Physical Properties and Failure Analysis for Bolts in the Diaphragm Valve by Micro Morphology and Metallographic Test

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Abstract. The reasons leading to the fracture of 17-4PH stainless steel bolts in the isolation valve of a power plant was analysed by means of morphology analysis, chemical analysis, hardness test, metallographic test, pitting corrosion test and intergranular corrosion test, SEM and other detection means. The results show that there are many corrosion pits on the surface of the valve stem in the seawater system, the corrosion pits is extend and propagation in intergranular cracking. The main reasons to valve stem fracture are the low corrosion resistance of the material and the improper aging process of heat treatment.

Keywords: 17-04 PH, erosion corrosion pits, intergranular cracking, aging process.

1. Introduction
The 17-4 precipitation hardness (PH) stainless steel is a kind of martensitic stainless steel containing low carbon content, high chromium content, copper, niobium and other elements. 17-4 PH has the characteristics of high strength, high hardness, good corrosion resistance to atmosphere and diluted acid or salt after solution and aging treatment. This alloy is more common than any other type of precipitation-hardened stainless steels, owing to its favorable combination of excellent mechanical properties and good corrosion resistance, as well as low cost, and it has been used in a variety of applications including oil field valve parts, chemical process equipment, aircraft fittings, fasteners, pump shafts, compressor impeller, nuclear reactor components, gears, paper mill equipment, missile fittings, and jet engine parts [1-4]. The reasons leading to fracture for the Lange bolts in the diaphragm valve of a power plant was analyzed in this paper, finds out the cause of failure, and provides an effective basis for the subsequent safe and stable operation of the power unit. The structure of the failure valve is shown in Fig.1. The valve is M-type straight through diaphragm valve, which working temperature and pressure are 115℃ and 2.0 MPa. There are four flange bolts in the valve, which the material is 17-04 PH, the specification is M6 × 40mm, and the implementation specification is RCC-M M5110.
Two failed bolts are examined. Experimental procedure consists of macroscopic inspection, material verification, metallographic analysis, tensile test at room temperature, hardness test, pitting corrosion isolation valve corrosion and intergranular corrosion test, SEM analysis, etc. And for these procedures, some specimens were prepared from the fracture bolts. The specimens’ location is shown in Fig. 2.

2. Observation results

2.1. Visual inspection
The two fracture bolts are shown in Fig. 3, and have similar fracture features. It can be seen that the bolts are kept upright without obvious bending deformation. The fracture is relatively flat which no obvious macroscopic plastic deformation nearby is, and the fracture surface has been generally corroded. A few corrosion pits was also visible on the bolts surface.
2.2. Chemical composition of the bolts
The chemical analysis on the fracture bolts are carried out, and its results are presented in Table 1. The result shows that the chemical compositions are coincided with standard specification X6CrNiCu17-04(17-04 PH) [3] except that the elements of Cr, Ni, Cu and Nb are in the lower limit of the standard, C is relatively high, P exceeds the standard.

|   | C  | Si | Mn | P     | S     | Cr   | Ni  | Cu  | Nb  |
|---|----|----|----|-------|-------|------|-----|-----|-----|
| 1#| 0.05| 0.44| 0.82| 0.034 | <0.01 | 15.70| 3.73| 3.21| 0.16|
| d | 7  | 0  | 0  | 5     | 0     | 0    | 0   | 0   | 0.15~0.4 |

2.3. Microstructure examination
Metallographic analysis of the bolts was undertaken on transversal cross-sections. As shown in Fig.4, the Microstructure of the bolts are composed of tempered martensite matrix and more dispersed precipitates, the original austenite grain boundary can be clearly seen. Fig. 5 exhibits the screw teeth are...
processed by roll forming and no obvious corrosion of screw teeth. Metallographic analysis of the bolts was undertaken on transversal cross-sections near the crack (as seen in Fig.2). The crack initiates on the outer surface of polished rod and propagates along the axial direction. The fracture is intergranular with obvious bifurcation characteristics in local position, which is a typical stress corrosion cracking.

Fig 4. Microstructure on transversal cross-sections of the bolts.
(a) The No.1 bolt, (b) The No.2 bolt.

Fig 5. Bolt thread of the Bolts.
(a) The No.1 bolt, (b) The No.2 bolt.

Fig 6. Microstructure of No.1 Bolt fracture.
(a) Fracture edge, (b) Middle Position, (c) Fracture edge.
2.4. Hardness

The Brinell hardness test results are shown in Table 2. The hardness of the broken bolts is between 461 and 490HB, which is much higher than the requirements of RCC-M 5110.

| Hardness | value (HB) |
|----------|------------|
| No.1     | 490        |
| No.2     | 461        |
| specified | ≥302HB     |

2.5. Pitting corrosion test and intergranular corrosion test

The samples were taken from the bolt section of the sample as shown in Fig. 2 for pitting corrosion test and intergranular corrosion test. The test results are shown in Table 3, Fig. 8 and Fig. 9. The length and width of the pitting pits are basically the same. The results of intergranular corrosion test showed that there were obvious intergranular corrosion cracks.

| Value (μm) | Length | Width | Depth |
|------------|--------|-------|-------|
| No.1       | 2353.46| 1138.94| 1890.1|
| No.2       | 2442.96| 2369.62| 1317.5|

Fig 7. Microstructure of No.1 Bolt fracture.
(a) Fracture edge, (b) Middle Position, (c) Fracture edge.

Fig 8. Pitting corrosion test of the Bolts. (a) The No.1 bolt, (b) The No.2 bolt.
2.6. SEM observation on fractures
The fracture and surface of the bolts were observed by micro morphology and analyzed by scanning electron microscopy (SEM). The results show as follows: Fig.6 gives fractographs of fracture region of the bolts. The intergranular characteristics on the fracture surface and obvious pitting corrosion on the bolts surface can be seen. Among them, the fracture surface are covered with obvious corrosion products, which the composition is Cl, S and other corrosive elements.

Fig 9. Intergranular corrosion test of the Bolts. (a) The No.1 bolt, (b) The No.2 bolt.

Fig 10. SEM of No.1 the fracture bolts. (a) The No.1 bolt, (b) The No.2 bolt.

Fig 11. SEM of the bolts surface. (a) The No.1 bolt, (b) The No.2 bolt.
3. Discussion

From the results of the morphology analysis and microscopic analysis, it is inferred that the bolts fracture presents intergranular stress corrosion cracking characteristics, which is absence of plastic deformation on fracture, the fracture surface is covered with a layer of corrosion products, the micro fracture is intergranular and the grain boundary is straight and a few bifurcated intergranular secondary cracks near the fracture.

According to the valve design drawings, the bolts material is precipitation hardening martensitic stainless steel (17-04 PH), which is mainly obtains high strength by precipitation of dispersed Cu rich phase and hard NbC particle phase through aging strengthening. According to RCC-M 5110 and ASME SA-564/ SA-564M, the heat treatment of 17-04 PH stainless steel should be as follows: austenitizing temperature is 1020-1060℃, aging hardening temperature is 595-620℃. The hardness of the material can reach more than 388HB when the aging hardening temperature is 480℃. It can be seen that the actual heat treatment process of the fracture bolts does not strictly comply with the requirements of RCC-M 5110 specification which the actual aging temperature of bolts should be lower than 480℃. The results showed that under the condition of the lower aging temperature, when aging temperature is lower than 480℃, which lead to high strength, temper brittleness, grain boundary weakening, significant reduction of stress corrosion resistance of the material under service conditions[4-7]. The results of corrosion test at sampling point and intergranular corrosion test also basically verify this point. In conclusion, the fracture property of the bolt is intergranular stress corrosion cracking.
4. Conclusions
After performing the cause analysis of the bolts, conclusions can be summarized as follows:

(1) The content of main alloy elements (Cr, Ni, Cu, Nb) is in the range of relative lower limit, the content of element C is relatively high, and the content of impurity element P exceeds the standard. The actual tensile strength is far higher than the lower limit required by the standard. The hardness test results also show a similar situation.

(2) The fracture property of the bolts are intergranular stress corrosion cracking, which is related to the improper heat treatment process.

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