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Using design of experiments in the evaluation of the microstructural characterization parameters with the LePera reagent in a multiphase steel

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Abstract

The performance of multiphase steels is closely related to their microconstituents. Thus, the characterization and effective control of the microstructure of these materials are crucial, and chemical attack processes are essential for this to occur. In this context, this study sought to employ the Taguchi method to evaluate the chemical etching process with the LePera reagent in order to investigate the influence of different process variables, in addition to the interactions between them, on the quality of the image obtained. The variables analyzed were: concentration of picral (CP) and sodium metabisulfite (CSM) solution, pre-attack (PA), drying mode (DM) and attack time (AT), in addition to the relative humidity and temperature of the environment. Therefore, a quality index (QI) was suggested for the images obtained, which, together with the analysis of variance, indicates the best route for microstructural characterization of TRIP800 steel (CP-4.0%; CSM-0.5%; PA-No; DM-Natural; AT-25s; Temperature (°C): 15I—25 and Relative humidity (%): 55I—90). In addition to indicating the variables that most influence the process. The influence of noise factors (temperature and relative humidity) was also proven.

1. Introduction

As a result of the environmental damage caused by the burning of fossil fuels, the automotive industry needs to increasingly reduce the footprint of products and minimize fuel consumption, thus reducing costs and any potential environmental damage. As such, next-generation cars should be lighter, more economical, safer and less polluting [1]. In light of these challenges, Advanced High Strength Steels (AHSS) have emerged since the 1990s. The main characteristic of these materials lies in their microstructure. AHSS have multiphase microstructures and may contain ferrite and different volume fractions of martensite, bainite and/or retained austenite in quantities sufficient to produce unique mechanical properties [2–4]. Thus, in relation to the common steels used, the AHSS allow the reduction of mass and/or thickness in the applications used, as they are much more resistant and even so, ductile [5, 6].

The performance of multiphase steels is closely related to their microconstituents. The characterization and effective control of the microstructure of these materials is therefore crucial, and the chemical attack processes are essential for this to occur. The chemical attack process on metals aims to reveal the microstructure of the material. This is only possible due to differences in chemical composition and crystallographic orientation that the different phases (austenite, ferrite, martensite, bainite, for example) have. Due to these differences, chemical attack can happen in two different ways: selective phase dissolution—where the phases will be corroded at different depths and by chemical deposition—where a thin film is formed, varying the thickness according to the substrate (phase) in which it is. In the first case, the attack is achieved with the aid of acidic solutions, such as a solution of nitric, hydrochloric and/or picric acid. In the second, salt solutions are used, mainly aqueous solutions of sodium or potassium metabisulfite and/or sodium thiosulfate. This process deserves attention because AHSS have a complex microstructure and knowledge of the composition and volumetric fractions of the present phases are important to understand how superior mechanical properties of these materials, such as...
the investigation of retained austenite-vital phase to gain mechanical properties in AHSS steels. The knowledge of its morphology, chemical composition and distribution are of great importance to evaluate the microstructure and properties of a multiphase steel [7]. In view of this, one of the reagents used to characterize the microstructure of these steels is the LePera reagent (1: 1%–4% pikral + 1% Na₂S₂O₅, in aqueous solution) [8]. Its advantage over other techniques is that the use of LePera reagent makes it possible to separate the phases present in the steel microstructure in different colors: from the matrix ferrite (blue), bainite (brown) and the constituents martensite + retained austenite (white), enabling the morphological characterization, distribution and volume fraction of these phases [9–11].

However, our current understanding of the mechanisms of the attack processes for each material is not precise enough to predict the compositions, times and appropriate conditions. As such, the various attacks with the reagents described in the literature have been developed through a method of trial and error, as described by [12–16], and widely employed by various researchers over the years, such as [2, 17–28], among others.

The reproducibility problem of the chemical attack process arises from the large number of variables/parameters involved, such as the material to be characterized, the mode of attack, the drying method, the time of exposure of the sample to the reagent, whether or not a pre-attack was performed, the concentration of reagents, relative humidity, temperature, among other factors [12, 14, 15, 17, 20, 22, 23]. These will significantly affect the outcome of this process, which makes it necessary to analyze the effects of these same factors on the metallographic characterization of the material under study [29–33]. As mentioned above, relative humidity and temperature have a great influence on the microstructural characterization process of steels, as this technique uses a controlled corrosion process driven by differences in electrochemical potential between surface areas with chemical or physical heterogeneities (phases of the microstructure). The reagents used for these provide an electrolytic action that results in different reduction-oxidation rates for different phases of the sample surface. In general, in corrosion processes, the increase in temperature and relative humidity causes an increase in the corrosion rate [34, 35], directly affecting, for example, chemical attack times. With a higher corrosion rate, a shorter chemical attack time would be needed to achieve the same degree of chemical attack.

In such situations—in which a large number of variables (factors) are involved—the use of Design of Experiments (DOE) techniques would make the study of these factors and their possible interactions broader and more efficient, in addition to reducing the number of experiments to be carried out. Among these techniques, the Taguchi method is widely employed in various industrial fields (for example, Chemical, Electrical, Mechanical, Human Performance, Software Testing, Quality Engineering and etc. described by [36] and it provides a simple, efficient and systematic approach to optimize the designs in relation to performance, quality and cost [37].

The Taguchi method serves as the foundation for a robust design based on three procedures: the orthogonal array—a set of tables containing information on how to determine the lowest possible number of experiments and their conditions for studying a process. The Signal-to-Noise (SN) ratio (equation (1)) [36]—the sensitivity to the variability in performance of the product/process. The loss function (equation (2)) [36]—a mathematical formula proposed by Taguchi to quantify—monetarily—the savings arising from the application of the planning of experiments [38–40].

\[
SN \text{ ratio} = \frac{\text{power of signal}}{\text{power of noise}} = \frac{(\text{sensitivity})^2}{(\text{variability})^2}
\]

\[
L = k(y - m)^2
\]

where \(L\) is the loss in dollars when the quality characteristic is equal to \(y\), \(y\) the value of the quality characteristic (i.e., length, width, concentration, surface finish, flatness, etc.), \(m\) the target value of \(y\), and \(k\) a constant for a given quality characteristic.
In recent years, the number of works using this technique in metallurgy for the optimization of processes and development of alloys has increased, including in studies with AHSS [40–45]. In this context, this study seeks to employ the Taguchi method to evaluate the chemical attack process with the reagent LePera in order to investigate the influence of different process variables, in addition to the interactions between them, on the quality of the image obtained after the chemical attack and the characterization of a TRIP steel.

2. Materials and methods

2.1. Materials
In this work, steels with multi-constituted structure TRIP800 for the automotive industry were used. The chemical composition of these materials is shown in table 1 and their mechanical properties in table 2. TRIP steels can be produced directly from hot strip rolling, in which the slow cooling of the coil ensures the carbon enrichment of the remaining austenite, or cold rolled and annealed [30–33, 46], as shown in figure 1.

2.2. Design of experiments-definition of the orthogonal array and linear graph
A planning of experiments was carried out in order to select the factors (controllable and non-controllable) of the processes and response variables as well as to design the experimental planning, execute the experiments and analyze the data.

The definition of the sources of variation of the processes (factors) under analysis was based on various literature references [12, 14, 15, 17, 20, 22, 23, 29, 47–60] and can be found in table 3 (inner array + interactions) and 4 (outer array). In addition to the factors described in table 3-Inner array—a study of the interactions between these factors was also performed. In table 3 (inner array) the controllable factors were chosen, those in which the levels can be adjusted by the experimenter during the conduct of the experiments. In table 4 (outer array), the uncontrollable factors or ‘noise’ are the factors that cause natural and uncontrollable variations, influencing the response variable. The factors relative humidity and temperature were considered noise because they suffer natural variations and, in many cases, are not subject to control.

The determination of the values related to the levels of the factor ‘Attack time’ was based on previous research [61], literature references, and—mainly—on previous experiments.

Based on the number of controllable factors, their levels and the interactions between them, the standard orthogonal array called L₁₆ was selected for the study of the controllable factors and L₄ for the analysis of the non-controllable factors (temperature and humidity). Since interactions between the factors were foreseen, linear graphs corresponding to these orthogonal arrays were also selected, figure 2.

The ratio SN represents the ratio of sensitivity to variability of product/process performance [39]. In order to minimize the variability of the process, the SN ratio should be maximized.

2.3. Preparation of the metallographic samples and execution of experiments
The techniques used to prepare the metallographic samples followed the processes standardized by the ASTM E 3–11 (2017) [62]. First, samples were taken from the longitudinal section parallel to the rolling direction of the TRIP800 steel sheets. Afterwards, the samples were subjected to hot embedding using phenolic resin (bakelite) for embedding metallographic samples. Water sandpaper with different grain sizes were used for the sanding process: #100, 220, 320, 400, 600, 1000 and 1200 mesh, successively, in a manual sander. Manual polishing was performed at 150 rpm rotation. OP-NAP polishing cloth, STRUERS fabrication and colloidal silica suspension for ferrous materials (Colloidal Silica Suspension, Non-Crystallizing-0.05 μm), ALLIED fabrication and distilled water were used. At this stage, the cleaning of the samples was done by lightly rubbing cotton on the surface of the sample under running water and drying with forced hot air. The attacks were carried out in accordance with the ASTM E407–07 (2015) standard [63]. The experiments were carried out in a metallography laboratory with a controlled environment. Thus, it was possible to control the temperature and humidity according to the experiment to be carried out, using a room conditioner. Through a temperature and relative humidity meter, the values of these variables were monitored and, if necessary, adjusted with the air conditioner in the laboratory.

The experiments were conducted randomly so as to reduce systematic errors. The trials were performed in quadruplicate in order to be able to estimate the experimental error and decrease the margin of error. The samples were photographed after the completion of each experiment. 15 images were captured randomly over the entire surface of the samples for the attacks with the LePera reagent. The images were photographed in bright field, using a NIKON MODELO EPIPHOT 200 optical microscope, coupled to a PC and an AXIO CAM 1CC3 ZEISS digital camera, using the AXIO VISIO-ZEISS software.
Table 1. Chemical composition of TRIP 800 steel (%wt).

|   | C  | Si | Mn | P  | S  | Cr | Ni | Al | Ti | V  |
|---|----|----|----|----|----|----|----|----|----|----|
| 0.205 | 0.870 | 0.280 | 0.027 | 0.003 | 0.015 | <0.001 | 0.040 | <0.001 | <0.001 |

Table 2. Mechanical properties of TRIP 800 steel measured in transverse (T), longitudinal (L) and 45° directions in relation to the rolling direction. \( \sigma_R \) is the ultimate tensile strength, \( \sigma_E \) the yield strength, \( \varepsilon \) the elongation, \( r \) anisotropy coefficient and \( n \) hardening coefficient.

| Material | \( \sigma_R \) (MPa) | \( \sigma_E \) (MPa) | \( \varepsilon \) (%) | \( r \) | \( n \) |
|----------|-----------------|-----------------|-----------------|--------|--------|
| TRIP 800 T | 855.0 | 538.1 | 23.4 | 1.00 | 0.18 |
| TRIP 800 L | 854.2 | 548.2 | 23.1 | 1.09 | 0.19 |
| TRIP 800 45° | 849.4 | 539.0 | 19.1 | 1.01 | 0.19 |

Table 3. Factors, levels of the inner orthogonal array and interactions between the factors.

| Factors | Level 1 | Level 2 |
|---------|---------|---------|
| A: Concentration of picral solution | 2% | 4% |
| B: Concentration of sodium metabisulphite solution | 0.50% | 1% |
| D: Pre-attack | No | Nital |
| H: Drying mode | Natural | Forced cold air |
| O: Attack time | 15 s | 25 s |

Interactions

| C: Concentration of picral solution versus Concentration of sodium metabisulphite solution |
| E: Concentration of picral solution versus Pre-attack |
| F: Concentration of sodium metabisulphite solution versus Pre-attack |
| G: Drying mode versus Attack time |
| I: Concentration of picral solution versus Drying mode |
| J: Concentration of sodium metabisulphite solution versus Drying mode |
| K: Pre-attack versus Attack time |
| L: Pre-attack versus Drying mode |
| M: Concentration of sodium metabisulphite solution versus Attack time |
| N: Concentration of picral solution versus Attack time |
2.4. Development and definition of the ‘quality index-QI’

In order to evaluate the images obtained in each experiment, an experimental scale was proposed and developed to classify the obtained photomicrographs, since the results of the attacks with colored reagents don’t always result in images where any phase/microconstituent can be distinguished. Four researchers in the field of Microstructural Characterization analyzed all the obtained images and assigned a score for each one of the experiments, which was called their QI (Quality Index). The researchers (authors of the article plus one researcher) compared the images of microstructures attacked with LePera reagent and classified them according to the classical references \[12, 14, 15, 17, 20, 22, 23, 29, 47–60\], that is, a qualitative assessment was carried out. Also, the ImageJ software was used to verify the possibility of phase quantification - percentage of ferrite, bainite and retained austenite + martensite. The images that enabled the complete characterization (phase count) received the highest quality index (5). As the quality of the images dropped-making it impossible to count all phases until the distinction of some phases (index 1)- the quality index would also drop, characterizing a quantitative assessment.

The QI values were divided into classes (1 to 5). The criterion for the development of this score was the degree of chemical attack of the samples under study, both for corrosive attack and chemical deposition. After classes were assigned to each performed experiment by each one of the four researchers, the average QI values were calculated and each experiment was classified this way. These categories were used as a parameter to perform the quantitative study of the effects of the factors involved in the process with the aid of the Taguchi techniques.

2.5. Performed statistical analyses

In addition to the developed quality index, the analysis of variance (ANOVA) technique was used to distinguish the relative influence of the factors on the variation of the results using the F statistic (ratio between the variance of the factor and the variance of the error) to measure the significance of the factor under analysis in relation to the variance of all factors included in the error term. The critical values of F were removed from the tables presented by \[64\]. A significance level of 5% was employed. In addition to the ANOVA, the contribution percentage of each factor was calculated.

3. Results and discussion

3.1. Quality index

Chart 1 summarizes the proposed QI scale and the criteria used to classify the images obtained in the performed experiments.

3.2. Statistical analyses

The mean effect of the factor, which corresponds to the mean response of the factor in each level of the experiment, was estimated with the STATISTICA software. The results related to the TRIP800 steel are shown in figure 3.

*The brown coloration in regions in which it is supposed to be ferritic matrix, may indicate excessive time of attack which resulted in additional layer deposition of reagent film or excessive erosion of phases due to corrosive attack. That is, this color is a consequence of the thickness of the film formed on the phase or of localized corrosion (erosion) of the ferritic matrix. Still, you can have a combination of factors.

Analysis of the SN ratio was also performed and the result is shown in figure 3. The results shown in the figure were obtained with the signal-to-noise ratio (SN) ‘bigger is better’. The highest SN ratio corresponds to the lowest variance of the response variable around the desired value. As can be observed in figure 4, the effect of the interaction between the concentration factors of the picral solution and attack mode in relation to the SN ratio is considered significant for the process and the parameters: concentration of the picral solution and drying mode also have a high probability of having a significant effect for the response variable. It should be noted that the
### Chart 1. Description of the quality indices used in the quantitative study of the effects of the factors on the chemical attack process with the reagent LePera.

| Quality Index | Micrograph | Description |
|---------------|------------|-------------|
| 5             | ![Micrograph](image) | Has well-defined contours of the grains and different shades that characterize the distinct phases present in these materials (blue: ferrite matrix, brown: bainite, white/clear: retained austenite + martensite). |
| 4             | ![Micrograph](image) | The brown color can be found in regions where the ferritic structure is supposed to be, which may indicate an excessive attack time resulting in the deposition of an additional layer of reagent film, or an excessive erosion of phases due to the corrosive attack*. |
| 3             | ![Micrograph](image) | Different shades can be distinguished, including shades of blue, indicating the occurrence of selective deposition of the LePera reagent. However, it is impossible to distinguish the contours of grains, nor to clearly identify the different phases present in the material. |
| 2             | ![Micrograph](image) | Presents characteristics of an excessively long corrosive attack. There are no indications of regions where a layer of reagent film was deposited, only an occasional occurrence suggesting this deposition (shades of blue). |
| 1             | ![Micrograph](image) | Insufficient corrosive attack which allows only for distinguishing the contours of grains, the phases present, or evidence of film deposition (shades of blue). |

*Chart 1. Description of the quality indices used in the quantitative study of the effects of the factors on the chemical attack process with the reagent LePera.*

### Figure 3. Graph of the effects and interactions of the controllable factors on the average quality index (QI) - Inner Array. (A): Concentration of picral; (B): Concentration of sodium metabisulphite solution; (C): Interaction between A and B; (D): Pre-attack; (E): Interaction between A and D; (F): Interaction between B and D; (G): Interaction between H and O; (H): Drying mode; (I): Interaction between A and H; (J): Interaction between B and H; (K): Interaction between D and O; (L): Interaction between D and H; (M): Interaction between B and O; (N): Interaction between A and O; (O): Attack time.
same variables that have significant influence on the process are the same ones whose variability must be controlled in order to minimize the effects on the response variable.

Figure 4 reveals that the picral concentration (A) and drying mode (I) factors stand out, with the factor concentration of the picral solution having the greatest impact on the response variable of the process. With a concentration of 2%, the average QI obtained was 1.74. When a concentration of 4% was used, however, the index rose to 2.26. The impact that the change in the concentration of this factor causes can be observed in figure 5.

The factors that differentiate the experiments represented in the images of figure 5 are the concentration of the picral solution and the attack time. Since the attack time factor and the effect of the interaction of this factor with the other factors are not significant, as can be seen in figure 3, it is assumed that the difference between the images obtained is due to the picral factor.

The difference in depth and, consequently, in the tonality of the sample in figure 5(a) shows that a corrosive chemical attack occurred. However, as can be seen, this was not enough to reveal the phases of the material. There is some indication of the formation of reagent film on the surface of the sample [15], as it is known, the formation of the film on the surface of the sample, indicating that the metal was attacked, depends on the reaction product is not the same as the metal surface. If this chemical system is applied to microscopic dimensions it becomes possible to contrast different microstructural components by their deposits and the produced color interference. The effect of the deposition etching coloration is essentially dependent on the thickness of the film formed, for example, blue (about 70 nm) and brown (greater than 70 nm) [11, 13]. In figure 5(b), on the other hand, different shades of blue and brown can be distinguished. It is not possible, however, to clearly make out the lighter shades (white), which would characterize the presence of the constituent martensite + retained austenite in the material.
Another factor whose variation also proved to be important for the improvement of the process was the drying mode. When the natural drying mode was used, the QI was 2.21, and when the sample was dried with forced air this index dropped to 1.79, figure 6.

Since the attack time was a factor that showed little variation in the response variable (change in QI from 1.98 to 2.02), it is once again assumed that the drying mode provided the difference between the images presented in figure 6. The outline of grains can be distinguished in the sample dried with forced air, figure 6(a), which is the result of a corrosive attack on the surface of the sample, probably as a result of the pre-attack, there was no formation of reagent film. The naturally dried sample of figure 6(b), on the other hand, has the characteristics of a super attacked sample, there is evidence of film formation on the surface of the material (regions that have a blue hue).

The other factors—concentration of the sodium metabisulfite solution, pre-attack and attack time—showed no pronounced variation. When the factor sodium metabisulfite was changed, the QI varied from 1.89 (concentration of 1%) to 2.12 (concentration of 0.5%). The performance of chemical attacks without a prior attack of the samples with nital resulted in a QI of 2.03, and when such a pre-attack was performed, the QI of the images was 1.97. The attack time was the factor that resulted in the lowest variation of the response variable, with a QI of 1.98 (at the low level of this factor—15s) and of 2.02 (at the high level of this factor—25s).

It is clear that the input variables of the process are not the only parameters to be considered to obtain images that allow for the characterization of the material, since the quality index never reaches its maximum value, regardless of the level of the factors that is employed, as shown in figure 3. This shows that to obtain photomicrographs with the ideal quality index (QI 5) or even for images considered close to the ideal (QI 4), as shown in Chart 2, it is necessary to consider not only the action of isolated factors, but also the combination that may occur between them.

As such, the effect of the interaction between the picral concentration and the pre-attack should also be considered. To confirm or reject these possibilities, tools were applied to perform an analysis of variance (ANOVA), table 5, which is useful to determine the influence of input parameters on the experimental results and enables the interpretation of the data.

| Experiment | A   | B   | D       | H      | O     | QI |
|------------|-----|-----|---------|--------|-------|----|
| 9          | 4.0%| 0.5%| No      | Natural| 25    | 4  |
| 11         | 4.0%| 0.5%| Nital2%/t = 2s| Natural| 15    | 4  |
| 12         | 4.0%| 0.5%| Nital2%/t = 2s| Forcéd air| 25    | 5  |
| 15         | 4.0%| 1.0%| Nital2%/t = 2s| Natural| 25    | 5  |

The analysis of variance for the QI values, therefore, reveals with 95% confidence that the following factors influence the performance of the process: the concentration of the picral solution and the effect of the interaction between the picral concentration and the drying mode. At the same time, the drying mode seems to have a highly significant effect. Through the statistical F test it is not possible to conclude with certainty about the main effects of the other variables. This occurs because the results show that there is a probability (p value close to the significance level) of disregarding the main effects of these factors as significant in the process.

Besides the important role of the interaction between factors, the presence of non-controllable factors (noise) may or may not have a significant influence on the response variable. As discussed so far, if there were no interactions among the factors in the process and non-controllable factors (noise), then the best combination would be:

- concentration of picral solution: level (2), i.e. 4%
- concentration of sodium metabisulfite solution: level (1), i.e. 0.5%
- pre-attack: level (1), i.e., no pre-attack;
drying mode: level (1), i.e., natural mode;
attack time: mode (2), i.e. 25s.

The equivalent combination (1, 2, 4, 8, 15) can be found in experiment 9. However, the QI values for this experiment were 1, 3 and 4, as illustrated in figure 7.

It should be noted that noise temperature and relative humidity are the only factors that differ in the experiments shown in figure 7. This justifies their choice as the non-controllable factors to be analyzed. As such, a statistical study of the cited parameters was performed and the results are shown in table 6 and figure 8.

Table 5. TRIP800 steel: ANOVA (in relation to the QI value averages) of the controllable factors and percentage of contribution.

| Factors | SS  | DF  | QM  | F   | p    | [%]  | % contrib |
|---------|-----|-----|-----|-----|------|------|-----------|
| A       | 4.25| 1   | 4.25| 5.21| 0.03 | 97.31| 20.95     |
| B       | 0.82| 1   | 0.82| 1.01| 0.32 | 67.93| 5.73      |
| C       | 0.27| 1   | 0.27| 0.33| 0.57 | 42.92| 2.99      |
| D       | 0.05| 1   | 0.05| 0.06| 0.81 | 19.03| 2.25      |
| E       | 1.45| 1   | 1.45| 1.77| 0.19 | 81.09| 6.89      |
| F       | 0.02| 1   | 0.02| 0.02| 0.88 | 12.31| 0.26      |
| G       | 0.19| 1   | 0.19| 0.23| 0.63 | 36.97| 1.61      |
| H       | 2.90| 1   | 2.90| 3.55| 0.06 | 93.46| 19.66     |
| I       | 3.88| 1   | 3.88| 4.75| 0.03 | 96.58| 27.49     |
| J       | 0.00| 1   | 0.00| 0.00| 0.97 | 2.75 | 0.11      |
| K       | 0.01| 1   | 0.01| 0.01| 0.93 | 6.86 | 0.57      |
| L       | 0.94| 1   | 0.94| 1.15| 0.29 | 71.12| 5.67      |
| M       | 0.01| 1   | 0.01| 0.01| 0.93 | 6.86 | 0.03      |
| N       | 0.07| 1   | 0.07| 0.09| 0.77 | 23.00| 0.08      |
| O       | 0.02| 1   | 0.02| 0.03| 0.86 | 13.66| 0.03      |
| Error   | 39.16| 48  | 0.82|      |      | 97.69|          |
According to the obtained results, one can see that the relative humidity factor has a significant percentage of contribution in the outcome of the process (55.42%), and the best results are obtained when this variable is at level 2 or high (relative humidity: 55 – 90%). The contribution of the temperature was not as relevant as the humidity (23.60%), with the best results being obtained when it was at a low level (15 – 25°C).

When comparing the results of table 4 with figure 3 and table 5, one can see that the four experiments with a QI equal to 4 and 5 (Chart 2) were attacked with picral at level (2), which demonstrates the impact of this
parameter on the response variable. Three of these tests were performed with the factors: drying mode at level (1), temperature at level (1) and relative humidity at level (2).

When analyzed in isolation, the attack time proves to cause negligible change to the response variable, since its contribution percentage is virtually nil.

The contribution of the factors under analysis (concentration of the picral solution, drying mode, and interaction between these two factors), stand out in relation to the others, as expected. When associated, the factors may change the QI of the obtained image despite their low contribution percentage. The factor concentration of the sodium metabisulfite solution had an unexpected result with a contribution percentage of 5.73% for the maximization of the response variable, and the best results were recorded when this factor was used at level (1), i.e., a concentration of 0.5%, as can be seen in figure 3 and based on the images of Chart 2.

When the attacks made on the samples listed in Chart 2 are compared, one can see that the concentration of the picral solution, the attack time and the pre-attack factor were employed at the same level in both trials (12 and 15). Both differ regarding the concentration of the sodium metabisulfite solution and the drying mode. The sample resulting from experiment 15 has a better contrast between the phases, possibly due to the drying mode used.

According to the results, statistically the best combination of parameters to achieve a good chemical attack is using the following levels of factors: concentration of picral solution: 4%. Concentration of sodium metabisulfite solution: 0,5%. Pre-attack: No. Drying mode: Natural. Attack time: 25 s. Temperature (°C): 15I—25 and Relative humidity (%): 55I—90. Using these factors at their respective levels, a chemical attack was performed and an image of the TRIP800 steel, figure 9, was obtained with good quality to differentiate the microstructures and quantify them. Micrographs of the TRIP800 steel microstructure were also obtained with the aid of the scanning electron microscopy technique. Figure 10 shows this microstructure where it is possible to differentiate the ferritic matrix (α) from the bainitic structure (αB) and from the retained austenite (γr).

4. Conclusions

The use of the Quality Index (QI) for the qualitative data analysis was efficient and effective. Among the variables under study, the concentration of the picral solution and the interaction between the concentration of the picral solution and the drying mode are the variables with more than 95% of probability of influencing the performance of the process. The drying mode variable was not statistically significant according to the ANOVA, but it had a contribution percentage of 19.66%.

Due to the sensitivity of the chemical attack process with the LePera reagent, even a variable with a low contribution percentage (10% or less) may affect the process variable when its level varies.

Although the noise factors (temperature and relative humidity) did not have a greater than 95% probability of influencing the performance of the process, these variables were shown to have a significant influence on the chemical attack process using the LePera reagent.

Best combination of parameters to achieve a good chemical attack on TRIP800: concentration of picral solution: 4%. Concentration of sodium metabisulfite solution: 0,5%. Pre-attack: No. Drying mode: Natural. Attack time: 25 s. Temperature (°C): 15I—25 and Relative humidity (%): 55I—90.
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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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