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INFLUENCE OF PHYSICAL AND CHEMICAL INTERACTIONS BETWEEN STEEL AND THEIR PRETREATMENT AND MACHINING CONDITIONS IN THE FORMATION OF SOFT SPOTS IN THERMOCHEMICAL TREATMENTS[1]

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Abstract

Soft spots or reduction of the hardened layer in localized areas on parts thermochemical treated (Carburizing, Carbonitriding and Nitriding) are relatively common and are typically derived from one inadequate cleaning before treatment. Improper cleaning allows the presence of surface contaminants from cutting fluids used in the machining processes, acting as a barrier to the penetration of Carbon and Nitrogen in the heat treatment processes. This paper presents the problem of formation of soft spots during thermochemical treatments of steel in a more complex way than normal, from the interaction of several parameters which contribute to the formation of a passivation barrier, reducing the penetration of C and N during treatment. These parameters were associated with the type of steel and its pretreatment before machining, and various parameters of machining operations as those fluids, tools, and cutting speed. Solutions to avoid this kind of problem are also presented.

Key words: Soft spots; Thermochemical treatments; Passivation barrier.

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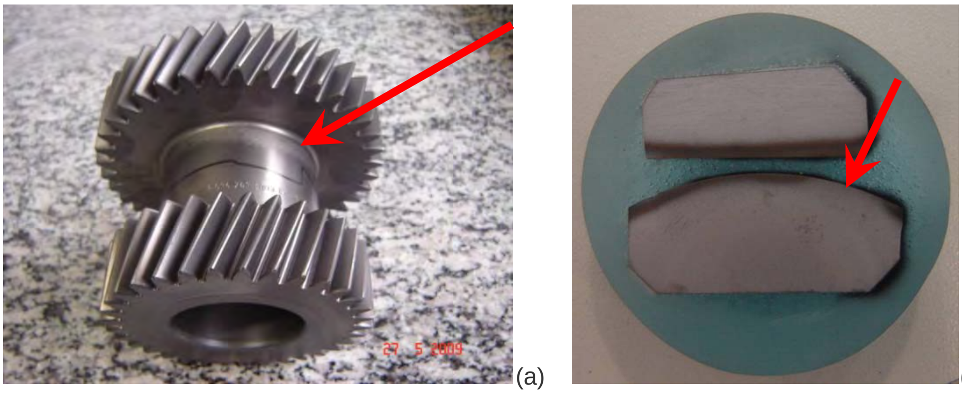
1 INTRODUCTION

Thermochemical treatments of steels as carburizing, carbonitriding and nitriding are fundamental processes in manufacturing parts such as shafts and gears for the mechanical industry. The mechanical strength properties, wear, contact and bending fatigue in these types of parts are critical to their performance.
Therefore it is very important to control the variables of heat treatment to get metallurgical properties such as surface and core hardness, case depth, and case and core microstructures within the engineering specifications as well as good repeatability of these parameters over process.
The thermochemical processes still most used in the world are those with controlled gaseous atmosphere. The most important variables in these processes are working temperatures, the duration of treatment and the treatment atmosphere control (in the case of carburizing, the carbon potential available in the atmosphere). Other variables also important in thermochemical treatment are the conditions of quenching (oil type, temperature, agitation, etc.) and the final process of tempering to stress relieving from quenching and the final adjustment of the hardness of the part.
Besides the process variables, another very important factor is the preparation of parts for treatment.[1,2] Parts and loading fixtures must be thoroughly cleaned before entering the furnace treatment. Usually parts and fixtures are washed in an heated alkaline solution and then dried before go into the furnace. Residues of alkaline solution or waste cutting fluids from machining can form protective films on parts which preclude the entry and diffusion of carbon or nitrogen during the thermochemical process. These protective films or barriers may locally reduce the treated layer depth and the surface hardness. This localized areas with low case depth and hardness can be called “soft spots”. A typical example of soft spot is showed in Figure 1.



(a) (b)

Figure 1. Soft spot on external diameter of gear, (a) area with porous aspect (visual spot), (b) sample from gear showing a lack of carburizing (no carbon penetration).

This paper presents the problem of formation of soft spots during thermochemical treatments of steel in a more complex way than normal. The interaction of several parameters which contribute to the formation of a passivation barrier, reducing the penetration of C and N during treatment was studied. These parameters were associated with the type of steel and its pretreatment before machining, and various parameters of machining operations as those fluids, tools, and cutting speed. Brinksmeier, Lucca and Walter[3] and Walter[4] studied the influence on the surface properties of the physical and chemical interactions between the cutting tool, part

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material, and metalworking fluid in machining processes. Unfortunately there is not much technical literature on this particular subject.

2 MATERIAL AND METHODS

2.1 Material

Two different steels were compared in this study. The choice of the steels was based on ease or difficulty of obtaining a microstructure most suitable for machinability. A part as showed at Figure 1 was used for tests. The Table 1 shows the nominal chemical composition (main elements) and the heat treatment after forging to obtain the desired microstructure suitable for machining.

Table 1. Nominal chemical composition and forging heat treating

Material	Nominal Chemical Composition (%)					Forging Heat Treating
	C	Mn	Cr	Ni	Mo	
A	0.24	1.05	0.50	-	0.15	Normalizing
B	0.21	0.85	0.83	-	0.43	Isothermal Annealing

2.2 Heat Treatment

In all experiments, the samples were carburized, quenched and tempered in continuous furnaces with controlled endothermic atmosphere. The parameters of heat treatment as carburizing time, carburizing temperature, quenching temperature, conditions of quenching (type of oil, temperature and agitation), and tempering temperature and time were kept constant in all experiments to have no influence on the results. A typical continuous furnace is showed at Figure 2.
During the experiments, the prewashing machine was strongly controlled to give to the parts, the best quality in cleanness to prevent any soft spots due to some impurities or fluid contamination from the machining area.

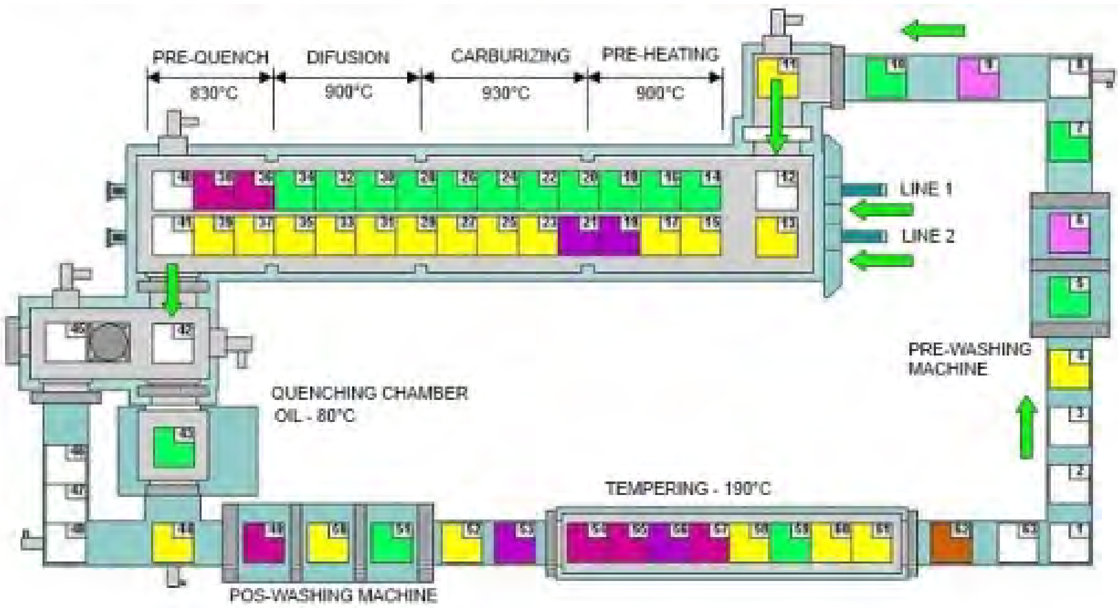

Figure 2. A typical continuous furnace used in the experiments integrated with prewashing machine, quenching chamber, post washing machine and tempering furnace.

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2.3 Process Map

A complete process map about the formation of soft spots was did and analyzed. Then, the main inputs and defined experiments were established to understand the problem completely.(5) Below a list of the experiments planned to compare the influence of each parameter and its interactions each other:

- Multi-vari analysis of dirt elements that came from oil and lubricants and the raw material (Figure 3).

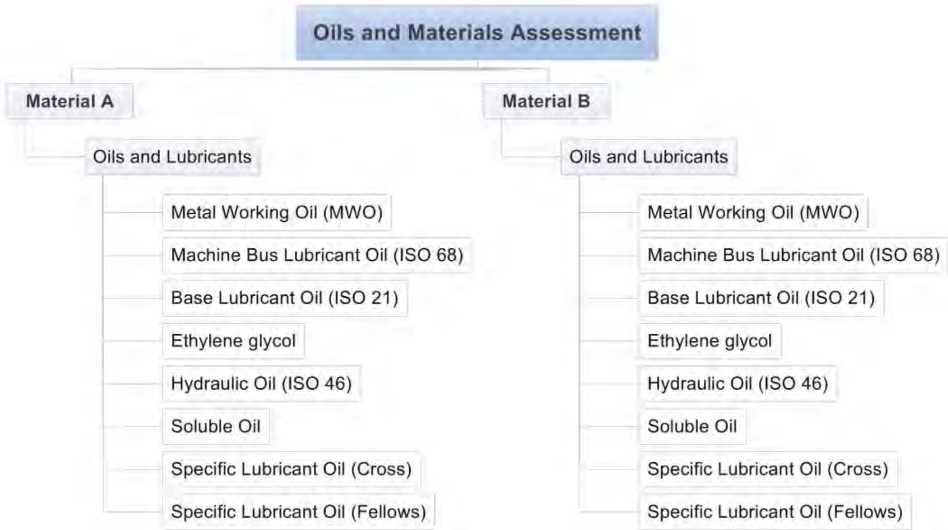

Figure 3. Steels and different oils and lubricants from machining evaluated.(5)

- A full-factorial 3 factor/2 level DOE of turning tools, finished surface machining, degreasing and their interactions (Figure 4).

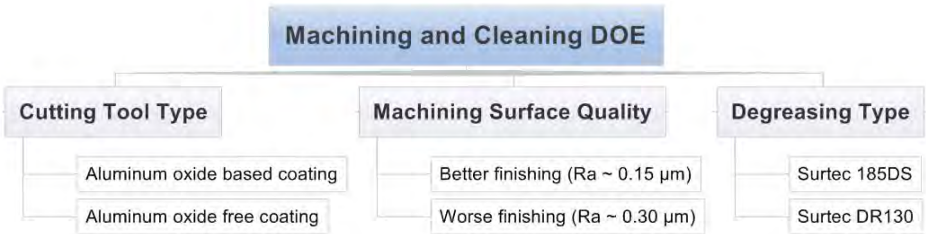

Figure 4. Interactions between DOE factors (turning tools, finished surface and degreasing) and its levels.(5)

- A full-factorial 2 factor/2 level DOE of heat treatment and raw material (Figure 5).

Figure 5. Interactions between DOE factors (materials and furnaces) and its levels.(5)

- A full factorial 2 factor/2 level DOE of forging microstructure and turning tools (Figure 6).

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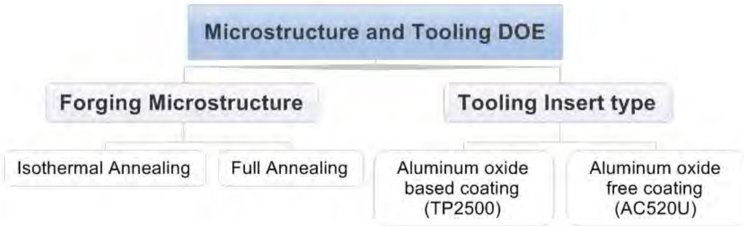

Figure 6. Interactions between DOE factors (forging microstructure and turning tools) and its levels.[5]

2.3.1 Identification of the carburizing inhibitor element

Were used two different techniques to try to identify the composition of the carbon inhibitor layer formed on some areas of the part:

- EDS: Energy dispersive spectroscopy;
- GDOS: Glow discharge optical spectroscopy.

2.3.2 Effective cleaning system to avoid soft spots

Different methods were used to eliminate the local lack of carburizing (soft spots):

- Double washing before the parts to the furnace;
- Shot blast the green parts (after machining);
- Pressure washing;
- Different alkaline degreasing;
- Manganese phosphate process before heat treatment;
- Acid degreasing the green parts (after machining).

2.3.3 Evaluation of soft spots

To ease the evaluation of all experiments, was initially given a visual analysis of soft spots that appear as a "stain" upon application of shot peening (after heat treatment). The blasting or peening visually presents a "stain" as shown in Figure 1. The location of the "stain" has low surface hardness (which is why we call these regions as soft spots) due to the lack of penetration of carbon. Then, after the visual determination of the regions with soft spots, hardness Rockwell C measurements were performed to confirm and evaluate the decrease of hardness.

3 RESULTS

For the interactions between oils and lubricants from machining and steels (Figure 3), the BoxPlot for Hardness is showed at Figure 7 and a Multi-Vari Chart for Hardness by Oil and Material is showed at Figure 8.

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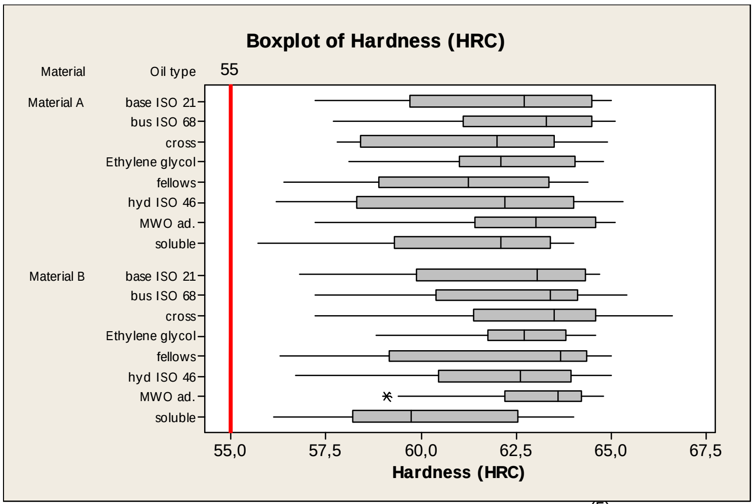

Figure 7. Boxplot of hardness (HRC).(5)

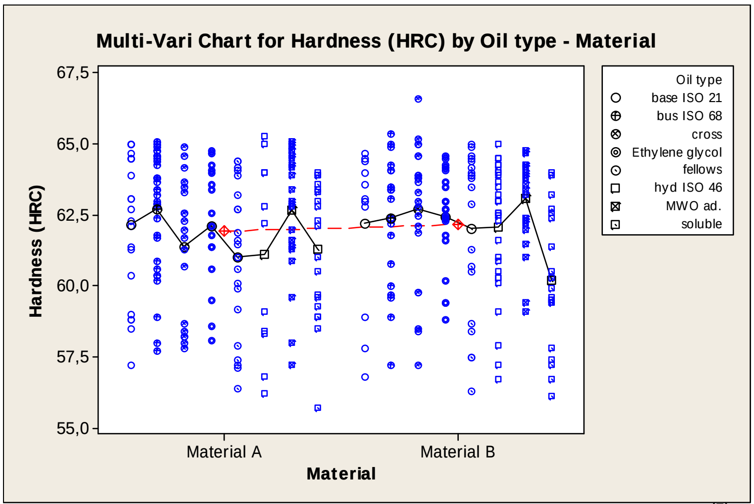

Figure 8. Multi-Vari chart for hardness by oil and material.(5)

For the interactions between turning tools, finished surface and degreasing (Figure 4), the Pareto Chart of the Effects is showed at Figure 9. The Pareto chart includes both defective fraction, p (%), and the Freeman-Tukey transformation of defective fraction, p (adm). This transformation was done in order to confirm the analysis by the experiment variance stabilization.(6) The Figure 10 shows the Main Effect Plot for defective fraction (% of rejected parts), and Figure 11 shows the Interaction Plot for % of rejected parts.

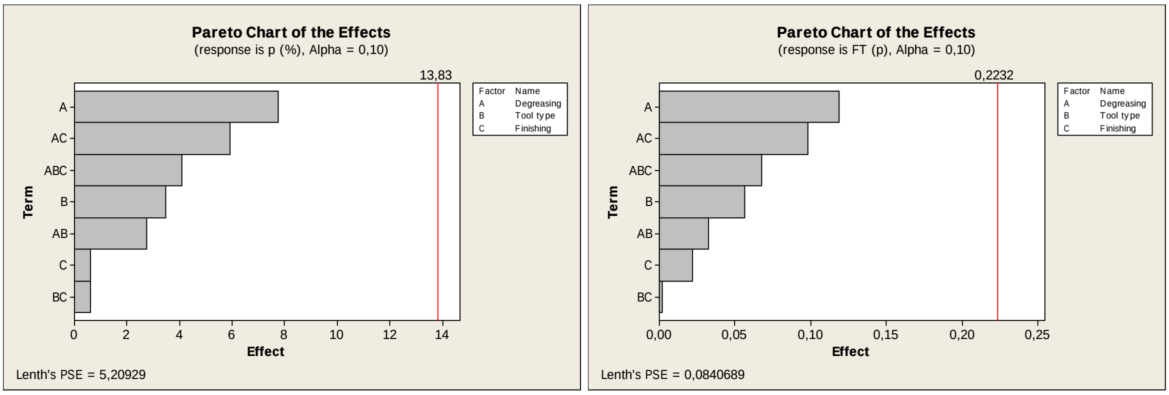

Figure 9. At left, Pareto chart of the effects of defective fraction (p, as % of rejected parts), and at right, Pareto chart of the effects of Freeman-Tukey transformation of defective fraction, p (adm).(5)

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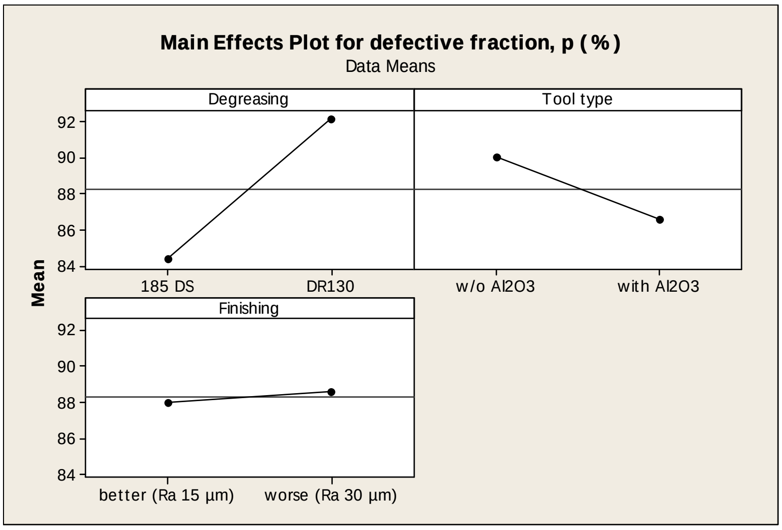

Figure 10. Main effect plot of defective fraction (% of rejected parts).(5)

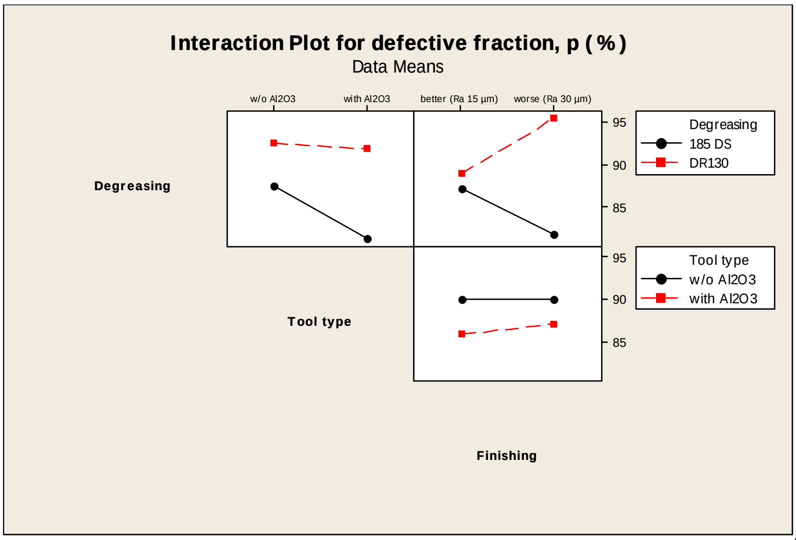

Figure 11. Interaction plot for defective fraction (% of rejected parts).(5)

For the interactions between heat treatment and raw material (Figure 5), the Main Effect Plot for defective fraction (% of rejected parts) is showed at Figure 12, and Figure 13 shows the Interaction Plot for defective fraction (% of rejected parts).

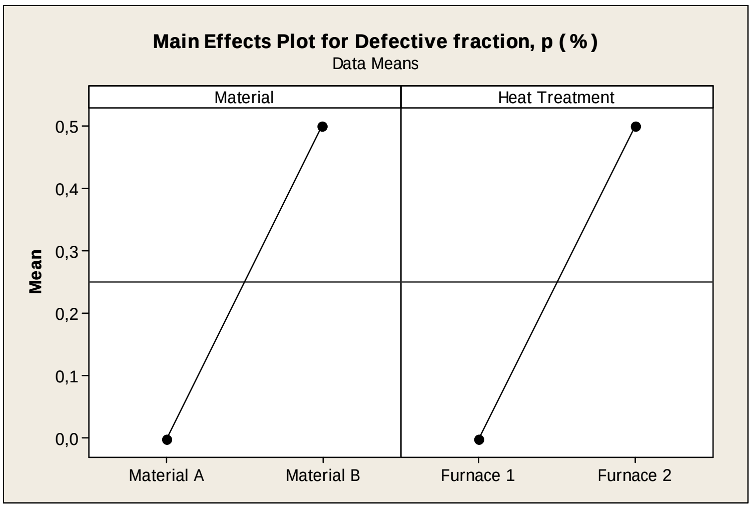

Figure 12. Main effects plot for defective fraction (% of rejected parts).(5)

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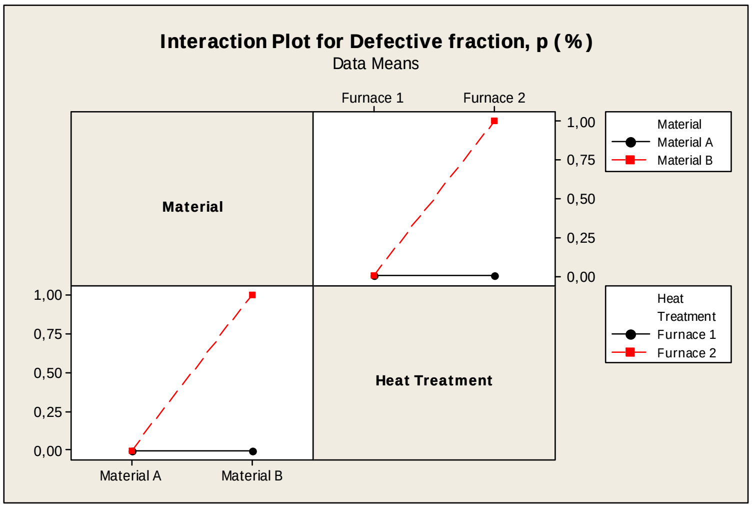
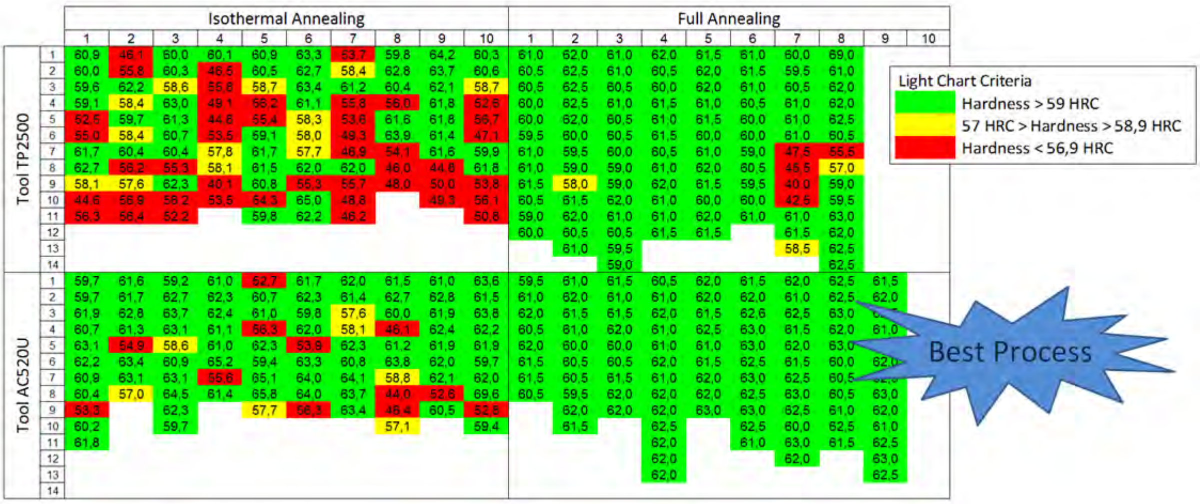


Figure 13. Interactions plot for defective fraction (% of rejected parts).(5)

For the interactions between forging microstructure and turning tools (Figure 6), the Light Chart for Hardness is showed at Figure 14, and the Figure 15 shows the Individual Value Plot for Hardness.



Tool TP2500

	Isothermal Annealing 1	2	3	4	5	6	7	8	9	10	Full Annealing 1	2	3	4	5	6	7	8	9	10
1	60,9	46,1	60,0	60,1	60,9	63,3	53,7	59,8	64,2	60,3	61,0	62,0	61,0	62,0	61,5	61,0	60,0	69,0		
2	60,0	55,8	60,3	46,5	60,5	62,7	58,4	62,8	63,7	60,6	60,5	62,5	61,0	60,5	62,0	61,5	59,5	61,0		
3	59,6	62,2	58,6	55,8	58,7	63,4	61,2	60,4	62,1	58,7	60,5	62,5	60,5	60,0	62,0	61,0	60,5	62,0		
4	59,1	58,4	63,0	49,1	56,2	61,1	55,8	56,0	61,8	52,6	60,0	62,5	61,0	61,5	61,0	60,5	61,0	62,0		
5	52,5	59,7	61,3	44,6	55,4	58,3	53,6	61,6	61,8	56,7	60,0	62,0	60,5	61,0	61,5	60,0	61,0	62,5		
6	55,0	58,4	60,7	53,5	59,1	58,0	49,3	63,9	61,4	47,1	59,5	60,0	60,5	61,5	60,0	60,0	61,0	60,5		
7	61,7	60,4	60,4	57,8	61,7	57,7	46,9	54,1	61,6	59,9	61,0	59,5	60,0	60,5	61,0	59,0	47,5	55,5		
8	62,7	56,2	55,3	58,1	61,5	62,0	62,0	46,0	44,8	61,8	61,0	59,0	59,0	61,0	62,0	60,5	45,5	57,0		
9	58,1	57,6	62,3	40,1	60,8	55,3	55,7	48,0	50,0	53,8	61,5	58,0	59,0	62,0	61,5	59,5	40,0	59,0		
10	44,6	56,9	58,2	53,5	54,3	65,0	48,8		49,3	56,1	60,5	61,5	62,0	61,0	60,0	60,0	42,5	59,5		
11	56,3	56,4	52,2		59,8	62,2	46,2			50,8	59,0	62,0	61,0	61,0	62,0	61,0	61,0	63,0		
12											60,0	60,5	60,5	61,5	61,5		61,5	62,0		
13												61,0	59,5				58,5	62,5		
14													59,0					62,5		

Tool AC520U

	Isothermal Annealing 1	2	3	4	5	6	7	8	9	10	Full Annealing 1	2	3	4	5	6	7	8	9	10
1	59,7	61,6	59,2	61,0	62,7	61,7	62,0	61,5	61,0	63,6	59,5	61,0	61,5	60,5	62,0	61,5	62,0	62,5	61,5	
2	59,7	61,7	62,7	62,3	60,7	62,3	61,4	62,7	62,8	61,5	61,0	62,0	61,0	61,0	62,0	62,0	61,0	62,5	62,0	
3	61,9	62,8	63,7	62,4	61,0	59,8	57,6	60,0	61,9	63,8	62,0	61,5	61,5	62,0	61,5	62,6	62,5	63,0	[illegible]	
4	60,7	61,3	63,1	61,1	56,3	62,0	58,1	46,1	62,4	62,2	60,5	61,0	62,0	61,0	62,5	63,0	61,5	62,0	61,0	
5	63,1	54,9	58,6	61,0	62,3	53,9	62,3	61,2	61,9	61,9	62,0	60,0	60,0	61,0	61,0	63,0	62,0	63,0	[illegible]	
6	62,2	63,4	60,9	65,2	59,4	63,3	60,8	63,8	62,0	59,7	61,5	60,5	60,5	62,0	61,5	62,5	61,5	60,0	62,0	
7	60,9	63,1	63,1	55,6	65,1	64,0	64,1	58,8	62,1	62,0	61,5	60,5	61,5	61,0	62,0	63,0	62,5	60,5	[illegible]	
8	60,4	57,0	64,5	61,4	65,8	64,0	63,7	44,0	52,6	69,6	60,5	59,5	62,0	62,0	62,0	62,5	63,0	60,5	63,0	
9	53,3		62,3		57,7	56,3	63,4	46,4	60,5	52,8		62,0	62,0	62,0	63,0	63,0	62,5	61,0	62,0	
10	60,2		59,7					57,1		59,4		61,5		62,5		62,5	60,0	62,5	61,0	
11	61,8													62,0		61,0	63,0	61,5	62,5	
12														62,0			62,0		63,0	
13														62,0					62,5	
14																				

Light Chart Criteria

Green	Hardness > 59 HRC
Yellow	57 HRC > Hardness > 58,9 HRC
Red	Hardness < 56,9 HRC

Best Process

Figure 14. Light chart for hardness.(5)

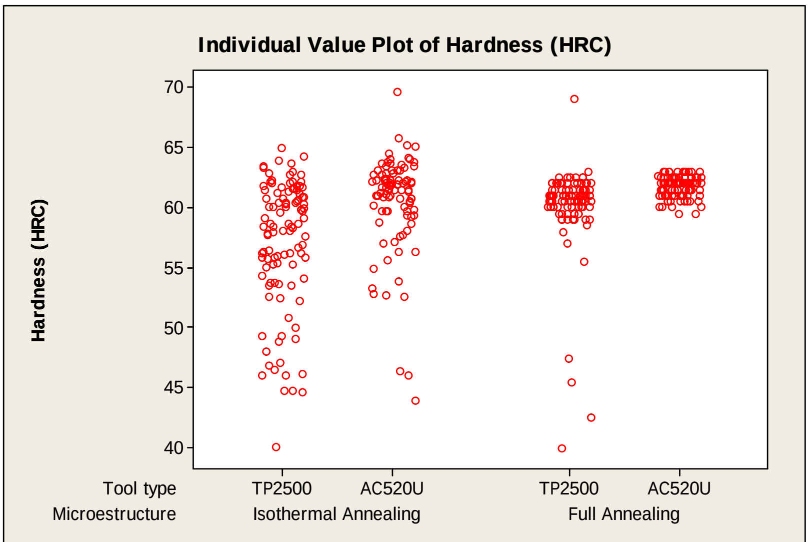


Figure 15. Individual value plot for hardness(5)

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The Figures 16 and 17 show EDS analysis on areas without and with soft spots, respectively.

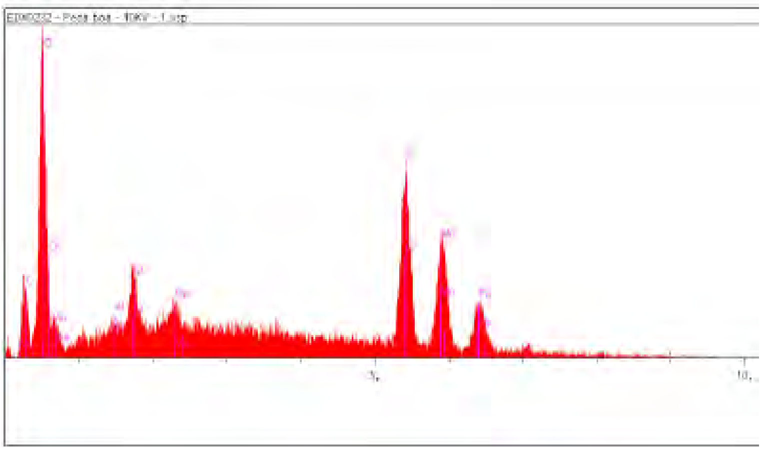

Figure 16. Area without soft spots.

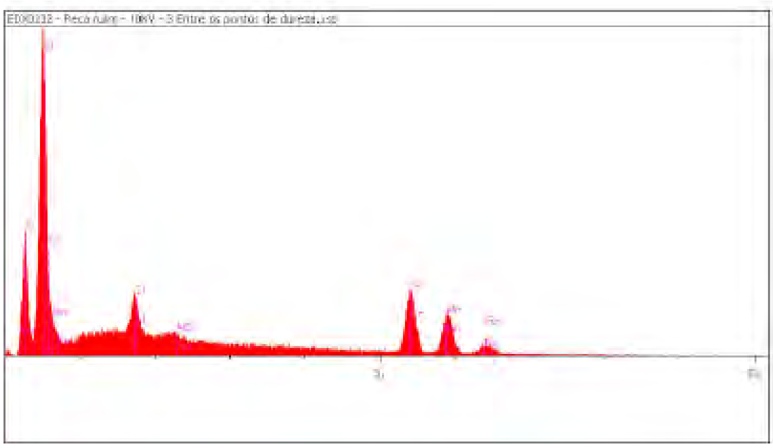

Figure 17. Area with soft spot.

The Figures 18 and 19 show GDOS analysis in two areas with soft spot in different depth.

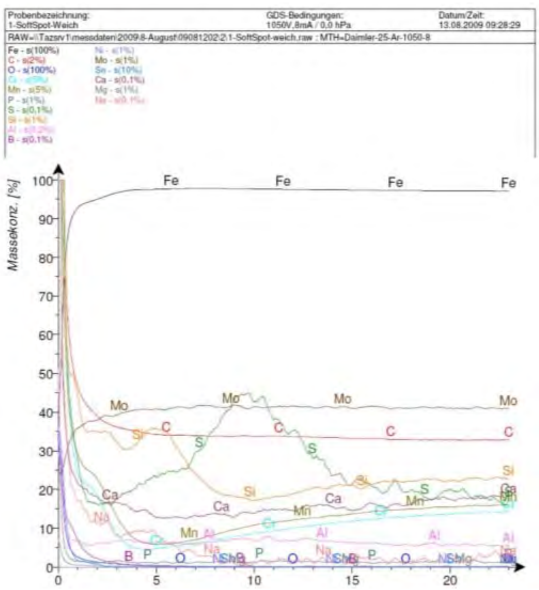

Figure 18. Area with soft spot (0 to 20 nm analysis).

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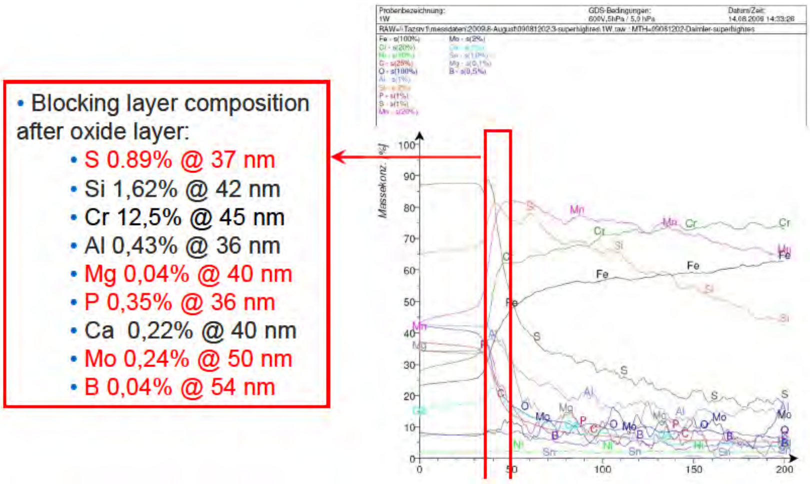

Figure 19. Area with soft spot (0 to 200 nm analysis).

The Figure 20 shows the results for each containment process.

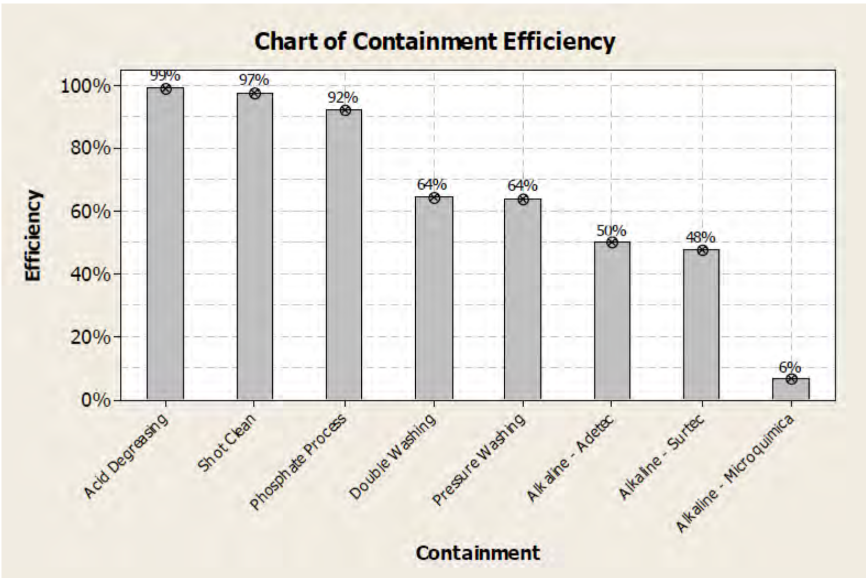

Figure 20. Containment efficiency chart.[5]

4 DISCUSSION

The objective of the experiment showed at Figure 3 was to check the influence between different oils and lubricants used in all steps of the process and two types of carburized steels (Table 1) commonly used in this process, and their interactions to form the defect called soft spots. By the analysis of Figures 7 and 8 is possible to conclude that there is no influence between the factors studied. In this experiment, all parts were pickling with acid and this action probably remove any possible inhibitor element to carburizing. This was the first indication that acid pickling can remove some kind of prejudicial contamination.

The experiment showed at Figure 4 was to analyze the influence between turning tools, machining surface finished and degreasing. By the Figure 9 (Pareto Chart) is possible to conclude that no factor and their levels studied are statistically significant. The Figure 10 (Main Effect Plot) shows that degreasing influenced the results in 8%, while the factor turning tools and surface finishing influenced 4% and 2% respectively. The Interaction Plot (Figure 11) shows strong interaction between the

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factors degreasing and surface finishing, weak interaction between the factors degreasing and turning tools, and no interaction between turning tools and surface finishing. Although there is no conclusive answers, this experiment indicated the degreasing could be a very important factor, mainly because other experiments showed bad results with parts with bad surface finishing.
The objective of the experiment showed at Figure 5 was to evaluate the factors heat treatment (two different continuous furnaces) and different steels as showed on Table 1. The results from Figures 12 and 13 can be considered no conclusive. But, parts manufactured with material A did not presented soft spots in the experiment.
The objective of this experiment (Figure 6) was evaluate the impact of the factors forging microstructure and turning tools for the formation of soft spots. Only the Material B was used in this experiment because it showed more sensitive for soft spot formation during carburizing in all experiments. The factor forging microstructure was tested in the levels Isothermal Annealing and Full Annealing. The microstructures for each treatments were analyzed and are showed at Figure 21.

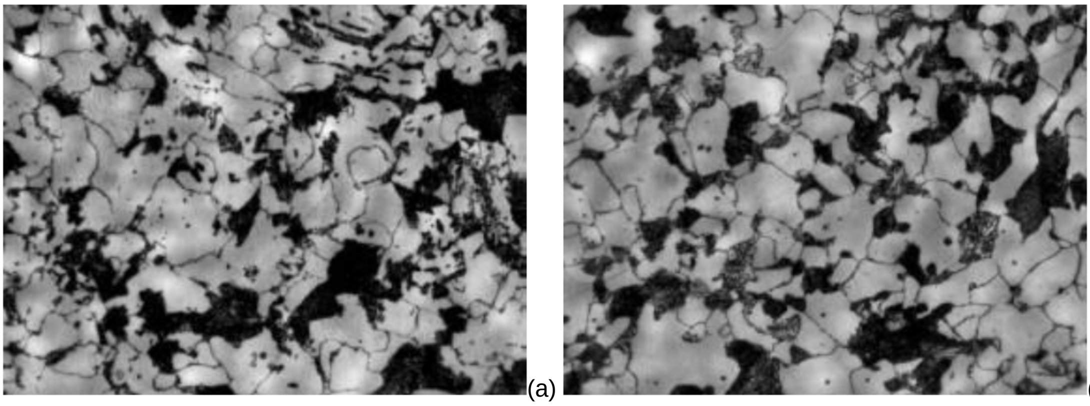
(a) (b)

Figure 21. Etch Nital 3.5%, 400x. (a) Isothermal annealing: ferrite, pearlite and some quantity of cementite, (b) Full annealing: ferrite, pearlite and small quantity of cementite.

The Light Chart for Hardness (Figure 14) shows all hardness measurement in each part in their respective experiment run. This chart was built based on surface hardness specification. Color green means hardness higher than 59 HRC, yellow means 57 – 58 HRC, and red means hardness less than 57 HRC. Through this chart is possible to see that the best process was reached with full annealing and turning tool A520U.
Figure 15 shows the “best process” that presented less variation on surface hardness when compared to others experiments in the study.
Other experiments using Material B focusing the forging microstructure showing a strong dependency of this factor. The microstructure from Isothermal or Full Annealing has strong variation lot by lot. And some regions of the parts usually present some colonies of bainite. Bainite has higher hardness compared to a mix of ferrite and pearlite, and this combination can play a different conditions during machining in areas with bainite. Parts with a very good forging microstructure (homogeneous ferrite and pearlite distribution, without colonies of bainite) do not present soft spots.
Through all experiments realized was possible to conclude that:

- there is an element (small layer) that inhibit the carbon penetration during carburizing;

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- this “element” came from an interaction between the type of raw material, oils and lubricants, machining parameters (as cutting speed) and type of turning tools used in green machining;
- this layer can be removed only with aggressive cleaning after machining using acid pickling, shot blasting or pre-oxidation process.

To try to identify the inhibitor elements two different techniques were used: EDS and GDOS. EDS analysis using a Scanning Electronic Microscope (Figures 16 and 17) did not show no effective element potentially to acting as a carburizing inhibitor. The x-ray in this analysis usually has a penetration of 1-2 µm from the surface. Then, using a GDOS technique that analyze from the surface depths is a nanometer scale (Figures 18 and 19) was possible to see a higher concentration of different chemical elements close the surface forming a “blocking layer”. This carbon blocking layer is basically formed by Mg, P,S, B and Mo. This technique to observe elements in a nanometer scale that can prevent carbon or nitrogen penetration is in accordance to Brinksmeir, Lucca and Walter[3] and Walter[4] works.
Based on these verifications, several aggressive cleaning process were studied to remove this carburizing blocking layer after machining. Figure 20 presents the results for each process studied. Acid cleaning presented 99% of efficiency, followed by shot blasting with 97% of efficiency.

5 CONCLUSION

Areas with soft spots present a significant reduction of carbon content and case depth when compared with areas without soft spots. This reduction on carbon content is accompanied by a reduction in surface hardness.
There is a contaminant layer on the part surface inhibiting the carburizing process. The elements that forming this blocking layer come from green machining.
The process to produce this blocking layer is due a combination of several factors: type of steel used; forging microstructure due to non-uniform and proper annealing process; high cut machining speed; and, type of turning tools (inserts).
This kind of inhibiter layer cannot be removed only using common alkaline washing process. It is necessary to use a more aggressive method to remove it.
The main aggressive methods to clean the parts could be by mechanical cleaning (Shot Blasting) or chemical cleaning (manganese or zinc phosphate, oxidizing before heat treatment or washing using a lightly acid degrease.

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