Influence of surface integrity on bonding process

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Abstract

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Adhesive bonding technology is successfully applied partly in the primary production, partly in the renovation in various fields of human activities. This fact emphasizes the importance of the partial factors research, which influence is essential for the quality, reliability and necessarily the usable life of bonded joints. The mechanical preparation of bonded surface is a varied process which influences directly the resultant bonded joint quality. The aim of the bonded surface mechanical preparation is the adhesion improvement in the adhesive – adherend interface. For mechanical preparation we use tools of defined cutting edge, by means of which we get the uniform surface texture, e.g. by milling. On the contrary such methods are more often used when the uniform surface cannot be reached. Shot blasting and grinding are significant representatives of these methods. The manual grinding can be significantly applied mainly in the agriculture. The absence of connected areas availability for the mechanical tooling and impossibility of their work clamping are the reasons for that. The breakwater in the sprinkler cistern restricting the liquid motion is the example.

Keywords: bonding; mechanical preparation; parameter *Ra*/*Rz*; renovation

In single industrial branches the production process varies. However, one element is common, namely the joint creation at the contemporary monitoring of the whole production process simplicity and effectiveness; the continual development and search for new perspective technologies are related to these. In this way the production process will be easier (Lancaster 2001).

The significant representative of jointing technologies is the adhesive bonding, which is the process of materials jointing with the help of the substance acting as the chemical factor according to Messler (2004); it is able to keep the materials together by means of surface attractive forces. Forces, which make the attraction possible, result from one or more sources. These sources are mostly originally chemical, but some can be mechanical or electrostatic. These forces are the cause of what is called adhesion, i.e. different materials bonding together (Messler 2004).

Attractive forces depend to a great extent on the bonded surface mechanical preparation and their task is to reach the definite integrity degree of the surface (Shahid, Hashim 2002). As the term "surface integrity" the complex of factors is meant which describes the machined or tooled surface properties. The parameters of surface roughness are ranked among significant factors influencing the surface integrity.

The surface roughness parameters are often used as the bonded joints model parameter and a number of scientists conducted a study of their effect on the bonded joints strength and durability using various adherends and adhesives (Katona, Batterman 1983; Matsui 1990; Sargent 1994; Gritchlow, Brewis 1995). The relation between roughness and

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adhesion is not simple. The optimum surface profile depends on the loading type and on the adhesive type (Shahid, Hashim 2002).

The adequate surface preparation of bonded parts gives rise to the adhesive strength increase (Chen et al. 1997; Uehara, Sakurai 2002; Bjorgum et al. 2003; Lunder et al. 2004; Prolongo et al. 2006). To obtain right and tenacious joint it is substantial to start with the adherend surface cleaning, because the adherends surface preparation is one of the most important operations in the bonding process (Messler 2004; Kašpar 2005). If the surface preparation is insufficient, the bonded joint necessarily and unpredictably breaks down in the area between the material and the adherend (Loctite 1998). At the right surface preparation every contingent failure will be always cohesive.

The surface preparation is made with the target to reach the maximal surface wettability of the chosen adhesive. In this way the ideal conditions for the adhesive – adherend contact and the initiation of adhesive bonds are created. The bonded surface wettability depends on the surface energy and on the surface integrity, which is represented above all by the roughness (Bumbálek et al. 1989, Packham 2003). The more the bonded joint surface participates in the adhesive bonds the higher is the bonded joint strength (Peterka 1980; Habenight 2002; Elbing et al. 2003). Harris and Beever (1999) do not agree with this statement. Sometimes it is mentioned that the greater roughness creates the greater surface for the connection. While these mechanisms interpret some of general characteristics of adhesion to the roughened surface, the more detailed analyses indicate that the roughing process can start the physical-chemical changes, which influence the surface energy and wettability. But Messler (2004) confirms the above mentioned theory and states that a measure of mechanical "encapsulation" contributes nearly always to the bonded joint strength increase. Wenzel (1936) draw in its works conclusions that the wettability and adhesive extension over the surface are rather more influenced by the effective surface area, which can really interact with the liquid i.e. adhesive than by the surface integrity characteristic. The importance and necessity of the surface roughness analyzing at the bonding technology application is presented in their works by a row of authors (Baker, Chester 1992; Sargent 1994; Gritchlow, Brewis 1995; Harris, Beevers 1999; Shahid, Hashim 2002; Packham 2003; Brožek, Müller 2004; Müller et al. 2007a b).

In compliance with this statement, Packham (2005) mentions that at surfaces of higher values of roughness parameters the increase in the surface area can lead to the adhesion relative increase as long as the surface roughness does not lower the contact between surfaces. With the bonded surface mechanical preparation, the adherend roughness increases and largely increases the bonded joint surface thanks to the greater surface effective area for the bond (Jennings 1972).

Gent and Lai (2003) also agree to the mentioned statement. Gent and Lai (2003) compared the adhesion of smooth and blasted steels and they observed the 2 to 3 times spalling energy increase, what they described to the surface increase.

Tamai and Aratanic (1972) found out that in frame of definite limits the specimen roughing evokes their wettability. It is possible to explain that the peaks of the slow eminence are barriers which avoid the drops spreading.

At adherends the surface energy influences the liquid running over the surface. In this way the surface wetting occurs. If the optimal surface wetting should be reached, the surface energy of the adhesive must be lesser that of the adherend.

The degree of the adherend surface coverage by the adhesive depends on its consistency (viscosity), on the surface cleanliness and on the surface asperity form. In the time of the adhesive application the adhesive viscosity must be so low so that the time of the asperity and cavity filling is sufficiently short compared with the time of the adhesive curing.

As a result of various technological operations use the asperities are formed on the bonded surface. The technological factors causing the asperities influence at the same time the surface properties. The form of asperities is very various (Bumbálek et al. 1989).

At the bonded surfaces mechanical preparation we start with two basic theories, namely of abrasive and erosive wears.

The abrasive wear is typical in the case of the bonded surface mechanical preparation using grinding methods. The grinding proceeds at the cut of big number of grains of irregular geometry, namely above all at the negative rake angle, relatively big edge radius, broken grain edge of different angle caused by the whole grains or their parts breaking off (Holešovský et al. 2007).

The abrasive wear is distinguished by the material particles separation and translocation at knurling and cutting using hard particles (Suchánek et al. 2007). The bonded abrasive particles get at relatively small moving of particles in the softer worn-out surface of a metal (Suchánek et al. 2007).

From the abrasion theory it follows the possibility of a surface roughness change at the influence of the load and path length change at the manual grinding by various persons. This presumption leads to the question of results comparability at the method application of the bonded surface preparation by the way of manual grinding. Also Messler (2004) draws attention to the possibility of different surface roughness reaching at the bonded surface preparation using abrasive cloth.

The erosive wear is characteristic for the cases of the bonded surface mechanical preparation by blasting. The erosive wear mechanism is similar to the abrasive wear, i.e. the knurling and cutting of material occurs (Pošta et al. 2002).

The erosive wear is caused by the impact of particles included in the streaming medium on the surface. If the impacted particle is of the sufficient energy, in dependence on the impact angle the replacement or detachment of material from the surface occur (Pošta et al. 2002). For the erosive wear the non-uniform surface failure is typical. The surface is undulating and corrugated (Pošta et al. 2002).

Blasting is a good method for the large surfaces cleaning. In this way the reached surface roughness offers good results of bonding if the very rough or on the contrary the very fine blasting material (abrasive material) is used (Loctite 1998).

Harris and Beevers (1999) targeted themselves to the dry blasting process using abrasive medium from aluminium of different grain size in order to discover and quantify the possible correlation between the surface roughness, surface energy and adhesion. At their research they elicited that the fine differences in the blasted grains types represent the measurable component of the surface characteristic. The primary surface integrity measuring confirmed their expectation that the coarse abrasive induces rougher surface and generally the more rough surfaces show themselves by the lower surface energies. The same conclusions are presented by (Varacalle et al. 2006; Chandler et al. 2009). They made partially clear the geometrical surface features effect on the drops widening and the contact angle change. Next they confirmed that the blasting process represents the chemical changes on the adherend surface which influences the surface energy. The same statements are supported by Shahid and Hashim (2002).

Other authors (Harris, Beevers 1999) found out that although the blasting conduces to the higher strength of bonded joints compared to other mechanical processes of adherend surfaces preparation, at blasting using rough and fine grains no difference of bonded joints strength was taken.

Considering the published pieces of knowledge and following evaluation of the dependence between the bonded joint strength characteristics and roughness parameters *Ra* and *Rz*, their relationship at single methods applicable at the bonded surface mechanical preparation were the aim of experiments.

MATERIAL AND METHODS

The influence of the bonded surface integrity was analyzed on the basis of laboratory experiments carried out according to the Czech technical standards. The comparability of foreign authors' results appeared as problematic. But it was excluded by laboratory experiments carried out by Marek (2002) who proved that the foreign and Czech standards engaged in problems of bonded joints destructive testing are comparable and offer comparable results. On the basis of this presumption it was possible to pick up again on research and conclusions published in the contemporary scientific papers. The carrying out of the tests according to the standard ČSN EN 1465 (1997) was the basis of bonded joints laboratory testing. According to the report elaborated by Broughton et al. (1999) from the Centre for Materials Measurement & Technology, Teddington, UK the above mentioned test is the most used method of destructive tests having the relationship to the concrete manufacturing program. The authors carried out tests according to the standard BS EN 1465: 1995 which is identical with the standard ČSN EN 1465 (1997).

The bonded surface mechanical preparation was carried out using methods recommended by literary sources, namely using manual grinding with abrasive cloth and blasting. Next, the face milling and rolling (i.e. surface without mechanical preparation) were evaluated.

Methods of manual grinding using various abrasive clothes are often used in renovation and on places where the machine preparation is not possible. The limiting factor of manual grinding with abrasive cloth is the action of many outer influences resulting in different surface roughness. This reason conduced to the use of two independent sets made by two persons. Each set contained 200 values of the abrasive cloth given grit. On prepared specimens the surface roughness parameters *Ra* and *Rz* were measured with the aim to determine if the surface roughness of these two sets was comparable.

For the comparability examination one of parametric tests was used, namely the *t*-test of the hypothesis about the conformity of two means tested for the significance level α = 0.05. Before the *t*-test execution the execution of the *F*-test was necessary. The *F*-test is the test of the hypothesis about two scatterings conformity, when we verify the scatterings conformity of the basic sets Ra^2 and Rz^2.

The surface of the standard test specimen was prepared using the abrasive cloths of P 40, P 120, P 180, P 240 and P 320 grit. Thanks to the identical material structure it was possible to consider the influence of different grits on the change of the mechanical prepared specimen surface roughness. The grinding was carried out perpendicular to the load force direction. The length of path was of 600 mm. The value of the load forcing the test specimen to the abrasive cloth was from 15 to 16 N.

The experiments intent on the bonded surface preparation by blasting put mind to the evaluation of the blasted corundum different grain size influence on the steel specimens surface roughness and on the influence on the bonded joints strength characteristics. Blasting is ranged among the most often used methods of surface preparation before the bonding technology application.

Before the bonding the specimens surface was mechanically prepared by the artificial corundum blasting. The used grits were F 40, F 80 and F 100. The specific grain size of the main fraction is determined by the standard FEPA "F" 42-D-86. The blasting was carried out using the pressure of 4 MPa. The nozzle slope to the specimen surface was of 90°, the distance between the nozzle and the specimen surface was of 100 mm.

The bonded surface preparation by milling is not the conventional method with regard to the time demands and high costs. In practice the technological operation of the surface mechanical preparation is in some cases totally omitted. This fact led to the inclusion of specimens, which surface was reached by the metallurgical semi-product fabrication, i.e. by the rolling process.

The laboratory experiments were carried out using the test specimens from the constructional carbon steel S235.J0 (old marking 11 373) made according to the standard ČSN EN 1465 (1997). The test assemblies are made by adhesive bonding of two adherends of dimensions 100 ± 0.25 × 25 ± 0.25 × 1.6 ± 0.1 mm. The lapping length was of 12.5 ± 0.25 mm. For bonding the constructional epoxy adhesive was used.

By the laboratory experiments it was proved that the optimal values at the use of epoxy adhesives were reached at the adhesive layer thickness about 0.1 mm (Messler 2004; Müller et al. 2006, 2008; Müller, Herák 2010). This fact led to this thickness use at next experiments.

After the bonded joint fracture the maximum force is read, the actual lapping length was measured and the type of fracture according to ČSN ISO 10365 (1995) was determined. Then the bonded joint surface S (1) and afterwards the bonded joint strength τ (2) are calculated.

$$S = l_u \times b \tag{1}$$

where:
S – bonded joint surface (mm^2)
l_u – lapping length (mm)
b – lapping width (mm)

$$\tau = \frac{F}{S} \tag{2}$$

where:
τ – tensile lap-shear strength (MPa)
F – acting force (N)
S – bonded joint surface (mm^2)

At the study of the fracture mechanism the fracture area will be qualitatively and quantitatively evaluated using the video analysis.

The mechanically prepared surfaces were evaluated by means of two surface roughness parameters according to the standard ČSN EN ISO 4287 (1999), namely:

- *Ra* – the arithmetic mean of the departures of the profile from the mean line (μm),
- *Rz* – sum of the height of the highest peaks and depths of the deepest valley in the range of sampling lengths (μm).

Before the surface roughness measuring the specimens were cleaned using the ultrasound in the perchlorethylene bath. The surface roughness parameters were measured using the profilometer Surftest 301. The roughness parameters were

Table 1. Statistical evaluation of two sampling sets congruence (abrasive cloth P 40)

	Roughness parameter			
	Ra		*Rz*	
Measurement variant	Person A	Person B	Person A	Person B
Arithmetic mean (μm)	2.382	2.451	16.242	16.676
Standard deviation (s)	0.439	0.428	2.586	2.549
Scattering (s^2)	0.193	0.183	6.686	6.498
Measuring number (m, n)	200	200	200	200
F-test	1.05		1.03	
$F_{0.05 – \text{tables}}$	1.26		1.26	
$F_{\alpha(0.05)} > F$	hypothesis H_0 confirmation		hypothesis H_0 confirmation	
t-test	1.58		1.69	
$t_{0.05 – \text{tables}}$	1.96		1.96	
$t < t_{\alpha(0.05)}$	hypothesis H_0 confirmation		hypothesis H_0 confirmation	

measured in five points of each specimen using the cut-off of 0.8 mm. The bonded joint strength was determined by the destructive test using the universal tensile-strength testing machine.

For the experiments evaluation the parameter *Ra*/*Rz* was used, where the relationship between the arithmetic mean of the departures and the average of five maximum peak-to-valley lengths shows itself. The statement of Tamai and Arantanic (1972) about the influence of considerable and occidental eminences, which create the barriers for wetting, led to the use of this criterion. Higher numerical values of this relationship eliminate contingent surface departures and decrease the contingent barriers. At the lower values of the parameter *Ra*/*Rz* it is possible to assume the parameter *Rz* values increase, which shows itself by the local occurrence of peak-to-valley lengths.

RESULTS AND DISCUSSION

Analyzing obtained data of *Ra* and *Rz* parameters measured at manual grinding methods by the use of abrasive cloth the statistical investigation was performed in order to express relevant conclusions and to neglect the outer acting factors influence limiting the resultant surface roughness.

At the *F*-test application following hypotheses were respected:

– H_0: $\sigma_1^2 = \sigma_2^2$, zero hypothesis asserts that no difference between scatters exists,

Table 2. Statistical evaluation of two sampling sets congruence (abrasive cloth P 120)

	Roughness parameter			
	Ra		*Rz*	
Measurement variant	Person A	Person B	Person A	Person B
Arithmetic mean (μm)	0.945	0.947	7.377	7.391
Standard deviation (s)	0.101	0.114	0.862	0.929
Scattering (s^2)	0.010	0.013	0.743	0.863
Measuring number (m, n)	200	200	200	200
F-test	0.79		0.86	
$F_{0.05 – \text{tables}}$	1.26		1.26	
$F_{\alpha(0.05)} > F$	hypothesis H_0 confirmation		hypothesis H_0 confirmation	
t-test	0.23		0.15	
$t_{0.05 – \text{tables}}$	1.96		1.96	
$t < t_{\alpha(0.05)}$	hypothesis H_0 confirmation		hypothesis H_0 confirmation	

Table 3. Statistical evaluation of two sampling sets congruence (abrasive cloth P 180)

	Roughness parameter			
	Ra		*Rz*	
Measurement variant	Person A	Person B	Person A	Person B
Arithmetic mean (μm)	0.741	0.740	5.903	5.806
Standard deviation (s)	0.157	0.141	1.111	1.165
Scattering (s^2)	0.024	0.020	1.235	1.356
Measuring number (m, n)	200	200	200	200
F-test	1.24		0.91	
$F_{0.05 - \text{tables}}$	1.26		1.26	
$F_{\alpha(0.05)} > F$	hypothesis H_0 confirmation		hypothesis H_0 confirmation	
t-test	0.05		0.86	
$t_{0.05 - \text{tables}}$	1.96		1.96	
$t < t_{\alpha(0.05)}$	hypothesis H_0 confirmation		hypothesis H_0 confirmation	

- H_1: $\sigma_1^2 \neq \sigma_2^2$, alternative hypothesis asserts that scatters are not identical.

At the *t*-test application following hypotheses were respected:

- H_0: $\mu_1 = \mu_2$, zero hypothesis asserts that no difference between means exists,
- H_1: $\mu_1 \neq \mu_2$, alternative hypothesis asserts that the means are not identical.

On the basis of carried out statistical tests confirming the H_0 hypothesis, presented in Tables 1 to 5 it is proved that the determined roughness parameters (*Ra* and *Rz*) of two independent sets are comparable, i.e. from statistical view it is not decisive whether the specimens were prepared by one or more persons.

This presumption enables following evaluation of the acquired data and the optimum conditions reaching at the manual grinding with the use of abrasive cloth.

The influence of the relationship between the *Ra* and *Rz* roughness parameters and the surface mechanical preparation is presented in Figs 1 and 2.

From the experimental results of P 40 grit presented in Figs 1 and 2, different influence of the surface mechanical preparation at the identical corundum grains grit keeping is evident.

Bonded joints were prepared according to the above mentioned standards and destructive tests. As the result of the destructive testing the destructive force and the bonded joint surface were determined according to the Eq. (1). These two parameters were used for the strength calculation according to the Eq. (2).

Table 4. Statistical evaluation of two sampling sets congruence (abrasive cloth P 240)

	Roughness parameter			
	Ra		*Rz*	
Measurement variant	Person A	Person B	Person A	Person B
Arithmetic mean (μm)	0.575	0.550	4.205	4.159
Standard deviation (s)	0.142	0.147	0.886	0.893
Scattering (s^2)	0.020	0.022	0.786	0.798
Measuring number (m, n)	200	200	200	200
F-test	0.93		0.99	
$F_{0.05 - \text{tables}}$	1.26		1.26	
$F_{\alpha(0.05)} > F$	hypothesis H_0 confirmation		hypothesis H_0 confirmation	
t-test	1.73		0.52	
$t_{0.05 - \text{tables}}$	1.96		1.96	
$t < t_{\alpha(0.05)}$	hypothesis H_0 confirmation		hypothesis H_0 confirmation	

Table 5. Statistical evaluation of two sampling sets congruence (abrasive cloth P 320)

	Roughness parameter			
	Ra		*Rz*	
Measurement variant	Person A	Person B	Person A	Person B
Arithmetic mean (μm)	0.375	0.385	2.952	2.991
Standard deviation (s)	0.095	0.104	0.683	0.622
Scattering (s^2)	0.009	0.011	0.466	0.387
Measuring number (m, n)	200	200	200	200
F-test	0.85		1.21	
$F_{0.05 - \text{tables}}$	1.26		1.26	
$F_{\alpha(0.05)} > F$	hypothesis H_0 confirmation		hypothesis H_0 confirmation	
t-test	1.04		0.61	
$t_{0.05 - \text{tables}}$	1.96		1.96	
$t < t_{\alpha(0.05)}$	hypothesis H_0 confirmation		hypothesis H_0 confirmation	

Single mechanical preparations were ordered ascending according to the *Ra*/*Rz* parameter and graphically demonstrated in Fig. 3. The relationship between the *Ra*/*Rz* parameter and the used surface mechanical preparation is represented by the polynomial curve of the 2nd degree, which corresponds best to the correlation field of measured points.

Parallel, bonded joints strength is graphically demonstrated. The relationship between the bonded joint strength and the used surface mechanical preparation is represented by the polynomial curve of the 2nd degree, too.

For the right evaluation, the intensity of the given dependence was also determined using the correlation analysis through the confidence coefficient R^2_{yx}. The confidence coefficient takes the values from 0 to 1. The more the value R^2_{yx} tends to number 1, the closer the given dependence is to the determined equation.

From the results presented in Fig. 3 the decreasing trend of the strength of different prepared bonded joints at the decreasing parameter *Ra*/*Rz* is evident.

The curves behavior presented in Fig. 3 is described by the Eqs (3) and (5). The Eq. (3) describes the dependence between the bonded surface mechanical preparation (*MP)* and the roughness parameter *Ra*/*Rz* presented in Fig. 3 and at the same time the confidence coefficient (4).

$$Ra/Rz = 0.0004 \times MP^2 + 0.0012 \times MP + 0.1279 \quad (3)$$

$$R_{Ra/Rz} = 0.994 \quad (4)$$

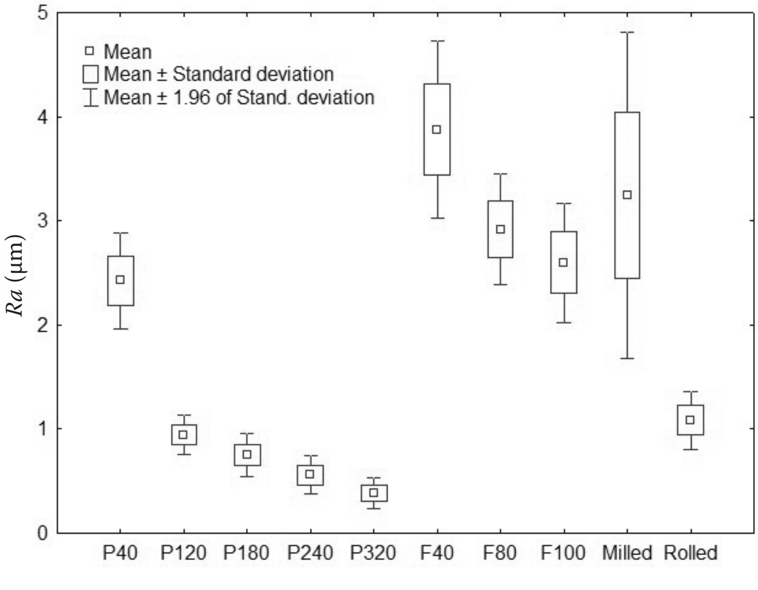

Fig. 1. Influence of surface mechanical preparation on the roughness parameter *Ra*

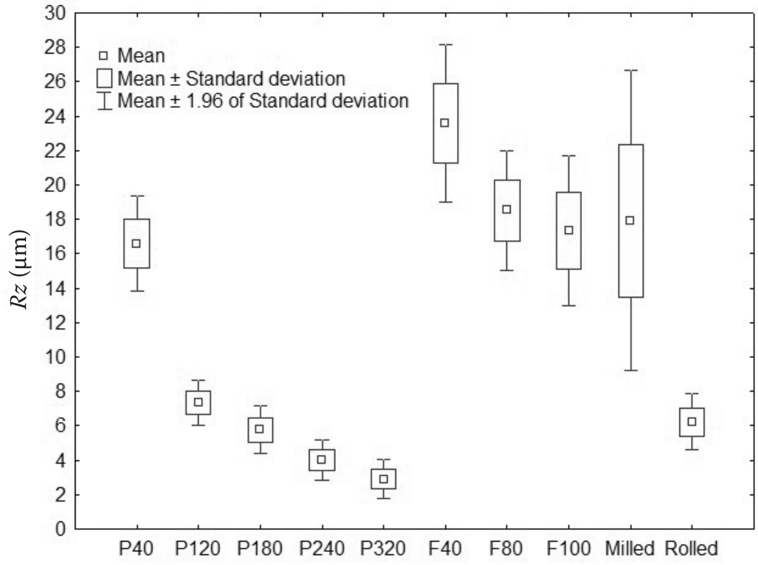

Fig. 2. Influence of surface mechanical preparation on the roughness parameter *Rz*

The Eq. (5) describes the relationship between the bonded surface mechanical preparation (*MP*) and the bonded joint strength (τ) presented in Fig. 3 and at the same time the confidence coefficient (6).

$$T = -0.1362 \times MP^2 + 0.6917 \times MP + 17.087 \quad (5)$$

$$R_\tau = 0.860 \quad (6)$$

The bonded joints test results show evident influence of different bonded surface mechanical preparatio. This fact contradicts the theory of Harris and Beevers (1999), which tells that at blasting using different grain size, no expressive differences of bonded joints strengths occur. The statement of Tamai and Aratanic (1972) about the influence of accidental and considerable peaks imposing the wetting barriers was not proved. The higher numerical values of the *Ra*/*Rz* parameter eliminate the pertinent profile departures and cut down the pertinent barriers. But from the results presented in Fig. 3 the decreasing trend of the bonded joints strength at the increasing *Ra*/*Rz* parameter is evident. The measurement results are supported by the fracture area evaluation, too. At all bonded joints the surface of which was mechanically prepared by blasting and manual grinding the fracture area was of cohesive type. The fracture area of bonded joints of rolled or milled surfaces was of adhesive type. This fact decreases the bonded joints strength, which is secondarily evident from Fig. 3.

CONCLUSION

At the bonded surface mechanical preparation the specific roughness occurs. The presumption of authors (Peterka 1980; Habenight 2002; Elbing et al. 2003) being related to the increasing function surface part thanks to increased values of surface roughness were confirmed. Higher values of bond-

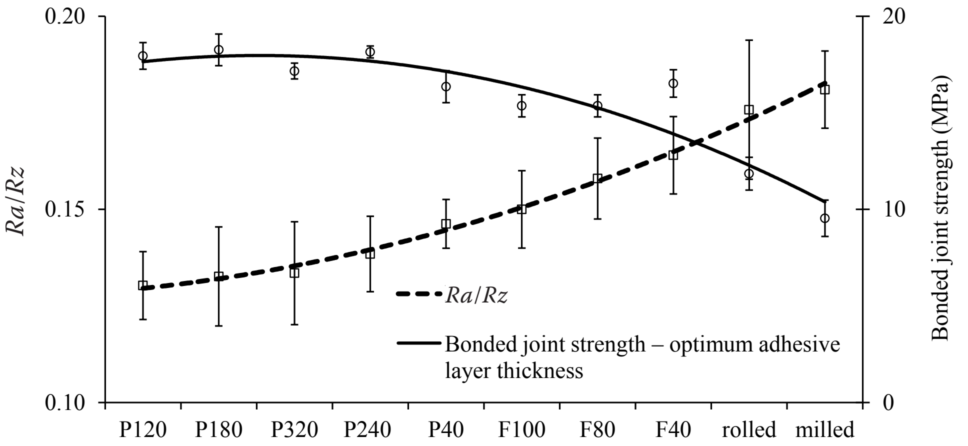

Fig. 3. Influence of the bonded joint surface mechanical preparation on the bonded joint strength and on the parameter *Ra*/*Rz* (Pxx – abrasive cloth grit, Fxx – abrasive grit at blasting)

ed joint strength were reached at lower parameter *Ra*/*Rz*, which proved about the increasing trend of the overlap surface.

Problems of bonded surface mechanical preparation using the manual grinding by abrasive cloth are on the research periphery today; minimum utilization in the lot manufacture is the reason. Yet, this method can be of use especially in various craftsman work. The generally spread premise that different results may be reached using the identical abrasive cloth is the next significant reason. This premise is presented e.g. by Messler 2004. At the manual grinding using abrasive cloth the hazard of different load acting on the abrasive cloth occurs, together with conjoined different quantity of abrasive grains. At manual grinding the abrasion theory shows the possible change of the surface roughness parameters at the load and path length change. By the statistical examination of two independent sets, this presumption was not met and it is possible to state that the determined roughness parameters *Ra* and *Rz* of specimens prepared by different persons are comparable. This conclusion is important especially for the objectivity of presented results concerning the manual grinding method and the mentioned knowledge acceptance in practice. This problem has not been experimentally solved so far. Only general premises and theories were taken into consideration.

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