59.08	HRC 58.92	HRC 58.83	HRC 58.83
3	%MC 7.92	%MC 7.92	%MC 8.13	%MC 7.88
	%M ₂ C 4.85	%M ₂ C 4.88	%M ₂ C 4.87	%M ₂ C 4.65
	HRC 58.83	HRC 58.87	HRC 58.75	HRC 58.50
4	%MC 7.55	%MC 7.18	%MC 7.78	%MC 7.68
	%M ₂ C 4.25	%M ₂ C 5.00	%M ₂ C 4.16	%M ₂ C 4.32
	HRC 58.00	HRC 58.55	HRC 57.85	HRC 57.55

Analysis area 2				
Cell	A	B	C	D
1	%MC 7.95	%MC 8.18	%MC 8.25	%MC 8.02
	%M ₂ C 5.04	%M ₂ C 4.68	%M ₂ C 4.35	%M ₂ C 4.97
	HRC 59.88	HRC 59.85	HRC 59.75	HRC 59.75
2	%MC 7.75	%MC 7.82	%MC 7.89	%MC 7.75
	%M ₂ C 4.13	%M ₂ C 4.78	%M ₂ C 4.96	%M ₂ C 5.08
	HRC 58.75	HRC 57.75	HRC 57.87	HRC 58.35
3	%MC 7.78	%MC 7.78	%MC 7.81	%MC 7.78
	%M ₂ C 4.16	%M ₂ C 4.32	%M ₂ C 4.55	%M ₂ C 4.35
	HRC 57.75	HRC 57.25	HRC 57.21	HRC 57.50
4	%MC 7.68	%MC 7.78	%MC 7.72	%MC 7.53
	%M ₂ C 4.32	%M ₂ C 4.25	%M ₂ C 4.85	%M ₂ C 4.23
	HRC 57.55	HRC 56.95	HRC 57.63	HRC 58.00
Analysis area 3				
Cell	A	B	C	D
1	%MC 8.75	%MC 8.48	%MC 8.25	%MC 8.18
	%M ₂ C 4.13	%M ₂ C 4.68	%M ₂ C 4.35	%M ₂ C 4.16
	HRC 59.75	HRC 59.85	HRC 59.75	HRC 59.35
2	%MC 7.92	%MC 8.02	%MC 7.91	%MC 8.25
	%M ₂ C 4.88	%M ₂ C 4.27	%M ₂ C 4.58	%M ₂ C 4.08
	HRC 58.87	HRC 58.75	HRC 58.21	HRC 58.35
3	%MC 7.73	%MC 7.73	%MC 7.79	%MC 7.48
	%M ₂ C 4.53	%M ₂ C 4.89	%M ₂ C 4.26	%M ₂ C 4.35
	HRC 58.24	HRC 57.93	HRC 57.17	HRC 57.50
4	%MC 7.53	%MC 7.82	%MC 7.47	%MC 7.55
	%M ₂ C 4.23	%M ₂ C 4.18	%M ₂ C 4.32	%M ₂ C 4.25
	HRC 58.00	HRC 57.15	HRC 57.02	HRC 58.00

The relationship of hardness with the percentages of MC and M₂C carbides is graphically shown in Figure 5, where different combinations

of hardness according to the precipitated carbides can be seen. The desirable hardness for a HSS steel resistant to wear would be the metallurgical combinations where the hardness is maximum, such as the one obtained in dark coloured areas, where the hardness is greater than 59.5 HRC, which is obtained for a combination of approximately 8.2-8.6% MC and 4.2-4.6% M_2C ; we can also observed other areas with the same hardness of 59.5 HRC, however the dimension of these are very small and the possibility of developing a heat treatment that meets these characteristics would be more complicated so the efficiency is discarded. The values of the hardness correspond to those obtained by Blaha et al. [10], who obtained hardness of 59.2 to 60.4 HRC for the four alloys that he analyzed in his study.

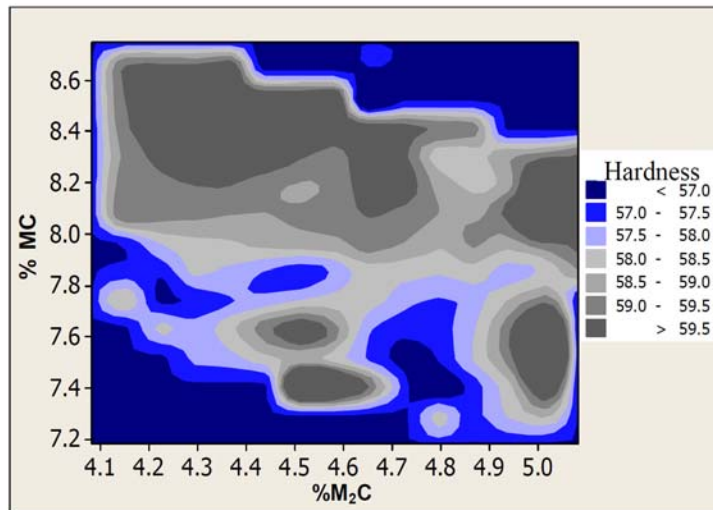


Figure 5. Graphic representation of the macro hardness according to the carbides percentages MC and M_2C .

It is interesting to compare the analysis of the total carbides with the results of Dae Jin Ha [5], where it confirms that the presence of MC carbides influences the hardness of its studied alloy, which has 25% in

the total of the different carbides, in our case the minimum values of the total of carbides were 11.74% with hardness of 58.00 HRC and the maximum was 13.16% with hardness of 59.85 HRC, confirming the theory that the higher percentage of carbides trend of increase of hardness is possible; however, Kim et al. [6] carried out his study with five different alloys in an interval of 12.0 to 17.8% of carbides, and confirms that carbides with high hardness affect the toughness and fracture resistance of HSS steels, therefore it recommends to control the refinement and percentage of carbides, and suggests to use alloying elements to improve the characteristics of the matrix.

By analyzing separately the type of carbides as a function of the total of them, we would expect that when there is a higher density of carbides with high micro hardness, the hardness of the alloy should be higher, in Figure 6 is shown the ratio of the total percentage of carbides MC presented in the analyzed cells of the HSS alloy, where it can be observed that the zones of maximum hardness greater than 59.5 HRC correspond to the approximate values of 8.2-8.6% MC and 12.5-13.1% of the total carbides.

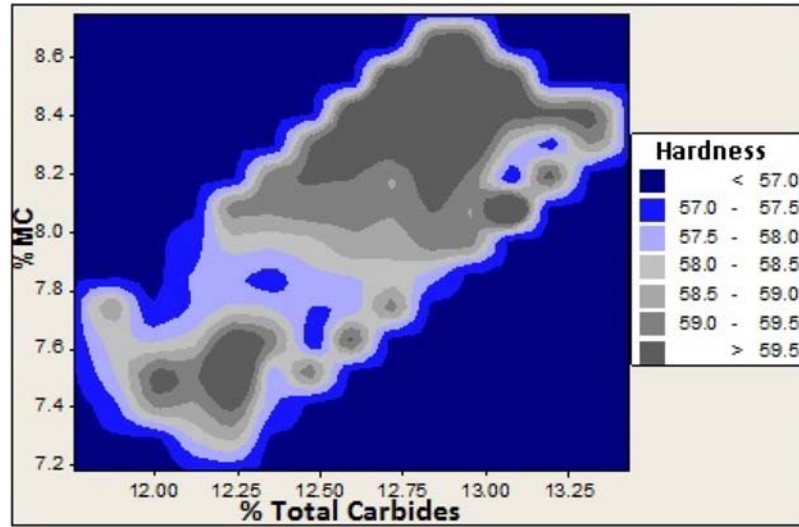


Figure 6. Graphic representation of macro hardness according to the %MC and the total percentage of carbides.

Comparison of the total percentage of carbides with respect to the M_2C carbide content, Figure 7 graphically shows that the hardness zone greater than 59.5 HRC corresponds to the range of 4.2-4.7% M_2C and 12.5-13.1% total carbides.

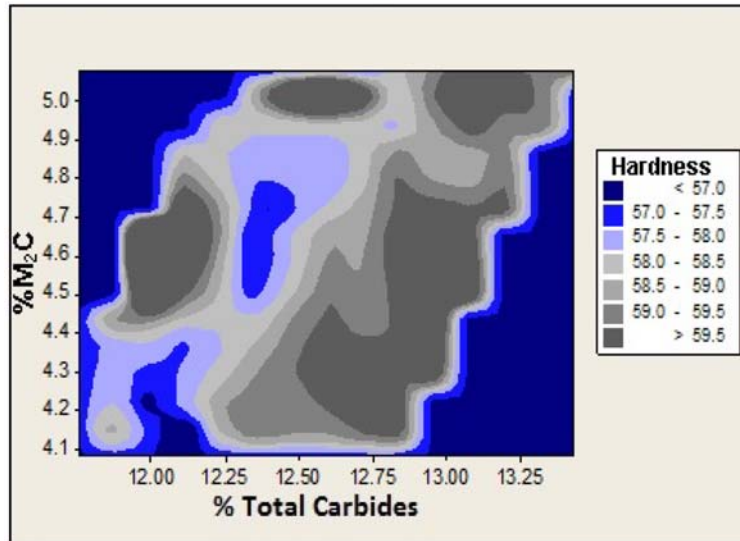


Figure 7. Graphic representation of the macro hardness according to % M_2C and the total percentage of carbides.

3.3. Effects in the carbides percentages of MC and M_2C according to the hardness drop

By analyzing the hardness values, it can be confirmed that there is a hardness drop of the surface of the piece with respect to the center of the same; Cells A1, B1, C1, and D1 (0-5mm) of each zone analyzed form the surface of the bar, while cells A4, B4, C4, and D4 (15-20mm) integrate the center of the piece. In the graph shown in Figure 8, the values of hardness can be observed for each of the cells, and the solid line represents the hardness drop, where we can see a downward trend as it moves towards the center of the piece, the average for cells with prefix one representing the surface of the piece is 59.56 HRC, while the center of the piece that form the cells with prefix four is 57.69 HRC.

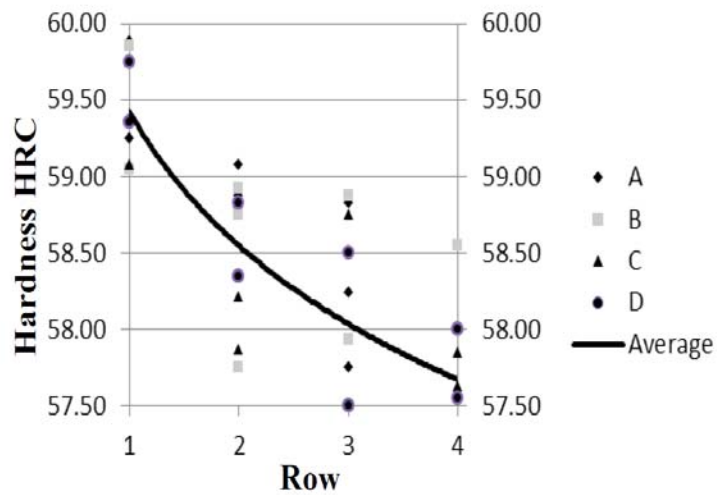


Figure 8. Graphic representation of hardness drop.

The hardness drop is due to the fact that the percentages of carbides MC and M_2C also decrease in the cells of the center of the analyzed bar, in Figures 9(a) and 9(b) the percentages of carbides can be observed for each of the cells analyzed and the continuous lines represent the downward trend of the carbide density, which is the same phenomenon for the carbide types MC and M_2C , and consequently the same tendency in the hardness drop of the material analyzed as shown in Figure 8.

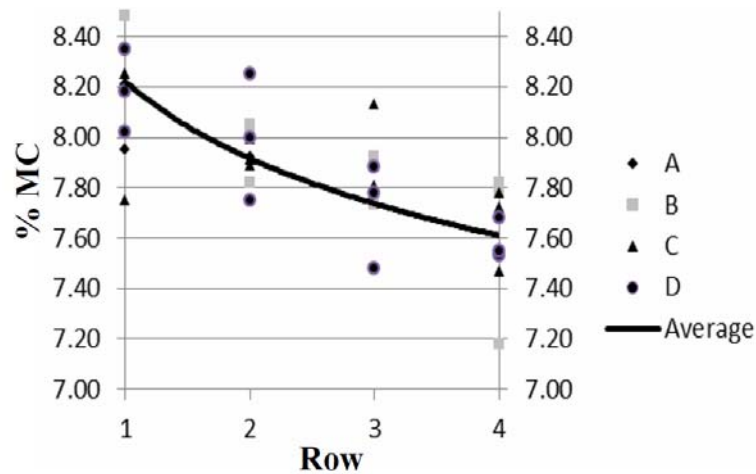


Figure 9(a). Graphic representation of the drop of %MC.

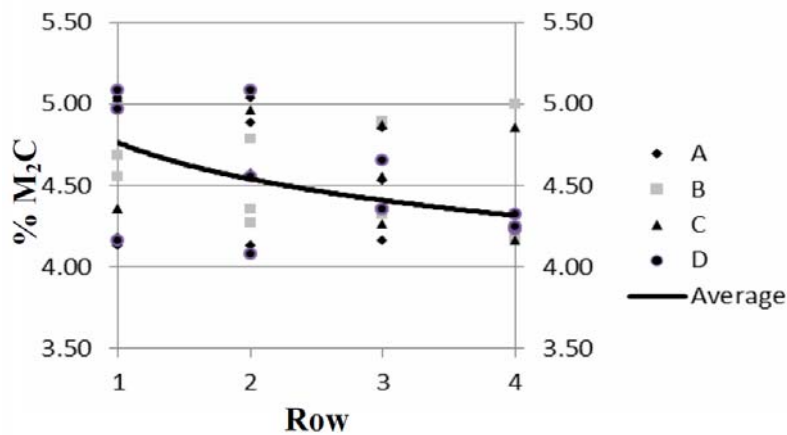


Figure 9(b). Graphic representation of the drop of %M₂C.

In the graphics of Figures 8 and 9, it is possible to appreciate the drop of the values according to the analyzed cells, for the three cases the trend is the same, the hardness and the percentage of carbides drop as it is analyzed towards the center of the piece; however, if we analyze the same values but in percentages, we would have the results shown graphically in Figure 10, where we can see that the lines from 1 to 3 (0-

15mm), the tendency in the hardness drop is proportional to the reduction of percentages of carbides, and row 4 (15-20mm) proportionality is no longer maintained, this coincides with the theory of De Colnet [8], where it states that for his study in the center of the roller, the size of the carbides is greater; so that proportionality is lost in the drop of hardness.

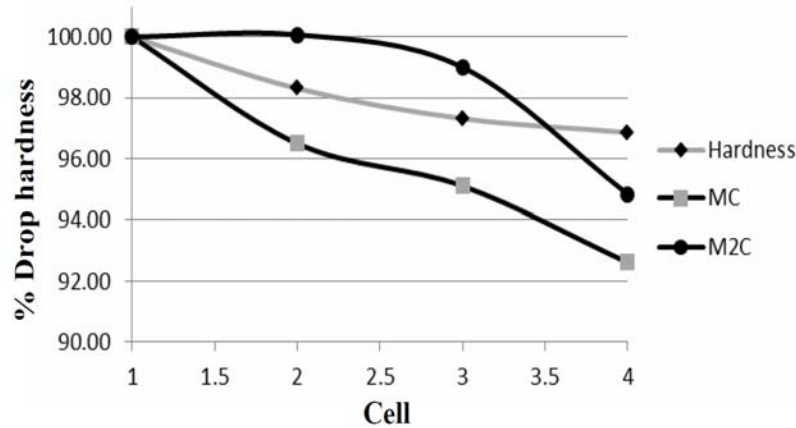


Figure 10. Graphic representation of % in the hardness drop of %MC and %M₂C according to analyzed fields.

4. Conclusion

The formation of carbides MC and M₂C considerably favours the increase of microstructural hardness that makes them very resistant to wear to HSS steels, in the analyzed graph in Figure 5, four zones of high hardness are observed for different combinations of percentages of carbides, however three of them are very small, so it can be concluded that the best combination of percentages of carbides is 8.2-8.6% MC and 4.2-4.6% M₂C. To obtain the maximum hardness of 59.5 HRC, which gives us a relation of 2% of MC carbides per 1% of M₂C carbides; the

maximum percentage of carbides does not guarantee to obtain the highest hardnesses, according to the graphs of Figures 6 and 7, these are obtained in the range of 12.5 to 13.1% of carbides so it is confirmed that the percentage configuration of MC carbides and M_2C influence the macro-hardness of the material, since values greater than 13.1% total carbides correspond to hardness less than 59 HRC, which is lower than the 59.5 HRC that were obtained with lower percentages of carbides that met with the condition of 2% MC by 1% M_2C .

There is a hardness drop of 1.87 HRC which represents 3.14%, and the reduction of carbides is 0.84% which represents 6.27% of the total. This indicates that the correlation that exists between the percentage in the reduction of carbides with the hardness is 2% to 1%; at the surface of the piece the maximum hardness is obtained and it decreases by 1%, and this same phenomenon occurs for the carbides which precipitate their maximum surface value and decrease by 2% for the cells of the center of the analyzed HSS material, with this reduction in the percentage of carbides combined with the theory of De Colnet [8], where it states that its industrial process solidifies faster on the surface of the roll than in the center, therefore it is possible that in our study the hardness drop and carbide percentages decrease from 2% to 1% of the surface compared to the center of the test piece.

References

- [1] S. H. Avner, *Introducción a la metalurgia física*, Segunda Edición McGrawhill, México D.F., 1994.
- [2] J. Colnet, E. Pirard, J. Lecomte-Beckers, P. Boeraeve and R. Ghfiri, Quantitative description of MC, M_2C and M_7C_3/M_6C carbides in high speed steel rolls, Tchoufang – Proceedings of MSMF-3 International Conference, Brno, Czech Republic (2001), 710-717.
- [3] Keun Chul Hwang, Sunghak Lee and Hui Choon Lee, Effects of alloying elements on microstructure and fracture properties of cast high speed steel rolls: Part I: Microstructural analysis, *Materials Science and Engineering A*, Elsevier Science SA 254(1-2) (1998), 282-295.

- [4] K. K. Chang, J. Park, L. Sunghak and K. Y. Chan, Effects of alloying elements on microstructure, hardness, and fracture toughness of centrifugally cast high-speed steel rolls, *Metallurgical and Materials Transactions* 36A (1) (2005), 87-97.
Doi:10.1007/s11661-005-0141-0
- [5] Dae Jin Ha, Effects of alloying elements on microstructure, hardness, wear resistance, and surface roughness of centrifugally cast high-speed steel rolls, *Metallurgical and Materials Transactions A, Korea* 40A (2009), 2568-2577.
DOI: 10.1007/s11661-009-0006-z
- [6] C. K. Kim, D.-G. Lee and S. Lee, Correlation of microstructure and fracture properties of five centrifugal cast high speed steel rolls, *Materials Science and Technology* 23(9) (2007), 1065-1074.
Doi:10.1179/174328407X213170
- [7] Kim Chang Kyu; Park, Il Jong, Jae Hwa Ryu and Sunghak Lee, Correlation of microstructure and thermal-fatigue properties of centrifugally cast high-speed steel rolls, *Metallurgical and Materials Transactions A* 35(2) (2004), 481-492.
Doi:10.1007/s11661-004-0359-2
- [8] L. De Colnet, E. Pirard, J. Tchoufang Tchuindjang, J. Lecomte Beckers, R. Gfhiri, P. Boeraeve and S. Cescotto, Quantitative description of MC, M₂C, M₆C and M₇C₃ carbides in high speed steel rolls, University of Liège, Liège, Belgium, In proceedings of the MSMF-3 international conference held in Brno, Krakowic (2001), 710-717.
- [9] J. W. Park, H. C. H. Lee and S. Lee, Composition, microstructure, hardness, and wear properties of high-speed steel rolls, *Metallurgical and Materials Transactions A, Korea* 30A (1999), 399-409.
Doi:10.1007/s11661-999-0329-9
- [10] J. Blaha, C. Kremaszky and E. A. Werner, Carbide distribution effects in cold work tool steels, *Proceedings of the 6th International Tooling Conference, Karlstad, Sweden, Karlstad University* (2002), 289-298.
- [11] M. Boccalini Jr. and A. Sinatora, Microstructure and wear resistance of high speed steel for rolling mills rolls, *Proceedings of the 6th International Tooling Conference, Karlstad, Sweden, Karlstad University* (2002), 509-524.
- [12] C. Rodenburg and W. M. Rainforth, A quantitative analysis of the influence of carbides size distributions on wear behaviour of high-speed steel in dry rolling/sliding contact, Elsevier Ltd., IMPPETUS, Department of Engineering Materials, University of Sheffield, *Acta Materialia* 55 (2007), 2443-2454.
Doi:10.1016/j.actamat.2006.11.039
- [13] K. L. Johnson, *Contact Mechanics*, Cambridge University Press, Cambridge, 1985.

- [14] W. Sitek, A mathematical model of the hardness of high-speed steels, Transactions of Famena XXXIV-3, Institute of Engineering Materials and Biomaterials Silesian University of Technology, Gliwice, Poland (2010), 39-46. UDC 669.14:539.53
- [15] ASTM E3-91, Standard practice for preparation of metallographic specimens, ASTM International (1991), 82-86.
- [16] ASTM E92-82, Standard test methods for Vickers hardness of metallic materials, ASTM International (1991), 260-268.
- [17] ASTM E18-89a, Standard test methods for Rockwell hardness and Rockwell superficial hardness of metallic materials, ASTM International (1991), 176-189.

