

## Assessment Of Mechanical Properties Of Biphased Steel By The Small Punch Test And Design Of Experiment Methods

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### Abstract

*The characterization of biphased steel structure led us to exploit the experimental results of a study which consists in the determination of local properties of different steels investigated [1]. The present work is a comparative study between the empirical relation (CENCWA 15627(part A) ) [2] and the two relations examined by S.D.Norris and J.D.Parker [3], concerning the estimation of the ratio of mechanical strength over maximum local with respect to the geometric properties of the indentation apparatus and the thickness of the specimen. We propose a mathematical model of type  $2^3$  factorial plan to express the ratio using the characteristic variables of the apparatus.*

*Key words: small punch test (SPT), mechanical resistance over maximum load, design of experiment, statistical tools.*

### 1. Introduction

During the small punch test, the material is subjected to a uni-axial stress, plastically deformed, cracked up to rupture. Small punch test (SPT), conducted in five DP steels show the ductile behavior of steels from plots of the exerted force on the specimen with respect to displacement. Design of experiment is a method to ameliorate the quality. The success of the original planning is its possibility to interpret experimental results with a minimum effort in experimentation. The minimization of the number of experiment reduces time and cost. The work performed to find the best correlation between mechanical resistance and maximum load ( $\frac{\sigma_u}{P_{max}}$ ) are conditioned by dimensional standards of indentation set up. This limits the application and exploitation of the computational methods for different experimental set up. The objective of this study is the verification of S.D.Norris and J.D.Parker's method, [3] for the determination of the mechanical ratio ( $\frac{\sigma_u}{P_{max}}$ ) with respect to geometrical parameters of the small punch test apparatus. The specimen used are made of dual phase steel which has a good deal between resistance/drawing. This emanate from the microstructure made of hard phase (martensite or bainite) dispersed in ductile ferritic matrix. The consolidation capability induced by the deformation of these steels is considerable. A statistical analysis of the small punch test has been made using design of experiment with a general linear model.

### 2. Description of the experimental device

The small punch test (SPT) is a method considered in practice to be non-destructive because of the reduced size of the specimen (10\*10mm<sup>2</sup>), and 0.5mm in thickness. The SPT can measure directly the mechanical properties of the materials [4]. The test consists in integrating the entire contour of the specimen which is maintained between two dies (3,4), then after to deform the specimen up to rupture using the punch(2) with spherical lead of 2.5mm in diameter. A extensometer (5) is used to determine the stress-strain plot through the lower die (3) (with 4mm in diameter)of the indentation apparatus as shown in figure 1:

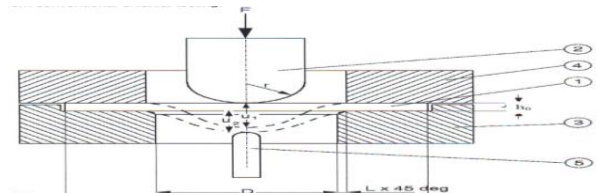


Figure 1. Small punch testing device [2]

### 2.1 Experimental results

Five steels with carbon content (<20%) have been used in the experimental study conducted by [1]. The main mechanical properties of these steels are grouped in table 1.

Table 1. Mechanical properties obtained by SPT [1]

Specimen	P <sub>max</sub> (N)	σ <sub>m</sub> (Mpa)	P <sub>y</sub> (N)	σ <sub>ys</sub> (Mpa)	d <sub>max</sub> (mm)
2	1941,75	700	382,75	460	1,325
3	1955,25	825	401,5	460	1,38
4	1979,75	870	476,25	632	1,365
5	1445,75	990	496	622	0,84
6	1689,25	1101	590,75	874	0,835

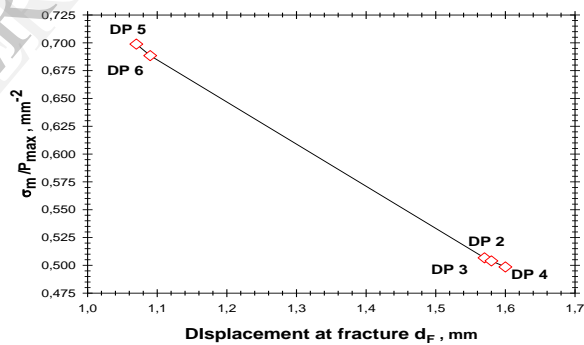


Figure 2. Characteristic curve of indentation tests of the DP steels

The values of the displacement at rupture have been extracted from figure 2. The statistical serie recording the ratio (mechanical resistance σ<sub>u</sub> over max load P<sub>max</sub>) of the five DP steels investigated in [1] are given in table 2.

Table 2. Maximum mechanical ratio ( $\frac{\sigma_u}{P_{max}}$ )

d <sub>F</sub> [mm]	1.57	1.58	1.60	1.07	1.09
σ <sub>m</sub> /P <sub>max</sub> [mm <sup>-2</sup> ]	0.36049	0.42194	0.43945	0.68476	0.65177

### 3. Method of determination of the mechanical ratio

**3.1 Normative method (cen cwa 15627)**

Replacing the geometrical characteristic by their values (R=  $\frac{D}{2}$  = 2mm, p =  $\frac{A}{2}$  = 1.25mm, t = 0.5mm).

In the emperical equation [2]

$$\frac{P_{max}}{\sigma_u} = 3.33.K_{sp}.R^{-0.2}.r^{1.2}.t \tag{3.1}$$

we obtain :  $\frac{P_{max}}{\sigma_u} = 1.891.K_{sp} \text{ (mm}^2\text{)}$  (3.1.1)

This confirm the demonstration of Yang Z and Wang Z.W, [5] using Newton’s polynomial development taken from Chakrabarty’s theory[6].

$$\frac{P_{max}}{\sigma_u} = 1.72476.\Delta - 0.05638. \Delta^2 - 0.17688. \Delta^3 \tag{3.2}$$

Thus  $\frac{P_{max}}{\sigma_u} = 1.891 \text{ mm}^2$  if  $\Delta=1.6 \text{ mm}$  [7].

if  $K_{sp}=1$  (CEN CWA 15627, Hyde and Sun., 2009) then

$$\frac{P_{max}}{\sigma_u} = 1.891$$

giving  $\frac{\sigma_u}{P_{max}} = 0.52882$  (3.3)

**3.2 S.D.Norris and J.D.Parker’s method**

**3.2.1 Nomenclature and equations** Equations (3.4) and (3.5) rad been examined by S.D.Norris and J.D.Parker[3].

$$\sigma_u = \frac{P_{max}}{t(2.22D - 0.9Cl + 0.56)} \tag{3.4}$$

$$\sigma_u = \frac{P_{max}}{t(0.14D - 0.9Cl + 2.17d_F + 0.6)} \tag{3.5}$$

with  $Cl=A-(d+2t)$  (3.6)

$\sigma_u$  : Ultimate strength (Mpa)

$P_{max}$  = Maximum load (N)

Cl : Clearance of the dies

t : thickness of specimen (0.5mm)

D : sphere diameter (indenter) (2.5mm)

A : Lower die diameter (4mm)

$d_F$  : Displacement at rupture (mm)

Considering equations (3.4) and (3.6) we obtain :

$$\frac{\sigma_u}{P_{max}} = \frac{1}{t \cdot (3.22D - 0.9A + 1.8t + 0.56)} \tag{3.7}$$

We obtain a constant ratio, by using the values of dimensions of the experimental set up we obtain :

$$\frac{\sigma_u}{P_{max}} = 0.3384 \tag{3.8}$$

From formula (3.5), we express the ratio ( $\frac{\sigma_u}{P_{max}}$ ) with respect to the displacement at rupture ( $d_F$ ) we obtain :

$$\frac{\sigma_u}{P_{max}} = \frac{1}{0.270 + 1.095d_F}$$

(3.9) Thus we obtain using the second formula the values of the ratio shown in table 3.

**Table 3. Maximum mechanical ratio of DP steels**

Désignation Steel	d <sub>F</sub> [mm]	$\frac{\sigma_u}{P_{MAX}}$ [mm <sup>-2</sup> ]
2	1.57	0.50673
3	1.58	0.50396
4	1.60	0.49850
5	1.07	0.69884
6	1.09	0.68850

**4. Proposed model by design of experiment**

The study of the small punch test apparatus can be schematized as follows :

We are interested in a quantity Y called response which dependent on a certain number of variables X<sub>1</sub>, X<sub>2</sub>, X<sub>3</sub> called factors.

The mathematical modeling consists in measuring the response Y for many valuesof variables X<sub>i</sub> by maintaining the fixed the other two variables. We iterate this method for each of the variables.

The design of experiment method proposes a factorial experimentation [8] i.e all the factors vary simultaneously. The handling of the results is made by multiple linear regression and variance analyses.

**4.1. Choice of the factors and experimental domain**

The factors are supposed to be the parameters that influence the response which characterizes the behavior of the phenomenon under investigation.

It is important to be able to attribute two levels for each factor, a low and a high level. The low level is coded by (-1) and the high level is coded by (+1). Table 4 shows the factors of the model

**Table 4. Designation of the factors**

N	Désignation	Abreviation
1	Constant	Cst
2	Thickness	t
3	Radius of lower die	R
4	Radius of indenter (sphere)	p
5	Thickness * Radius of lower die	t*R
6	Thickness * Radius of indenter	t*p
7	Radius of lower die * Radius of indenter	R*p
8	Radius of lower die * Radius of indenter * Thickness	R*p*t

The plan for which each of the three factors has only two levels is called 2<sup>3</sup> plan. Thus the corresponding table is :

**Table 5. Choice of the level of the factors**

Factors	Units	Type	level
t	mm	Quantitatif	0.20 - 0.50
R	mm	Quantitatif	0.75 - 2.00
p	mm	Quantitatif	0.50 - 1.25

## 4.2 Experimental matrix

The matrix of experiment is a table which indicates the number of experiment to carry out, their order and the way to vary the factors.

**Table 6. matrix of experiment**

Experience	t	R	p	$\frac{\sigma_m}{P_{max}}$
1	0.2	0.75	0.5	3.25
2	0.5	0.75	0.5	1.30
3	0.2	2	0.5	3.96
4	0.5	2	0.5	1.58
5	0.2	0.75	1.25	1.08
6	0.5	0.75	1.25	0.43
7	0.2	2	1.25	1.32
8	0.5	2	1.25	0.53

This table is composed of high (+1) and low (-1) levels. The response Y expresses the ratio of ultimate stress to ultimate load we use an experimental plan of  $2^3$  using a factorial matrix with Yates algorithm [8]. The results are given in table :

**Table 7. Presentation of experimental plan  $2^3$ (annex 1)**

**4.2.1 Estimation of coefficients of the model** The coefficient of the model are obtained using model 6.0. It can be observed that  $a_1, a_2$  and  $a_3$  are well the average effects of factors  $X_1, X_2$  and  $X_3$ . The coefficient  $a_0$  is the theoretical response at the center of the domain of variation of the factors.

The model is written as:

$$Y = a_0 + a_1.X_1 + a_2.X_2 + a_3.X_3 + a_{12}.X_1.X_2 + a_{23}.X_2.X_3 + a_{13}.X_1.X_3 + a_{123}.X_1.X_2.X_3 \quad (4.1)$$

$$\text{Or } Y = 1.682 - 0.721X_1 + 0.164X_2 - 0.842X_3 - 0.070X_1X_2 - 0.082X_2X_3 + 0.360X_1X_3 + 0.036X_1X_2X_3 \quad (4.2)$$

The variables  $X_1, X_2, X_3$  designate the variables t, R, respectively. The model can be expressed as:

$$\frac{\sigma_m}{P_{max}} = 1.682 - 0.721.t + 0.164.R - 0.842.p - 0.070.t.R - 0.082.R.p + 0.360.t.p + 0.036.t.R.p \quad (4.3)$$

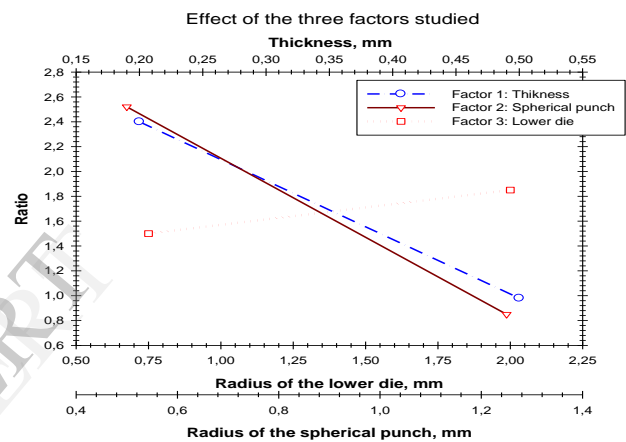
$$\text{or } \frac{\sigma_u}{P_{max}} = \text{fct}(t, R, r, t.R, R.p, t.p, t.R.p)$$

## 4.3 Graphical analysis of the results

Yates method has been used for the calculation of the effects. The originality of this method is that can allow directly to find the general formula e of the average effects [8].

**4.3.1 Analysis of the effect of parameters on the mechanical ratio** A graphical illustration of the results allows easy interpretation of the obtained information.

Figure 3 : shows the effect of each parameter on the mechanical ratio

**Figure 3. Effects of the factors on mechanicals ratio**

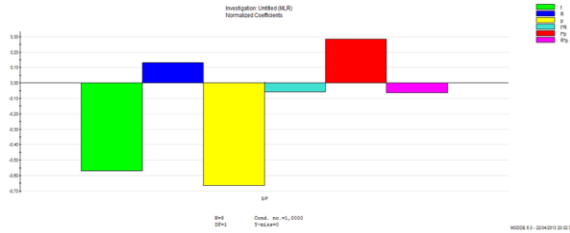
The plots of the average effects of the parameters give immediate observation of in important factors. The spherical indenter has the highest effect on the maximum variation of load indentation, then the effect of the lower die which has low influence on the reponse.

## 4.3.2 Analyse of interaction effects

**Figure 4. Graphical representation of interaction between the three factors (annex 1).**

In each of these graphs, the existence of interaction is detected when the lines are not parallel. In the case under investigation the interactions are relatively low. A low interaction between the indenter and the lower die can be observed: more the size of the spherical indenter is important, more the variation of the thickness allows to increase the load of indentation.

**4.3.3 Pareto's diagram** It is possible to decompose the variation of the response from the contribution of factor in a model.



**Figure 5. Pareto's diagram of the three factor and their contributions**

Pareto's diagram, [9] is a complement of the plot of the average effects. The contribution and the analysis of pareto's diagram for obtaining a maximum mechanical ratio put into evidence the predominance of the role of the factor size of the spherical indenter (p) which appears first with the longest histogram. From the histogram we can notice that the factor indenter (p) and the thickness (t) explain the 70% of the variation of the response. The contribution of the third factor the diameter of lower die is very low and represents only 07.41%.

**4.4 Variance analysis**

The variance analysis (ANOVA) allows to compare the variance of cumulated values of the model with that of residuals. This analysis is a statistical test Gaudoin [10] (Fisher-Snedecor).

**Table 8. ANOVA**

	1	2	3	4	5	6	7
1	<b>Rapport</b>	DF	SS	MS	F	p	SD
2				(variance)			
3	Total	8	33,8051	4,22564			
4	Constant	1	22,6128	22,6128			
5							
6	Total Corrected	7	11,1923	1,5989			1,26448
7	Regression	6	11,1818	1,86363	177,277	0,057	1,36615
8	Residual	1	0,0105125	0,0105125			0,102531
9							
10	Lack of Fit (Model Error)	--	--	--	--	--	--
11	Pure Error (Replicate Error)	--	--	--			
12							
13							
14							
15	N = 8	Q2 = 0,940		Cond. no. = 1,0000			
16	DF = 1	R2 = 0,999		Y-miss = 0			
17		R2 Adj. = 0,993		RSD = 0,1025			

**4.5 Test of the signification of coefficients**

The coefficients of the factors and those of interactions necessitate test of signification. The statistical calculations which allow if the effects are significant, to calculate the confidence intervals or to validate the model linearity make intervene the residuals i.e the difference

between the experimental value and the value predicted by the model, and an estimator of variance of residuals. For the estimation and the significance the effects of coefficients a student's test is used [8].

**Table. 9. Residuals**

	1	2	3	4	5
1	<b>mechanical ratio</b>	Observed	Predicted	Obs - Pred	Conf. int(±)
2	1	3,25	3,28625	-0,0362499	1,21863
3	2	1,3	1,26375	0,0362499	1,21863
4	3	3,96	3,92375	0,0362499	1,21863
5	4	1,58	1,61625	-0,0362499	1,21863
6	5	1,08	1,04375	0,0362504	1,21863
7	6	0,43	0,46625	-0,0362502	1,21863
8	7	1,32	1,35625	-0,0362498	1,21863
9	8	0,53	0,49375	0,0362497	1,21863
10					
11	N = 8	Q2 = 0,940		Cond. no. = 1,0000	
12	DF = 1	R2 = 0,999		Y-miss = 0	
13		R2 Adj. = 0,993		RSD = 0,1025	
14				Conf. lev. = 0,95	

The results and the values of the statistical test with the coefficients are grouped in table10 :

**Table. 10. Ponderation of the effects**

Variable	Effect	Result
Constant	$a_0 = 1.682$	Significant
t	$a_1 = -0.721$	Significant
R	$a_2 = 0.164$	Significant
p	$a_3 = -0.842$	Significant
t.R	$a_{12} = -0.070$	Non Significant
R.p	$a_{23} = -0.082$	Non Significant
t.p	$a_{13} = 0.360$	Significant
$X_1, X_2, X_3$	$a_{123} = 0.036$	Non Significant

This table shows that the variables t, R, p and the interaction (t.p) are significant.

It is clear that the average of the response given by  $a_0$  is the most dominant, which explains that the effect of all the factors is important. It can be observed that the action of indenter (p) is the most significant before the thickness of the specimen (t) and before the diameter of the lower die. Only the interaction shown by (t.p) is significant. The interactions shown by (t.R) and (R.p) which are not significant this means that the effect is not, with the given risk, significantly different from 0. That is the variables associated to  $a_{12}$ , and  $a_{23}$  have no effect on the response. The result of the test recommend to take a model of the form:

$$\frac{\sigma_m}{\sigma_{max}} = 1.682 - 0.721.t + 0.164.R - 0.842.p + 0.360.t.p \quad (4.4)$$

## 4.6 Response surface

These responses are presented the two different ways. The first in space with a curved surface, the second is the projection of the surface on a plan called iso-response, [9]:

**Figure 6. Contour of response of the variation of mechanical ratio (annex 2).**

The first curve represented by the blue zone which is the inferior part of the graph corresponds to a lighter influence of the parameters as shown by the small variation of response which less than  $0.95 \text{ mm}^{-2}$ . Even with greater size of the indenter up to a radius of 1.25 mm, one note that this is due to the mon significant effect of the lower die less than 0.8 mm.

The analysis of the rest of the zones (yellow, orange and red). The whole are under the form of a triangle with a pick representing a critical point to which all points converge. This means that the behavior of the material changes to another state. This can be a plastic deformation or rupture.

One note that this zone corresponds to an important variation of the response with respect to the preceding cases. We reach a maximum ratio of  $2.75 \text{ mm}^{-2}$  which comes from the interaction of two parameters, the increase of the matrix radius up to 2 mm and the decrease of the radius of the punch at least 0.55mm.

We also note that this zone correspond to an important variation reaching a maximum ratio of  $2.49 \text{ mm}^{-2}$  due to the interaction of two parameters increase of matrix radius up to 1.6 mm and the decrease of the specimen thickness at least 0.2 mm.

The analysis of the third curve shows that the simultaneous decrease of two parameters shows that the variation of the response is inversely proportional simultaneous decrease of the thickness and the punch radius can give a maximum increase of the response that reaches  $3.27 \text{ mm}^{-2}$  for 0.25 mm thickness and 0.6 mm punch radius.

It is clear for an increase of the thickness and the punch radius the variation of the response is alternative and instable. It has been noticed that for a constant thickness, the increase of punch radius lowers the response. For a constant punch radius the increase of the thickness increases the response.

The simultaneous increase of these two parameters results into a compensation.

## 5. Results and discussion

**Table. 11. Gives a summary of the obtained results (annex 2)**

- The variance and standard deviation: The couple variance and standard deviation [8] are estimated as (0.017, 0.13) for the first case and (0.011, 0.11) for the third case. Both values of the couples are too close for the two cases. The average deviation of value of the ratio is small and the variance low. Thus the scores are concentrated around the calculated average.

-The coefficient of variation : First case  $X=0.52816, S= 0.13$  and  $CV1= 25.6\%$  and third case  $X= 0.56440, S= 0.11$  and  $CV3=18.50\%$

The values of the series of the ratios of the third case have less deviation from their center than that of the first case. They are less dispersed and are homogeneous. The degree of dispersion of the two distribution stoy closer with a 5% tolerance.

The two relative dispersion converge to the same center average value of the first case 0.52816 and 0.56440 of the third case with a tolerance of 3.6%.

- The average of the ratios : From the statistici analysis of the first study (Cardenas and al., 2009) first case, the average is 0.52816 which is conform to the normative method (4<sup>th</sup> case) with an average value of 0.52882. The calculation of the ratio by Norris and Parker's method using the second formula gives a value of 0.56440 closer to the normative value and that of the first case with only a tolerance of 6% .

- The experimental design : The experimental design gives a new expression by giving a ratio estimated of 0.54700 with 3% deviation from that of the normative method.

Through the experimental design we observe :  
- The increase of the thickness of the indentation specimen decreases.

- The increase of the size of the sphere lowers the response.

- The interaction of the increase of the size of the sphere with the decrease of the thickness of the specimen increase the response.

- The simultaneous increase of these two parameters results in a compensation.

The ratio of the ultimate stress over the maximum load is equal to 0.33840 for the second case using Norris and Parker's first formula which shows a value far from the average calculated values with an important deviation of 36%.

## 6. Conclusion

The statistical tools have allowed to analyse the expression of the variation of the mechanical properties with respect to the dimensional parameters of small punch test device's. We have shown that S.D.Norris and J.D.Parker's second formula is applied to the obtained results (1).

The characterization of the five DP steels by (SPT) gives results conform to the normative method .

This can be explain by the fact that the ratio is a function of the displacement at rupture.

It has been verified that S.D.Norris and J.D.Parker's first formula which is an empirical formula expressing the ratio wich respect to the dimensions of small punch test device's appratus and the thickness of the specimen gives a constant result far from the normative method.

The first formula does not take into consideration the mechanical properties of the steels but only the parameters R, r and t.

## References

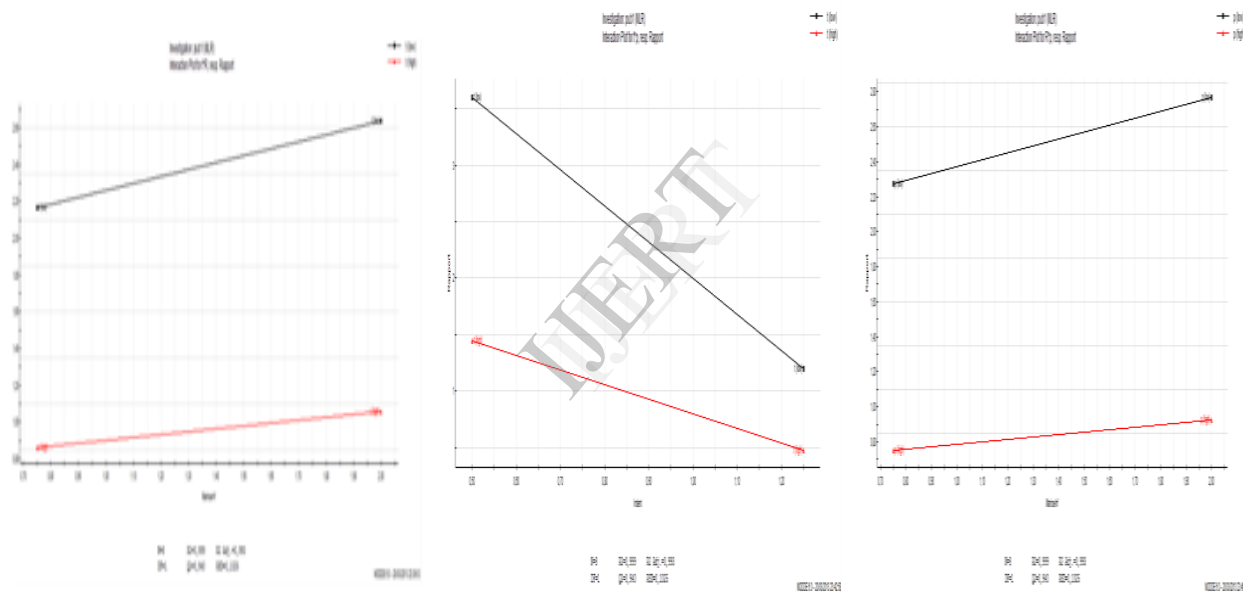
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**Annex 1**

**Table 7. Presentation of experimental plan 2<sup>3</sup>**

Experience	Average	X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	X <sub>1</sub> . X <sub>2</sub>	X <sub>2</sub> . X <sub>3</sub>	X <sub>1</sub> . X <sub>3</sub>	X <sub>1</sub> . X <sub>2</sub> . X <sub>3</sub>	Y <sub>exp</sub>
1	+1	-1	-1	-1	+1	+1	+1	-1	3.25
2	+1	+1	-1	-1	-1	+1	-1	+1	1.30
3	+1	-1	+1	-1	-1	-1	+1	+1	3.96
4	+1	+1	+1	-1	+1	-1	-1	-1	1.58
5	+1	-1	-1	+1	+1	-1	-1	+1	1.08
6	+1	+1	-1	+1	-1	-1	+1	-1	0.43
7	+1	-1	+1	+1	-1	+1	-1	-1	1.32
8	+1	+1	+1	+1	+1	+1	+1	+1	0.53
Divisor	8	8	8	8					
Effects	a <sub>0</sub> =1.68	a <sub>1</sub> =- 0.72	a <sub>2</sub> =0.16	a <sub>3</sub> =- 0.84	a <sub>12</sub> =-0.070	a <sub>23</sub> =-0.082	a <sub>13</sub> = 0.360	a <sub>123</sub> = 0.036	

**4.3.2 Analyse of interaction effects**



**Fig. 4. Graphical representation of interaction between the three factors**



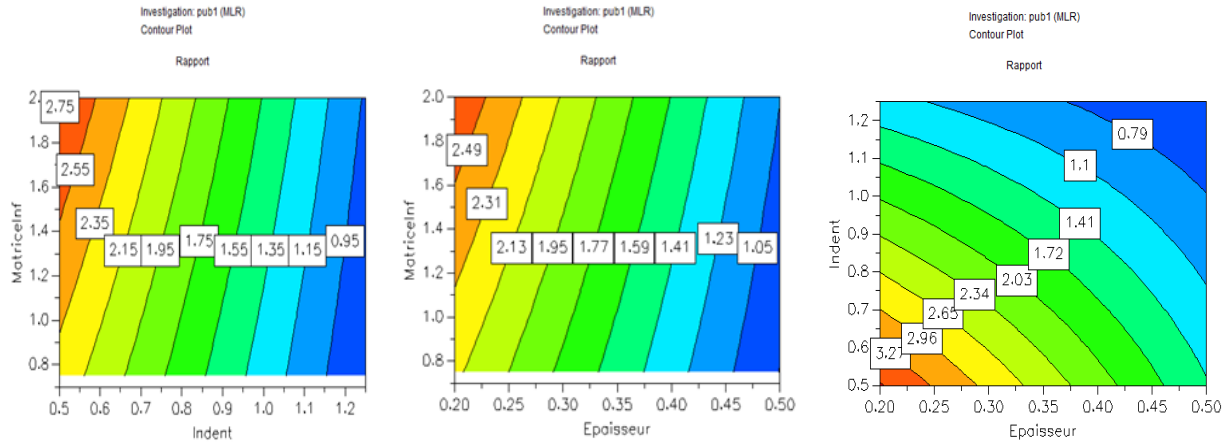


Figure 6. Contour of response of the variation of mechanical ratio.

Table. 11. Gives a summary of the obtained results

	Ratio $\frac{\sigma_m}{P_{max}}$ [mm <sup>2</sup> ]		Empirical Formula	Standard deviation S	Coefficient of variation CV	Absolut average deviation EMA
	Géométric Characteristic of SPT device	Méchanical Properties of Material				
1/ Statistics of study [1]		0.52816	Résultats expérimentaux (Traction/SPT)	0.13	25.6 %	0.12
2/ Norris and Parker formula (3.4)	0.33840		$\frac{1}{t * (2.32D - 0.9Cl + 0.56)}$			
3/ Norris and Parker Formula (3.5)		0.56440	$\frac{P_{max}}{t * (0.14D - 0.82Cl + 2.17d_F + 0.6)}$	0.11	18.5 %	0.10
4/ Normative Method	0.52882		$3.33 \cdot K_{sp} \cdot R^{-0.2} \cdot t^{1.2} \cdot t$			
5/ Design of experiment 2 <sup>3</sup>	0.54700		$1.682 - 0.721.t + 0.164.R - 0.842.p - 0.070.t.R - 0.082.R.p + 0.360.t.p$ Or $K_{sp}(1.682 - 0.721.t + 0.164.R - 0.842.p + 0.360.t.p)$			