DEGRADATION LIMITS OF BONDING TECHNOLOGY DEPENDING ON DESTinations EUrope AND INDONESIA

Miroslav Müller, David Herák, Petr Valušek

The production process is diverse in single technological branches, but they all have one element in common, that is the joint creation, which is influenced by the environment. The influence of operating conditions and degradation processes was examined in Central Europe, Indonesia and Laboratory during 8, 16 and 26 weeks. From the results the simulation was worked out serving for the bonded joint strength prediction for a longer time interval. Secondly the curing process influence on the bonded joint was evaluated. The test results did not confirm the substantial influence of different climatic and geographic conditions on the bonded joint strength.

Keywords: degradation, destructive testing, epoxy, steels

Granice razgradnje u tehnologiji spojavanja ovisno o odredištima Europa i Indonezija

Proizvodni proces je različit u pojedinim tehnološkim granama, ali sve imaju jedan zajednički element, izradu spoja, na što djeluje okolina. Procjećavao se utjecaj radnih uvjeta i procesa razgradnje u Srednjoj Europi, Indoneziji i Laboratoriju tijekom 8, 16 i 26 tjedana. Rezultati su poslužili za izradu simulacije kako bi se predvidjela čvrstoća spoja za duži vremenski period. Zatim se procjenjivao utjecaj postupka očvršćivanja na vezni spoj. Rezultati ispitivanja nisu potvrdili da postoji bitan utjecaj različitih klimatskih i geografskih uvjeta na čvrstoću spoja.

Ključne riječi: čelici, epoksid, ispitivanje s uništavanjem materijala, razgradnja

1 Introduction

The long-time life span expectancy is a significant problem limiting greater utilization of adhesives [1]. It is beyond all doubt that the environment can be harmful to the bonded joint [2]. Doyle and Pethrick [3], Sargent [4] claim logically that the change of environment can have effect on the adhesive physical properties. By the degradation process the bonded joint initial strength can be decreasing [5, 6].

The initial strength decrease is caused by the adhesive ageing process in the area near the joint boundary. The failure mechanism change is the proof of this fact.

Also Ning Su et al. [7] were interested in the bonded joint ageing by the effect of weather conditions. They evaluated the influence of increased/lowered temperature during 8 years. They found out that some adhesives kept their strength also in conditions when they were subjected to the natural environment, i.e. to the potential variation of temperature and relative humidity. They also found that joints cured at increased temperature were of higher strength during the whole service life.

The bonded joints exposed to the "natural environment" conditions are attacked by many degradation factors, among which the effect of heat, humidity, atmospheric oxygen, ozone and microorganisms are the most remarkable [8].

Messler [9] states that if the bonded joints are subjected to the effect of weather influences, adhesives lose their strength on average during 6 months up to 1 year very rapidly. Further he states that from the viewpoint of the development prediction it is important to watch the strength changes during the first 6 months in the given environment.

On the basis of the study by the authors Herák et al. [10] and Messler [9] it was possible to continue the research directed to the potential and to degradation limits of the bonding technology in dependence on the application in Europe and Indonesia.

The experimental determination of different climatic and geographic environmental influences on strength characteristics and failure behaviour was the aim of the laboratory testing.

2 Methodology

2.1 Materials and adhesives

The bonded joints laboratory testing was carried out according to the standard CSN EN 1465 (Adhesives – Determination of Tensile Lap-shear Strength of Rigid-to-Rigid Bonded Assemblies) [11]. The test samples were made from the unalloyed structural steel S235J0 using the two-component epoxy adhesives BM (Beta epoxy metal/made in Holland).

2.2 Specification of geographic and climatic environment of testing

Central Europe is situated in the mild climatic zone of the northern hemisphere. It is the transition zone. Warm summer with rainfalls alternates with winter with lasting snow cover. Europe is especially characteristic by industrial production, and its products are exported all around the world. But at the present time this influence goes missing.

Indonesia is of tropical climate, which is without fluctuations during the whole year. The division into classic times of the year does not exist here. The division is related to the dry and rainy seasons. The average air humidity ranges from 70 to 100 %. In consideration of the volcanic activity and Indonesia climate conditions the soil is very fertile. Regarding the region level of development and number of inhabitants (the fourth most populous state) agriculture is put on the dominant position [2, 12].
At the present orientation it is primarily possible to assume the import of agricultural technology, which is nowadays solved by the import above all from the Asian region, i.e. from China. In countries of the Third World the import and application of new advanced technologies depends on the level of education, political situation and environmental specifics. The use of modern technologies including service is very limited. It is necessary to pay attention to low failure rate, possibility of simple renovation and absence of complicated mechanisms. The bonding technology meets the above mentioned requirements.

**Laboratory environment** serves as some kind of standard with constant temperature $22 \pm 2$ °C and relative humidity $50 \pm 5$ %. The producers of adhesives and of products containing bonded joints relate their published results to the laboratory environment and that is why the laboratory environment was included in the experimental program.

### 2.3 Bonding procedure

The bonded material surface was mechanically prepared by corundum blasting and was chemically cleaned. The adhesive layer thickness was constant, namely of 0,1 mm. The bonded joints were cured during 24 hours. The curing process of the first set was at the laboratory temperature, i.e. $22 \pm 2$ °C, of the second set at the increased temperature, i.e. $70 \pm 2$ °C.

The cured bonded joints were left according to the standard CSN EN ISO 9142 [13] for 8, 16 and 26 weeks in the environment of Central Europe (Czech Republic), Indonesia (North Sumatra) and Laboratory. The destructive testing was carried out in laboratories of CULS in Prague using the universal tensile strength testing machine of the max. tensile load 50 kN.

The transport by plane of the bonded joints from Indonesia lasted 48 hours and samples were kept all the time in vacuum packing. After the bonded joints destruction the tensile lap-shear strength was calculated according to the equation (1) and the failure type was determined according to ISO 13065 [14].

$$\tau = \frac{F}{S} = \frac{F}{l_u \cdot b},$$

where:
- $\tau$ – tensile lap-shear strength (MPa)
- $F$ – tensile force (N)
- $S$ – bonded joint surface ($\text{mm}^2$)
- $l_u$ – bonded joint lapping length (mm)
- $b$ – bonded joint lapping width (mm).

### 3 Results

The influence of the environment and the exposure time on the degradation process and connected bonded joint strength (adhesive BM) are presented in Fig. 1. Graphic presentation of dependence is given by the linear function, which meets most the correlation field.

The experiments confirmed the logical, expected result of the increased negative influence of the environment on the bonded joints strength. This assumption was fulfilled in all paired variants of the experiment. The average strength decrease of the bonded joints placed in laboratory after 26 weeks was not considerable. At the set cured at increased temperature of $70$ °C the decrease was 3,8 %, while the results ranged from 2,4 to 2,5 %. From the above mentioned the minimum time influence on the bonded joint strength is evident. The more substantial decrease occurred at the bonded joints exposed to the environment of Indonesia. After 26 weeks the average strength decrease ranged from 14,2 to 14,6 %. At the bonded joints placed in Europe the strength decrease ranged from 11,6 to 12,6 % after the same time interval.

---

**Figure 1** Environment and exposure time influences on the bonded joint strength
The increased temperature showed to be the substantial factor influencing the bonded joint strength. The bonded joint strength increased 18.5% on the average. At increased temperature the bonded joints showed lower values of the results scattering. So the bonded joint curing at increased temperature is the substantial factor which eliminates the ageing process.

In the laboratory conditions the bonded joints showed the surface failure of the cohesive type, i.e. the destruction inside the adhesive layer. By the surface picture analysis the diffuse seepage of the surrounding humidity was not discovered. The bonded joints of the destination Indonesia cured at the temperatures of 22 and 70 °C showed already after 16 weeks of exposure the failure of the mixed adhesive-cohesive type. By the picture analysis the surrounding humidity seepage of about 10% was visible. In the bonded joints location the relative humidity ranged from 80 to 90%. The bonded joints placed in Europe and cured at 22 °C showed after 26 weeks the failure of the mixed adhesive-cohesive type i.e. the failure was in the interface of adhesive and adherent (adhesive type) as well as in the layer of the adhesive (cohesive type). The diffuse seepage was not found. The bonded joints placed in Europe and cured at 70 °C showed during the whole interval the failure of cohesive type.

The function equations (2) to (7) describing the relevant functions presented in Fig. 1 characterize the dependence of the bonded joint strength \( \tau (\text{MPa}) \) on the exposure time \( t (\text{week}) \). Their validity is in the interval 0 to 26 weeks. The coefficient of determination ranged from 0.84 to 1.00, which proves the high correlation, i.e. the minimum difference exists between the destination and the real values.

\[
\tau_{\text{Indonesia/22 °C}} = -0.10 \cdot t + 18.06, \quad (2)
\]
\[
\tau_{\text{Europe/22 °C}} = -0.08 \cdot t + 18.01, \quad (3)
\]
\[
\tau_{\text{Laboratory/22 °C}} = -0.02 \cdot t + 17.89, \quad (4)
\]
\[
\tau_{\text{Indonesia/70 °C}} = -0.11 \cdot t + 21.40, \quad (5)
\]
\[
\tau_{\text{Europe/70 °C}} = -0.10 \cdot t + 21.31, \quad (6)
\]
\[
\tau_{\text{Laboratory/70 °C}} = -0.03 \cdot t + 21.16. \quad (7)
\]

The basic measurement of the interval 0 to 26 weeks was used in the predictive model creation. By the substitution in the function equations (2) to (7) the predictive dependence of the 100 weeks interval was created. For the comparison of the presupposed and real values the reference tests of the same exposure time in the European environment and in the Laboratory were carried out. In Indonesia the reference tests were not carried out because of operational reasons. The correlation results of the real and predictive values are presented in Fig. 2. The prediction of strength values was verified in the following weeks: 39, 51, 64, 77, 90 and 100 (for Laboratory 22 °C and 70 °C and Europe 22 °C and 70 °C). According to function equations the bonded joints cured at 22 °C in the destination Europe would reach the zero strength result theoretically after 217 weeks, cured at 70 °C after 225 weeks. In the destination Indonesia the bonded joints cured at 22 °C would reach the zero strength result after 178 weeks, cured at 70 °C after 180 weeks.

From the statistical analysis ANOVA which was calculated in the MathCAD 14 software for level of significance 0.05, it was seen that the values of \( F_{\text{crit}} \) (critical value compares a pair of models) were higher than \( F_{\text{ratio}} \) values (value of the F – test) for all measured experiments and amounts of \( P_{\text{value}} \) (significance level at which the hypothesis of equality of models can be rejected) were higher than significance level 0.05. This shows that fitted lines can be used for fitting measured values since the relationships between measured values and fitted lines values were statistically significant. All values of \( F_{\text{crit}}, F_{\text{ratio}} \) and \( P \) index are presented in table (Tab. 1), as well as \( R^2 \) which is the coefficient of determination.

But the prediction of the bonded joint strength long-term development is contrary to the above mentioned knowledge limited by many influences and it cannot be determined decidedly from the data acquired during 26
weeks in the Indonesia region. But the stated results of the carried out laboratory experiments will help in the basic idea of the bonded joints strength characteristics development.

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Statistical analysis of measured experiments</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$F_{stat}$</td>
</tr>
<tr>
<td>L 22</td>
<td>$6.35 \times 10^{-5}$</td>
</tr>
<tr>
<td>L 70</td>
<td>$2.92 \times 10^{-5}$</td>
</tr>
<tr>
<td>E 22</td>
<td>$3.77 \times 10^{-5}$</td>
</tr>
<tr>
<td>E 70</td>
<td>$1.33 \times 10^{-5}$</td>
</tr>
</tbody>
</table>

4 Discussion

From the experimental results presented in Fig. 2 minimum deviation from predictive values is evident. The linearity of the bonded joints strength decrease after a definite period is not in direct correlation with the Lancaster results [15]. He studied at similar tests the levelling of the strength decrease of bonded joints exposed to various media and contaminants [15].

Different resistance of particular adhesives to the environment was also studied by authors [7], who subjected bonded joints to a given environment for a much longer time, namely for 8 years. But the author [9] states that the time needed for the determination of the bonded joint strength change owing to the ageing process caused by outer influences is already from six months. The critical period of the strength potential decrease of the bonded joint is during this time. At the experimental program oriented on the long-term ageing influence on the bonded joint strength this presumption was confirmed.

The negative influences of the climatic environments of Europe and Indonesia are more considerable than the ones in the laboratory. From the viewpoint of the application this factor is negligible. For the minimization of the negative influences of environment the bonded joints curing at increased temperature proved to be the most suitable. For experiments the curing temperature of 70 °C was determined in accordance with the results in papers [16, 17] and with own experiments.

From the above mentioned follows the recommendation to use bonded joints cured at increased temperatures for the application in unfavourable conditions. This statement is in accordance with the results published by authors [13], who argue that joints cured at increased temperature are in total of higher strength during their whole lifetime.

It is possible to agree with the statement of Duchacek [7] and Faires [18] about strength changes owing to geography and connected climatic conditions. But today the statement of geographic conditions does not characterize fully the given problems in consideration of the substantial climatic changes.

The experiments showed that the environment influence is mostly negative. It is characterized by the bonded joints strength decrease and by the potential appearance of changes. The above mentioned negative changes are accompanied by the failure type change of the bonded joint, namely by the change of the cohesive type in the adhesive-cohesive up to the adhesive type. The authors [2, 17] came to the same conclusion.

5 Conclusion

The carried out experiments led to the following conclusions:

- The possibilities of the elimination of the environment negative influences by the effect of the curing process. Bonded joints cured at increased temperatures, namely at 70 °C, showed, compared to bonded joints cured at laboratory conditions (22 °C), higher values of the initial strength. This trend was mostly kept also after a longer time.
- The fact of the minimization of the strength differences with respect to the world climatic and geographic conditions is significant for producers and importers of machines and devices containing bonded joints.
- There is a significant strength difference between the laboratory conditions and the real destination conditions of application owing to the temperature and relative humidity fluctuations.

Acknowledgement

The paper is supported by the project of foreign developing cooperation (ZRS) 136/05 – 09/MZe/B entitled "Building up consultation and advice centre for the sphere of agricultural and environmental engineering at the university UNITA in Tarutung".

6 References


Authors' addresses

Miroslav Müller, Assoc. prof., Ing., Ph.D.
Department of Material Science and Manufacturing Technology
Faculty of Engineering, Czech University of Life Science
Kamýcká 129, 165 21 Prague, Czech Republic
muller@tf.czu.cz

David Herák, Assoc. prof., Ing., Ph.D.
Department of Mechanical Engineering
Faculty of Engineering, Czech University of Life Science
Kamýcká 129, 165 21 Prague, Czech Republic
herak@tf.czu.cz

Petr Valášek, Ing., Ph.D.
Department of Material Science and Manufacturing Technology
Faculty of Engineering, Czech University of Life Science
Kamýcká 129, 165 21 Prague, Czech Republic
valasek@tf.czu.cz