# Application of Response Surface Methodology in the Analysis of Weldability Test Results on X80 Steel

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Abstract: Engineering research is bases on experiments that give answers how different influential process factors can impact an outcome. Therefore, the research plot has to be designed in order to capture a valid analysis of the results. Many research project use research methodologies as a help to reach the answers in a technical point of view and to determinate the laws of the controlled input variables on the experimental outcome. This paper will show a realistic usage of the response surface methodology with an CCD experimental plan for 2 influence factors on a welding example using FCAW welding process in order to investigate the hydrogen input and the critical implant stress upon changing the heat input and preheat temperature and to define the weldability of API 5L X80 steel welds.

Keywords: central composite design; diffused hydrogen content; heat input; implant test; preheating; response surface methodology; Weldability X80 steel; welding

#### 1 INTRODUCTION

Experimental tests are conducted in order to determine the laws, whereby certain input variables are intentionally modified to determine the output result of the change and draw valid, systematic, and objective conclusions. In order to reach a valid conclusion, it is necessary to conduct valid research, a valid experiment, and valid analysis of the test results, which is achieved by applying the appropriate methodology.

The methodology of scientific research and the technology of scientific and development research represent the intellectual logistics of modern educational and scientific industries [1]. In this paper, the methodology of the scientific research paper will be approached from a technical point of view, which includes data collection by conducting a standardized experiment and analysis of the obtained results varied in the experiment as independent influencing variables.



Figure 1 The general model of the processing system [2]

In the engineering profession, research is based on experimental testing of great importance for the field of product development, production improvement, and production technologies. The general model of the processing system is based on input data that change within the process due to the action of controlled and uncontrolled variables and output data, as shown in Fig. 1.

For a meaningful design of experimental research, it is necessary to define the relationship between the observed phenomenon and influential factors through the determination of the strategy of the experimental paper.

The scientific approach to the research paper requires the use of appropriate models of the experimental plan in order to be able to determine as precisely as possible the regularity that appears in the observed model and which excludes the occurrence of noise or error. Since financial resources must be planned for each new scientific research paper, a scientific research paper is in most cases a large risky investment. Well-developed plans and models of experimental research in a scientific research paper can also achieve great savings.

#### 1.1 Influence of Hydrogen Content on the Weldability of Steal

In this paper, the weldability of API 5L X80 steel was tested with respect to the occurrence of diffuse hydrogen according to the following influential parameters: preheating temperature and heat input.

API 5L X80 steel mainly finds application in pipeline construction. According to literature, statistics showed that API 5L X80 steel has been used in global pipelines exceeding 300 000.00 km because modern TMCP technology makes it possible to produce an excellent combination of strength, toughness and weldability combining the proper alloying [3]. The pipeline for transporting, for example, the gaseous medium is installed in the ground, whereby during the service life the pipeline may be exposed to hydrogen absorption from localized corrosion mechanisms and as a by-product of cathodic protection. Although API 5L X80 steels are characterized by good toughness in the event that a larger amount of hydrogen is absorbed under certain conditions, there may be a loss of toughness and hardening of the material. In this case, hydrogen fragility occurs. The

literature indicates that during the service life of the pipeline was observed a sixfold increase in the hydrogen content on the outer walls of the pipeline in the first two years of operation, while tenfold higher amounts of hydrogen were estimated for 15 years of operation [4]. Namely, one of the common phenomena that can occur as a manufacturing error with welding technology is the appearance of cold cracks. Cold cracks occur at temperatures below 300 °C and can appear immediately, after several hours or even days after welding. Cold cracks correlate with material microstructure, material stress, and hydrogen content.

During the service life, the appearance of cold cracks on a welded pipeline or other welded steel structure could result in great material damage and damage dangerous to man and his environment.

Numerous literature sources mention different methods of preventing such material degradation or controlling the conditions in which cold cracks can occur [4-7].

The biggest obstacle is the lack of a universally applicable model for determining the amount of hydrogen sufficient to initiate a cold crack and that this model applies to all steels. Namely, all phenomena that occur as a consequence of the action of hydrogen on the degradation of materials are confirmed or not confirmed by the so far proposed theories on the mechanisms of action of hydrogen.

This paper presents one method determined through a scientific research paper with which the amount of hydrogen at which cold cracks can occur can be determined. In this paper, the tests were performed on steel supplied in the form of a plate produced by a Thermo Mechanical Controlled Process (TMCP).

The welding technology used was Flux Cored Arc Welding (FCAW). During welding, two flux-filled wires were used, which differ in flux content and composition. Both types of flux can be found in practical applications. The composition of the flux in the wires was of rutile and basic character.

The centrally composite experimental plan was used as the selected experimental plan. Therefore, for each wire, 14 experiment states were determined, of which 6 repetitions were in the centre.

The following were chosen as influential independent variables: heat input (*E*, kJ/cm) and preheating temperature ( $T_p$ , °C). Since these two variables impact the amount of diffused hydrogen, the goal of this paper was to show how the amount of diffused hydrogen is generated in the base material trough this chosen welding procedure.

For the purposes of the research, test tubes for measuring the amount of diffused hydrogen and for the Implant test were made.

The obtained results were subjected to detailed analysis and statistical processing that determined the prediction of the action of individual independent variables, and a mathematical model for predicting the amount of diffused hydrogen and critical Implant stress was developed.

Based on all experimental data, the critical amount of diffused hydrogen expressed in ml H2/100 g of welds was determined, as well as the optimal welding parameters in

which the probability of cold cracks as a result of hydrogen action is lower.

# 2 DESIGN OF THE EXPERIMENT AND THE EXPERIMENTAL PLAN

For successful process optimization, it is necessary to define the relationship between the observed phenomenon and the influencing factors. The starting point of the experiment in this paper was the choice of approach in defining the experimental plan, with the aim of controlling the influencing factors in the researched process. Since it is a nonlinear system, the statistical method of the response surface was used.

Response Surface Methodology (RSM) is a set of mathematical and statistical methods that model and analyze the effects of independent variables on the observed response [6]. Once the lawfulness or relationship of independent variables is established through a mathematical form i.e. response function, such phenomenon description form can serve for drawing concrete conclusions about the nature of the phenomenon and be a good basis for optimization by known optimization methods [8].

In this paper, the hydrogen input response and critical implant stress were observed, and the independent influencing variables were heat input and temperature. This type of experiment design is most commonly used in the technical sciences. The basic idea of the response surface methodology is to obtain the relationship of influential (independent) factors to the dependent variable (response) through the response function. In this case, the RSM prerequisite is also satisfied because there are two independent variables ( $x_1$  and  $x_2$ ) and response (y). In this paper are analyzed two responses that are based on two independent variables ( $y_1$ ;  $y_2$ ). The process result is formulated according to the following calculation:

$$y = f(x_1, x_2) + \varepsilon \tag{1}$$

where  $\varepsilon$  is the error or noise that occurs in the response *y*.

For this case, the set equation is:

$$H_{\rm D}, R_{\rm ik} = f(E, T_{\rm p}) - \varepsilon \tag{2}$$

Where is:  $H_D$  – amount of diffuse hydrogen, ppm;  $R_{ik}$  – critical implant stress, MPa; E – heat input, kJ/cm;  $T_p$  – preheating temperature.

Considering the initial settings, a centrally composite experimental plan was selected, which also belongs to the class of experimental plans that most often appear in the response surface methodology.

The observed values were determined at three levels, at three different heat inputs, and at three different preheating temperatures, the concept of a centrally composite experimental plan sets a total of five preheating temperature levels and five heat input levels, as shown in Fig. 2.

Fig. 2 shows the experiment states of the centrally composite experimental plan, with the largest number of

repetitions in the centre. A total of 6 repetitions were performed in the centre. This test plan was used for both welding wires, rutile and basic, and for testing the hydrogen input with a glycerol method, and for testing the X80 steel's susceptibility to cold cracks by the Implant test. All points, except the centre, were performed with only one measurement.



Figure 2 Display of welding parameters in accordance with the experimental conditions and CCD experimental plan for 2 factors [7]

 
 Table 1 Display of experiment state and corresponding welding parameters by experiment stages for basic and rutile wire

Experiment state		Heat Input,	Preheat	
Rutile wire	Basic wire	kJ/cm	temperature, °C	
RUT1	BAS1	12	20	
RUT 2	BAS 2	8	45	
RUT 3	BAS 3	12	100	
RUT 4	BAS 4	12	100	
RUT 5	BAS 5	12	100	
RUT 6	BAS 6	12	100	
RUT 7	BAS 7	12	100	
RUT 8	BAS 8	12	100	
RUT 9	BAS 9	16	45	
RUT 10	BAS 10	6	100	
RUT 11	BAS 11	18	100	
RUT 12	BAS 12	8	160	
<b>RUT 13</b>	BAS 13	16	160	
RUT 14	BAS 14	12	180	

The central point provides information on the nonlinearity in the response, while the axial points provide

the possibility of efficient estimation of the second-order parameters.

Fig. 2 shows the independent variables, E and  $T_p$ , heat input, and preheating temperature.

A total of 14 experiment states were performed for both wires used, and for the glycerine measurement method, and the Implant test. Tab. 1 shows the centrally composite experimental plan to determine the experiment state according to which the experimental paper was carried out. The electrode tip was 15 mm, and the welding was conducted with a neutral electrode angle 90°.

Welding parameters such as current type and current polarity, current amount, wire speed, voltage, wire diameter, shielding gas flow, welding speed, electrode inclination, and heat input were determined. Welding parameters are shown in Tab. 2. The wire diameter was 1.2 mm, welding current and polarity was DC+, gas flow was 18 l/min, and the weldment was conducted with a neutral electrode angle 90°.

Heat input is calculated according to the calculation [9]:

$$E = \frac{U \cdot I}{v_z} \cdot \eta \tag{3}$$

Where is: U – welding voltage, V; I – welding current, A;  $v_z$  – welding speed, mm/s;  $\eta$  – degree of utilization of the welding process.

Table 2 Display of welding parameters						
	Welding	Voltago II	Welding	Heat input,		
	current, A	voltage, O	speed, cm/s	kJ/cm		
BASIC WIRE						
1	162	24	30	6.22		
2	164	24	25	7.56		
3	200	26	20	12.48		
4	240	27.5	19	16.67		
5	268	28.5	20	18.33		
RUTILE WIRE						
1	180	23.5	27	6.68		
2	180	23.5	23	8.83		
3	220	24	20	12.67		
4	250	25.5	19	16.11		
5	270	27	20	17.50		

After the experimental part of the research, the obtained data need to be subjected to statistical processing and analysis. For this purpose, a program called Design Expert, version 7.1.4 was used which confirmed that the selected experimental plan is satisfying. As mentioned earlier, the experiment was developed in 14 experiment states, of which were 6 repetitions in the center. The scope of the experiment is large because four tubes for one experiment state were used to measure the amount of diffused hydrogen, and a minimum of five tubes was used to determine the critical Implant stress. Such an approach ensures the reduction of the possibility of error or noise that could occur due to human factors, or some other irregularities caused by the environment. Each test was performed under controlled laboratory conditions with monitoring of ambient temperature and humidity so that excessive deviations would not affect the results.

After entering the measured values according to the selected concept of the experimental plan, analysis of the results in the Design Expert program 7.1.4. is performed in several steps for each response surface. This is followed by an analysis of variance (ANOVA), post-ANOVA analysis of individual coefficients, and statistical data processing of residues and points that are not within the limits. It is necessary to validate the model using various diagnostic tools. If the model is appropriate, graphical analysis is performed.

In this particular case, transformations of experimental data were carried out in order to obtain an overview of the operation of the model, but they were not applied because the inverse observation of the data can give a distorted image.

#### 3 STATISTICAL ANALYSIS AND DEVELOPMENT OF A MATHEMATICAL MODEL

By establishing a centrally composite experiment plan with a total of 14 states, of which are 6 repetitions in the centre, the results used in the development of the mathematical model according to the response values of diffuse hydrogen,  $H_D$ , and critical implant stress  $R_{ik}$  were recorded.

### 3.1 Mathematical Models for Rutile Wire 3.1.1 $H_D$ Diffuse Hydrogen Content for Rutile Wire

The ANOVA analysis gave an equation with the following significant members: A,  $A^2$  and  $B^2$ . The value of F is 23.53, which implies that the model is significant and that there is only 0.01% of the possibility for the insignificance of the model due to error or noise. The value "Prob > F" that is less than 0.05 implies that significant factors are A (0.0242),  $A^2$  (0.0292), and  $B^2$  (<0.0001). The value "Adeq Precision" defines the relationship between noise and signal and it is satisfactory if it is more than 4. In this case, it is 12,576, which indicates that the signal is appropriate. The value of the predicted  $R^2$  is 0.5970, while the value of the adjusted  $R^2$ is 0.8739. The difference is 0.27. The reference amount that would indicate a good match between the data and the model is 0.2, and the value of 0.27 is negligible in this case, so it can be assessed that the match between the data and the model is good. The model diagnostics determined that all data are within the critical test values.

The final calculation for  $H_D$  for rutile-type flux cored arc welding

$$H_{\rm D} = 6.18458 + 5.09862 \times 10^{-3} \cdot T_{\rm p} - 0.61113 \cdot E - -3.46125 \times 10^{-5} \cdot T_{\rm p}^2 - 0.024621 \cdot E^2$$
(4)

Where is:  $H_D$  – amount of diffuse hydrogen, ml H<sub>2</sub>/100 g weld;  $T_p$  – preheating temperature, °C; E – heat input, kJ/cm.

Fig. 3 shows the graphical display of the response surface and a contour display of the isoquants for the diffuse hydrogen content according to the performed experiment states.,



Figure 3 (a) H<sub>D</sub> response surface display for rutile wire; (b) contour display of the isoquants for the diffuse hydrogen content

According to Fig. 3, it is visible that the largest amount of diffused hydrogen was determined in boundary conditions, i.e. the process indicates stability in the center where the smallest values of the amount of diffused hydrogen were measured. Namely, a cold crack was noticed on metallographic samples tested by implant testing, on a sample welded at  $T_p = 45$  °C and E = 16 kJ/cm, and on a sample welded at preheating temperature  $T_p = 20$  °C and E =12 kJ/cm. The cold crack arise through the HAZ to the WM, which can be seen in Fig. 4. Fig. 4 is therefore directly related to the response surface shown in Fig. 3 where it can be seen that the largest amount of diffused hydrogen was found at the lowest preheating temperatures. The literature [11] indicates the possibility of cold cracks if the preheating temperature is lower than 50 °C. Fig. 4b also shows an inclusion in the weld that can attract enough hydrogen and act as hydrogen trap with a potential to develop a hydrogen crack. These inclusions are usually not considered as welding defects, but they can produce cold cracking if critical conditions occur. This particular inclusion shown on Fig. 4b has an uneven surface of approximative  $1120 \,\mu\text{m}^2$  and is made of O, Mg and Si. Because of his uneven surface this inclusion can act as a

hydrogen trap. Ref. [12] proves how micro vacancy can play an impact role on the crack initiation, especially if there are conditions for stress corrosion cracking.





Figure 4 (a) Display of a crack located in the area of weld metal and coarsegrained HAZ; (b) display of a spectrum with inclusion suspectable to hydrogen accumulation and cracking in the weld metal.

#### 3.1.2 Rik Critical Implant Stress for Rutile Wire

The reduction of the model was performed. It was determined that member B (0.0239) is significant, however, the interaction of AB (0.0530) can also be assessed as significant.

The simplified quadratic model's ANOVA value is 4.11, meaning that there is a 3.85% possibility that the model's significance is the result of error or noise.

Significant model components are indicated by the value "Prob > F" being less than 0.0500.

The value "*Adeq Precision*" determinates the signal-tonoise ratio and the result is 7.622 which is more than 4 and proves that there is a corresponding signal.

Model diagnostics do not indicate excessive deviation of results beyond critical values.

The final term for *RIK* for rutile-type flux cored arc welding is:

$$R_{\rm 1K} = 630.93664 - 1.77887 \cdot T_{\rm p} - 8.50624 \cdot E - -0.15985 \cdot T_{\rm p} \cdot E$$
(5)

Where is:  $R_{ik}$  – critical implant strain, MPa;  $T_p$  – preheating temperature, °C; E – heat input, kJ/cm.

Fig. 5 shows the results of the response surface analysis, isoquant display, and optimization  $H_D$  and  $R_{ik}$ .



**Figure 5** (a)  $R_{IK}$  response surface for rutile wire; (b) display of  $R_{IK}$  isoquants for rutile wire; (c) optimization of  $H_D$  for  $H_D = 2.3 - 2.9$  ml H<sub>2</sub>/100 g weld and  $R_{IK} = 557$  MPa.

Fig. 5 shows that the maximum  $R_{ik}$  is the highest at the maximum E and  $T_{p,i}$ , so the optimization of  $H_D$  and  $R_{ik}$  is performed according to the criteria of minimum  $H_D$  and maximum  $R_{ik}$ . Here, also, the phenomenon of more diffuse hydrogen being measured at lower preheating temperatures is visible, and the observed appearance of cold cracks indicates a direct interaction between the amount of hydrogen and the state of the Implant stress.

## 3.2 Mathematical Models for the Basic Wire 3.2.1 $H_D$ Diffuse Hydrogen Content for Base Wire

By analysis of variance was obtained a model with active members A, AB,  $A^2$ ,  $B^2$ ,  $AB^2$ . This model was accepted due to the decrease in the value of  $R^2$ .

The proposed quadratic model's ANOVA results in a F value of 134.87, indicating that it is significant and that there is only a 0.01% chance that it was produced accidentally or by noise.

Value "*Prob>F*" which is less than 0.0500 indicates that A - preheating temperature (<0.0001) is a significant member, while B - heat input is less significant and that its action is seen in combination with member A (preheating temperature). However, due to the retention of the hierarchy of the form of the equation, the action of member B (heat input) whose "*Prob>F*" value is 0.5156 is accepted since based on the literature this member cannot be completely excluded.

Value "*Adeq Precision*" determinates the relationship between noise and corresponding signal and if the mentioned value is greater than 4 then such a state is considered desirable. In this case, the value of "*Adeq Precision*" is 32.457, which indicates the appropriate signal. This model can be used to determine the spatial design.

The predicted  $R^2$  value of 0.8672 is consistent with the adjusted value of  $R^2$  that is 0.9841. Since this value in the amount of 0.1169 is less than 0.2, this indicates a good match between the model and the data.

The final model for  $H_D$  for flux cored arc welding when the electrode is a wire with a basic flux:

$$H_{\rm D} = 3.98784 - 0.036367 \cdot T_{\rm p} - 0.55250 \cdot E +$$
  
+4.6383×10<sup>-3</sup> ·  $T_{\rm p} \cdot E + 3.59294 \times 10^{-5} \cdot T_{\rm p}^{2} +$   
+0.021108 ·  $E^{2} - 1.74195 \times 10^{-4} \cdot T_{\rm p} \cdot E$  (6)

Figs. 6a and 6b show the response surfaces and isoquants for the diffuse hydrogen content.

The Fig. 6 shows that the highest concentrations of diffuse hydrogen are expected at the lowest heat input and the lowest preheating temperature. However, the center is a more relevant landmark because the stability of the process is visible, indicating a rather low content of hydrogen intake. No cold cracks were observed on the samples welded with wire filled with basic character. Although, in any case, after the implementation of the optimization, it would be recommended to carry out preheating at temperatures around 100 °C and at E = 8 to 16 kJ/cm. All measured values of diffuse hydrogen are within the permitted limits, below 5 ml H<sub>2</sub>/100 g weld. However, the literature states that 1 ml of H<sub>2</sub>/100 g of weld is sufficient for the appearance of cold cracks on API 5L X80 grade steels [13, 14].



Figure 6 (a) Display of the response surface for HD when welding with wire filled with basic flux; (b) showing the isoquants of diffuse hydrogen content for a wire filled with a basic flux.

#### 3.2.2 *R*<sub>ik</sub> Critical Implant Stress for Base Wire

The chemical composition of the base wire is accountable for the dehydrogenization of the weld metal as indicated by the low values of the measured amount of diffused hydrogen, especially in the centre of the central composite test plan in which the measured values are 0.2 ml  $H_2/100$  g weld. Phenomena related to the occurrence of cold cracks at such a low hydrogen content have not been described in the literature. The base wire guarantees better mechanical properties and is mainly used to make more demanding steel structures.

No mathematical model was established for this experiment state because the deviations between the measured values were very small and possible deviations could be determined with more sophisticated testing equipment. However, the results presented in this way indicate a good resistance of the material to the occurrence of cold cracks and the possibility of applying the base and additional material in edge and harsher conditions with a satisfactory outcome in members of satisfactory weldability.

#### 4 CONCLUSIONS

It has been determined by the methods of scientific research that the results of measuring the amount of diffused hydrogen by the glycerol method according to JIS Z 3118: 1992 and the performed analysis and developed mathematical models indicate lawfulness. With two distinct experiment variables, the developed centrally composite experimental design (RSM method) generated 14 experiment states with 6 repetitions in the middle.

The presented results show that the centre almost always had the same value, which indicates the existence of physical law and repeatability. The ideal center for working conditions is one with six repetitions, a heat input of 12 kJ/cm, and a preheating temperature of 100 °C. No cold cracks were observed in this area, and this area is described as an area of stable conditions. Mathematical models indicate that heat input has a stronger influence on the hydrogen diffusion behaviour model while the preheating temperature is viewed as a secondary influence quantity. The chemical composition of the additional material, i.e. the chemical composition of the flux in the filled wires, is responsible for the diversity of mathematical models. Namely, elements from the different chemical compositions of the flux form compounds of the size of 1 to 3 µm, which indicate the heterogeneity of the structure and thus the different possible paths of hydrogen diffusion through the material.

The obtained mathematical models correspond to a wider range of welding parameters for the tested steel and additional material. Also, in the case of rutile wire, boundary conditions were determined at which there is an increased possibility of the appearance of cold cracks in the area where the preheating temperature is less than 50 °C, and conditions were determined in which the stability of the process is indicated. With the help of a detailed methodology of scientific research, this research established the dependence of the amount of diffused hydrogen on the welding parameters, heat input, and preheating temperature.

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