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Evaluation of TG202 inhibitor for tubing steels in 15% hydrochloric acid by electrochemical noise technology

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Abstract

Acid fracturing is an effective technology for increasing oil and gas production. However, the acid will cause serious corrosion to the tubing. In this paper, the inhibition performance of the TG202 inhibitor for acidizing of high temperature and high-pressure gas wells on N80 carbon steel and 13Cr martensitic stainless-steel tubing in 15% hydrochloric acid was studied by electrochemical noise technology. The results showed that with the increase of TG202 inhibitor content, the noise resistance increased and the corrosion rate of tubing steel decreased. Under the same condition, the order of corrosion rate of tubing steels: 13Cr > HP-13Cr > N80 > P110. The pitting corrosion of HP-13Cr and 13Cr is significant. The research showed that the TG202 inhibitor had a protective effect on tubing during acidizing. The inhibition mechanism of the TG202 inhibitor was discussed.

1. Introduction

Acidizing process is one of the main means to improve oil and gas production, but acid injection can cause serious corrosion of tubing. Corrosion inhibitors are frequently added in the actual acidizing process in order to reduce the corrosion of tubing and to improve their service performance [1–5]. The reaction between acid and metal matrix, the adsorption of corrosion inhibitor on the metal surface and the film-forming process is all unsteady processes. And, the traditional polarization curve test method has great limitations for the study of the film-forming and adsorption process of corrosion inhibitors. Electrochemical noise (abbreviated as EN) technology is a phenomenon of spontaneous potential or current fluctuation on electrode surface during corrosion process, which provides a lot of information about system evolution, including the change of system from quantity to quality. The EN technology is studied based on potential or current fluctuation information, which is an in situ, non-destructive and non-interference electrochemical detection method [6], and can effectively measure the corrosion behavior of tubing in the acidification process.

There are many viewpoints on the formation mechanism of the EN, but it is generally believed that the EN of uniform corrosion comes from the formation and disappearance of micro-batteries on the metal surface, or the formation and stripping of hydrogen bubbles on the metal surface. The characteristics of current noise are generated by high frequency and small amplitude, which generally has no obvious noise peak and follows a typical normal distribution (also known as Gaussian distribution). While, the current noise generated by local corrosion (such as pitting corrosion, etc) contains an obvious transient peak of noise which is randomly and follows Poisson distribution [7–10]. The time-domain analysis and frequency domain analysis are the key to researching the corrosion behavior of metal through electrochemical noise analysis [6, 11–17]. Time-domain refers to the change of potential or current noise with time, and frequency domain refers to the change of spontaneous signal of the system with frequency. Aballe et al [18] and Dong et al [19] introduced the
electrochemical noise technology to the study of corrosion inhibitor mechanism and discussed the corrosion inhibition mechanism in-depth with the use of time-domain analysis and frequency domain analysis. Meanwhile, Zhang et al. [20, 21], Dong et al. [22] and Homburg et al. [23, 24] systematically summarized the advantages and disadvantages of different electrochemical noise data processing as well as the corrosion behavior and corrosion mechanism represented by various methods, so that electrochemical noise technology has been further applied in the field of electrochemical corrosion.

In recent years, Zhao et al. [4] and Zhang et al. [5] have explored the action mechanism of the TG201 corrosion inhibitor for N80 and HP-13Cr stainless steel from two perspectives of failure mechanism and corrosion inhibition mechanism, respectively. The results show that the TG201 has excellent inhibitory efficiency on N80 steel and HP-13Cr stainless steel. However, with the increase of bottom-hole temperature, the adsorption and desorption process of the TG201 inhibitor is unstable. Therefore, a new TG202 ultra-high temperature corrosion inhibitor was developed by CNPC Tubular Goods Research Institute. In order to clarify the electrochemical corrosion performance of the corrosion inhibitor, this paper used electrochemical noise technology to study the corrosion inhibition behavior of the TG202 inhibitor on tubing steel of different materials in 15% hydrochloric acid, aiming at tubing materials commonly used in oil and gas exploitation (N80, P110, 13Cr and HP-13Cr stainless steel).

2. Experiments

2.1. Materials and specimen preparation
The type of the TG202 is a quinoline derivative corrosion inhibitor with the molecular formula shown in Chart 1. The corrosion inhibitor mainly consists two-component, the main agent A is quinoline quaternary ammonium salt, and component B is the Mannich base compound and synergist. Quinoline quaternary ammonium salt was prepared by heating 0.5 mol 8-hydroxyquinoline and 0.5 mol benzyl chloride to 160 °C for 6 h, and then adding N-N dimethylformamide after cooling to prepare quinoline quaternary ammonium salt solution with an effective content of 50% [25]. In addition, a small amount of Cu$^{+}$ was added to form a stable chelate with 8-hydroxyquinoline, so as to improve the corrosion inhibition efficiency. Mannich base was prepared by mixing 0.1 mol aniline and 20 ml absolute ethanol, dropping concentrated hydrochloric acid to make the pH value of the solution 3, adding 0.1 mol acetoephone and 0.11 mol formaldehyde solution, stirring at room temperature for 24 h, filtering, recrystallizing the crude product with acetone and vacuum drying [26].

Coupons with dimensions of 10 mm × 10 mm × 3 mm were cut from tubing steels for which the chemical compositions are shown in table 1. They were ground with SiC sandpapers up to 2000#, then rinsed with distilled water and anhydrous alcohol, followed by acetone degreasing. Finally, 1 cm² of the surface area was exposed to the 15% hydrochloric acid and the rest were covered with epoxy resin.

2.2. Electrochemical noise measurement
EG&G M273A electrochemical workstation was used in the electrochemical experiment, with dual working electrodes and reference electrodes. Two identical samples were used as working electrodes and saturated calomel electrodes as reference electrodes. During the test, the frequency was 5 Hz and the test time was 1000 s.

Table 1. Chemical composition of tubing steels (wt%).

| Steel | C   | Mn  | Si  | S   | Cr  | Mo | V  | Ti  | Cu  | Ni  | P   |
|-------|-----|-----|-----|-----|-----|----|----|-----|-----|-----|-----|
| N80   | 0.31| 0.92| 0.19| 0.008| 0.20| —  | —  | —   | —   | 0.006| 0.005| 0.01|
| P110  | 0.24| 1.19| 0.22| 0.004| 0.036| 0.021| 0.06| 0.011| 0.019| 0.028| 0.013|
| HP13Cr| 0.011| 0.48| 0.30| 0.0035| 13.46| 1.93| 0.051| 0.096| 0.36| 6.19 | 0.023|
| 13Cr  | 0.18| 0.48| 0.33| 0.001| 12.94| 0.013| 0.048| 0.016| 0.13| 0.10 | 0.020|

Chart 1. The Molecular structure of the TG202 inhibitor ((a): quinoline quaternary ammonium, (b): Mannich base)
The localized index (abbreviated as LI), also called as the pitting index, is a metric used to evaluate the sensitivity of samples to uniform or localized attack. The Localized index values less than 0.01 being more vulnerable to uniform corrosion and those LI values larger than 0.1 being more sensitive to localized to localized attack [27]. Meanwhile, the LI value can be determined according to the current noise standard deviation, root-mean-square current value, elapsed time and current density at the corresponding time [27].

2.3. Surface analysis
To further understand the morphology of the steel surface after the addition of TG202 inhibitor, the corroded specimens were investigated by a scanning electron microscope (JEOL JSM-6390A).

3. Results

3.1. Time domain analysis
3.1.1. Effect of TG202 inhibitor concentration
The electrochemical current noise (abbreviated as ECN) and electrochemical potential noise (abbreviated as EPN) of N80 in 15% hydrochloric acid with different concentrations of TG202 inhibitor were shown in figure 1. As can be seen, the fluctuation of current potential is obvious without the addition of corrosion inhibitor,
indicating obvious corrosion behavior on the surface of N80 steel. When the concentration of TG202 inhibitor increased to 0.1%, the corrosion noise potential of N80 tended to be gentle, and the intensity of the transient peak of current noise decreased gradually, suggesting that corrosion inhibitor played a role in inhibiting corrosion. Additionally, when the concentration of the TG202 inhibitor reached to 0.20%, a large number of high-frequency transient peaks still appeared in the noise current, implying that the addition of corrosion inhibitor in hydrochloric acid suppressed the anodic reaction to a certain extent and forms corrosion product film on the surface of N80 steel [21].

The noise resistance $R_n$ is the ratio of the noise potential to the noise current to the variance, but due to the influence of the reference electrode and the external environment, the noise potential will appear a low frequency or DC trend. However, such drift has little relationship with matrix corrosion. We remove dc drift from the original ECN signal by using a polynomial method of 5th order. The results of potential standard deviation, current standard deviation, LI and noise resistance are shown in table 2. LI values of the specimens below 0.01 with the increase of inhibitor concentration, which belongs to uniform corrosion. Since $R_n$ is inversely proportional to the corrosion rate [28], the increase of the noise resistance from 0.29 to 32.96, indicated the reduced corrosion rate of N80 steel in the presence of the TG202 inhibitor, where the corrosion inhibitor may form a protective film on the steel surface.

3.1.2. Effect of tubing steel

The current noise power density curve of samples with different materials (N80, P110, 13Cr and HP-13Cr) added with 0.1% TG202 inhibitor in 15% hydrochloric acid at 90 °C is shown in figure 2. The noise potential of the four materials is in the order of HP-13Cr > 13Cr > N80 > P110. It can be seen that the surface noise amplitude of HP-13Cr stainless steel and 13Cr stainless steel is the smallest, and there is an obvious high-frequency current transient peak, corresponding to metastable pitting and passivation process on the sample surface. However, the current amplitude on the surface of N80 steel and P110 steel is obvious, and the lifetime of the corresponding transient peak is obviously higher than that of HP-13Cr and 13Cr stainless steel, illustrating that the corrosion process on the surface of N80 and P110 steel is a mainly slow process controlled by diffusion [29, 30]. The noise resistance (table 3) indicates that the corrosion rate of different tubing steels at the same experimental condition decreases as the following order: 13Cr, HP13Cr, N80, and P110. Probably due to the relatively poorer corrosion resistance of carbon steels (N80 and P110), it was prone to form a compact film on the surface when they were in contact with an acidic solution. Therefore, the current noise fluctuates greatly. Due to the existence of passive film on the surface of stainless steel (13Cr and HP-13Cr), when they contact with an acid solution, the dissolution process of the passive film first occurs, resulting in local pitting corrosion; After that, the inhibitor film is covered on the substrate surface, but it is difficult to completely cover the sample surface, resulting in a large corrosion rate, that is, an obvious high-frequency transient peak is formed on the current noise spectrum. Moreover, the LI value of the 13Cr sample is higher than that of HP-13Cr, indicating that its localized corrosion tendency is higher than that of HP-13Cr. As a result, the passivation film stability of HP-13Cr stainless steel is obviously higher than that of 13Cr, its corrosion rate is much lower than that of 13Cr.

3.2. Frequency domain analysis

3.2.1. Effect of TG202 inhibitor concentration

In order to further explore the inhibition effect of corrosion inhibitors, fast Fourier transform (FFT) was performed on the time-domain spectrum, and the transformation formula is shown by equation (1) [28, 31]:

$$P(\omega) = \frac{1}{\sqrt{2\pi}} \left( \int S(t) e^{-j\omega t} dt \right)^2$$

where $P(\omega)$ is a spectrum of energy density (frequency density, power spectral density), $V^2 \cdot Hz^{-1}$ or $A^2 \cdot Hz^{-1}$; $\omega$ is the angular frequency, $Hz$, and $S(t)$ is the measurement signal.

The characteristic parameters of power spectrum noise were obtained by Fourier transform of potential and current time noise, including height $W$ of the horizontal part of the curve (white noise level), frequency $f_0$ of the
turning point of the curve (cut-off frequency), slope $K$ of the high-frequency linear part of the curve. It is generally believed that the larger the $K$, $W$ and $f_c$ values in the current power spectrum are, the corrosion is mainly local corrosion; the smaller the $K$, $W$ and $f_c$ values are, the corrosion is uniform corrosion or in passivation state [6, 10]. Some scholars also believe that the $K$, $W$ and $f_c$ values in the current power spectrum cannot be used to distinguish whether the corrosion is local or uniform [6, 11, 12]. However, both represent the larger $K$, $W$ and $f_c$ values in the current power spectrum, which is proportional to the corrosion degree [32–35].

After FFT conversion, the power spectral density (PSD) of current and potential with or without corrosion inhibitor was obtained, as shown in figures 3 and 4. The value $W$ (white noise) in the horizontal section of the PSD curve is used to evaluate the corrosion resistance of the material, and the slope $K$ in the linear part of the

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**Figure 3.** Potential power spectral density for N80 steel inhibiting efficiency in 15% hydrochloric acid at 90 °C with different concentrations of TG202 inhibitor.

**Figure 4.** Current power spectral density for N80 steel inhibiting efficiency in 15% hydrochloric acid at 90 °C with different concentrations of TG202 inhibitor.
high-frequency band is used to describe the corrosion intensity and corrosion tendency of the corroded electrode surface. The fitting parameters are shown in table 4. As can be seen from table 4, the $K$ values of current noise and voltage noise are very small in the whole corrosion process, ranging from $-0.36$ to $-1.50$ dB/° and $-5.26$ to $-0.67$ dB/°, respectively, which are almost smaller than $-20$ dB/° [32], indicating that uniform corrosion is likely to occur. In the presence of the TG202 inhibitor, the decreased $W$ of current noise from $1.72 \times 10^{-24}$ to $1.2 \times 10^{-25}$ and the increased potential noise from $6.23 \times 10^{-12}$ to $3.23 \times 10^{-10}$, reveals that the corrosion resistance of N80 in acid solution was improved after the addition of TG202 inhibitor.

### 3.2.2. Effect of tubing steel

At the same condition (90 °C, 15% hydrochloric acid and 0.1% TG202 inhibitor), the potential and circuit PSD maps for different tubing steels are shown in figures 5 and 6, and the linear fitting parameters are listed in table 5. It can be seen that the slopes of stainless steels (13Cr and HP–13Cr) are significantly higher than those of carbon steels (N80 and P110), indicating that stainless steels have a greater corrosion tendency at the same condition. The PSD slopes of 13Cr and HP–13Cr stainless steel are both greater than $-2$, suggesting that obvious pitting behavior occurs on the electrode surface. Furthermore, the changes of $W$ in PSD$_{A}$ and PSD$_{E}$ are consistent, indicating that the corrosion resistance of tubing steels increases in the following order: 13Cr, HP13Cr, N80, and P110. This is in accordance with the time domain analysis results.
3.3. Surface analysis

Figure 7 shows the microscopic morphology of corroded specimens in 15% hydrochloric acid solution. It can be seen that the corrosion of N80 and P110 specimens is severe, and the morphology of the corrosion changes obviously, mostly uniform corrosion. On the surface of 13Cr and HP-13Cr, a small number of local pits, mostly due to localized corrosion. Figure 8 illustrates the SEM morphology of corroded N80 specimens in the cases with TG202 inhibitor. The corrosion products on the surface of the specimen are significantly reduced from 0 to 0.05% TG202 inhibitor. Meanwhile, the surface of the specimen is uniformly distributed with granular...
corrosion products in the presence of 0.1% TG202 inhibitor. The surface has only scattered corrosion products with 0.2% inhibitor, indicating that with the increase of the concentration of the TG202 inhibitor, the contact between acid and metal matrix was isolated. This is consistent with the time-domain analysis results.

4. Discussion

The corrosion of metal in hydrochloric acid solution is an electrochemical corrosion process \[8, 36, 37\]. The area with lower potential is the anodic area for corrosion and iron dissolution, while the area with higher potential is the cathodic area for hydrogen evolution reaction. The reaction equations are shown as equations (2) and (3):

$$\text{Anodic reaction: } Fe \rightarrow Fe^{2+} + 2e^- \tag{2}$$

$$\text{Cathodic reaction: } 2H^+ + 2e^- \rightarrow H_2 \tag{3}$$

Figure 9 shows the schematic diagram of the action mechanism of the TG202 inhibitor. Firstly, the anions in the HCl solution adsorb on the steel surface. Due to the presence of lone pairs electrons in the heteroatoms, quinoline quaternary ammonium salts and Mannich base are susceptible to protonation. The conjugated bond in the quaternary ammonium salt molecule is coordinated with the empty orbital of the iron atom to form a single adsorption film on the iron surface. The added Cu\(^{+}\) can form a very stable chelate with 8-hydroxyquinoline, which has a strong binding force with the metal surface, and can form a very dense chelate film on the metal surface to prevent the movement of corrosion ions to the metal surface, so as to improve the corrosion inhibition effect of quinoline quaternary ammonium salt \[25\]. On the other hand, the Mannich base compounds in component B rely on the polar groups in the molecules to produce certain adsorption with the metal surface, and the O atoms and N atoms in the molecules will also interact with the added Cu\(^{+}\) to form chelates so as to greatly improve the stability of the corrosion inhibitor film \[26\].

In addition, synergists in corrosion inhibitors also play a great role. For the polar group –CHO in synergist molecules, its central atom O has two pairs of lone pair electrons, which form a coordination bond with the d electron orbital of Fe. Adsorption on the iron surface can inhibit metal corrosion, protonation and form cations in acid solution, which plays a certain protective role for the cathode of iron, making the iron surface positively charged locally and repelling H\(^+\) in the solution, while the addition of iodide can also make its characteristic adsorption and enhance its adsorption capacity \[38\]. Due to the small molecule of synergist, it can effectively fill the gap existing when other corrosion inhibitors are adsorbed on the steel surface, so that the whole material surface can be effectively covered with a dense corrosion inhibitor protective film. At the same time, synergist is a small molecule, can effectively fill other corrosion inhibitor adsorption on the surface of the iron gap, and further the entire iron surface can effectively cover a layer of dense corrosion inhibitor protective film to endow excellent corrosion inhibition effect on the steel.

**Table 5.** Power spectral density parameters for different steel inhibiting efficiency in 15% hydrochloric acid at 90 °C with 0.1% TG202 inhibitor.

| Tubing steel | Potential W/V²·Hz⁻¹ | K | f₀/Hz | Current WA⁻²·Hz⁻¹ | K | f₀/Hz |
|--------------|----------------------|---|-------|-------------------|---|-------|
| 13Cr         | 6.54 × 10⁻¹¹        | −1.90 | 5.26 | 5.56 × 10⁻⁷⁵      | −0.33 | 5.06 |
| HP13Cr       | 7.39 × 10⁻¹¹        | −1.50 | 6.59 | 5.15 × 10⁻⁷⁵      | −0.61 | 5.02 |
| N80          | 8.27 × 10⁻¹¹        | −2.14 | 6.89 | 3.30 × 10⁻⁷⁵      | −0.47 | 4.87 |
| P110         | 1.26 × 10⁻¹⁰        | −2.25 | 7.39 | 1.14 × 10⁻⁷⁵      | −1.4  | 4.53 |

Figure 9. Corrosion inhibition diagram of TG202 inhibitor.
5. Conclusions

The inhibition performance of the TG202 inhibitor on several tubing sheets of steel in 15% hydrochloric acid was evaluated by electrochemical noise technology. Based on the results, the following conclusions can be made.

(1) In 15% hydrochloric acid at 90 °C, carbon steels (N80 and P110) mainly suffered from uniform corrosion, while stainless steels (13Cr and HP-13Cr) tended to form corrosion pits.

(2) TG202 inhibitor can effectively reduce the corrosion of tubing steels caused by 15% hydrochloric acid, mainly ascribed to the coupling effects of Cu+ chelated quinoline quaternary ammonium salt polymer and synergist modified Mannich base compound.

(3) Under the same conditions, the inhibition effect of TG202 inhibitor on 15% hydrochloric acid corrosion is dependent on the tubing steels, and its inhibition performance on carbon steels is relatively more prominent.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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