

**APPLICATION OF RESPONSE SURFACE METHODOLOGY TO MODEL DEPENDENCE OF
STRENGTH AND DEFORMABILITY OF LIMESTONE ROCK ON INTACT SPECIMENS
SHAPE DEVIATIONS**

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APPLICATION OF RESPONSE SURFACE METHODOLOGY TO MODEL DEPENDENCE OF STRENGTH AND DEFORMABILITY OF LIMESTONE ROCK ON INTACT SPECIMENS SHAPE DEVIATIONS

ABSTRACT

It is known from earlier and recent research that mechanical properties of intact rock like uniaxial compressive strength (UCS), Young's modulus (E) and Poisson's ratio (ν) are influenced by shape deviations of test specimens as deviations from the ideal cylinder. That impact can be significant at certain level of inaccuracy of specimen preparing, and it should be objectively evaluated and controlled in testing. The effects of intact specimens side straightness, ends flatness, ends parallelism and perpendicularity to the specimen axis, on UCS, E, and ν , measured in several actual ways during laboratory compression tests, were determined by previous research on 90 homogeneous specimens of limestone. In this paper, we subject these new experimental results to Response Surface Methodology (RSM) to model the mentioned dependence, to identify significant connections of variables and to evaluate the conclusions obtained directly from experimental phase. This study indicates how the responses of the model (outputs) – parameters UCS/E/ ν of limestone rock depend on factors (inputs) – parameters of specimen shape deviations like flatness R, parallelism P and perpendicularity O, assuming modern test machines with spherically seated upper platen. Starting from general RSM model with three factors (R, P, and O), we investigated the multiple linear regression models of specific mechanical property (UCS, E, and ν). Using the NCSS program ("NCSS 2004 and PASS 2005") in several steps, final models with very high coefficients of determination were developed for properties with the most evident effects of specimen shape deviations: nine models for strength UCS and UCS₅₀ (equivalent UCS for specimen with 50 mm diameter), and three models for ν_L (Poisson's ratio calculated from axial deformations measured on the entire specimen length). These statistical models with their response surfaces further strengthen and confirm the results and conclusions from the experimental phase. The critical values of R, P, and O are established using an additional statistical analysis to determine the lower and upper engineering limits. These findings set the basis for the new eligibility criteria for specimens of limestone (and similar rock with UCS about 100-150 MPa) in further testing of strength and deformability.

KEYWORDS

Multiple regressions, Limestone, Intact rock, Strength, Deformability, Specimens, Shape deviations

INTRODUCTION

Mechanical properties of intact rock like uniaxial compressive strength (UCS), Young's modulus (E) and Poisson's ratio (ν) are important input rock parameters required and determined for rock mechanical studies in most civil and mining projects. Depending on the rock type and UCS category, these mechanical properties are influenced by shape deviations of test specimens as deviations from the ideal cylinder (Hudson & Harrison, 2000; Thuro, Plinninger, Zäh, & Schütz, 2001; Vrkljan, 2006). It is known, from laboratory work and rare studies on rock (Hoskins & Horino, 1968; Podnieks, Chamberlain, & Thill, 1972), and other studies on concrete cylinders (Gonnerman, 1924; Richardson, 2000), that the inaccuracies during preparation of cylindrical specimen in the form of 'micro' deviations from flatness, perpendicularity and parallelism typically lead to the determination of smaller 'macro' properties - strength and various moduli. That impact can be significant at certain level of inaccuracy of specimen preparing, and should not be ignored. It also means that it should be objectively evaluated and controlled in testing.

The middle UCS category of approximately 100-150 MPa observed here is relatively common (limestones, dolomites, some sandstones), and is located between the two extremes. The first extreme is weak rocks with UCS up to 50 MPa, where the influence of small shape deviations is insignificant (Pells & Ferry, 1983). The other extreme is rocks with UCS even above 300 MPa (e.g. granite), where this influence is unquestionable and regulated by strict requirements (ISRM, 1979, 1983, 1999, 2007; ASTM D 4543-08; ASTM D 7012-10). Therefore, it can be assumed that for rocks with middle values of mechanical properties the requirements for sample shaping should be somewhere between the extremes. Previous test results (by observing the results in an accredited laboratory for a prolonged period) also show clear indications that the specimen requirements for rocks with UCS about 100-150 MPa are to some extent overstated. Also, taking into account the diversity of requirements for specimens in documents for testing and standards (ASTM D 4543-08; ASTM D 7012-10; EN 1997-2:2007; ISRM, 1978a, 1979, 1983, 1999, 2007; Mutchler, 2004), which are of various strictness and sometimes very difficult to achieve, there was a need for further clarification of the effects of specimens shape deviations in following testing. The aforementioned was a motive for the extensive previous research (Štambuk Cvitanović, 2012), which produced the experimental results as the input data for the RSM.

In previous research 90 samples from the same block of rock were tested for homogeneity, according to the variability of density ρ (kg m^{-3}), velocity of longitudinal (primary) elastic waves by ultrasonic technique v_p (m/s) (Woeber, Katz, & Ahrens, 1963) and Schmidt Rebound Hardness HR (ISRM, 1978b). Specimens were formed and divided into groups with different predefined shape irregularities: ID – “ideal” specimens (without shape deviations, basic criteria ASTM D 4543), A – specimens intentionally made only with non-parallelism of upper end, B – only non-flatness of the upper end, C – combination of A and B (both non-parallelism and non-flatness of the upper end), and D – only non-perpendicularity of the lower end. For these specimens (non-)flatness $R=W$ (mm) is defined as the maximum surface profile height or peak-to-peak amplitude of the surface waviness. (Non-)parallelism $P=\Delta\phi$ ($^\circ$) is angle between planes of upper end and lower end (the angular difference), and perpendicularity $O=\phi''$ ($^\circ$) is expressed as slope of lower specimen end. In addition, with the perpendicularity is associated the relative deviation of coaxial alignment $|P_o/D|$, where P_o is total displacement of the specimen axis from the global y-axis of the test machine, due to non-perpendicularity of the lower specimen end and side straightness deviations (measured at the level of specimen upper end), and D is specimen diameter. Surface profiles and waviness were accurately measured with new purpose-developed equipment. In the described manner, for all specimens the final input parameters for further research R , P , O were calculated and accepted. All specimens were tested according to ASTM D 7012-10, using servo-controlled test machine in the accredited laboratory, which means that all technical requirements were under constant supervision according to standard ISO/IEC 17025 (2005) for the competence of laboratories. Axial and radial displacements and strains were measured on several actual ways, using LVDT's and strain gauges, with axial deformations measured both in the middle of the specimen height and on the entire length of the specimen. From the recorded stress-strain curves, with deformations measured in several ways, UCS/E/ ν values were determined (a total of 9 different parameters of strength and deformability). E and ν parameters were calculated for the stress level of 0.5 UCS, from approximately linear portion of the curve.

These uniaxial experimental results for UCS/E/ ν dependence on R/P/O represent so called “natural models” of behavior, and are correlated with Lower Engineering Limits (LEL) and Upper Engineering Limits (UEL). LEL and UEL were determined so that they reflect variability that would normally be present for a given rock type/UCS category, with controlled probabilities of errors of the 1st and 2nd kind α , β (U.S. Department of Transportation, 1977). Using LEL/UEL limits and established natural models for mechanical properties, the critical values (tolerances) of R/P/O were obtained.

RESPONSE SURFACE METHODOLOGY

In order to assess the reliability of natural models obtained directly from experimental phase, the additional analysis using Response Surface Methodology (RSM) is undertaken. We used RSM to model the dependence of mechanical properties of limestone rock on shape deviations of intact specimens, to

identify significant connections or any new connection of input and output variables, and to evaluate the conclusions.

General Form of Response Surface Model

The influence of intact specimen factors R/P/O on mechanical properties UCS/E/v is estimated by RSM, in this case using the multiple regression analysis. RSM is a collection of mathematical and statistical techniques that are used to analyze the task in which a response of interest is influenced by several variables.

The general form of the RSM model with three factors is (Montgomery, 2001):

$$y = B_0 + B_1x_1 + B_2x_2 + B_3x_3 + B_4x_1x_2 + B_5x_1x_3 + B_6x_2x_3 + B_7x_1^2 + B_8x_2^2 + B_9x_3^2 + \varepsilon \quad (1)$$

where:

y – dependent variable, response of the model

x_1, x_2, x_3 – independent variables, factors

$B_0...B_9$ – model parameters, regression coefficients

ε – error

If the products and squares of independent variables in (1) are replaced with new variables, the equation:

$$y = B_0 + B_1x_1 + B_2x_2 + B_3x_3 + B_4x_4 + B_5x_5 + B_6x_6 + B_7x_7 + B_8x_8 + B_9x_9 + \varepsilon \quad (2)$$

is representing a *multiple linear regression model*, because the model parameters are linear regardless of the surface response shape which model produces. Model of specific mechanical property is:

$$\begin{bmatrix} y_1 \\ y_2 \\ \cdot \\ \cdot \\ \cdot \\ y_n \end{bmatrix} = \begin{bmatrix} 1 & x_{11} & x_{12} & \cdot & \cdot & x_{1k} \\ 1 & x_{21} & x_{22} & \cdot & \cdot & x_{2k} \\ \cdot & \cdot & \cdot & \cdot & \cdot & \cdot \\ \cdot & \cdot & \cdot & \cdot & \cdot & \cdot \\ \cdot & \cdot & \cdot & \cdot & \cdot & \cdot \\ 1 & x_{n1} & x_{n2} & \cdot & \cdot & x_{nk} \end{bmatrix} \begin{bmatrix} B_0 \\ B_1 \\ \cdot \\ \cdot \\ \cdot \\ B_k \end{bmatrix} + \begin{bmatrix} \varepsilon_1 \\ \varepsilon_2 \\ \cdot \\ \cdot \\ \cdot \\ \varepsilon_n \end{bmatrix} \quad (3)$$

where $k = 9$ and $n = 90$.

Multiple linear regression analysis was performed using the computer program "NCSS 2004 and PASS 2005" (Hintze, 2004) in several steps:

1. "Full" model that contains all combinations of input variables - members from the relations (1) and (2), with natural factors R, P, O (without a transformation of variables);
2. Full model with factors on which a transformation of variables is applied, or dependent variables are normalized;
3. Assessment of the influence of individual members in the full model; based on their statistical significance, dominant members were selected / rejected insignificant members;
4. Model (with natural or transformed variables) with a reduced number of parameters.

After exploring all of statistically significant dependencies, final models were developed for UCS, UCS₅₀ and v_L , i.e. those properties for which the greatest effects of specimen shape deviations were established.

RESULTS OF RSM ANALYSIS

Multiple Regression Statistical Models for Strength

Using the program NCSS and analysing many variants, 9 models for strength were developed:

- (i) 3 models (Model 1, Model 2, and Model 3) for UCS, with the dependent variable $DS = (UCS - \bar{X}) / \bar{X}$, where DS (Delta Strength) is the change in strength UCS compared to the reference mean value \bar{X} ($\bar{X}=127.5$ MPa is the average UCS obtained from specimens without influence of shape deviations);
- (ii) 3 models (Model 4, Model 5, and Model 6) with the dependent variable UCS/\bar{X} which is the uniaxial compressive strength normalized by mean value;
- (iii) 3 models for UCS₅₀ (Model 7, Model 8, and Model 9), analogue to models (ii). UCS₅₀ is defined as equivalent UCS for specimen with 50 mm diameter: $UCS_{50}=UCS/(50/D)^{0.18}$ (Hoek & Brown, 1980).

Models 1, 4 and 7 represent the linear regression (UCS or UCS₅₀ dependence on one dominant variable R), while the rest of the models are multivariate linear regression (connect UCS or UCS₅₀ with more independent variables). This paper presents Models 4, 5 and 6 in the briefest form. The coefficient of determination R^2 is marked *italic* to distinguish it from the flatness R.

Model 4

Equation:

$$UCS / \bar{X} = 1.0249 - 1.3784 R \quad (4)$$

provides strength UCS normalized by mean value \bar{X} depending on flatness $R=W$ (mm). It is a model with reduced number of parameters and the coefficient of determination $R^2=0.86$ (Fig. 1). Full model showed that the only significant variables are R, R^2 and PR (no perpendicularity O!). After reduced analysis, just flatness R remained as a statistically significant variable (Fig. 2).

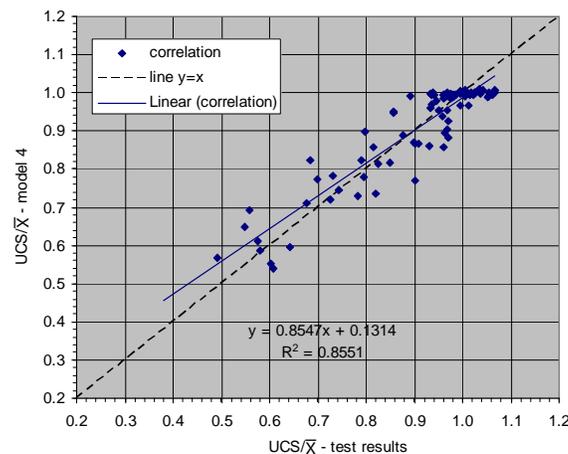


Figure 1 – Correlation between test results and Model 4

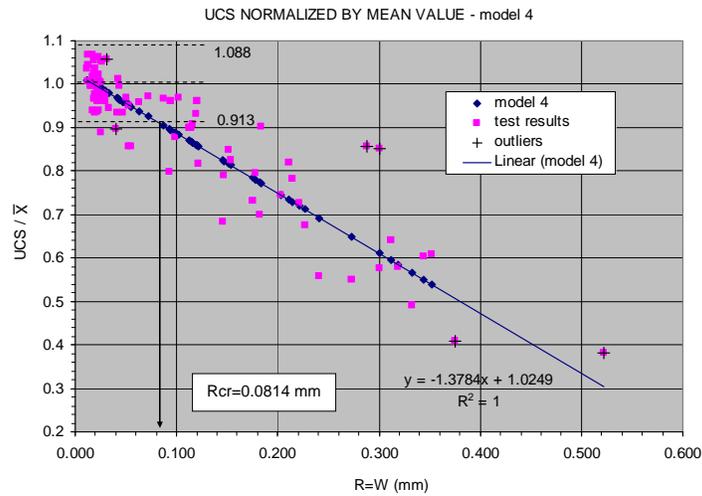


Figure 2 – UCS normalized by mean value \bar{X} for Model 4 (results obtained from tests and model); value UCS/\bar{X} on the LEL limit is $116.4/127.5=0.913$, for which the critical flatness is $R_{cr}=0.081$ mm

Model 5

Equation:

$$UCS / \bar{X} = 0.3482 + 0.0890 \ln P - 0.3524 \ln R + 0.0253 \ln R \ln P - 0.0484 (\ln R)^2 \quad (5)$$

determines the normalized strength UCS/\bar{X} , depending on flatness $R=W$ (mm) and parallelism $P=\Delta\phi$ (°). The model is reduced (containing a reduced number of parameters), with transformed input variables and coefficient of determination $R^2=0.88$. Includes all test results ($N = 90$) and it is shown in Fig. 3, Fig. 4, and Fig. 5. Strength UCS and its normalized value UCS/\bar{X} do not depend on O , but on members with the R and P . For UCS/\bar{X} on the lower limit engineering ($116.4/127.5=0.913$) corresponding critical flatness is within $R_{cr}=0.07-0.09$ mm depending on P , Fig. 3. In Fig.4, for samples from this study (present P) $R_{cr}=0.084$ mm is obtained. Response surface in Fig. 5 is drawn according to equation (5), using program "LAB Fit" (Silva & Silva, 2011).

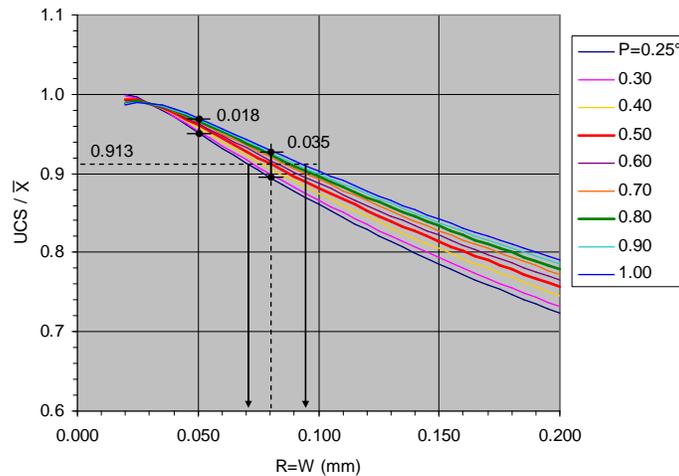


Figure 3 – Curves with $P = \text{const.}$ for Model 5; for R to 0.08 mm and the widest range of expected P from 0.25° to 1° corresponding variations USC/\bar{X} will be max. 3.5% (really and less)

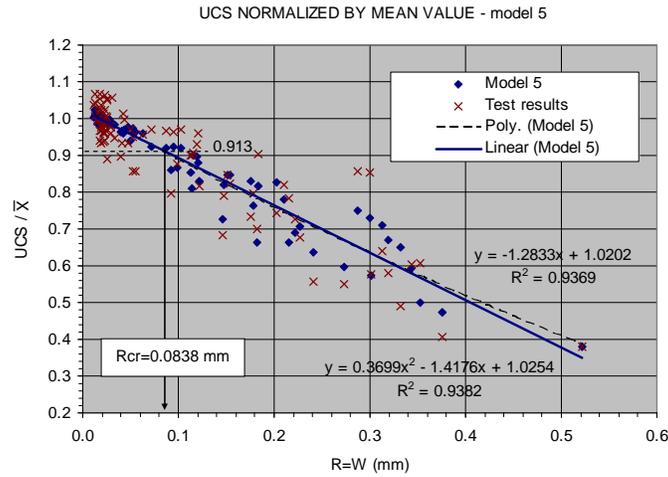


Figure 4 – Normalized strength UCS/\bar{X} depending on flatness R for Model 5 (results obtained from tests and model)

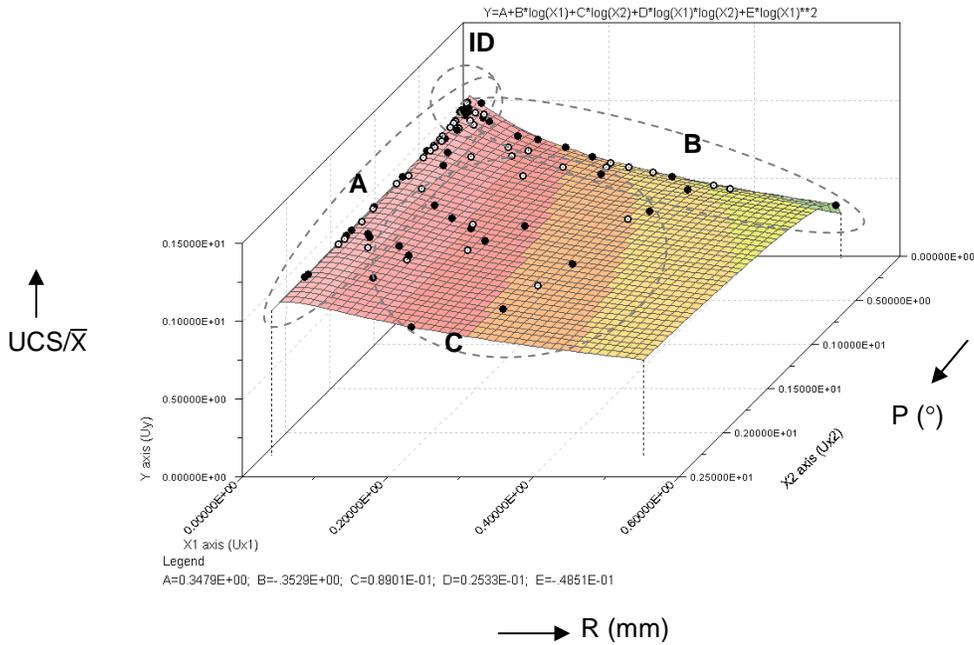


Figure 5 – Surface of the model for Model 5; areas that belong to the groups of specimens A, B, C, and ID from the natural models are indicated

From Fig. 5 it is evident that UCS does not depend on P (small R, group A), but there is an obvious dependence of UCS on R (small P, group B). The influence of P members appears only at higher R (hence in group A in the natural models this influence was not recorded). In group C, there is distortion at the edges of the surface (at higher R and P) which is assumed to represent some unexplored “shape effect” due to the already large deviations in the shape of the specimen. For the area of interest (R up to 0.1 mm, P to 1°) this influence is insignificant.

Model 6

Equation:

$$UCS / \bar{X} = 1.0240 - 1.5261 R + 0.2363 R P \quad (6)$$

gives normalized strength UCS/\bar{X} depending on flatness R and parallelism P in a simplified manner. The model shown in Figs. 6 and 7, with $R^2=0.89$ and specimens of groups A+B+C (N=68 sets of data) included, was obtained after a full regression analysis (significant variables R, RP and R^2), and then reduced analysis.

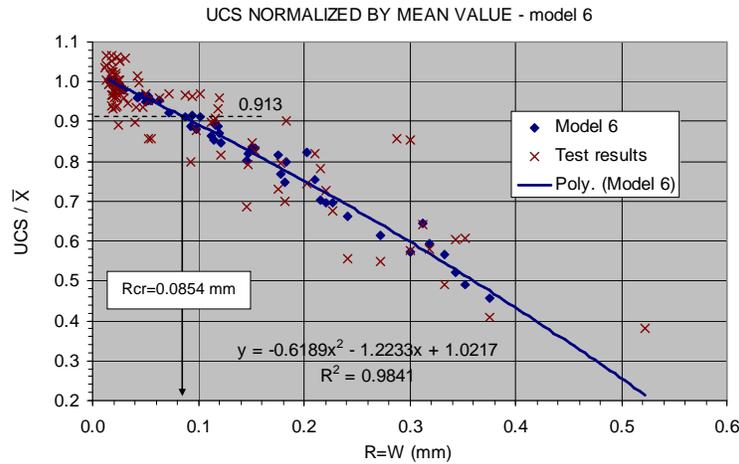


Figure 6 – Relation of normalized strength UCS/\bar{X} and flatness R for Model 6

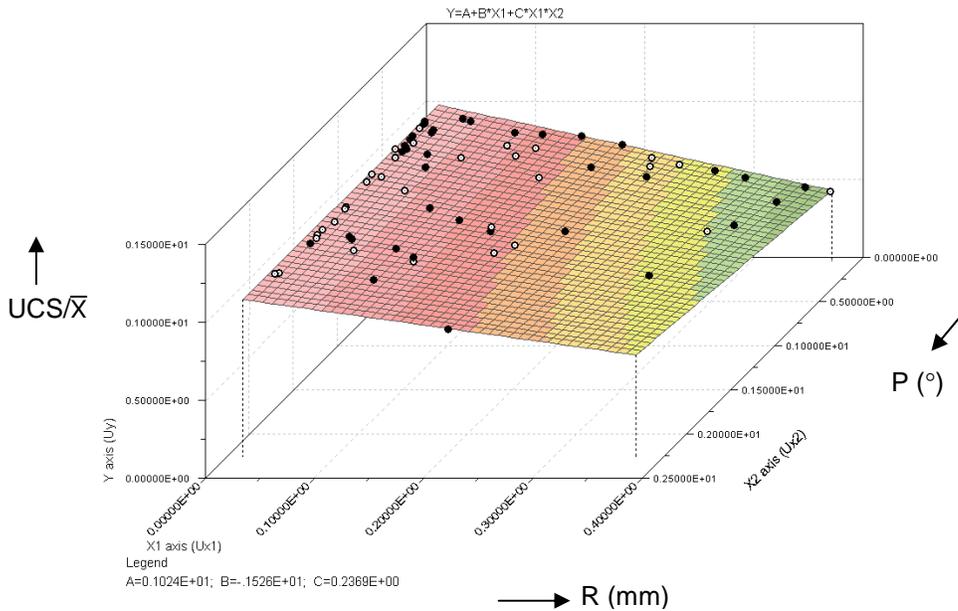


Figure 7 – Model 6: surface of the model; differences between model 6 and given surface (residuals) are not greater than 0.007, and the average value is $1.21 \cdot 10^{-10}$

Critical flatness for present P values is $R_{cr}=0.085$ mm (Fig. 6), and if in equation (6) $P=\text{const.}=0.25^\circ$ to $P=0.8^\circ$ is taken, the corresponding R_{cr} is in the range 0.076-0.083 mm. Surface of the model (Fig. 7) according to the equation (6) is simpler, but also reflects what has already been said about P.

Multiple Regression Statistical Models for Poisson's Ratio

Poisson's ratio was experimentally determined for 46 specimens. Using NCSS program three models for v_L were established – Models P1, P2, and P3. Model P1 (significant members only those with R, O, R^2 and O^2) confirms the cognition from the natural models that for the Poisson's ratio of greater importance is O than P, as well as the amount of critical flatness $R_{cr}=0.09$ mm. It gives the appearance of possible surface, according to which R should be kept within 0.05 mm when v_L will be determined (recommendation). Model P2 gives v_L dependence on the dominant input parameter R (“strip” with O to 0.5°), and again is $R_{cr}=0.09$ mm. P3 is a general model for the case of excessive side non-straightness, which except R also includes the relative deviation of coaxial alignment $|P_o/D|$. The covered range of $|P_o/D|$ is to 8%.

Review of Statistical Models and Evaluation of Results

Review of statistical models is given in Table 1, and the example of compatibility of results obtained with natural and statistical models in Fig. 8.

Table 1 – Review of statistical models

Model	Independent variables	Dependent variable	N=number of data (groups)	Coeff. of determination R^2	EQUATION (model, connection of variables)
1	R		84	0.86	$DS = 0.0249 - 1.3784 R$
2	$\ln R, \ln P$	$DS = \frac{UCS - \bar{X}}{\bar{X}}$	90	0.88	$DS = -0.6518 + 0.0890 \ln P - 0.3524 \ln R + 0.0253 \ln R \ln P - 0.0484 (\ln R)^2$
3	R, P		68	0.89	$DS = 0.0240 - 1.5261 R + 0.2363 R P$
4	R		84	0.86	$UCS/\bar{X} = 1.0249 - 1.3784 R$
5	$\ln R, \ln P$	UCS/\bar{X}	90	0.88	$UCS/\bar{X} = 0.3482 + 0.0890 \ln P - 0.3524 \ln R + 0.0253 \ln R \ln P - 0.0484 (\ln R)^2$
6	R, P		68	0.89	$UCS/\bar{X} = 1.0240 - 1.5261 R + 0.2363 R P$
7	R		84	0.86	$UCS_{50}/\bar{X} = 1.0262 - 1.3989 R$
8	$\ln R, \ln P$	UCS_{50}/\bar{X}	90	0.88	$UCS_{50}/\bar{X} = 0.3448 + 0.0870 \ln P - 0.3503 \ln R + 0.0244 \ln R \ln P - 0.0474 (\ln R)^2$
9	R, P		68	0.89	$UCS_{50}/\bar{X} = 1.0260 - 1.5449 R + 0.2272 R P$
P1	R, O		46	0.71	$v_L/\bar{X} = 0.9658 - 2.8451 R + 0.5033 O + 4.9736 R^2 - 0.2617 O^2$
P2	R	v_L/\bar{X}	44	0.66	$v_L/\bar{X} = 1.0798 - 3.0205 R + 5.4034 R^2$
P3	R, $ P_o/D $		46	0.66	$v_L/\bar{X} = 1.0070 - 3.2037 R + 8.5698 P_o/D + 5.8538 R^2 - 132.6015 P_o/D ^2$

Taking into account all the other results from the models (which can not be shown), it is interesting to mention the critical values (recommendations) for P and O, with values $P_{cr}=0.8^\circ$ for UCS and E generally (0.5° for v, v_L), and $O_{cr}=0.5^\circ$ for UCS and E generally (0.3° for v, v_L). R_{cr} of the order 0.08 to

0.10 mm is also appropriate for all Young's moduli E. Considering the conditions in the experiments conducted, the sum of the non-flatness on both specimen ends should not exceed 0.13 mm.

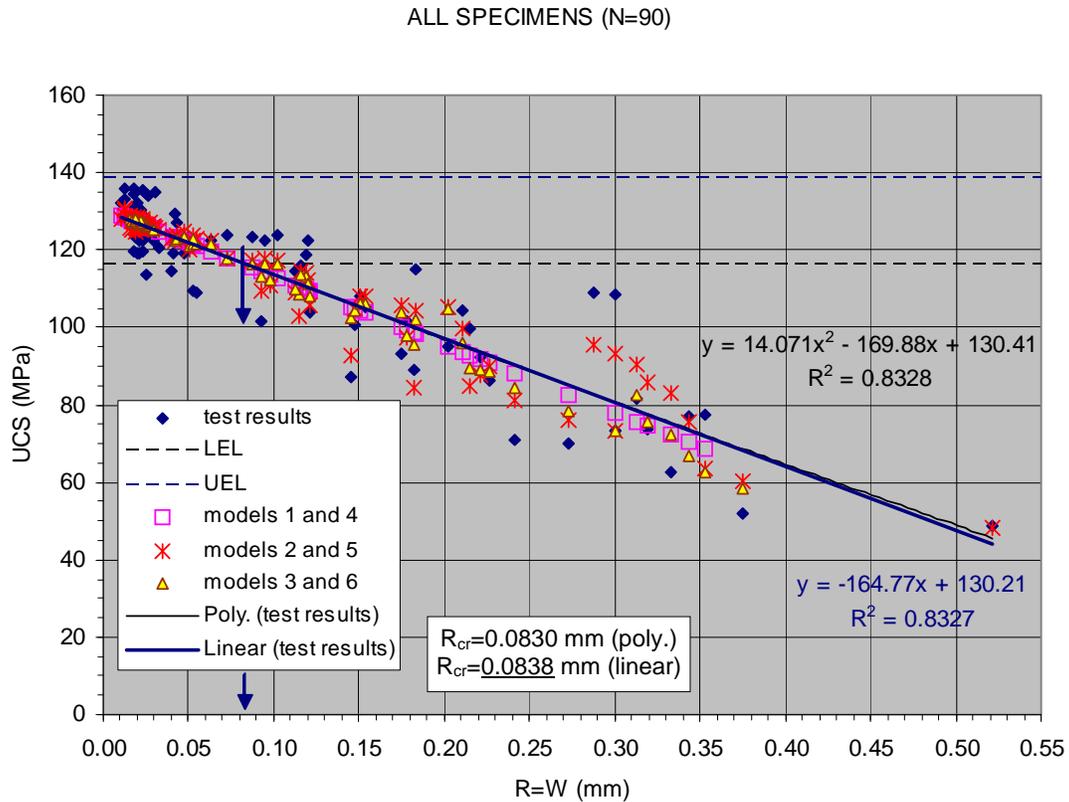


Figure 8 – Relationship UCS-R obtained experimentally and using RSM models

DISCUSSION OF THE RESULTS AND CONCLUSIONS

Both natural and statistical models for UCS and UCS₅₀ provide critical flatness $R_{cr}=0.08$ mm. For ν_L natural models give $R_{cr}=0.08-0.12$ mm, and 0.09 mm when all the specimens are drawn together, the same as statistical models. From statistical models the additional recommendation $R \leq 0.05$ mm is given if Poisson's ratio is going to be determined as ν_L value (and only ν_L). According to the statistical models, UCS depends also on P, but the presented surfaces show that this influence exists only for large R. Also, UCS does not depend on O, which corresponds to the results of group D in natural models. For Poisson's ratio statistical models suggest that O is more critical than P (equations with only R and O). This is also described by results of uniaxial tests. Young's modulus E is the least sensitive parameter.

Lower limit for UCS (LEL=116.4 MPa) was also confirmed using the NCSS program. All engineering limits were determined with controlled risks α and β (probabilities of errors of the 1st and 2nd kind), and proven in relation to measurement uncertainty and the data from ASTM interlaboratory testing program (published in ASTM D 7012-10).

All statistical models confirm the results and conclusions from the natural models. Obviously the models of behavior for the effects of specimens shape deviations in further testing have been successfully established. These findings set the basis for the new eligibility criteria for specimens of limestone (and similar rock with UCS about 100-150 MPa) in further testing of strength and deformability.

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