

## Low-cyclic fatigue test of adhesive bond reinforced with glass fibre fabric

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**Abstract.** Epoxy resins are widely used polymers, which are popular due to their workability, high tensile strength and a chemical resistance. The glass fibre fabric interlayer was used for improving the tensile and the quasi-static lap shear strength of joints bonded with an epoxy adhesive.

The aim of the experiment is to clarify a fatigue behaviour (low-cyclic tests of the fatigue) of structural two-component epoxy adhesive applied to a constructional steel S235J0. The fabric was composed from type E glass fibres in a plain weave. For optimization of properties of the composite bond it was used various weights in grams of the fabric in the extent of 80, 110, 160 g m<sup>-2</sup> for the fabric treated by a wax, where this treatment is determined for better spinning of fibres at the production of the fabric, and weights of grams of 80, 110, 163 g m<sup>-2</sup> at the fabric with a chemical dressing determined for improving the adhesion between the fibres and the epoxy resin. The specimens for quasi-static and lap shear strength tests were made in accordance with EN 1465:2009. The difference of the saturation of the various types of fabrics with the epoxy adhesive was observed with SEM (Scanning Electron Microscopy). It is obvious from the experiment results that it came to the improvement of the quasi-static loading at all adhesive bonds reinforced with glass fibres. The adhesive bonds specimens A110, A160, B110 and B160 resisted to required 200 cycles at 80% loading. The test specimens without the fabric showed worse properties.

**Key words:** adhesive bond, low-cycle fatigue, lap-shear strength, two-component epoxy adhesive.

### INTRODUCTION

Adhesive bonds are often used in constructions exposed to a cyclic stress. An application of an adhesive bonding technology is limited by a cyclic loading of an adhesive bond (Messler, 2004; Šleger & Müller, 2015).

The adhesive bonding technology is used as a connecting element, e.g. in the constructions of bodies of agricultural machines, automobiles, trains, etc. The machine is exposed to considerable vibrations in the case of a drive of the agricultural machines at the soil processing.

The low cycle fatigue of the adhesive bond can lead to a failure of the bonds (Kelly, 2006; Hafiz et al., 2010). The low-cyclic fatigue of the adhesive bonds has to be analysed for determining limits of the given adhesive bonding technology application (Kelly, 2006; Hafiz et al., 2010).

Service conditions can often involve an exposure to the cyclic fatigue, which is probably the most destructive form of a mechanical loading. The fatigue damage is an irreversible process which can occur at relatively low stress levels due to the presence of high peel and shear stresses at the overlap edges. These stresses reduce both the static strength and the fatigue life of bonded structures (Broughton et al., 1999).

Epoxy resins are widely used polymers, which are popular due to their workability, high tensile strength and a chemical resistance. The glass fibre fabric interlayer was used for improving the tensile and the quasi-static lap shear strength of joints bonded with the epoxy adhesive.

A specific problem with adhesive applications is bad integrity of used adhesive and bonded material caused by defects in the adhesive layer and construction faults (Hafiz et al., 2010). The bond with the irregular thickness of the adhesive layer can't provide a regular deformation in the adhesive layer under the loading (Messler, 2004; Müller, 2014). This leads to a crack propagation in the adhesive layer under the cyclic loading (Messler, 2004; Hafiz et al., 2010; Müller, 2014), and causes the bond failure (especially the adhesion failure).

To ensure the regular adhesive layer thickness several methods are used, one of them is using particles (Messler, 2004; Müller, 2011; Naito et al., 2012) or fabrics as fillers. It can be expected that using the fibre fabric to make the regular adhesive layer thickness will lead to an improvement of the adhesive bond durability under the cyclic loading.

Glass fibres in a form of the fabric were used within the experiment to reach even layer of the adhesive. Mechanical properties of fibre composite materials depend on a composition of particular layers, their orientation, a specific weight and a chemical treatment (Karbhari & Abanilla, 2007; Maheri, 2010) influencing a wettability. The wettability of the surface is very important aspect of adhesive applications (Rudawska, 2014), that's why the fabrics treated by a wax and a chemical dressing were used.

## **MATERIALS AND METHODS**

The aim of the experiment is to clarify a fatigue behaviour (low-cyclic tests of the fatigue) of the structural two-component epoxy adhesive applied to a constructional steel S235J0. The aim of the research was to evaluate a service life of the adhesive bond in terms of its fatigue stressing at the quasi-static shear test. The two-component epoxy adhesive cured at the laboratory temperature  $22 \pm 2$  °C was used for bonding test samples.

The fabric was composed from the type E glass fibres in a plain weave. For an optimization of properties of the composite bond it was used various plain weights in grams of the fabric in the extent of 80, 110, 160 g m<sup>-2</sup> for the fabric treated by a wax (marked as A), where this treatment is determined for better spinning of fibres at the production of the fabric, and weights of grams of 80, 110, 160 g m<sup>-2</sup> (marked as B) at the fabric with a chemical glaze determined for improving the adhesion between the fibres

and the epoxy resin. The specimens for quasi-static and lap shear strength tests were made in accordance with ČSN EN 1465 (2009).

The surface of the adhesive bonded material was treated by a mechanical treatment – grit blasted F 80 ( $\text{Al}_2\text{O}_3$ ) and a chemical treatment. The chemical treatment was performed in a bath of acetone (dimethylketon). Acetone is used for greasing of adhesive bonded surfaces. It is a colourless liquid which is used as a dissolving agent of organic substances.

The blasting was performed in a manual blasting chamber ITB 65 with a foot control of a compressed air. On a reciprocating compressor the pressure was set to 3.5 MPa.

Roughness parameters  $R_a$  and  $R_z$  were measured on the surface of adherents designed for the bonding. Roughness parameters were measured with a portable profilometer Mitutoyo SurfTest 301. A limit wavelength cut-off was set at 0.8 mm.

After the described surface preparation method, the adhesive material was applied and the adhesive bond was loaded with a weight of  $495 \pm 5$  g under laboratory conditions with the temperature  $22 \pm 2$  °C. The lapping was according to the standard  $12.5 \pm 0.25$  mm.

One batch of samples was bonded only with the adhesive. The fabric with glass fibres was applied into the adhesive bond in other series and so called ‘composite layer’ came into being. Parameters warp and weft at used fabrics were following:  $80 \text{ g m}^{-2}$  12 x 12 cm,  $110 \text{ g m}^{-2}$  16 x 15 cm and  $160 \text{ g m}^{-2}$  12 x 12 cm.

Laboratory tests were performed using the universal tensile strength testing machine LABTest 5.50ST (a sensing unit AST type KAF 50 kN, an evaluating software Test & Motion). The failure type according to ISO 10365 was determined at the adhesive bonds.

Six test specimens were tested in each batch. The reference value of the adhesive bond strength was determined for each tested adhesive according to the standard ČSN EN 1465. The upper and lower limits for low-cyclic tests were calculated from the average value.

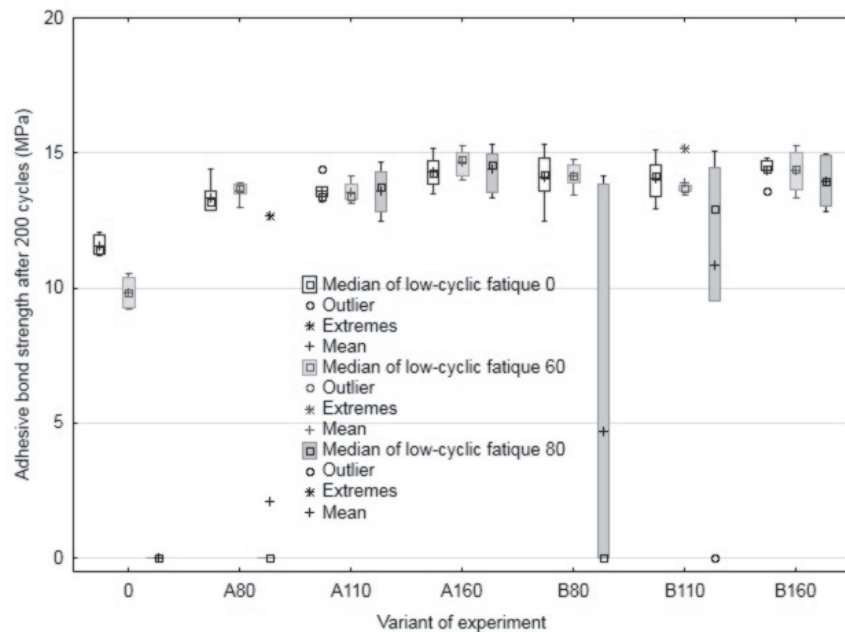
The test specimens were cyclically loaded in a such way the loading tension pulsed between the minimum value determined from the reference strength of the adhesive bond without the fabric (i.e. 5%) and chosen percentage value 60% and 80% from the reference strength of the adhesive bond without the fabric (average maximum strength values determined according to the ČSN EN 1465). The loading speed was always set at  $6 \text{ mm min}^{-1}$ . The endurance at the maximum and minimum force was 1 s. The number of cycles was 200. In a case that the failure did not occur during the cycling, the cycling was automatically stopped after 200 cycles. In the case that the adhesive bond was not destructive damaged after 200 cycles, the adhesive bond was subsequently broken, i.e. the testing machine developed a force by the speed  $6 \text{ mm min}^{-1}$  as long as the test specimens were broken.

Statistical hypotheses were also tested at measured sets of data by means of the program STATISTICA. A validity of the zero hypothesis ( $H_0$ ) shows that there is no statistically significant difference ( $p > 0.05$ ) among tested sets of data. On the contrary, the hypothesis  $H_1$  denies the zero hypothesis and it says that there is a statistically significant difference among tested sets of data or a dependence among variables ( $p < 0.05$ ).

The difference of the saturation of the various types of fabrics with the epoxy adhesive was observed with SEM (Scanning Electron Microscopy).

## RESULTS AND DISCUSSION

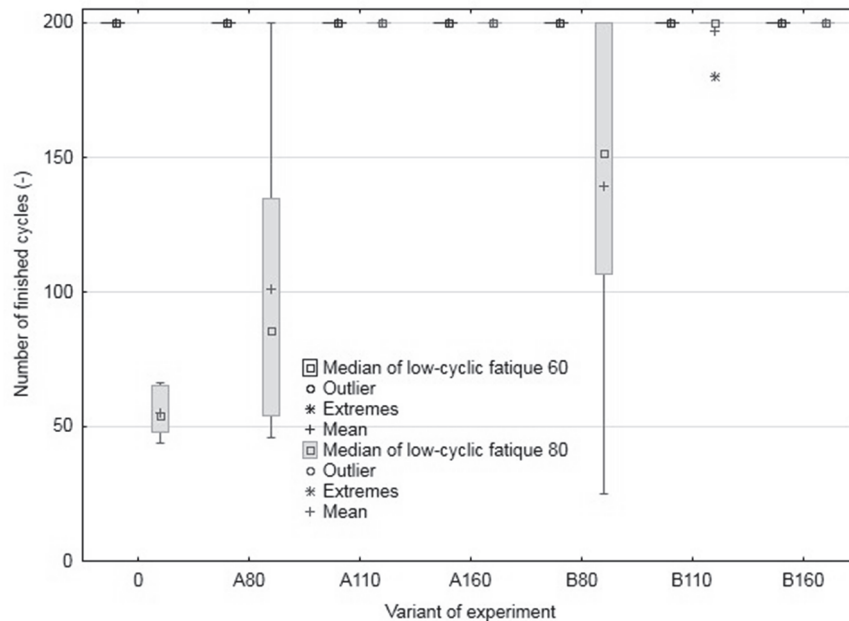
The Fig. 1 presents the strength results after the cyclic loading. The results show that examined specimens reach variable values of the static bond strength (marked as 0 cycles) in the interval 11.6 to 14.4 MPa. The adhesive bonds reinforced with various types of the fabric reach significantly better values of the static strength than the bonds without the fabric. The increase of the static strength was about 20%. More significant increase of the static strength was observed in the bonds reinforced with the fabric marked as the type B, i.e. with the chemical dressing.



**Figure 1.** Influence of low-cycle fatigue on adhesive bond strength.

Other results showed in Fig. 1 represent the values of the bond strength after the quasi-static testing, i.e. the low cycle fatigue after reaching 200 cycles. It is evident from the results that there is no significant change in the bond strength after the quasi-static testing at 60% of the reference strength. An average decrease of the bond strength was 1.3%. A significant decrease was observed at adhesive bonds without the glass fibre fabric, it is 15.5%.

The significant changes of the maximum bond strength were observed in the quasi-static testing at 80% of the reference strength. Only adhesive bonds reinforced with glass fibre fabrics of types A110, A160 and B160 reached 200 cycles (Fig. 2). Other variants of adhesive bonds were destroyed before reaching 200 cycles and the final bond strength couldn't be measured. The number of cycles absorbed by adhesive bonds is presented in Fig. 2.



**Figure 2.** Influence of low-cycle fatigue of adhesive bonds on number of finished cycles

Adhesive bonds which weren't reinforced with glass fibre fabrics were able to absorb  $55 \pm 9$  cycles of 200. The results show that adhesive bonds without fabrics badly resist to the low-cycle fatigue under the repeated stress at 80% of the reference strength.

In terms of the influence of the quasi-static testing on the bond strength, the results of ANOVA F-test are following:

The hypothesis  $H_0$  was not confirmed when comparing all variants of the low-cycle fatigue of the adhesive bond under 60% ( $p = 0.0000$ ) and 80% ( $p = 0.0000$ ) in the significant level 0.05, i.e. there is a difference among single tested variants of the adhesive bonds 0, A80, A110, A160, B80, B110, B160. It was demonstrated that the type and the plain weight of the glass fibre fabric used as the reinforcing layer has the effect on the adhesive bond strength under the quasi-static loading.

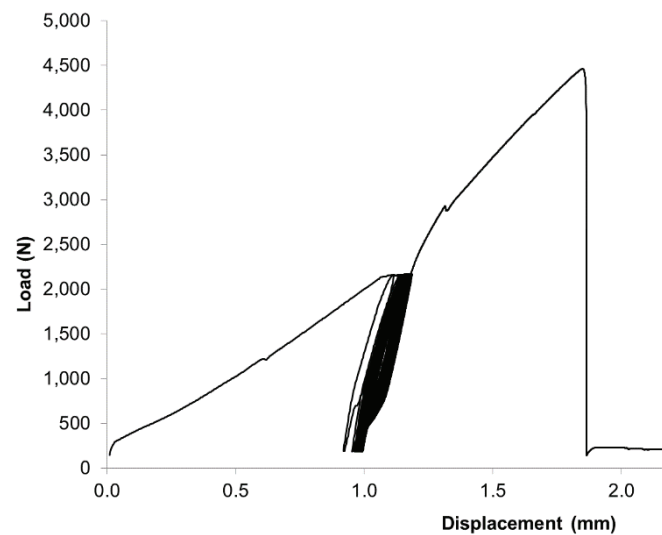
The hypothesis  $H_0$  was confirmed when comparing the low-cycle fatigue of the adhesive bond under 0, 60% and 80% for experiments A110 ( $p = 0.9722$ ), A160 ( $p = 0.6141$ ), B110 ( $p = 0.1988$ ) and B160 ( $p = 0.5072$ ), i.e. there is no difference among single tested variants in the significance level 0.05. It is not statistically proved any effect of the type and the plain weight of the glass fibre fabric used as the reinforcing layer on the low-cycle fatigue strength of the adhesive bond.

The hypothesis  $H_0$  was not confirmed for the low-cycle fatigue under 80% load, i.e. there is a difference among single tested variants 0, A80, A110, A160, B80, B110 and B160 depending on the reached number of cycles. It was demonstrated that the type and the plain weight of the glass fibre fabric used as the reinforcing layer has the effect on the adhesive bond strength under the quasi-static loading in the significance level 0.05.

The hypothesis  $H_0$  was confirmed for the low-cycle fatigue at 60% load, i.e. there is no difference among single tested variants 0, A80, A110, A160, B80, B110 and B160 depending on the reached number of cycles. It is not statistically proved any effect of the

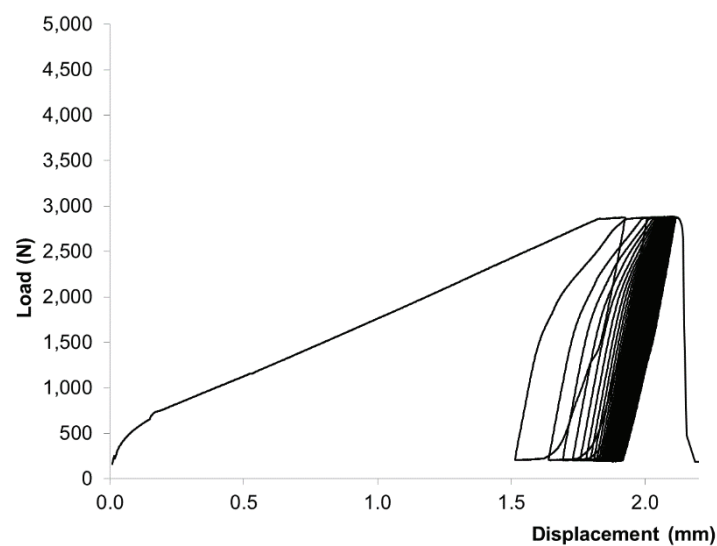
type or the plain weight of the glass fibre fabric on the low-cycle fatigue strength of the adhesive bond.

The testing process is presented on the diagrams below. Fig. 3 represents the quasi-static testing at 60% of the reference strength of the adhesive bond. If the test reaches 200 cycles, the bond is then destroyed by the static loading.



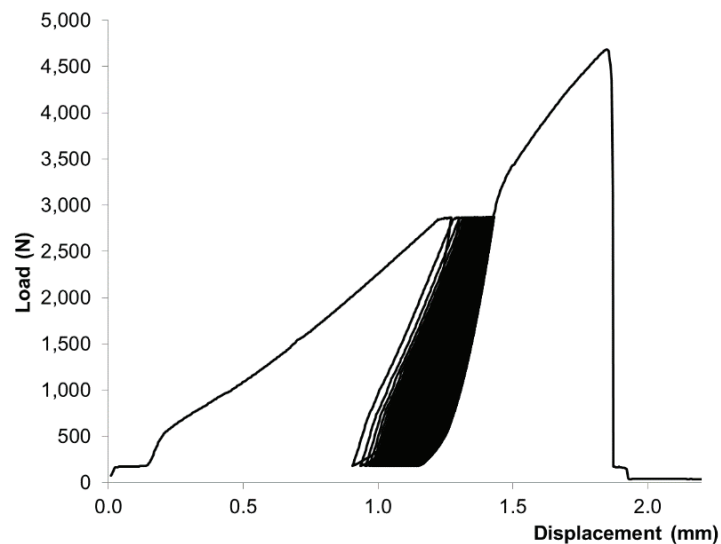
**Figure 3.** Quasi-static testing (60%, A110, 200 cycles).

Fig. 4 shows the typical quasi-static testing proces at 80% load of the reference strength before reaching 200 cycles, i.e. the bond was destroyed before reaching 200 cycles.



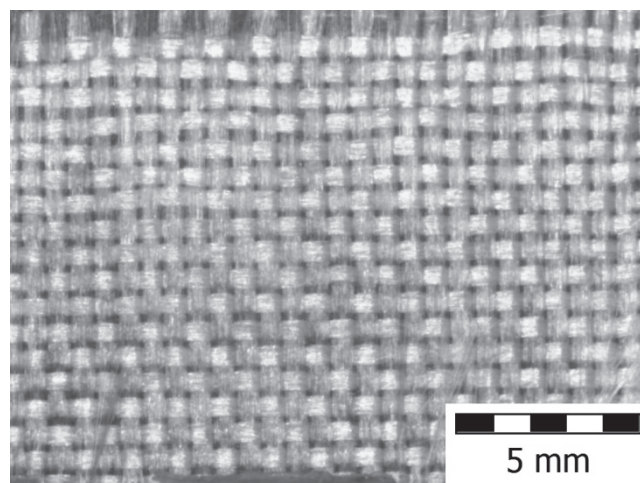
**Figure 4.** Quasi-static testing (80%, A80, 46 cycles).

Fig. 5 shows typical quasi-static testing proces at 80% load of the reference strength, i.e. the bond reached 200 cycles.



**Figure 5.** Quasi-static testing (80%, A110, 200 cycles).

The effect of the quasi-static loading on the fracture surface wasn't proved. Only the adhesion type of the fracture surface was observed (Fig. 6). The glass fibre fabric wetted with the adhesive is visible in Fig. 6. This layer was divided from the adhesive bonded material.



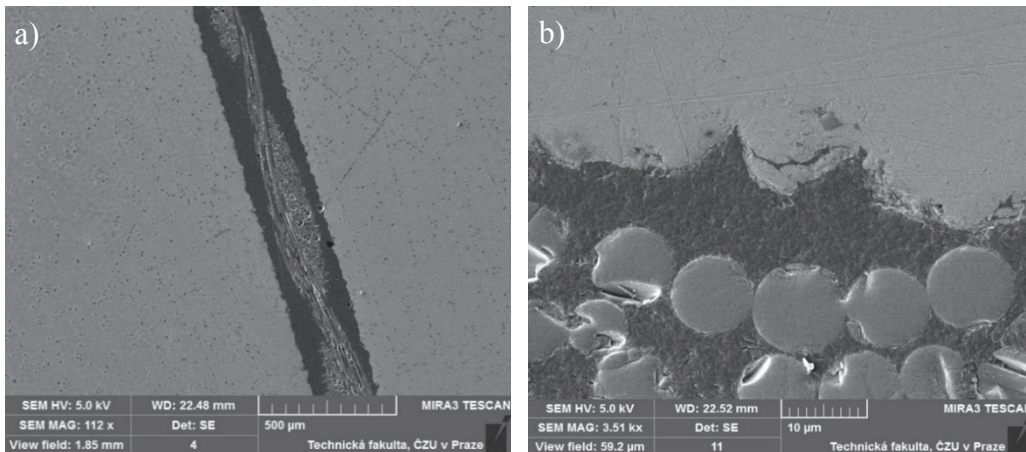
**Figure 6.** Typical fracture surface – cohesive type of failure of adhesive bond.

The results show that the quasi-static loading with high loads (Broughton et al., 1999; Šleger & Müller, 2015) (f.e. 80% of the reference strength) can lead to early failure of the adhesive bond at low number of cycles. The reason is a cumulative effect of the



cyclic shear stress. The experiment results proved that this cumulative effect can be reduced using the glass fibre fabric layer in the adhesive bond.

Fig. 7 (a, b) show a cut through the composite adhesive bond with the use of the electron microscope Tescan Mira 3. Fig. 7a presents a layout of the glass fibre layer in the adhesive bond cut. They are crossed fibres. Using the electron microscopy within the experimental research a good interaction of the glass fibres with the matrix in the form of the epoxy was proved.



**Figure 7.** SEM images of cut of composite adhesive bond – type B160.

It was proved that the adhesive wetted the inserted layer of the glass fibre fabric. The failure of the cohesiveness between the adhesive and the reinforcement which is notified by Ashcroft et al. (Ashcroft et al., 2001) in their research was not confirmed within the research.

Fig. 7a shows the regular thickness of the adhesive layer in the bond, the violence of the measured thickness was only 6% from the average value 185 µm. The layer thickness at adhesive bonds without the glass fibre fabric interlayer wasn't regular. The ireegular layer of the adhesive decreases the strength of the adhesive bond (Kotousov, 2007; Grant et al., 2009; Müller, 2014).

## CONCLUSIONS

The experiment results proved the improvement of the bond strength under the quasi-static loading at all adhesive bonds reinforced with the glass fibre fabric. Adhesive bonds reinforced with the glass fibre fabric of types A110, A160, B110 (197 cycles) and B160 succesfully reached 200 cycles under 80% load. The test specimens without the fabric showed worse properties. Adhesive bonds reinforced with the glass fibre fabric of the type A80 resisted to  $101 \pm 53$  cycles and B80 resisted to  $139 \pm 61$  cycles of the quasi-static loading under 80% load. The test specimens without the fabric resisted to only  $55 \pm 9$  cycles of the quasi-static loading under 80% load.

Good interaction between the glass fibres and the epoxy adhesive matrix was confirmed based on conclusions from the mechanical testing and microscopy results.



The effect of various plain weights of the glass fibre fabric on the quasi-static strength of adhesive bonds wasn't confirmed.

A use of the interlayer from the glass fibre fabrics increases the endurance to the low-cycle fatigue comparing to the normal adhesive bond. The glass fibre fabric interlayer acts against the crack propagation in the adhesive bond. It can be assumed that the fabric geometry and the plain weight have the effect on blocking of the crack propagation too.

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