Mechanical Properties and Fracture Analysis of High Strength Bolts in Nuclear Power Plant

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Abstract. A high-strength bolt of a steel structure of a nuclear power plant failed and fell off. In order to determine the cause of the bolt's fracture, a series of tests such as macro inspection, physical and chemical performance test, and microscopic analysis of the fracture were carried out on the failed bolt. The results show that the cause of bolt fracture is hydrogen embrittlement. Hydrogen infiltrated into the bolt during the phosphating process and the corrosion process during use, and then hydrogen continued to accumulate eventually causing hydrogen embrittlement fracture. Dehydrogenation treatment should be performed immediately after the phosphorylation treatment, and the anti-corrosion method of the bolt should be optimized, which is an effective measure to prevent the hydrogen embrittlement of high strength bolts.

Keywords: ML20MnTiB, High strength bolt, Hydrogen embrittlement, Corrosion, Fracture.

1. Introduction
Connecting with high-strength bolts has the advantages of short construction working period, higher quality compared with welding and convenient to operate [1]. It is widely used in the connection of various steel structures in nuclear power plants [2]. In recent years, there have been many failure cases of high-strength bolts of the steel structures in nuclear power plants, including high-strength bolts of gantry steel structure installed in the open-air and high-strength bolts of roof truss installed indoors [3-4]. The fracture of high-strength steel bolts not only affects the stability and safety of the steel structure, but also has a great risk of falling off and hurting people.

In this case, high-strength bolts of a gantry steel structure in nuclear power plant had serviced 5 years and failed. The material of the failed bolts is ML20MnTiB structural alloy steel, which has excellent cold deformation performance, high strength and great toughness [5]. The specification of the failed bolts is M24 and the strength grade is 10.9S grade. Fabrication process included acid washing the round steel, cold drawing, cold heading, thread machining, heat treatment, phosphorylation treatment, packaging and storage. A certain number of bolts on the gantry site were found to failed or missing, as is shown in Figure 1. In case of the bolt's fracture, macroscopic inspection, chemical composition analysis, hardness test, metallographic analysis and scanning electron microscope analysis were used to analyze the cause of the fracture and preventive measures would be proposed.
2. Observation results

2.1. Visual inspection
A total of 4 bolts were missing at the site. One of the bolts was relatively intact and selected for analysis. The photo of the failed bolt is shown in Figure 2. The surface and fracture of the failed bolt was severely corroded. The total length of the bolt is about 116mm and the fracture surface is located at the bottom of the first thread from the polished rod side. The main section is about 64mm away from the supporting surface of the bolt head. The fracture surface was flush and there was no obvious plastic deformation. Obvious extended radiation ridge features can be seen on the propagation zone, and the radiation converges on the other side of the fracture surface, indicating that this area is the initiation zone of cracks.

2.2. Chemical analysis
The chemical contents of the failed bolt are given in Table 1. It is found that the chemical analysis of specimen agrees with the specification of ML20MnTiB in the standard GB/T 6478-2015 “Steel for cold heading and cold extruding”.

![Figure 1. Photo of bolt missing position](image1)

![Figure 2. Macroscopic photos of fracture bolt](image2)
Table 1. Chemical composition of valve disc and valve seat

|        | C   | Si   | Mn   | P   | S   | Ti   | B   |
|--------|-----|------|------|-----|-----|------|-----|
| Failed bolt | 0.21 | 0.28 | 1.47 | <0.010 | <0.010 | 0.051 | 0.002 |
| Standard value | 0.18–0.23 | 0.10–0.30 | 1.30–1.60 | ≤0.025 | ≤0.025 | 0.04–0.10 | 0.0008–0.0035 |

2.3. Mechanical properties

The Rockwell hardness test and tensile performance test at room temperature results performed on the failed bolt are shown in Table 2 and Table 3. The results show that the Rockwell hardness value and room temperature tensile properties of the bolt sampling meet the standard GB/T 3632-2008 “Sets of torshear type high strength bolt hexagon nut and plain washer for steel structures” requirements for 10.9S grade bolts.

Table 2. Results of the hardness test

| Specimen       | Tested position               | Test values /HRC | Average value |
|----------------|-------------------------------|------------------|---------------|
| Failed bolt    | Within 1/2 radius of cross section | 34.5             | 34.5          | 34.0 | 34.5 |
| Standard value (10.9S) |                           |                  | 33–39         |

Table 3. Results of the tensile test

| Specimen       | Yield strength Rm (MPa) | Tensile strength Rp0.2 (MPa) | Elongation, min, δ (%) | Reduction of area, min, ψ (%) |
|----------------|-------------------------|-------------------------------|------------------------|-------------------------------|
| Failed bolt    | 1151                    | 1077                          | 15.5                   | 62.5                          |
| Standard value (10.9S) | 1040–1240                  | ≥940                         | ≥10                    | ≥42                           |

2.4. Microstructure examination

The metallographic photo of the specimen of the failed bolt is shown in Figure 3. The results show that the microstructure of the fractured bolt is tempered martensitic and there are no obvious features of decarburization or carburization on the surface of the thread. The thread processing quality is evaluated as good and there is no obvious folding or other processing defects. Besides, there are numbers of corrosion pits on the surface and fracture surface of the bolt and there is no secondary crack near the fracture surface.

![Microstructure of the surface cross-section specimen](image1)

![Microstructure of the internal cross-section specimen](image2)
(c) Microstructure of the fracture surface of the longitudinal-section specimen
d(d) Microstructure of thread bottom of the longitudinal-section specimen

Figure 3. Microstructure metallography at different positions

2.5. SEM and EDS analysis

Figure 4. SEM and EDS on the corrosion products of bolt fracture surface

The SEM and EDS analysis results of the bolt show that there are many corrosion products on the fracture surface. The original fracture surface is covered by corrosion products so that it is difficult to
observe. The corrosion products are mainly Fe and O elements and a small number of corrosive elements such as Cl. After chemical cleaning of the corrosion products on the fracture surface, typical intergranular cracking characteristics can be observed microscopically in the initiation zone and the propagation zone of the fracture. The characteristics of quasi-cleavage and a small number of dimples can be observed in the rapid expansion zone. Finally, typical dimple features can be observed in the termination zone.

Figure 5. Micro morphology of bolt fracture after chemical cleaning

3. Discussion
According to the macro and micro characteristics of the failed bolt, the bolt’s surface and fracture are covered with plenty of corrosion products, the corrosion products are mainly oxides of Fe, and there are a small number of corrosive elements such as Cl in partial areas. Mean well, the macroscopic observation to the fracture surface showed that it was relatively flat without obvious plastic deformation, and the characteristics of extended radial ridges were visible. The microscopic manifestations were mainly: cracking along the grain boundary → expanding along the grain boundary → expanding with the characteristics of intergranular cracks and a small number of dimples expanding with the characteristics
of quasi-cleavage and a small amount of dimple → termination with dimple. No secondary cracks were observed in the metallography of the fracture surface. Based on the analysis of the fracture characteristics, the fracture bolt does not have the typical characteristics of fatigue fracture, overload fracture or stress corrosion fracture and it is more consistent with the fracture characteristics of hydrogen embrittlement cracking [6-7].

Based on the physical and chemical analysis results of the fractured bolt, chemical composition, Rockwell hardness and room temperature tensile properties all meet the standard requirements and the microstructure and processing quality are normal. In various microstructures, the general order of sensitivity to hydrogen embrittlement from large to small is martensite, upper bainite (coarse bainite), lower bainite (fine bainite), sorbite, pearlite, and austenite. [8] While the microstructure of the fractured bolt is tempered martensite with less slip system in the structure and more sensitive to hydrogen embrittlement.

Regarding the source of hydrogen, it can be analysed from the processing technology, service environment and macro characteristics of the failed bolt. On the one hand, the bolt underwent phosphorylation. During being phosphate, iron in the anode area dissolved into Fe²⁺ and released electrons at the same time, and a great amount of hydrogen was released in the cathode area. Hydrogen was formed during this process and part of the hydrogen atomic was absorbed by the material. Dehydrogenation treatment was not performed after phosphorylation, so the infiltrated hydrogen cannot be removed. On the other hand, the service environment is an open-air marine atmosphere. The surface and fracture surface of the bolt was serially corroded, indicating that hydrogen produced by the corrosive effect during service was also one of the sources of hydrogen embrittlement fracture of the bolt. In summary, the residual hydrogen in the bolt and the hydrogen generated during the corrosion process accumulated at the surface corrosion pits on the bottom of the first thread which has higher stress concentration. Combining with the hydrogen-induced additional stress and the service static load, the bolt would be eventually caused Hydrogen embrittlement fracture.

4. Conclusion

Through the experimental analysis of the fractured bolt, it is concluded that the bolts are highly sensitive to hydrogen embrittlement and hydrogen is introduced in the phosphorylation treatment and the corrosion process under service conditions. With the continuous accumulation of hydrogen, hydrogen embrittlement fracture was finally caused.

According to the reasons of bolt fracture, it is recommended to conduct a general survey of the same type of bolts on site. If there is obvious corrosion or other abnormal conditions, sampling or replacement should be carried out. For the bolts that continue to be used, an optimized anti-corrosion plan should be considered such as using multi-layer coating with better anti-corrosion performance. [9,10] For replaced bolts, it should be ensured that their indicators meet the standard requirements. It is recommended to follow the requirements of GB/T 11376-1997 “Phosphate conversion coatings for metals”. Dehydrogenation treatment should be performed immediately after phosphorylation, and hydrogen embrittlement sensitivity test should be carried out on the bolts before go into service.

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References

[1] Sun Hongpeng. High-Strength Bolted Connections and Failure Analysis of Boiler Steel Structrues. Steel Construction; 2012, 6 (27): 50 - 53.
[2] YE Sheng-yuan, FANG Zhen-lin, QUAN Shu-li, XU Dao-song,LI Xia. Cracking Reason of the Head of ML20MnTiB High-strength Bolt. Physical Testing and Chemical Analysis(Part A: Physical Testing); 2009, 45: 78 - 80.
[3] Zhao Xun, Su Rongrong. Corrosion Analysis of High Strength Bolts in Gantry Frame of Nuclear
[4] Zhang Zhouyong, Wang Jun, Liu hongqun, Liu Zhong. Fracture Analysis of Internal Bolts in Auxiliary Boiler of Nuclear Power Plant. Heat Treatment of Metals; 2019, 44 (S1): 421 - 424.

[5] PAN Zhi-jun. Analysis on Fracture Crack of High Strength Bolt ML20MnTiB. Research On Iron and Steel; 2005, 6 (147): 22 - 25.

[6] Zhong Qunpeng, Li Helin, Zhang Zheng. China Materials Engineering Canon (Volume 1, Chapter 5, material failure analysis). Beijing: Chemical Industry Press; 2006.

[7] ZHANG Jingbao, HU Yang, GAO Guoqing, YANG Chuan. Fracture Analysis of 40Cr Steel Bolt for Tension Wheel of Vehicle. Materials for Mechanical Engineering; 2016, 40 (10): 108 - 110.

[8] Sun Xiaoyan. Hydrogen Embrittlement of Alloy Steel Bolt. Aerospace Standardization; 2012, 01: 5 - 13.

[9] ZHANG Jianbin, YANG Xiaogang. Application of Multilayer Petrolatum Coated Anti-corrosion Technology to Key Parts of Steel Structure in a Coastal Power Plant. Corrosion & Protection; 2011, 32 (11): 896 - 904.

[10] Hou Baorong. Corrosion Behavior of Steel Facilities in Splash Zone and New Coating Protection Technology. Corrosion & Protection; 2007, 28 (4): 174 - 175.