

Study on intrinsic sulfidation of iron oxides and oxidation behavior of sulfidation products

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Received December 27, 2017; Accepted January 31, 2018

The study simulated the sulfur corrosion process in an oil tank to evaluate intrinsic sulfidation of iron oxides and oxidation behavior of sulfidation products at room temperature. The methods of SEM and EDS were used to characterize the microstructure and surface state of the sulfidation products of Fe₂O₃, Fe₃O₄ and Fe(OH)₃, which are the dominant components of rust. The surface chemical state of sulfidation and oxidation products was characterized by XPS measurement to analyze the oxidation degree and oxidation mechanism of the sulfidation products. The results showed that three kinds of iron oxides can produce the same kind of iron-sulfur compounds after sulfidation, but the microstructure and elemental distributions of these iron-sulfur compounds are different. The main components of the sulfur corrosion products of the three iron oxides are identical. The sulfur corrosion process of iron oxides converts iron oxides into FeS₂ and FeSO₄, which is accompanied by the formation and transformation of FeS and S₈. At room temperature the sulfidation products of iron-sulfur compounds are finally oxidized to Fe₂O₃ and FeSO₄.

Keywords: iron oxides; sulfidation; oxidation; iron-sulfur compounds; XPS.

INTRODUCTION

In the process of oil refining, storage and transportation, the active sulfur in the oil will react with the rust (the dominant components are iron oxides such as Fe₂O₃, Fe₃O₄ and Fe(OH)₃) on the inner wall of the device, which can generate iron-sulfur compounds with a certain pyrophoricity. Fire and explosion accidents caused by self-ignition of corrosion products of sulfur occasionally happen [1-3]. Many scholars have extensively investigated the mechanisms of spontaneous combustion of iron-sulfur compounds [4-8].

Corrosion of iron in H₂S containing solutions is a general problem in crude oil and natural gas production, and is generally referred to as sour corrosion. Aqueous H₂S solutions promote corrosion of steels [9-15], but the exact nature and mechanisms of corrosion strongly depend on the reaction conditions [16-18]. While the process has been widely investigated for pure iron and carbon steels, there is still a lack of understanding of the reaction path of the corrosion products. The chemistry of the corrosion products formed during H₂S-triggered corrosion is rather complex, as there are many different solids consisting only of iron and sulfur. X-ray photoemission spectroscopy (XPS) has been used to confirm the presence of the reaction products [19-22]. On the other hand, the corrosion product can be rapidly oxidized in the air and release heat. Different types of materials for the

formation of sulfur compounds and changed sulfidation environment will make the sulfidation products have different oxidation pyrophoricity.

Although there were many investigations about the sour corrosion and oxidation process of sulfidation products, it is not totally clear what happens in the course of the sulfidation process and oxidation process. The objective of this work was to investigate intrinsic sulfidation behaviors of iron oxides in H₂S, N₂ and O₂ gas atmosphere and the oxidation behaviors of sulfidation products at room temperature. In this paper, dry Fe₂O₃, Fe₃O₄ and Fe(OH)₃ were used by reacting with a mixture of sulfidation gas at a certain temperature, to simulate the sulfur corrosion process in an oil tank containing sulfur. Following the oxidation process of the sulfidation products, the chemical morphology of the reaction products was identified by XPS. It would be highly desirable to understand the sulfidation mechanism of iron oxides and the oxidation mechanism of sulfidation products in order to better understand the sulfidation process and oxidation process. The experimental results provided a theoretical basis for the prevention of spontaneous combustion of sulfur compounds.

EXPERIMENTAL

Experimental method

The scheme of the experimental apparatus of the sulfidation and oxidation tests is shown in Fig. 1. In the experiments, three groups of three-necked

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flasks were installed in parallel to a gas bag and filled with Fe_2O_3 , Fe_3O_4 and $\text{Fe}(\text{OH})_3$, separately. This is to make sure that the sulfidation processes of the three iron oxides take place under the same conditions.

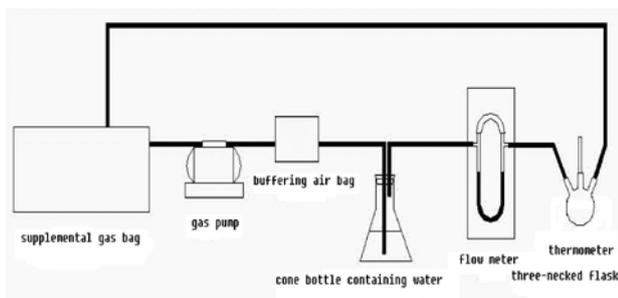


Fig. 1. Apparatus for the sulfidation and oxidation processes

Desired concentration of sulfidation gas (volume ratio of $\text{H}_2\text{S}:\text{O}_2:\text{N}_2 = 5:1:4$) was pre-configured and the gas was stored in the gas bag. Quantities of Fe_2O_3 , Fe_3O_4 and $\text{Fe}(\text{OH})_3$ samples were placed in the three-necked flasks separately. The mixture of sulfidation gas in the gas bag was pumped by a gas pump and passed through the buffering air bag, water containing cone bottle and flow meter which can detect and control flow, and then entered the three-necked flasks. The sulfidation reaction of iron oxides was carried out in the three-necked flasks. The sulfidation products were divided in two, one part was directly exposed to the air; the other part was sealed with ethanol. EDS and XPS analysis were carried out using the ethanol sealed sulfidation products. The sulfidation products directly exposed to the air were used for the oxidation process. Open both sides of the stopper, let air get into the reaction flask and react with corrosion products. The temperature was measured by a thermocouple thermometer inside the reactor. The final products were analysed by XPS.

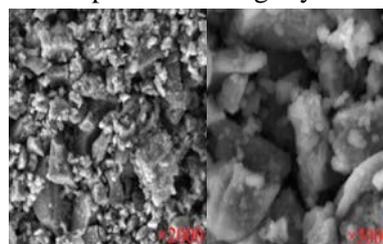
Characterization method

The sulfided and oxidized samples were analysed by scanning electron microscopy with energy-dispersive X-ray spectroscopy (DEM-EDS) at the acceleration voltage of 30 KV and X-ray photoelectron spectroscopy (XPS) technique with an ESCALAB 250 spectrometer using monochromatic $\text{Al K}\alpha$ X-ray (1486.6 eV).

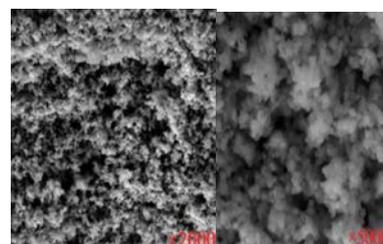
RESULTS AND DISCUSSION

DEM-EDS characterization of sulfidation products

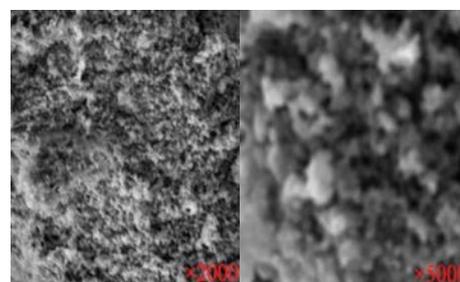
The spontaneous oxidation reaction of the sulfidation products has a strong relationship with their microstructure, and the specific surface area is different, which leads to different contact areas with oxygen. The results of sulfidation products of Fe_2O_3 , Fe_3O_4 and $\text{Fe}(\text{OH})_3$ characterized by SEM technique are shown in Fig. 2. As can be seen, there are some differences in the microstructure of the three sulfidation products. The sulfidation product of $\text{Fe}(\text{OH})_3$ mostly consists of sheets or blocks which are more regular, and the particle size is relatively large, the specific surface area is relatively small, and is disadvantageous for the adsorption and diffusion of O_2 molecules. The particles of the sulfidation products of Fe_3O_4 and Fe_2O_3 are fine, porous, loose and the surface area is big. The small particles of the Fe_3O_4 sulfidation products are polygons, and the pore structure is more obvious, but the Fe_2O_3 sulfidation products are approximate pellets and slightly denser.



(a) sulfidation products of $\text{Fe}(\text{OH})_3$



(b) sulfidation product of Fe_3O_4

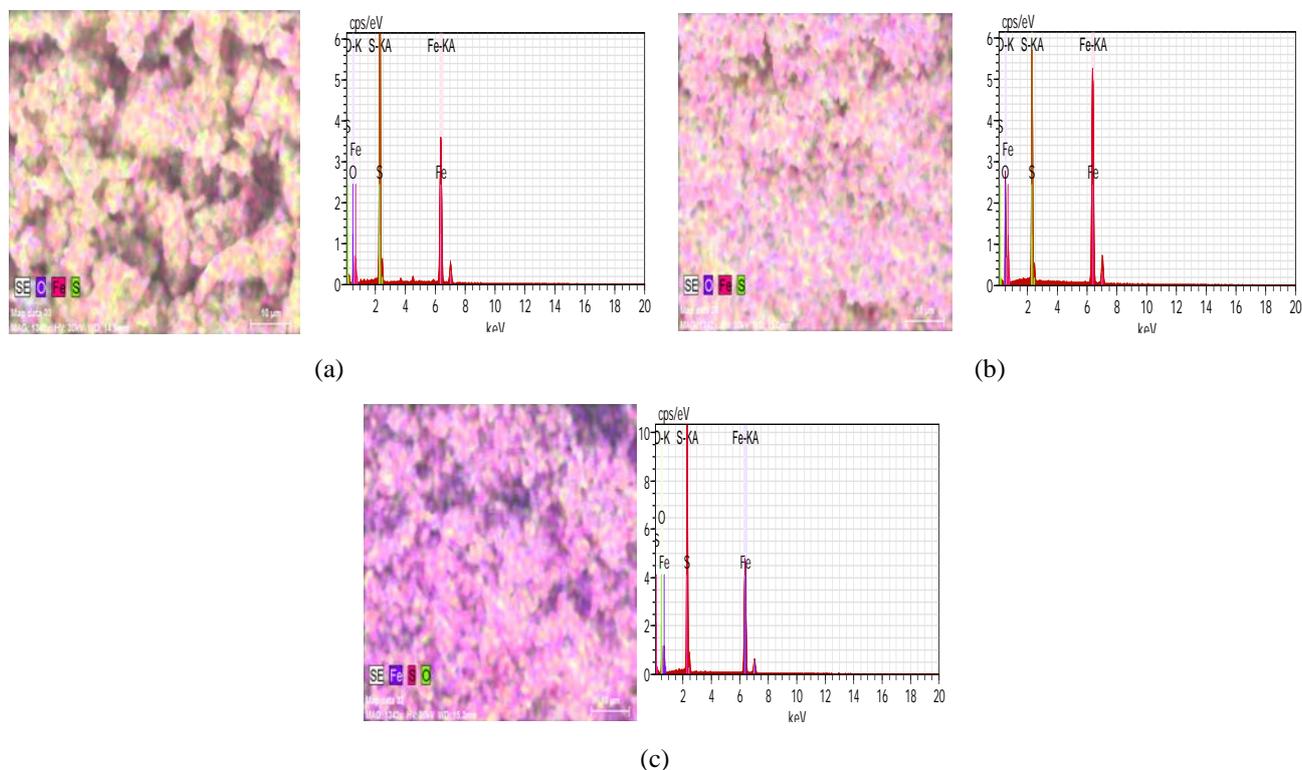


(c) sulfidation product of Fe_2O_3

Fig. 2. SEM photographs of sulfidation products, Left: 500 \times , Right: 2000 \times

Table 1. Contents of Fe, S, O in samples (% , mass fraction)

| Sample | Fe | S | O |
|-----------------------------------|--------|--------|--------|
| Sulfided $\text{Fe}(\text{OH})_3$ | 24.76% | 38.54% | 36.70% |
| Sulfided Fe_3O_4 | 22.24% | 16.82% | 60.94% |
| Sulfided Fe_2O_3 | 25.29% | 39.08% | 35.63% |

**Fig. 3.** EDS of sulfidation products (a) $\text{Fe}(\text{OH})_3$ (b) Fe_3O_4 and (c) Fe_2O_3

The scanning results of Fe, S, O in the sulfide products are shown in Fig. 3. The distribution of Fe and S on the surface of the $\text{Fe}(\text{OH})_3$ sulfidation products is more uniform, but that of O is less so. For the Fe_3O_4 sulfidation products, the distribution of various elements on the surface was more average, except for S which was more dispersed. However, the distribution of S on the surface of Fe_2O_3 sulfidation products was more concentrated, and Fe was mainly distributed in pores. The results showed that the element type of the three kinds of sulfidation products was the same, but the distribution was different. The contents of Fe, S and O in the sample are listed in Table 1.

XPS characterization of S

The binding energies of S 2p in the sulfidation products before and after oxidation are shown in Fig. 4. The binding energy at 161~161.7 eV most likely belonged to FeS. The binding energy at 161.7~163.4 eV most likely belonged to FeS_2 . The peaks at 163.3~164.5 eV were considered to be S_8 . The peaks at 168.3~169.4 eV were considered to be SO_4^{2-} . The results of curve fitting are presented in

Table 2. It can be seen from Table 2 that before oxidation the S atoms in the three kinds of sulfidation products were mainly FeS_2 , which accounted for 42.66%, 53.98% and 31.45%, respectively. Small amounts of reduced FeS, S_8 and partially SO_4^{2-} were also contained. The SO_4^{2-} in the sulfidation product after oxidation was the main form of S in the sample, and the atomic contents were by 29.71%, 17.28% and 37.39% higher than those before oxidation. After the oxidation process, the contents of FeS_2 and FeS decreased significantly, or even disappeared.

From the content of S, the SO_4^{2-} -component in the sulfide product after oxidation sharply increased, while the FeS_2 and FeS contents in the reduced state were obviously reduced and the S content increased in some varieties and decreased in others. It was illustrated that the low-valence S was oxidized to the high-valence SO_4^{2-} in the oxidation process. However, some of the S was only oxidized to S_8 , indicating that S of different sulfide products was oxidized to a different degree.

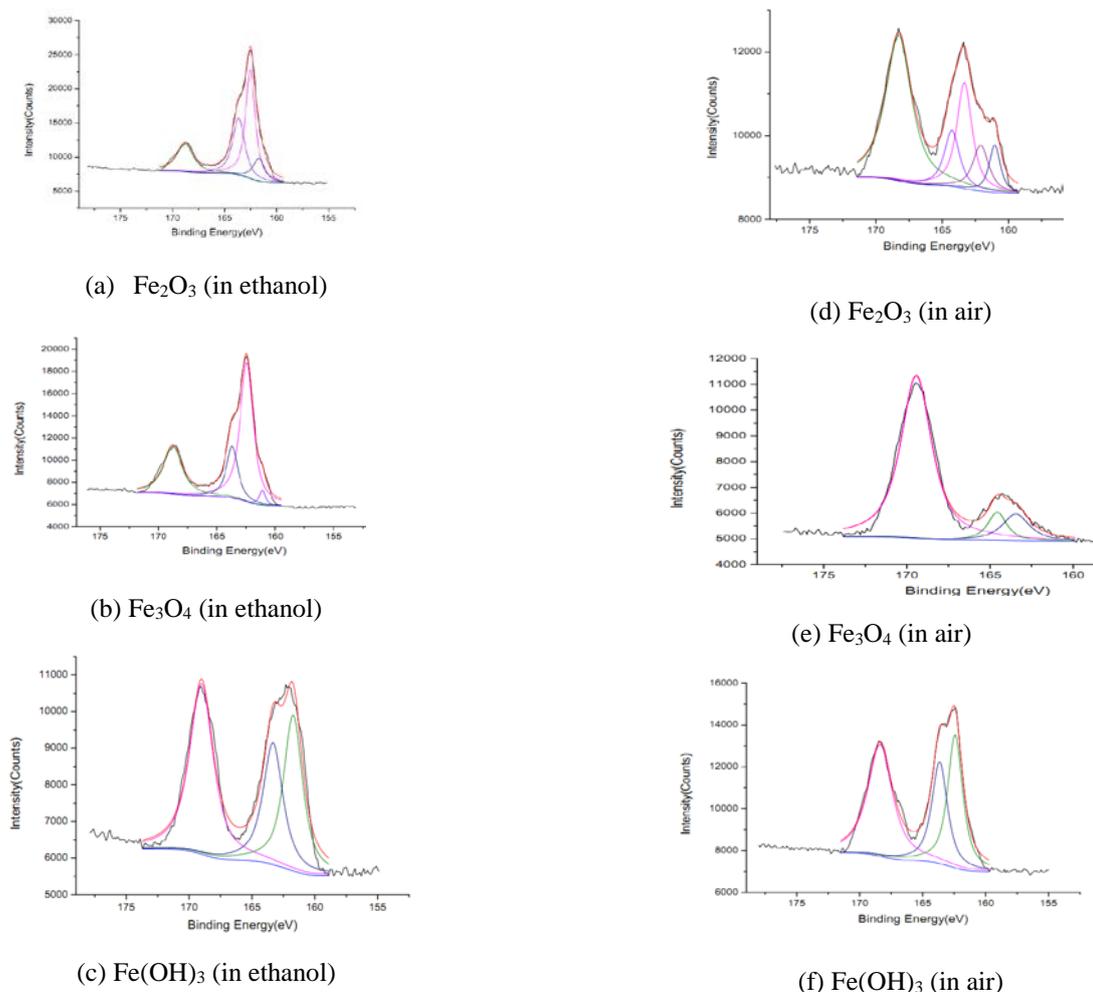


Fig. 4. XPS spectra of S 2p of sulfidation products.

XPS characterization of Fe

The binding energy of Fe 2p before and after oxidation of the three ferrosulfide compounds is shown in Fig. 5. As can be seen, the binding energy of Fe 2p existing in the form of FeS_x (mainly FeS₂) was 706.7 ~ 708.3 eV, the binding energy of Fe 2p existing in the form of Fe₂O₃ was 711.1 ~ 711.9 eV, and the binding energy of Fe atom bonded to SO₄²⁻ was 711.1 ~ 711.9 eV. The results of curve fitting are shown in Table 3. It can be seen that before oxidation, the species of Fe in the three sulfidation products were FeS₂, FeO, Fe₂O₃, and FeSO₄ in addition to their own non-sulfured iron oxides.

After being fully oxidized in the air, the form of Fe changed little, but the content of each component changed greatly. Comparing the three sulfidation products (Fe₂O₃, Fe₃O₄ and Fe(OH)₃) before and after oxidation, the contents of Fe in the form of Fe₂O₃ increased from 49.50%, 26.81% and 26.28% to 82.68%, 46.91% and 33.69%, respectively. The contents of Fe in the form of FeSO₄ increased from 5.82%, 12.48% and 17.78% to 11.27%, 14.31% and 26.60%, respectively. With the progress of oxidation, more and more Fe₂O₃ and

FeSO₄ were produced. However, the content of FeS_x and FeS₂ was greatly reduced during the oxidation process. Only 6.05% of Fe was found to be FeS_x in the oxidation product of Fe₂O₃ sulfidation product, and the existence of FeS_x was not detected in the other two oxidation products.

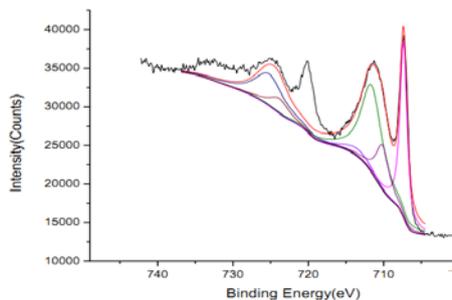
Analysis of the sulfidation behavior of iron oxides

In the process of iron oxide sulfidation, the reaction process of iron oxide (FO) and pure H₂S is very straightforward [23]: FO $\xrightarrow{H_2S}$ FeS+S+H₂O. The FeS produced by the reaction further reacts with S to form FeS₂: FeS \xrightarrow{S} FeS₂. The change in the content of each component from the sample can be used to estimate the trend of mutual conversion between the various components.

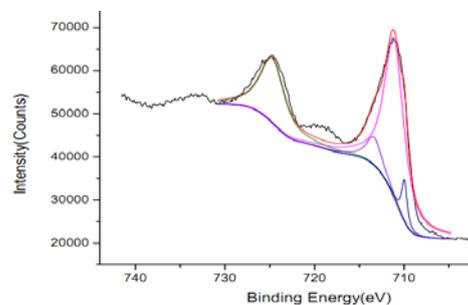
Through the analysis above, in the actual reaction process, the remaining FeS is very little and even almost undetectable, but the content of FeS₂ is much higher. This is presumably due to the progress of the sulfidation process; H₂S is continuously oxidized to elemental S, the composition of the system, the temperature and other conditions changes will lead to the reaction of elemental S and FeS to form FeS₂ as the main

Table 2. Results of curve fitting of S 2p of sulfidation products

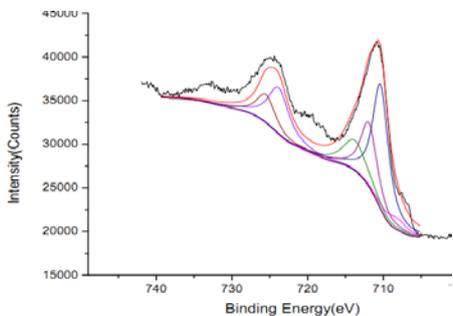
| Sample | Before oxidation (in ethanol) | | | After oxidation (in air) | | |
|-----------------------------------|-------------------------------|-----------------------|--------------------|--------------------------|-----------------------|--------------------|
| | BE | S atom percentage (%) | Chemical state | BE | S atom percentage (%) | Chemical state |
| Sulfided Fe_2O_3 | 161.7 | 10.93 | FeS | 161 | 6.13 | FeS |
| | 162.5 | 42.66 | FeS_2 | 162.0, 163.3 | 36.49 | FeS_2 |
| | 163.6 | 27.97 | S_8 | 164.3 | 9.23 | S_8 |
| | 168.8 | 18.44 | SO_4^{2-} | 168.3 | 48.15 | SO_4^{2-} |
| Sulfided Fe_3O_4 | 161 | 2.59 | FeS | 162.4 | 31.37 | FeS_2 |
| | 162.4 | 53.98 | FeS_2 | 163.6 | 25.11 | S_8 |
| | 163.7 | 17.19 | S_8 | 168.3 | 43.52 | SO_4^{2-} |
| | 168.7 | 26.24 | SO_4^{2-} | | | |
| Sulfided $\text{Fe}(\text{OH})_3$ | 161.7 | 31.45 | FeS_2 | 163.4 | 11.41 | FeS_2 |
| | 163.3 | 25.75 | S_8 | 164.5 | 8.40 | S_8 |
| | 169.0 | 42.80 | SO_4^{2-} | 169.4 | 80.19 | SO_4^{2-} |



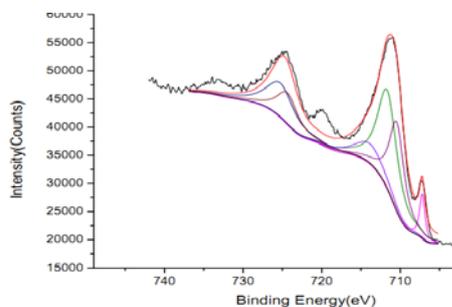
(a) Fe_2O_3 (in ethanol)



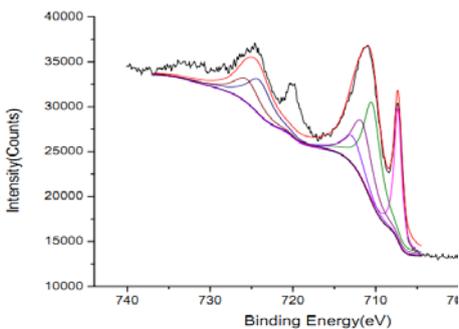
(d) Fe_2O_3 (in air)



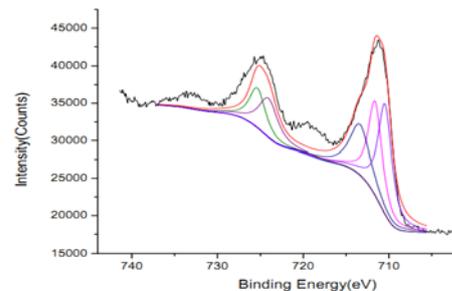
(b) Fe_3O_4 (in ethanol)



(e) Fe_3O_4 (in air)



(c) $\text{Fe}(\text{OH})_3$ (in ethanol)



(f) $\text{Fe}(\text{OH})_3$ (in air)

Fig. 5. XPS spectra of Fe 2p of sulfidation products.

Table 3 Results of curve fitting of Fe 2p of sulfidation products

| Sample | In ethanol | | | In air | | |
|--------------------------------|--------------|------------------------|--------------------------------|--------|------------------------|--------------------------------|
| | BE | Fe atom percentage (%) | Chemical state | BE | Fe atom percentage (%) | Chemical state |
| Fe ₂ O ₃ | 707.3 | 34.40 | FeS ₂ | 710.0 | 6.05 | FeS _x |
| | 710.1 | 10.28 | FeO | | | |
| | 711.5 | 49.50 | Fe ₂ O ₃ | 711.1 | 82.68 | Fe ₂ O ₃ |
| | 713.4 | 5.82 | FeSO ₄ | 713.0 | 11.27 | FeSO ₄ |
| Fe ₃ O ₄ | 707.3 | 22.22 | FeS ₂ | 707.0 | 5.98 | FeS ₂ |
| | 710.4 | 38.49 | Fe ₃ O ₄ | 710.4 | 32.80 | Fe ₃ O ₄ |
| | 711.6 | 26.81 | Fe ₂ O ₃ | 711.6 | 46.91 | Fe ₂ O ₃ |
| | 712.8 | 12.48 | FeSO ₄ | 713.8 | 14.31 | FeSO ₄ |
| Fe(OH) ₃ | 706.7, 708.3 | 6.63 | FeS _x | | | |
| | 710.3 | 49.31 | FeO(OH) | 710.4 | 39.71 | FeO(OH) |
| | 711.9 | 26.28 | Fe ₂ O ₃ | 711.6 | 33.69 | Fe ₂ O ₃ |
| | 713.7 | 17.78 | FeSO ₄ | 713.4 | 26.60 | FeSO ₄ |

reaction, which makes the content of FeS₂ in the sulfidation product greatly increase. The main component in the iron oxide-sulfide products is FeS₂, and contains some intermediate products, such as FeS, and elemental S. The XPS result shows that some FeSO₄ is formed in the sulfide products, and a small amount of FeO is detected in the Fe₂O₃ sulfidation product. It is presumed that because of the presence of a small amount of O₂ in the sulfidation system, resulting FeS oxidizing reaction occurs: FeS $\xrightarrow{O_2}$ FeSO₄, FeO. Due to the relatively small amount of oxygen in the oxidation system, the contents of FeSO₄ and FeO in the sulfide products are not high.

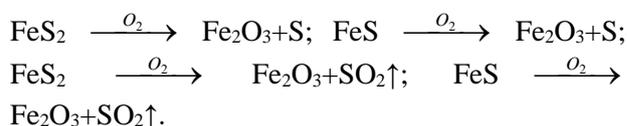
Analysis of the oxidation behavior of sulfidation products

The previous studies have shown that iron sulfide compounds such as FeS are pulverized and ground and can be slowly oxidized at room temperature [24]. But the tendency of spontaneous oxidation of FeS will be weakened after the temperature rises to 50°C. In addition, the FeS has the tendency to be converted into FeS₂, so the FeS₂ has stronger spontaneous oxidation ability during exothermic oxidation.

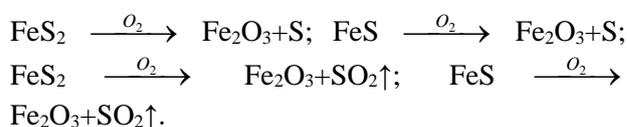
Buckley and Woods studied the surface of pyrite (FeS₂, FeS, etc.) exposed to oxygen by XPS [25]. It was concluded that FeSO₄ was generated quickly and stably, and S₈ was formed during the oxidation process. After the prolonged oxidation, with the disappearance of elemental S, the iron oxides appeared.

At room temperature, O₂ in the environment can oxidize the S atom of the sulfur compounds into elemental S and release heat. The concentration of the heat makes the oxidation reaction further strengthened, and the S atom of the iron-sulfur compound is oxidized to SO₂, while releasing heat.

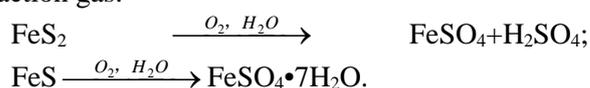
This is also the reason for the irritating odor generated in the experiment.



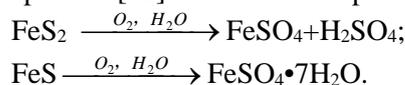
Due to a substantial increase in the content of Fe₂O₃ after oxidation, whereas, the content of other iron oxide is almost unchanged, it is considered that the oxide of iron produced after oxidation is mainly Fe₂O₃, which mainly involves the reaction:



After the oxidation reaction, the content of FeSO₄ in the product was significantly increased, indicating that the S atom was further oxidized to the higher valence SO₄²⁻ except for the above occurring oxidation reaction. During the experiments, a small amount of droplets were generated on the inner wall of the reacted three-necked flasks, which was due to the condensation of the water vapor carried in the reaction gas.



It was found that the presence of H₂O greatly increased the spontaneous combustion of sulfur compounds [26]. The oxidation process is:



From the results of XPS it follows that when the sulfidation product is oxidized at room temperature, the content of elemental sulfur in the oxidation product is dramatically reduced, but the amounts of Fe₂O₃ and FeSO₄ obviously increase. It is shown

that the generation of sulfur is only in the initiation process of oxidation reaction. With the progress of oxidation reaction, the reducing substances such as FeS₂, FeS and S in the sulfidation product are gradually oxidized to Fe₂O₃ and FeSO₄.

CONCLUSIONS

In order to simulate the sulfur corrosion process in the oil tank, a method for vulcanizing the wet H₂S gas containing a small amount of oxygen with three kinds of iron oxides, Fe₂O₃, Fe₃O₄ and Fe(OH)₃, was proposed. The results of SEM and EDS showed that various iron oxides can produce the same kind of iron-sulfur compounds after sulfidation, only their microstructure and elemental distribution are different. The analysis by XPS indicated that the different iron oxides have the same sulfur corrosion products, mainly FeS₂, FeS, elemental S and a small amount of FeSO₄. The oxidation process of iron oxides is the process of converting iron oxides into FeS₂ and FeSO₄, which are accompanied by the formation and transformation of FeS and S₈. The presence of a small amount of oxygen in the sulfide gas is the main reason for the formation of FeSO₄. In addition, the oxidation reaction involved oxygen which promoted the conversion of FeS to FeS₂ which has a stronger ability of spontaneous oxidation.

At room temperature, the oxidation process of the sulfidation products is a process of gradually oxidizing the reducing substances, such as FeS₂, FeS, S, etc. Through temperature monitoring it is found that the oxidation exothermic process belongs to the low-temperature oxidation process, which required a temperature below 300 °C. However, the presence of H₂O promotes the oxidation reaction, and the reducing substances in the vulcanized product are finally oxidized to Fe₂O₃ and FeSO₄.

Acknowledgments: This work is financially supported by the Natural Science Foundation of China(Grant No.21171083)and the Natural Science Foundation of Liaoning Province, China (Grant No. 201202125).

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Изследване на сулфидирането на железни оксиди и поведението на сулфидираните продукти при окисление

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Постъпила на 27 декември 2017, приета на 31 януари 2018

(Резюме)

С цел да се оцени сулфидирането на железните оксиди и окислителното поведение на продуктите на сулфидирането при стайна температура, в настоящата статия е симулиран серният корозионен процес в маслен контейнер. Микроструктурата и състоянието на повърхността на продуктите на сулфидирането на Fe_2O_3 , Fe_3O_4 и $\text{Fe}(\text{OH})_3$, които са основните компоненти на ръждата, са характеризирани чрез SEM и EDS. Химичният състав на повърхността на сулфидираните и окислените продукти е характеризирани чрез XPS с оглед да се изясни степента на окисление и механизмът на окисление на продуктите на сулфидирането. Установено е, че трите вида оксиди на желязото дават един и същ вид съединения на желязото и сярата след сулфидиране, но с различна микроструктура и разпределение на елементите. Основните компоненти на серните корозионни продукти на трите железни оксида са идентични. Серният корозионен процес на железните оксиди ги превръща съответно в FeS_2 and FeSO_4 , което се съпровожда с образуване и трансформация на FeS и S_8 . При стайна температура окислителният процес на продуктите на сулфидирането завършва с превръщане на съединенията между желязото и сярата в Fe_2O_3 и FeSO_4 .