INTERNATIONAL JOURNAL OF INNOVATIVE RESEARCH & DEVELOPMENT

ISSN 2278 – 0211 (Online)

Research on Decarburization of Steel Wire SAE 51B35 during Heat Treatment

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Abstract:
Statistical methods have been used to improve processes and accelerate technological development. The aim of this study was to investigate the influence of the variables of heat treatment process of steel wires SAE 51B35 and seek the best condition of certain process parameters adjustment to reduce the depth of decarburization superficial. Four factors were identified as potentially important to the quality of the material. Each of these factors was studied at two levels and all experimental races were held in the production environment. To conduct this survey of classic way would require a large number of experiments, which involve high cost, however, by using the method of design of experiments, structured in a fractional saturated array, it was possible to reduce the financial cost. The study made it possible to conclude what the factors and interactions were causing decarburization problem and what are the best settings to minimize the problem.

Keywords: *Design of experiments, fractional saturated array, heat treatment in SAE steel wire 51B35*

1. Introduction

Statistical studies are not used only for data analysis, but primarily for the planning of experiments in which these are collected. For more sophisticated than is the analysis that, the lack of planning can cause great failure and in many cases, much time can be lost until the researcher realizes that he should have planned their experiments properly.

The use of Fractional Saturated arrays in design of experiments allows the investigation of the influence of factors and their interactions in variable-response behaviour of a given process. The use of the method decreases the cost of testing, without great damage to the conclusions reached (ROSA et al., 2009).

2. Literature Review

2.1. Decarburization

The decarburization is a phenomenon that can occur during heat treatment of steels and involves the loss of carbon in the surface of the material. The decarburization is related to the microstructure of the material and, consequently, with its properties. The main consequences of decarburization are the loss of surface hardness, tensile strength, wear resistance and fatigue strength due to the depletion of carbon from the surface, and may disqualify the material for those functions it normally would play. The decarburization is more serious for applications where the material is not subjected to surface treatment, as for example, carburizing.

According to Tschiptschin (1980), the decarburization can occur for a variety of situations, depending on the specific characteristics of the thermal treatment. The loss of carbon from the surface of the material happens as a result of factors such as temperature and treatment time, furnace atmosphere (presence of oxidizing gases such as oxygen, carbon dioxide and water vapor), carbon steel alloy elements. It can occur through chemical reactions with hydrogen or material with iron oxides, in this last case, forming the slag which is the top layer of rust. By comparison with standards, you can sort the decarburization in three basic types (HERNANDEZ JR.; FONSECA; DICK, 2010):

- Type 1: superficial Region with measurable thickness with ferrite and carbides, free under this layer of ferrite, Pearlite fraction increases with the distance from the surface;
- Type 2: Occurs on the surface a loss exceeding 50% average value of the carbon content of the steel, but without the complete decarburization of this region; and

- Type 3: Occurs on the surface a loss less than 50% of the average carbon content of steel.

Surface Oxidation is made up of three iron oxides (shown in Figure 1), so this factor has been the main factor chosen because there was a chance that the oxidation reaction with the internal temperature of the oven during heat treatment and reduce the carbon from the surface layer of steel wire.

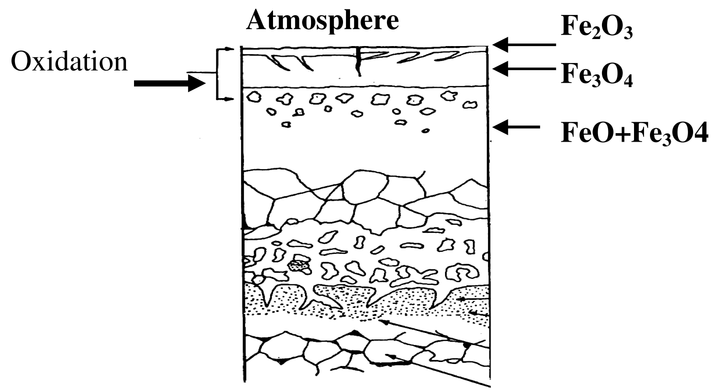

Figure 1: Draw of Oxidation (Tschiptschin, 1980).

2.2. Design of Experiments

According to Lima et al. (2011), Silva and Silva (2008) and Granato et al. (2011), the design of experiments (DOE) is very adequate to study several process factors and their interactions complexity in order to solve problems by means of statistical analysis.

The factorial planning is widely used in experiments involving several factors where it is necessary to study the effect of all of them on one or more answers (MONTGOMERY, 2013).

According to Neto et al. (2007), to perform a factorial planning, you must specify the levels at which each factor should be studied and the more important of these special cases is called 2^k factorial planning, which uses two-level factors k each. In this type of experiment, a full replica requires 2 x 2 x 2 x ......2 = 2^k observations.

According to Benyounis and Olabi (2008), the application of design of experiments can also be performed using fractional arrays, providing a significant reduction in the quantity of experiments. The application of fractional array implies, however, prejudice to the statistical analysis, because it confuses the effects of factors with the interactions.

3. Material and Method of Research

3.1. Drawn Steel Wire

The drawn steel wire is a product widely used in mechanical construction, which is the raw material used for the manufacture of various products such as: screws; chains; bearings and covers for sails.

This product has a great demand of consumers in Brazil and in the world, because they are used in machines in various sectors and especially by the automotive industry.

The material used in this work was the SAE 51B35 steel wire, cold drawn, with 12.85mm diameter, round. Chemical analysis was carried out in the chemical laboratory of the company funding the research, using optical emission spectrometer ARL brand. The results are presented in table 1.

C %	Ni %	Si %	Mn %	P %	Mo %	Al %	Cu %	S%	Cr%	B %
0.35	0.07	0.25	0.46	0.021	0.020	0.022	0.15	0.002	0.97	0.0017

Table 1 : Chemical composition of SAE 51B35 steel wire used in the research.

3.2. Characteristics of Heat Treatment Furnace

For this study, we used a high-convection Bell type oven, with heat treatment capacity for 20 tons of wire per cycle.

The principle of operation of Bell type furnaces boils down basically in the heating and cooling of material loaded at the base, protected by the canopy of protection, with internal pressure always positive. This pressure is obtained by injection of inert protective gas (N_2) with flow rate of 200 m³/h in the first 90 minutes of purging and 300 m³/h after the initial bleed. The goal of maintaining the positive internal pressure is to prevent the entry of oxygen inside the base (canopy).

According to Hernandez Jr.; Fonseca; Dick (2010) the heat treatment is widely used in medium and high carbon steels in order to produce a structure of globular carbide in a ferritic structure array. This structure provides the reduction of hardness, the increase in ductility and Machinability.

Loads of steel (SAE 51B35) used in this research were treated according to the X and Y, whose time and temperature settings are represented in table 2.

	Cycle X			**Cycle Y**		
	Temperature (Degrees Centigrade)		Time (Minutes)	Temperature (Degrees Centigrade)		Time (Minutes)
	Start	End		Start	End	
1	25	760	1320	25	765	1485

Table 2 : Characteristics of thermal treatment cycles.

3.3. Selection of Factors, Response Variable and Choice of Array of Design of Experiments

For the selection of the factors raised the possible causes that could influence the decarburization of the wire, being selected the following:

- Oxidation, assuming for the sake of argument that iron contained oxides, somehow during the thermal treatment, could react with the surface layer of steel wire by subtracting the carbon.
- Moisture, assuming the oxygen emitted by the sample in wet condition, because the moisture caused by the wet material would evaporate after heating and oxygen released could react chemically with the surface layer of steel due to high temperature in the oven;
- Heat treatment cycle, assuming the time and the temperature had influence on decarburization;
- Dew point, assuming that the amount of oxygen inside the oven had influence on decarburization. This factor characterized by measuring the internal pressure inside heat treatment furnace. The values indicate measurements performed by the specific equipment of this heat treatment furnace;
- Interaction between russets and heat treatment cycle, assuming the oxides react with the time and temperature;
- Interaction between russets and dew point, supposing the oxides interact with oxygen inside the oven (if there was oxygen in your interior).

The selection of the levels of the factors was based on the actual condition of the process (the minimum and maximum for all factors).

The choice of Fractional Saturated Array best suited for the situation was based on number of studied factors (Oxidation, Heat treatment cycle, Moisture and Dew point). As in this case it would be necessary to study four factors and suspected-whether the existence of two interactions, was chosen a Fractional Saturated Array with 7 columns with the corresponding spaces for inclusion of factors or interactions (table 3).

Experiments	**Columns**						
	A	**B**	**C**	**AB**	**AC**	**BC**	**ABC**
1	-	-	-	+	+	+	-
2	+	-	-	-	-	+	+
3	-	+	-	-	+	-	+
4	+	+	-	+	-	-	-
5	-	-	+	+	-	-	+
6	+	-	+	-	+	-	-
7	-	+	+	-	-	+	-
8	+	+	+	+	+	+	+

Table 3 : Fractional Saturated array with 8 and 7 factors experiments/interactions

The - and + signs inside the Table 3 refer respectively to the low and high levels of each factor. The allocation of each factor follows the following order:

- Column A: Put the factor Oxidation;
- Column B: Put the factor Heat treatment cycle;
- Column C: Put the factor Moisture;
- Column AB: Put the factor Dew point. It is important to remember that in this method, the use of the column AB would generate the interaction between the factors A and B respectively, but in this case, the placement of the Factor Dew point in this column will generate, confounding between the Factor Dew point and interaction AB, being this situation already provided for the use of this method of Fractional Saturated Array, with the intention of obtaining the 50% reduction in costs with experimentation because a complete array would, in this case, the double of experiments and in proportion would double the costs associated.
- Column AC: Used to calculate the value of the interaction between the oxidation Factor and Moisture;
- BC column: Used to calculate the value of the interaction between the factors heat treatment cycle and Moisture;

● ABC column: will be used to calculate the experimental error, which is necessary for calculation of the significance of the factors and interactions.
All settings of the factors and their respective levels of adjustment are presented in table 4.

Fatores	**A-** Oxidation	**B-** Heat treatment cycle	**C-** Moisture	**AB-** Dew point	**AC-** Interaction between A e C	**BC-** Interaction between B e C
Nível -	With Oxidation	X	With Moisture	-35		
Nível +	without Oxidation	Y	Without Moisture	-25		

Table 4: Factors and their levels

3.4. Preparation of Samples for Testing
The raw material (SAE 51B35 drawn steel wire) selected for realization of experiments was obtained from the same manufacturing batch to the lower variation possible in relation to the decarburization, since it would hardly be possible to obtain in this case materials exempt from this feature.
The sample was sent to the laboratory of the company funding the research to measure the initial decarburization and to her this analysis an average value of 0,03mm deep. The table 5 describes the experiments performed.

Experiments	**Description**
01	With Oxidation, Cycle X, With Moisture, -25.
02	without Oxidation, Cycle X, With Moisture, -35.
03	With Oxidation, Cycle Y, With Moisture, -35.
04	without Oxidation, Cycle Y, With Moisture, -25.
05	With Oxidation, Cycle X, Without Moisture, -25.
06	without Oxidation, Cycle X, Without Moisture, -35.
07	With Oxidation, Cycle Y, Without Moisture, -35.
08	without Oxidation, Cycle Y, Without Moisture, -25.

Table 5 : Description of the experiments

The results of the decarburization of each experimental condition of Fractional Saturated Array are shown in table 6.

Experiments	**Decarburization (mm)**									
01				0.200						
02				0.073						
03				0.213						
04				0.105						
05				0.125						
06				0.090						
07				0.140						
08				0.095						

Table 6 : Results of the response variable (Decarburization)

4. Data Analysis and Results

4.1. Influence of the Factors
Analyzing the table 7 is found that the influential factor causing decarburization is the oxidation factor (A), since this factor presents the P value less than 0.05 for 95% confidence level (default used). It was also possible to see the influence of the moisture Factor and the interaction between the factors moisture and oxidation. The Figure 2 shows the Pareto chart that the column related to this factor and the interaction exceeded the critical value established for the 95% confidence level. In this way, through the application of fractional saturated array, proved the influence of surface oxidation of the material when subjected to the heat treatment, noting the presence of interaction between this superficial oxidation and the temperature inside the furnace heat treatment. This influence was established due to observation of the results which indicated that the material with the oxidized surface layer causes the highest oxidation when heat treated. With the calculation of the coefficients shown in table 7 was made possible the creation of the mathematical model of decarburization process. However, in table 7 the software generated the model considering all the factors studied, but will only be considered for the final model the factors and interactions considered influential on decarburization, other factors will be eliminated from the model by their significance statistically proven to the 95% confidence level. The mathematical model of decarburization is described in the formula M_1:

Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value
Constant		0,13012	0,00113	115,67	0,006
Oxidation	-0,07875	-0,03938	0,00113	-35,00	0,018
Heat treatment cycle	0,01625	0,00813	0,00113	7,22	0,088
Moisture	-0,03525	-0,01763	0,00113	-15,67	0,041
Dew point	-0,00725	-0,00363	0,00113	-3,22	0,192
Oxidation*Moisture	0,03875	0,01938	0,00113	17,22	0,037
Oxidation*Dew point	-0,00625	-0,00312	0,00113	-2,78	0,220

Regression Equation in Uncoded Units

Decarburization = 0,13012 - 0,03938 Oxidation + 0,00813 Heat treatment cycle
- 0,01763 Moisture - 0,00363 Dew point + 0,01938 Oxidation*Moisture
- 0,00312 Oxidation*Dew point

Table 7 : Statistical analysis for evaluation of the influence of the factors (Calculated in Minitab software)

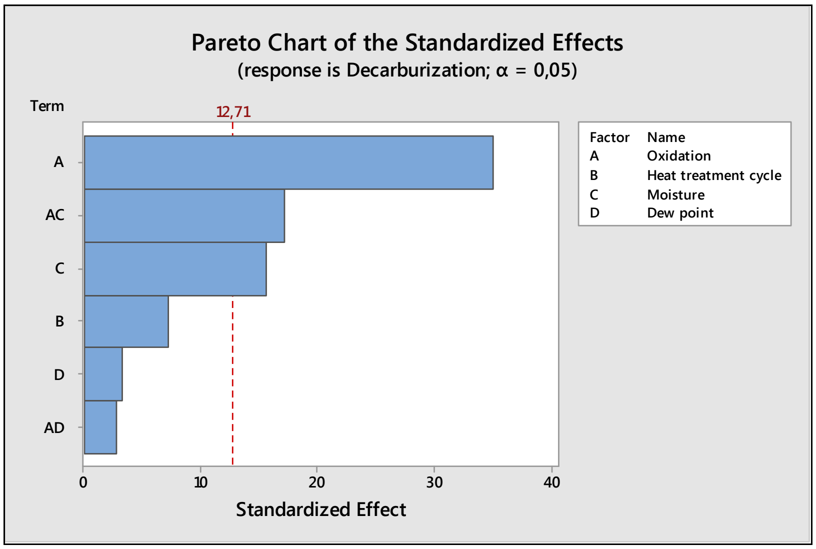

Figure 2 : Pareto chart for test of significance of the factors and interactions investigated

Observing figure 3 it is possible to perceive in which fits the influential factors (rust and moisture) increase or decrease the decarburization. Both factors increase the average decarburization when are set on level (-) which means the material with rust and moisture (wet). The opposite also occurs, if getting a little when the material not decarburization has rust and lacks moisture.

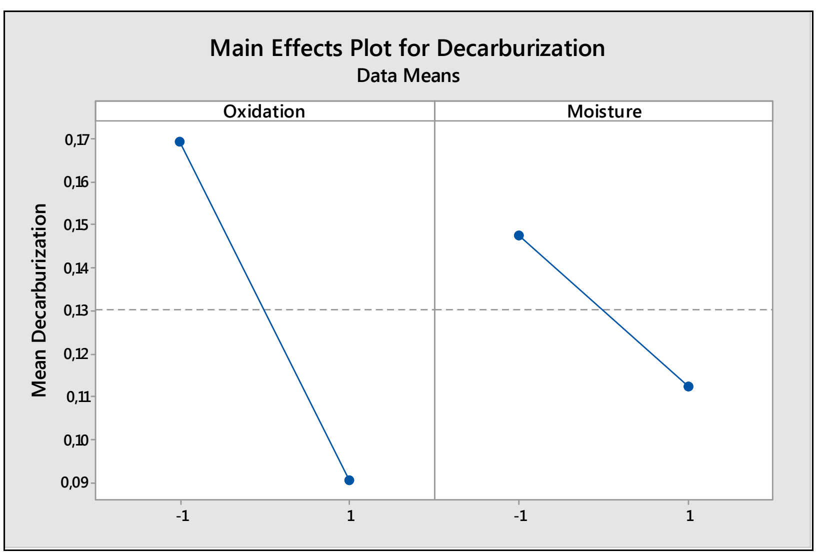

Figure 3 : Influence of settings of the factors on the decarburization

Decarburization = 0.13012 - 0.03938 (oxidation) - 0.01763 (moisture) + 0.01938 (oxidation x moisture)

M_1

5. Conclusion

It was concluded that the oxidation factor is the factor causing decarburization of drawn steel wire SAE 51B35 during the heat treatment process. It was also possible to see the influence of the moisture Factor and the interaction between the factors moisture and oxidation. It is believed that the influence of the moisture factor on decarburization is precisely because with the increase in the internal temperature of the oven there will be the evaporation of the water contained in the material (cause of the moisture) and in this case, the oxygen may remain for some time in the oven and react chemically (by high temperature), with the top layer of the steel-wire and subtract the carbon of your surface. In addition, with proof of the influence of oxidation and moisture factors one can imagine that the occurrence of these two factors acting simultaneously could further enhance the decarburization for dealing with two causative factors of this phenomenon, that justifies the proven influence of the interaction between these factors.

This conclusion indicated the need to better plan the operational practice of this process, causing the steel wires are previously sandblasted with steel shot or chemically pickled for removal of surface oxidation before heat treatment.

There was also the possibility of reduction of decarburization depth by more than 50% during the heat treatment. However, it is necessary to standardize the oxidation removal before the material be treated thermally. In addition, it is important whenever possible keep the material stored in appropriate locations and protected against the action of rain, thus preventing this is material with moisture (wet) and be placed in the oven to heat treatment in this condition, as this reduced enough the decarburization caused during heat treatment.

It was observed in the experimental results obtained by fractional saturated array used in planning experiments that in experiment 3 the decarburization was too large to 0.213mm, while in experiment 2 was only 0.073mm. For the significant reduction of decarburization of steel wire during the heat treatment is necessary the total removal of surface oxidation of the material, because it was found in this study that the superficial oxidation associated with the temperature in the heat treatment cycle causes the significant reduction of the chemical element carbon. It is important to note that the decarburization above 0.10 mm for steel wire results in your disqualification for their mechanical applications required. Therefore, very important to standardize the removal of oxidation of the surface layer of the wire where the wire is subjected to heat treatment.

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