High-strength mechanical joints under combined mechanical and medial loads

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Abstract. This paper introduces a test method for joined hot-dip galvanized steelwork test specimens. The specimens joined by means of a lock bolt system were subjected to a simultaneously combined mechanical and medial loading. This was achieved by combining a vibration testing machine and a condensation chamber that was expanded by an electrolyte dosing unit. The electrolyte was dosed on one side of the specimen in the area around the lock bolt head. The zinc dissolution is locally accelerated in the area around the joining element possibly due to superimposed bimetal and crevice corrosion. In addition, the electrolyte enters the space between the joined steel sheets and initiates corrosion processes there, too. Besides the expected zinc ions, potassium ions were also detected in the collected electrolyte by means of elemental analysis. These are originally part of the friction-increasing coating.

1. Introduction

The ambition to increase efficiency in the field of wind energy leads to the construction of wind turbines with large hub heights. The most common tower concept are currently steel tube towers which are erected in segmental construction methods. However, in regard to large hub heights transporting the segments (diameter > 4,2 m) is a significant problem considering infrastructure [1]. In consequence, concrete-steel hybrid towers are used as an alternative on the one hand. On the other hand, design concepts based on a separated steelwork supporting structure, such as lattice towers or lengthwise separated steel tube towers are tested (cf. figure 1). These usually consist of bar profiles or shell profiles and are assembled on-site by means of mechanical joining technology, using high-tensile prestressed bolts or lockbolts, for example. The new construction methods reduce costs for transport and at the same time offer the possibility of saving material. Contrariwise the assembly costs on-site increase. The semi-finished parts are typically batch-galvanized [2], specifying the assembly costs on-site increase. The semi-finished parts are typically batch-galvanized [2], specifying the corrosion resistance by means of the zinc-film thicknesses. According to DIN EN 1090-2 [3] an alkali silicate zinc-dust coating is applied [4]. This leads to an increase in the coefficient of friction and a better corrosion protection for the slip-resistant prestressed joints. Typically, it is directly applied before joining and the joints themselves are not overcoated.
High-tensile prestressed bolts or lockbolt systems are used as joining elements. The bolts are usually hot-dip galvanized, as is the case with the semi-finished parts, and have an average film thickness of 50 µm in accordance with DIN EN 10684 [5]. Lockbolt joints have metallic coatings as well as organic coatings [6]. The durability of these protection systems is determined by means of laboratory test methods, such as the neutral salt spray test. Thus, for both corrosion protection systems sufficient durability for steelwork applications is theoretically verified. In regard to dynamically loaded constructions such as wind turbines the joints are executed as slip-resistant prestressed joints [7]. From an economic point of view it is crucial for such supporting structures to reach sufficient service life after construction. Due to increased loading a maximum of 20 years is the basis for calculation regarding wind turbines [8]. These service lives can only be accomplished if the joints resist the combined mechanical and medial loads on-site and a quantitative estimation of the effects of combined mechanical and medial loads can currently be done exclusively by means of mere conservative inspections of real constructions or prolonged natural weathering tests.

Figure 1. Examples for wind turbine supporting structures. Left: Lattice tower, Butzkies Stahlbau GmbH. Center: Lattice tower, Stieblich Hallenbau GmbH. Right: Steel tube tower separated lengthwise, SENVION S.A.

Figure 2. Effect of combined loads on mechanical joints after 5 years of operation [Source: construction inspection on 2016-10-25; wind turbine by the Butzkies Stahlbau GmbH in Neuenbrook]
The planning periods to be concluded from this (> 5 years) are not practice-oriented. For the estimation of the effect of combined mechanical and medial loads on the functional capability of mechanical joints in steel constructions there is currently no validated laboratory test method available. Construction inspections (as illustrated in figure 2) show, that despite nominally high corrosion protection durability (> 15 years) of the individual joint partners (semi-finished parts, anti-friction surface, joining element) already after a few years significant corrosion occurs on the assembled components. In accordance with the current state of the art no laboratory loading regimen on combined mechanical and medial loads for high-strength joints in steel constructions exists. Previously performed laboratory loading cycles refer to automotive and railway vehicle engineering, e.g. [9–13]. During these examinations three different test procedures with the following configurations had been carried out:

1. Pre-corrosion and subsequent mechanical loading
2. Alternating medial and mechanical loading
3. Simultaneous medial and mechanical loading

Only in test configuration 3 it is possible for gaps to occur, leading to forced electrolyte entry and, consequently, promoting corrosion processes on the joints in a practical manner. Until the electrolyte is dried this local continuous moisture load leads to significant corrosion increase [14]. It is to be emphasized that regarding simultaneous medial and mechanical loads a low test frequency shall be set, since the corrosion processes are dependent on time [11] and take place slowly. The consequences of test frequencies that are too high during combined loading would be corrosion influences that are too low. In addition, moisture entering crevices during test frequencies that are too high can lead to a modified tension distribution (local pressure increases due to electrolyte concentrations) in the contact area of the joint. As a consequence, test specimens prematurely fail during laboratory testing and, thus, real conditions cannot be reproduced.

This paper presents the first developing steps of a laboratory test method for simultaneous medial and mechanical loading of steelwork test specimens. This method is intended to help with the practice-oriented design of corrosion protection systems and their application for high-strength mechanical joints for supporting structures of wind turbines. During a test period of 3 weeks a (locally limited) corrosion corresponding to a duration of 5 years in corrosivity category C4 [15] is to be accomplished.

2. Experimental

2.1. Test chamber for combined loading

The combined mechanical and medial loading tests have been carried out in a test installation by the Institute for Corrosion Protection Dresden GmbH (IKS). The installation is a special construction consisting of a 100 kN horizontal vibration test device (DynaMess, Aachen) with hydrostatically supported test cylinder and a condensation-water chamber S450-S (Weisstechnik). In order to achieve the high corrosion requirements mentioned above, the installation had to be extended by a salt dosing unit. Since due to installation-specific reasons the application of salt spray is not possible, it was specified to drip the electrolyte onto the surface of the (inclined) test specimen by means of a cannula during the vibration test in condensation-water atmosphere. An approach with similar electrolyte placing is described in [16]. The build-up of the complete installation is given in figure 3. The electrolyte is conveyed onto the test specimen from a storage container outside the chamber through a tube and a cannula. The volumetric flow of the salt solution is controlled with a dosing valve. During the load tests the electrolyte solution dripping from the test specimen is conveyed into a collection container by means of a funnel.

2.2. Preparation of test specimens

The geometry of the test specimen is given in figure 4 and is following the standard DIN EN 1090-2 [3]. In the underlying research project these test specimens are used for the validation of the test system as well as for the determination of the failure behavior under combined loading. Due to the limited
dimensions of the test chamber an implementation conforming to the standard to 100 % was not possible at the IKS.

![Test Chamber of the IKS](image)

**Figure 3.** Test chamber of the IKS for the combined mechanical and medial loading (special construction consisting of 100 kN horizontal vibration tester, condensation-water chamber and electrolyte dosing unit) and alignment of the test specimen. [Image source: IKS]

The test specimens consist of 3 semi-finished parts with the substrate S355J2+N, which is hot-dip galvanized. The surfaces are mechanically pre-treated at the joints and have an alkali silicate zinc dust coating (Interzinc 697). The semi-finished parts are slip-resistant prestressed and are joined with a joining element. Latter consists of a lockbolts of the type MBT-DT16-35J45 (substrate non-galvanized steel + two-coat corrosion protection system) as well as a locking ring of the type MBTC-R16J46 (substrate hot-dip galvanized steel, single-coat corrosion protection system). The prestress was 116 kN. An industrial partner manufactured the semi-finished parts and applied the alkali silicate zinc-dust system. The preparation of the joining elements for the prestress determination (during mechanical loading) and the joining of all joints was done at the Fraunhofer IGP.

![Reduced Geometry](image)

**Figure 4.** Reduced geometry (side view) of the test specimens following DIN EN ISO 1090-2. 3 semi-finished parts (80 mm with each) slip-resistant prestressed by a lockbolt system.
2.3. Parameters of the mechanical load
In 2015 the Fraunhofer IGP implemented a comprehensive monitoring of the developed maintenance-free wind turbine lattice tower [17]. The positions of the acceleration sensors and the used DMS bolts, serving the exact absorption of load spectra, are given in figure 5. The detailed analysis of the real mechanical load is used as the basis for the combined laboratory loading at IKS.

The frequencies of the first bending vibrations of the installed acceleration sensors are roughly about 1 Hz. The frequency for the vibrating mechanical loading of the test specimens results from this. The other parameters of the mechanical load were estimated on the basis of studies on current calculation models [18] as well as publications on prestressed steel construction connections [19]. As amplitude of the mechanical load a highest load of 25 kN and an R-correlation of 0,1 has been specified [20]. Within the elaborated loading cycle of 3 weeks $10^6$ load changes (LC) are pursued.

2.4. Parameters of the medial load
The atmospheric zinc removal over a period of 5 years in corrosivity category C4 can be estimated with the help of the correlations and calculation models, which are explained in more detail in [15, 21]. Depending on which model is used, the zinc removal is about $11,7 \pm 3,9 \mu m$. In order to estimate the amount of salt solution to drip onto the test specimen in one location within 3 weeks, a strip-galvanized steel sheet (Z100; zinc film approx. 14 µm), inclined by $15^\circ$ to the vertical, has been exposed to a condensation-water atmosphere (relative humidity $\approx 99 \%$, $9 = 40 \pm 3 \, ^{\circ}C$) with a 5% by mass NaCl solution (the same is used for neutral salt spray tests). The electrolyte consumption was specified to $25 \pm 1 \, ml$ per hour. After 24 hours the $14 \mu m$ thick zinc film was removed and red-rust formation occurred. Consequently, it was detected that the 24-hour loading is above the intended value. For this reason, at first, a total duration of salt exposure of 15 h was specified, corresponding to a local minimum zinc removal of $8,75 \mu m$. This value lies at the bottom of the above-mentioned range. The salt has been dosed for 1 h during each of the 15 days of loading.

2.5. Combined loading
The mechanical and medial test parameters determined in 2.3. and 2.4. were now combined in a test program. It was specified that in each of the 3 test weeks the test specimens are exposed to condensation-water alternating climate (“AHT” in accordance with [22]; 8h relative humidity $\approx 100 \%$ and $40 \pm 3^{\circ}C$
and 16 h drying at 18-28°C and ambient humidity) for approx. 104 hours. During this time the vibration stress is applied simultaneously (f = 1 Hz; R = 0.1; F = 25 kN; approx. 374000 LC each week). In order to accomplish the required total LC (>1x10^6), an overall loading duration of 3 cycles (= 3 weeks) is required. The dosing of the NaCl solution was distributed to 3x5 days and was carried out in each case in the middle of the damp phases for one hour on the bolt side of the test specimen.

![Figure 6](image-url)  
**Figure 6.** Upper: Sequence of the combined test program during a one-week cycle. Lower: Measured values of atmospheric temperature and humidity as well as specimen temperature during a day cycle. The electrolyte dripping from the test specimen was conveyed into a collection container and filled up to a volume of 50 ml using deionized water. Subsequently, these solutions have been acidified and analyzed regarding their cation content.

A one-week loading cycle is given in the upper part of figure 6, the alignment of the test specimens can be taken from figure 3. The lower part of Figure 6 shows the development of the chamber temperature and humidity as well as the temperature of the sample over a 24-hour cycle. It has to be mentioned, that the sample’s temperature profile does differ from that of the atmosphere. In the beginning the chamber heats up a little faster. The humidity rises very quick so that the dew point is reached after around 30 min. Returning to room clime after 8 h one can observe that the sample cools down very slowly and falls below the required temperature of 28°C after 4 hours.

### 2.6. Characterization methods

A digital camera Canon EOS 2000D and a flatbed scanner EPSON PERFECTION 4990 PHOTO were used for the macroscopic images. The light-microscopic images were recorded using an inverted light-microscope Zeiss Axio Observer.Z1M and a stereomicroscope Zeiss Discovery.V20. Further, the 3D laser-scanning microscope Keyence VK-X130 K was used. The FE-SEM images and EDX analyses were recorded with a field-emission electron microscope Zeiss SIGMA VP with 2 XFlash 5030 EDX detectors (Bruker Nano). For the chemical analyses a Varian 725-ES optical emission spectrometer was available.
3. Results and discussion

3.1. Condition of the test specimens and the joining elements after the end of stress application

Figure 7 exemplifies a test specimen prior to and after combined mechanical and medial loading for 3 cycles (= 3 weeks). The run-off marks of the salt solution are clearly visible, as well as the deposition and corrosion products formed in these locations. Only a rather small percentage of the sheet surface is affected by corrosion due to the locally limited electrolyte deposition; however, it is clearly visible that the drained off NaCl solution distributes over the underside of the test specimen and almost entirely wets it.

[Image of test specimen before and after stress application]

After severing the bolt, the test specimen was disassembled. It was observed that parts of the double layer bolt coating remained adhered to the galvanized steel surface of the sheet after disassembling the test specimen (figure 8 top right section). This was not the case for a merely joined but unloaded test specimen (not shown), meaning that the humidity during the test affects the adhering phenomenon. In the area around the removed bolt head, where the electrolyte flowed by, circular accumulations of corrosion products are clearly visible, which are mostly separated from one another. On the joining elements (figure 8 bottom section) macroscopic corrosion could not be found. The cause for the heterogeneous corrosion around the bolt head is not clear yet. We suspect that sporadic coating defects in the edge area of the bolt head may result in local bimetal corrosion, which would lead to a locally limited but accelerated zinc dissolution.
Figure 9 shows such a point. In the area close to the edge the base coating (phosphorus-free, zinc-containing) is missing and the thickness of the top coating (phosphorus-containing, zinc-free) is rather small. The thin Zn and P-containing layer on the bolt surface corresponds to the zinc phosphating. The base coating typically contains certain corrosion protection pigments. If this coating is not present the corrosion protection efficiency is reduced.

It was then examined whether the bolt coating or the crevice were infiltrated by the electrolyte. For this purpose, a cross-section of a metal sheet with adhering bolt coating was analyzed in the FE-SEM-EDX after the loading test and disassembling. The results are shown in figure 10. From left to right the images are arranged in the following way: Outer area with the circular accumulations of corrosion products; Crevice area under the bolt; Crevice end with a little indent in the galvanization caused by the bolt; Area under the bolt. The qualitative EDX maps of the elements zinc, oxygen, chlorine and phosphorous are shown below the FE-SEM images.
In the outer area mainly loose corrosion products were identified and, as one could expect, mainly zinc and oxygen were detected; the contrast in the maps of chlorine and phosphorous are very weak. In case of chlorine a very little intensification is noticeable in the region of the corrosion front. In the crevice, the chlorine map comes to a higher contrast meaning that Chlorine has penetrated in that zone. A noticeably attack on the zinc surface can also be seen in that images. In the transition area containing the bolt indentation, no attack on the zinc surface was detectable and the chlorine signals drop to the background noise level. In the phosphorous map the signal intensities increase in the area corresponding to the adherent (phosphorous containing) top coat of the bolt head. In the inner area, the galvanizing was affected by weak corrosion in some places; However, chlorine could not be detected there. Some parts of the top coat were found again. In summary, we suggest that bimetal and crevice corrosion are superimposed in the area around the bolt head.

**Figure 10.** Cross section FE-SEM-EDX-Analyses in the region with increased zinc corrosion (cf. Figure 8). From left to right: Outer area; Crevice area; Crevice end with bolt indentation; Area under the bolt. Fields of view [µm]: 435 x 300, 435 x 300, 210 x 145, 360 x 250, resp. Upper line: FE-SEM images (HV: 15 kV). Red, Blue, Purple and Green: Zn-Kα, O-Kα, Cl-Kα and P-Kα EDX intensities.
Since the locking ring side was not loaded by the electrolyte and was only exposed to humidity, we will discuss it only in a short way. Due to a typically occurring weak deformation of the locking ring flange during the bolting process a small crevice is formed, in which condensation water seeps into. This entry is limited to a depth of around 3.5 mm with the present flange width of approx. 7 mm. The opening angle of the crevice is approx. 1.5° (see figure 11). This value was determined by evaluating 2 profiles from 3D laser-scanning microscope images taken from opposite parts on the locking ring flange. Macroscopically, there was only a slight and facile corrosion of the galvanized layer detectable.

![Inclination profiles of a locking ring flange determined by means of laser-scanning microscopy. Dimensions [µm]: Left: 6286.8 x 205.3, Right: 6288.2 x 183.2.](image1)

**Figure 11.** Inclination profiles of a locking ring flange determined by means of laser-scanning microscopy. Dimensions [µm]: Left: 6286.8 x 205.3, Right: 6288.2 x 183.2.

3.2. Condition of the inner interfaces and of the alkali silicate zinc-dust coating after the end of stress application

After disassembling the test specimens, it is noticeable that the electrolyte enters the spaces between the joined sheet stack (figure 12) starting from the bottom edge (framed in red) and rising at the sides (framed in blue). This occurs mainly due to the capillary effect of gap between the layers. In addition, the quickly drying alkali silicate zinc-dust coatings were applied by means of a brush, resulting in a wavelike texture. Due to this, small channels form in the intermediate areas, promoting entry of the salt solution. A further reason is the formation of cracks in the layers, since the reference value of 75 µm dry film thickness specified in the manufacturer’s data sheet is only difficult to match under practical conditions. The resulting cracks should further increase the entry of the electrolyte.

![View of the inner contact surfaces of two sheets (left: middle sheet, right: outer sheet of the specimen) after 3 loading cycles. Parts of the electrolyte enter the space between the sheets. In the areas where the coating was partially pulled off, a dark discoloration occurs. These can also be found in the opposite areas with adherent coating.](image2)

**Figure 12.** View of the inner contact surfaces of two sheets (left: middle sheet, right: outer sheet of the specimen) after 3 loading cycles. Parts of the electrolyte enter the space between the sheets. In the areas where the coating was partially pulled off, a dark discoloration occurs. These can also be found in the opposite areas with adherent coating.

Furthermore, it is noticeable that the coating pulls off of the outer sheets. The pulled-off parts largely adhere to the opposite side of the center sheet. In locations in which the coating pulls off, the galvanized surface often shows dark discoloration. Such a dark discoloration was analyzed by means of FE-SEM-
EDX, both in top view (figure 13) as well as on the cross-section (figure 14). From the analysis we can derive that an attack occurred on the galvanized zinc layer. A local oxygen content of approx. 27 percent by mass could be verified. Because the mass fraction of oxygen in pure zinc oxide is only approx. 20% and the concentration of other metals was found to be comparably low, the formation of zinc hydroxides appears plausible.

![Figure 13](image)

**Figure 13.** Left: FE-SEM-EDX analysis on a dark region with delaminated alkali silicate zinc-dust coating. The table below shows the results of the EDX-Quantification for 3 surface segments (marked by colors) given in percent by mass. Right: The elements O, Zn and Si are distributed very heterogeneously.

| C  | O  | Al | Si | Fe | Zn | Pb |
|----|----|----|----|----|----|----|
| 2,7| 7,2| 0,3| 0,2| 0,6| 87,7| 1,2|
| 3,8| 26,9| 0,2| 0,9| 0,6| 61,1| 0,6|
| 2,3| 15,9| 0,2| 3,9| 1,5| 76,0| 0,2|

From the cross-section analysis it was deduced that the attack on the zinc layer reaches down up to a depth of 20 µm (lower Zn Kα and increased O Kα signal intensities). On the other hand, the analysis finds that the attack can also occur beneath the applied alkali silicate zinc-dust coating. This is most likely possible because of the formation of little gaps due to a decreased adhesion between the coating and the surface of the galvanized steel.

![Figure 14](image)

**Figure 14.** SEM-EDX images on the cross-section of an area, in which the zinc coating was attacked. The area with the sphere-shaped formations is the zinc-dust coating. Corrosion attack occurred beneath it, reaching in up to a depth of approx. 25 µm.
3.3. Element analysis of the collected electrolyte dripping from the test specimen

During loading a test specimen, the electrolyte was conveyed into a collection container by means of a funnel. The collected solution was filled up to a volume of 50 ml using deionized water and was acidified using 1 % nitric acid, in order to stabilize it. After completion of the test program all test specimens of the batch have been analyzed by means of ICP-OES [23]. An exemplified curve of the concentrations of the elements K, Zn and Fe is given in figure 15.

![Figure 15. Results of the element analyses by means of ICP-OES.](image)

It was found that a comparatively high release of potassium occurs at the beginning of the test series. This can either be caused by flushing of the excess potassium silicate or by its decomposition. Even on day 15 potassium has the highest concentration of the 3 diagrammed elements. The value of the potassium concentration decreases from initially approx. 180 mg/l to approx. 17 mg/l towards the end of the test. The discharge of zinc is almost constant, the concentration varies between 2 and 12 mg/l. The concentration values of iron are 2 magnitudes below that and are, in consequence, close to the detection limit. We consider the release of the potassium ions as another proof of the electrolyte activity in the spaces between the joined sheets.

4. Summary and conclusion

In this paper we describe the development of a test method allowing for combined mechanical and medial tests on practice-oriented, joined steelwork test specimens. This was accomplished by combining a vibration test in a closed chamber atmosphere with alternating humidity. During the damp phases an electrolyte solution was specifically placed on the area around the bolt head of the joining element. This method enables the detection of weak points in the joined structure, in the material selection (e.g., zinc coating, joining elements) as well as in the preparation process.

The first results indicate a heterogeneous corrosion in the area around the edges of the bolt head, if the electrolyte is dosed on the bolt head side of the sample. This behavior we attribute to a superposition of crevice corrosion with bimetallic corrosion due to local coating defects of the bolt head.

We further found out that the electrolyte enters the spaces between the joined sheet stack mainly caused by the capillary effect of the layers. The reason for that we see in the wavelike texture and the micro cracking coming from the brush application and the high coating thickness. We discovered that the penetrating electrolyte leads to a corrosion of the galvanized surface. Further evidence of electrolyte penetration was derived from the result of the chemical analysis of the electrolyte that dripped from the sample. From the beginning of the experiment potassium is released. This element originates from the zinc dust coating which is only applied to the inner surfaces of the galvanized steel sheets.

Future tasks will be the medial loading on a larger area on both sides of the test specimen (bolt head and locking ring) in order to enable a more realistic corrosive attack over the whole sample. Examinations are already conducted in this regard. Other focal points are the preparation and loading of test specimens, on which the alkali silicate zinc-dust coating has been applied by means of spraying and/or by rolling, and the determination of slip factors.
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