Analysis of compressor valves bolts failure

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1. Introduction

Fracture, which is a most severe form of material damage, is under influence of numerous factors, such as: mechanical stresses, temperature, composition and properties of atmosphere, shapes and dimensions of a part or construction; structure and properties of material and quality of surface [1]. In order to determine the cause of fracture, in this paper, the specimens of bolts that were broken during the exploitation and also, used but unbroken bolts were tested. Their malfunction can cause a major expenses due to production downtime, safety hazards due to effluence of process fluids and/or disturbance of the process parameters in the later stages of the refining process. In petroleum industry, the compressors are used as the high pressure systems. Their malfunction is sometimes connected to failure of bolts that hold the valves of the compressor. Nasirpouri et al. [2] have recently published the study that deals with the failure of the high strength steel bolts achieved from the petroleum industry. Their case study is the closest to this work that could be found in literature. The corrosion behavior of carbon steels in the H_2S containing environments was studied by Tang et al. [3]. One of the most common failures in the industry would happen due to the brittle fracture known as sulphide stress cracking when the surrounding environment contains sulphur compounds such as H_2S [2, 3].The salts and sulfide compounds dissolved in crude oil can provoke the formation of a corrosive aqueous solution whose chemical composition involves the presence of both hydrochloric acid (HCl) and hydrogen sulfide (H_2S). This corrosion aqueous solution is very aggressive causing varied damages on carbon steel during plant operation. Several previous studies [4-7] have been performed related to the corrosion process of iron and steel in H_2S solutions. It should be noted that there is large number of studies that consider transformations of mackinawite (which is often found in petroleum industry deposits) under the influence of various conditions, such as water solutions [8] or ambient air [9]. The important aspect of Kidam-Kurme study [10] is that takes into account only the root causes of failures. In the case of the high strength steel bolts failure analysis in the presence of the H_2S-containing environment, any process responsible for formation of initial cracks before or during service can be considered as the main contributor. If the initial cracks are formed during service, then the working conditions (corrosive environment and/or loading conditions) can be considered as the main contributor to failure. The structure and other properties of the high strength steel bolts will either facilitate or delay the crack initiation process, but they will not prevent it and thus they can be considered as the sub-contributors to failure. If the initial cracks are formed before service, then the working conditions can be considered as the sub-contributors to failure. The aim of this study is to identify the main and sub-contributors of bolts failures.

2. Materials and Methods

Two used but unbroken bolts and two broken bolts are made of hot-rolled, quenched and tempered ASTM A 193 B7 steel with approximately 7.8 mm in diameter, 24 and 40 mm in length were achieved from oil refinery (Figure 1). Their average chemical composition was determined by use of spectrophotometry (Table 1). The as-received bolts were in rolled and quenched and tempered condition according to mechanical properties (Table 2).

Table 1. Chemical composition of A 193 B7 [mas. %].

C	0.37 ÷ 0.49	Mo	0.15 ÷ 0.25
Mn	0.65 ÷ 1.10	P	< 0.035
Si	0.15 ÷ 0.35	S	< 0.040
Cr	0.70 ÷ 1.20	Fe	rest

Table 2. Mechanical properties of A 193 B7 at room temperature.

Rm, [MPa]	min. 860
Rp, [MPa]	min. 720
A_5, [%]	min. 16

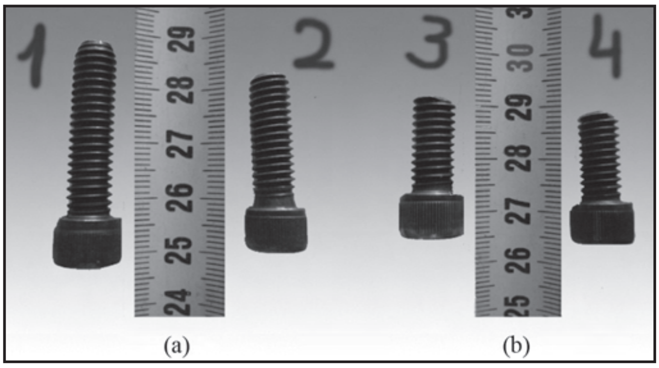


Figure 1. As-received used unbroken (a) and broken (b) bolts from compressor valve.

In order to estimate the cause of their failure they were firstly subjected to detailed visual examination and radiographic measurements by γ-rays, according to ISO 5579 standard. From these measurements and appearance of complex fractured surfaces, as well as presence of bolt deposits, it was decided to estimate the quality of broken bolts and to perform chemical analysis of bolt's deposit and fractured surfaces of broken bolts. It was done to perform the deposit analysis separately from bolts analysis and learn more about the aggresive environment that have been in contact with the surface of the bolts during service. The phases in the deposit powders were analyzed by using the X-ray powder diffractometry (XRPD) and the chemistry of the deposits particles by using the scanning electron microscopy with energy dispersive system (SEM-EDS). The measurements were performed on mechanically homogenized mixtures of bolts deposit powders. The XRPD analysis was performed with powder diffractometer PHILIPS PW 1710 (40 kV, 30 mA, CuKα). The 2θ-scanning range was between 4° and 90° and the scanning speed was 0.02°/0.5s. Obtained diffracttion patterns of deposits were compared with available literature and JCPDS standards. The SEM-EDS analysis was performed by using the JEOL JSM–6610LV scanning electron microscope connected with the INCA350 energy-dispersion X-ray analysis unit. The acceleration voltage of 20 kV and the tungsten filament were used. Before SEM-EDS analysis, the deposite powder particles were placed on the specific sample holders with a carbon-based sticky conductive surface and subsequently they were gold-coated in a vacuum chamber of a sputter coater device. The bulk properties of the recieved bolts materials were assessed through chemical analysis, primary and secondary structure analysis, information about presence of impurities and/or micro-porosity and fractography. Additionally, the fracture resistance is estimated from results of fractography of fractured surfaces of originally broken bolts. The chemical analysis of bolt deposit and fractured surfaces has given the information about presence of corrosion environment. The samples for structure analysis were taken from different parts of broken and unbroken bolts by cutting in radial direction. Subsequently, they were grinded by SiC abrasive paper and polished down to 1 μm using Al_2O_3 suspension. Primary structure was revealed by use of Oberhoffer's reagent, while secondary structure with 2% Nital. The bolts structures in radial and axial direction were studied by using the light optical microscopy. Fractography, fractured surfaces chemistry and deposit chemistry were done by use of Scanning Electron Microscopy with Energy Dispersive System (SEM-EDS).

3. Results and Discussion

3.1. Visual examination and radiography measurements

Visual examination of used unbroken bolts hasn't shown any irregularities presence. Broken bolts were deposite-coated. The color of deposite was predominantly black. The light-gray and brown-red areas (probably the result of oxidation in ambient air) were occasionally observed mixed with black deposite. Generally, all bolts had a complex topography of fractured surfaces. Common for all bolts was that the fracture was initiated at the thread roots. After initiation, cracks propagated through the stud in radial direction, sometimes abrubtly changing the planes of propagation to the final fracture. Radiography measurements have shown that all broken bolts had mechanical demages in the stud. The measurements of used unbroken bolts didn't reveal any presence of defects / imperfections / discountinuities.

3.2. Deposit analysis

Using XRPD analyze the presence of corrosion products is detected, which are very poorly crystallized and which occur in corrosion environments where H_2S is present. These are iron sulfides (FeS-makinawite, Fe_3S_4-centered cubic iron sulfide and Fe_3O_4-magnetite) and elementary sulfur S8. In the case of deposits obtained from used unbroken bolts (Figure 2a) only two crystalline phases are equally represented: iron sulfide, makinawite, $Fe_{1+x}S$, x = 0.057-0.064, (JCPDS 89-2738), and less present crystalline phase is sulfur, S_8 (JCPDS 83-2283). Makinawite is the layered sulfide, nonstoichiometric compound. Sulphur occurs as α-S, the most common rhombic allotropic modification, which was built by eight ring of sulfur atoms, and therefore it is marked as S_8. The most common corrosion phase (about 60%) is elemental sulfur, S_8 in the case of deposits obtained from broken bolts (Figure 2b) (JCPDS 83-2283). The other crystalline phases are presented each by approximately 20%. These are makinawite and iron oxide, magnetite, Fe_3O_4, (JCPDS 19-0629). Reflections of magnetite are very broad and poorly defined. There is a possibility that some magnetite transferred to iron oxide hydroxide goethite α-FeO(OH) (JCPDS 29-0713), but this can not be said with certainty. SEM-EDS analysis of the deposit is made in the peripheral parts of the bolts in order to determine

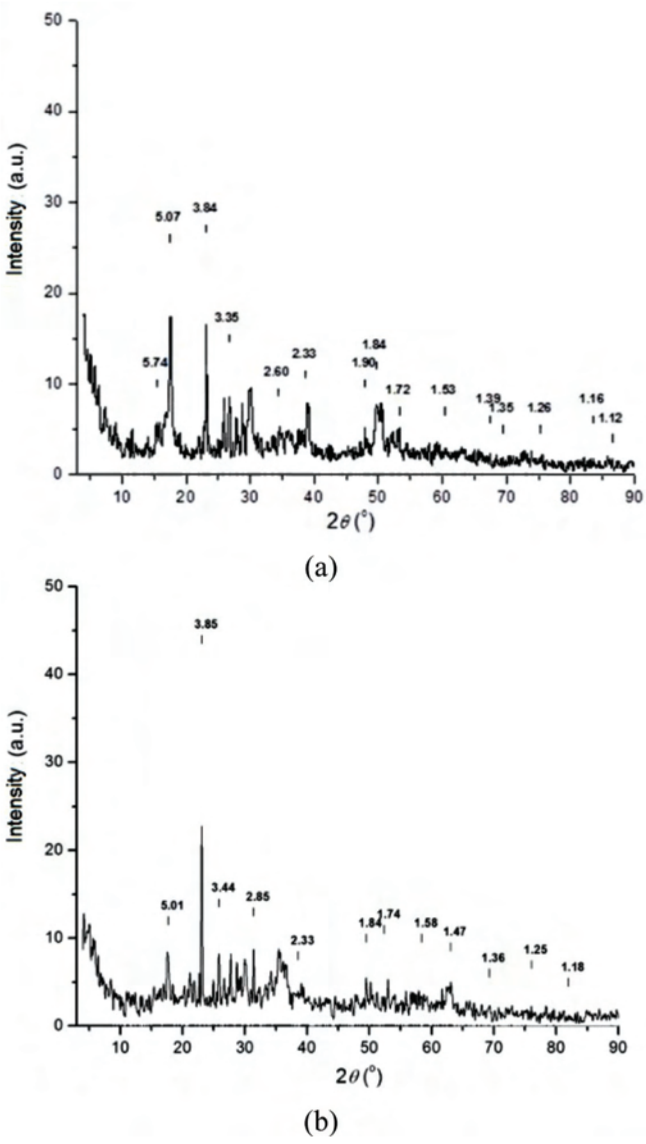
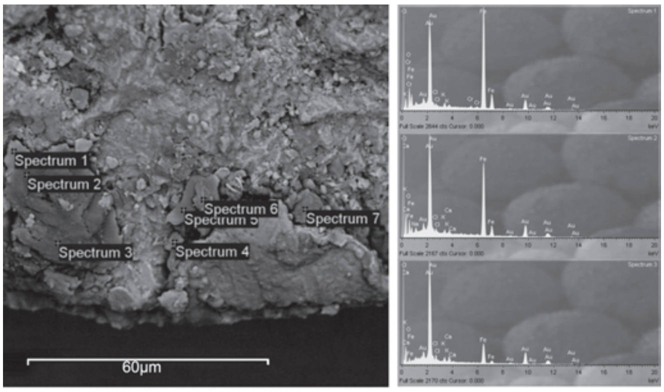


Figure 2. X-ray diffraction patterns of deposits from compressor valve: (a) diffraction pattern corresponds to mixture of deposit powders taken from used unbroken bolts; (b) diffraction patterns correspond to deposit powders taken from different as-received broken bolts.

their relationship to possible corrosion processes prevailing during operation of bolts. The tests were performed on the peripheral areas of broken bolts and the results are shown in Figure 3 and Table 3. EDS analysis of the samples indicated an increase in the presence of sulfur, oxygen, chlorine, calcium and potassium. Appearance spectrum with detected sulfur is similar to EDS spectrum obtained in the analysis of fracture surfaces of broken bolts. This suggests a possible link between the process of corrosion and fracture of bolt materials during operation. Additionally, the presence of oxygen were detected in all cases where the sulphur was found.



(a) (b)

Figure 3. (a) Fracture surface of broken bolt SEM image, (b) EDS spectrums of corrosion products.

Table 3. EMPA analysis, mas.%.

	C	O	S	Cl	Ca	K	Na	Cr	Fe
Spect 1	24.47	17.34	-	0.58	-	0.85	-	0.50	57.71
Spect 2	41.74	17.19	1.07	0.74	0.79	1.02	1.9	0.52	38.51
Spect 3	58.79	14.31	1.43	2.15	0.45	0.52	-	0.36	22.50

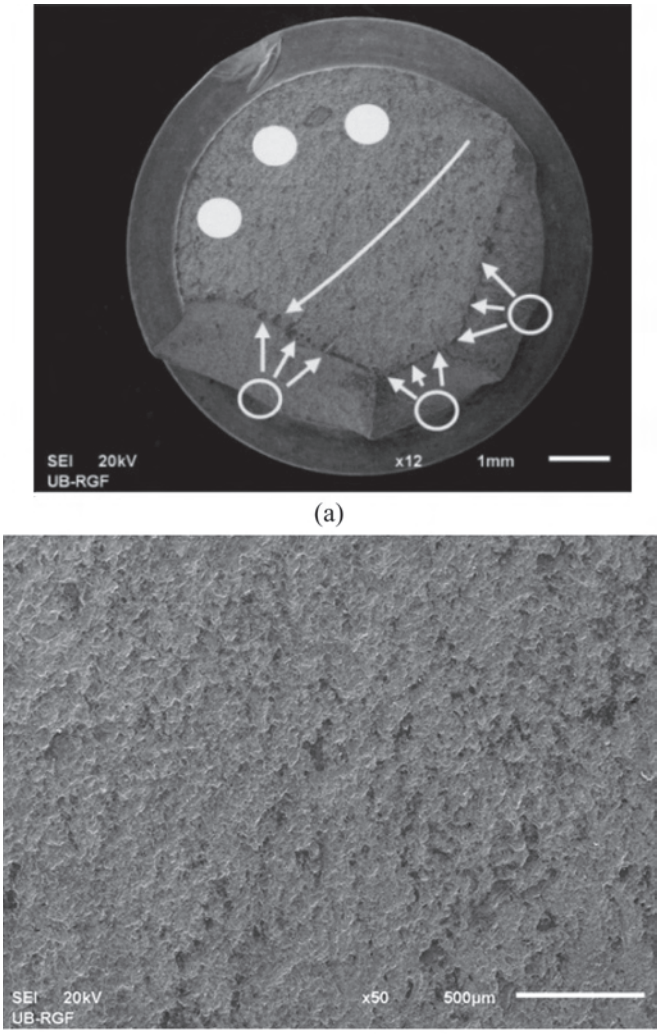
3.3. Bolts analysis

The representative average chemical compositions of materials of unbroken and broken bolts are given in Table 4. The chemical compositions of bolts are in limits with requirements of ASTM standard. However, the manganese content of broken bolts is approximately ≈0.2 wt.% lower in comparison to used unbroken bolts, and the sulphur and phosphorus content is higher by a factor of 13. Although the content of S meets the ASTM requirement, the difference in sulphur content between bolts is significant.

Table 4. The average chemical composition of bolt samples [wt.%]

Chemical elements	Material designation		
	ASTM A 193 B7	Unbroken	Broken
C	0.37-0.49	0.40	0.41
Si	0.15-0.35	0.24	0.33
Mn	0.65-1.10	0.878	0.615
P	< 0.04	0.001	0.013
S	< 0.04	0.001	0.013
Cr	0.70-1.20	1.123	0.959
Mo	0.15-0.25	0.177	0.151
Fe	balance	balance	balance

The secondary structure of unbroken and broken bolts from compressor valve is tempered martensite. The broken bolts have the lowest homogeneity of primary structure and this observation is consistent with chemical analysis where the broken bolts have the highest sulphur and phosphorus content (Table 4).



(a)

(b)

Figure 4. Fractography analysis of the broken bolt. (a), (b) the central zone. Surfaces were covered with gold coating (thickness of about 20 nm), scanning electron microscope (SEM) image of secondary electrons.

In Figure 4 are given the representative results of fractography analysis. The crack initiation sites analysis is mostly performed on used unbroken and on broken bolts. In this analysis, the used unbroken bolts are observed as candidates that should fail by fracture during their service but didn't. Therefore, in this case, there was a high probability to detect corrosion products that didn't develop into cracks or cracks that have been initiated at thread root sites that didn't grow to a critical size for final fracture to happen. Fracture surfaces of broken bolts shown in Figure 4 indicate that the effect of complex fractures incurred operating conditions (stresses and corrosive environments). Most of the fracture surface is checkmated, indicating the predominant character of ductile fracture. In all cases the initiation of fracture is created in the root of the bolt and in multiple locations, as shown in the open circles (○). Straight arrow indicates the direction of movement of cracks and fracture mechanism of brittle quasi-cleavage. Parts of the fracture surface indicating the final fracture are shown by full circles (●). These flat parts of the fracture surface indicate brittle fracture. The fracture micro-mechanism wasn't determined because of the presence of this surface layer, which was created during the operation of bolts

4. Conclusions

The used unbroken and broken bolts were achieved from oil refinery. The used and broken bolts were deposit-coated. In order to determine the cause of their failure, the bolts and deposit properties were analyzed separately. The deposit analysis showed the presence of mackinawite, elementary sulphur and pyrite of which the mackinawite and pyrite confirm that the bolts have been in contact with aggresive H2S-containing water condensate during service. The crack initiation sites analysis has shown the presence of possible corrosion pits and numerous initiated microscopic cracks on the thread root locations of the bolts that didn't fail during service (used bolts). For these reasons, the corrosion process was designated as the main contributor to bolts failure. This finding was also confirmed after fractography analysis of broken bolts fractured surfaces from which it was clearly observable that fracture has been initiated at the thread root locations. The bolts material properties and the loading conditions were designated as the sub-contributors to bolts failure. The existent inhomogeneities of primary structure has mostly affected the appearance of the fractured surfaces. The recommendation is to deal with the influence of the corrosive environment rather than with the replacement of the bolts material. The loading conditions of the bolted connections need to be thouroughly analyzed.

Acknowledgements

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