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Effect of grain boundary engineering on corrosion behavior of nickel-based alloy 825 in sulfur environment

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Abstract

Grain boundary engineering (GBE) by appropriate deformation and heat treatment processes was applied to nickel-based alloy 825 due to its excellent corrosion resistance. The microstructure of nickel-based alloy was analyzed by electron backscatter diffraction (EBSD) and the effect of GBE on the corrosion resistance in high sulfur-containing environments was studied by electrochemical method. The results show that the ratio of low Σ value coincidence site lattice (CSL) grain boundary is obviously improved from 47.1% to 65.5%. The special angle grain boundaries Σ3, Σ9, Σ27 and so on are randomly distributed on the network of the large angle grain boundaries, which destroy the connectivity of the original grain boundary network, and effectively block the corrosion cracking of the material along them. The corrosion resistance of nickel-based alloy 825 in high sulfur environments is enhanced verified by polarization curve and scanning electrochemical microscope, providing references for corrosion protection during the exploitation of high sulfur gas reservoirs.

1. Introduction

With the demand for oil and natural gas increasing, high sulfur gas reservoirs have been found. However, the existence of sulfur element in the high sulfur gas fields which induced corrosion of tubing will seriously affect the normal exploitation of the oil and gas fields. Therefore, how to safely and efficiently exploit and transport is a crucial issue for sulfur-containing gas reservoirs [1–3]. Nickel-based alloys can be used under severe corrosion conditions due to their high nickel contents and Cr, Mo and other elements of the complex. Research shows that the structure of the alloy has a significant effect on its corrosion resistance [4–7].

In polycrystalline materials, the grain boundary is the transition area where grains contact with each other, where atomic arrangements distorted and have high free energy. Therefore, its corrosion occurs easily in service [6]. The design and control of grain boundary was proposed by Watanabe T. [7, 8], which is gradually developed as ‘Grain Boundary Engineering (GBE)’. GBE refers to the adjustment of polycrystalline grain boundary network in FCC metals with medium and low stacking fault energy by appropriate deformation and heat treatment processes. The GBE approach was applied to reduce the susceptibility to grain-boundary attack [8]. And GBE has a high potential to suppress intergranular degradation and improve corrosion resistance [9]. As a face centered cubic alloy with low stacking fault energy, the GBE can be used to improve the grain boundary ratio of low Σ CSL, inhibit the precipitation of carbides and improve the grain boundary properties of nickel-based alloy. Here, the value of Σ is defined the reciprocal density of coincidence. The low Σ CSL are usually characterized by small volume, highly ordered atomic arrangement and resistance to intergranular corrosion and low Σ CSL refers to Σ ≤ 29.

Xia et al. [10–12] carried out grain boundary engineering treatment on nickel-based alloy. GBE approach has been shown to be effective in enhancing the properties in a variety of low-to-medium stacking fault energy materials [13]. It was found that not only the grain boundary ratio of low Σ CSL was increased, but also the
microstructure of grain clusters with $\Sigma^{3n}$ orientation relationship was formed. They cooperated with each other to block the connectivity of grain boundaries and reduce the corrosion tendency of the matrix material, which well controlled the occurrence of intergranular corrosion, thus inhibiting the initiation and expansion of corrosion. Krupp et al. \cite{14} carried out grain boundary engineering treatment on IN718 alloy. It was found that long time isothermal heat treatment can increase the twin density and increase the proportion of special angle grain boundary, thus reducing the intergranular fracture trend of the alloy. Kim \cite{15} found that special angle grain boundaries $\Sigma^{1}, \Sigma^{3}$ and $\Sigma^{5}$ in NiAl intermetallic compound can effectively prevent crack propagation, while $\Sigma^{7}, \Sigma^{11}, \Sigma^{13}, \Sigma^{21}$ and $\Sigma^{23}$ are relatively weak. Moreover, annealing treatment is more effective than deformation heat treatment in increasing the ratio of special angle grain boundaries, which can produce higher volume fraction of low $\Sigma$ CSL grain boundaries. Moreover it was reported that through small amount of deformation by cold drawing using a draw-bench on a production line and subsequent short time annealing at high temperature, the proportion of low $\Sigma$ coincidence site lattice (CSL) grain boundaries of the Alloy 690 tube can be enhanced to about 75% which mainly were of $\Sigma^{3n}(n = 1, 2, 3, \ldots)$ type \cite{16}. Furthermore, the grain boundary engineering treatment increases fatigue life due to grain refinement \cite{17}.

The changes of the grain boundary have an important influence on the corrosion resistance of the alloy, but it is rarely reported about the relationship between the quantitative change of grain boundaries and the corrosion resistance of nickel-based alloys. Moreover, it is necessary to further improve the performance of the nickel-based alloy for long-term service in a harsh sulfur-containing environment. So, the present work will attempt to explore the effect of heat treatment on grain boundary character distribution (GBCD) of nickel-based alloy 825 by electron backscatter diffraction (EBSD), and the effect of grain boundary changes on the corrosion behavior was investigated by the electrochemical test for the feasibility of improving corrosion resistance by GBE.

### 1.1. Material and experimental

The experimental material is nickel-based alloy Incoloy 825. The elements composition is shown in Table 1. The alloy was solution treated at 1000 °C for 1 h before water cooling, then annealed at 700 °C for 8 h with air cooled. EBSD analysis was carried out on the alloy samples before and after GBE. During the test, 10% perchloric acid alcohol solution was selected to electropolish the samples after mechanical polishing. The polishing temperature was $-20$ °C, the polishing voltage was 20 V, the polishing current was 0.1 A and the polishing time was 50 s. After polishing, the grain boundary character distribution of the samples was obtained by orientation imaging microscopy system attached to a TESCAN MIRA3 field emission scanning electron microscope. The scanning area was $750 \mu m \times 750 \mu m$, and then the data was processed by channel 5 software. The polarization curves of nickel-based alloy 825 before and after heat treatment were measured under the condition of saturated CO$_2$/H$_2$S at 90 °C. The scanning speed of electrochemical polarization curve was 1 mV s$^{-1}$, and the test range was relative open circuit potential $-500 \sim 800$ mv. The corrosion morphology of Incoloy 825 before and after heat treatment was analyzed by scanning electron microscope (SEM) under the condition of saturated CO$_2$/H$_2$S.
Figure 2. The XRD pattern.

Figure 3. Grain orientation image: (a) before GBE; (b) after GBE, and orientation difference distribution: (c) before GBE; (d) after GBE. (The black line in figure is the theoretical curve).
suspended sulfur at 90 °C for 30 days. The corrosion current of tiny areas of Incoloy825 corroded before and after heat treatment was analyzed by scanning electrochemical microscope (SECM) CHI900C. The size of SECM sample was 30 mm × 3 mm × 3 mm, and the scanning range was 400 μm × 400 μm. The scanning speed was 5 μm s⁻¹, the probe potential was 0.5 V, and the substrate potential was open circuit.

2. Results and discussion

2.1. Microstructure characterization

The microstructure and the XRD of nickel-based alloy 825 before and after GBE was shown in figure 1. After heat treatment, the grain sizes of nickel-based alloy 825 changed and became more uniform and regular as we can see from (b) compared with the microstructure before the treatment. The XRD pattern (figure 2) shows that the height of the diffraction peak after treatment is accordingly higher, indicating that the crystallinity of the alloy after GBE treatment gets better, the grains are more uniform, and the lattice distortion of the grains in the alloy is reduced.

The figures 3(a) and (b) depict that the nickel-based alloy 825 grain orientation is randomly distributed in all directions, but more are inclined to {001} and {111} planes. The {101} and {111} planes are the dense surfaces of the atoms for the face-centered cubic structure (FCC), while {001} is the sub-dense surface of the atoms. As a typical FCC lattice-arranged alloy, the most sensitive crystal surface to the point of corrosion of the three most typical low-index crystal surfaces is near the {001}, while {111} and {101} are the crystal faces which have strong resistance to the point of corrosion. Figures 3(a) and (b) also illustrate that there are many fine grains with different orientations in austenite grains. The orientation differences of these grains are relatively small, which may be due to the heat treatment in the process of leaving the factory are not complete. Therefore, the alloy has many network substructures which are not connected. The ratio of the small angle grain boundaries of the non-heat treated nickel-based alloy 825 is 86%, the sub-grain boundary (orientation angle is less than 2 degrees) accounted for 49%, and the high angle grain boundaries (orientation angle is more than 10 degrees) accounted for only 14%. Small angle grain boundaries have a lower interfacial energy so it is stable and less prone to have
intergranular corrosion. Thus, compared with other stainless steel and alloy, nickel-base alloy 825 has better corrosion resistance.

As shown in figures 3(c) and (d), the crystal faces begin to become more prone to \{101\} and \{111\} planes, and the pitting susceptibility of the nickel-based alloys is reduced compared with the untreated samples. Moreover, the grain sizes of the treated alloy become uniform and regular, and many twin structures appear at the same time. The precondition of the grain boundary engineering optimization is the generation of a large number of annealing twins. As nickel-based alloy 825 has low stacking fault energy, in the annealing process the general high angle grain boundary migration occurs continuously, and stacking fault occurs in the \{111\} plane. Thus, the annealing twins are produced in different directions, and these twins meet to form a \(\Sigma 3\) boundary. This is the process of \(\Sigma 3 + \Sigma 3 \rightarrow \Sigma 9, \Sigma 3 + \Sigma 9 \rightarrow \Sigma 27\) forming twin chains [9]: At the same time, the formation of a large number of twins can improve the ductility and the toughness of the material. The essential characteristic of the GB-engineered microstructure is formation of many large twin-boundaries as a result of multiple-twinning, which results in the formation of large grain-clusters [18]. Figure 3(d) illustrates that the grain orientation difference of the alloy has changed greatly since the small angle grain boundaries have

\[ \text{Figure 5. Corrosion Morphology of Nickel-Based Alloy 825 in Different Sulfur State at 90 °C for 30 days in different sulfur conditions: (a) before GBE without sulfur; (b) before GBE with suspended sulfur; (c) before GBE with deposited sulfur; (d) after GBE without sulfur; (e) after GBE with suspended sulfur; (f) after GBE with deposited sulfur.} \]
developed into large angle grain boundaries [19–21], whose ratio reached 90%. It is mainly due to the increase of special large angle grain boundaries after GBE.

$\Sigma 3$ has a higher occupancy rate in the special large angle grain boundaries. Since the relatively low energy of the $\Sigma 3$ grain boundary, its stable self-property, less segregation of impurities, and its much lower mobility can significantly improve the corrosion resistance of the alloy. The strong corrosion resistance of nickel-based alloy 825 depends on the highly ordered arrangement of the grain boundary atoms and the reduction of the interfacial energy at the special grain boundaries. From EBSD-grain boundary reconstructed images and statistical diagrams of low $\Sigma$ CSL grain boundaries (figure 4), the proportion of low $\Sigma$ CSL grain boundaries in Incoloy825 nickel base alloy is 47.1% before GBE, most of which are $\Sigma 3$ twin boundary (44.3%). Relatively, the proportion after GBE is significantly increased to 65.5%, $\Sigma 3$ twin boundary reaches 61.5%. We can observe that the connectivity of the original network can be destroyed when the special angle grain boundary is randomly distributed on the grain boundary network which are composed of the general large grain boundaries, thus effectively blocking the expansion of other failure modes, such as intergranular corrosion cracking and transgranular corrosion [9].

From figures 4(c) and (d), the proportion of special large angle grain boundaries is significantly increased after GBE, especially $\Sigma 3$ from 1.6% to 48.8%. The study found that the correlation factors such as the increase of the number of $\Sigma 3$ grain boundaries affect the corrosion resistance of the alloy [22]. The higher the proportions of $\Sigma 3$ in the special grain boundaries are, the weaker the intergranular corrosion is, and the stronger the corrosion resistance of the alloy is. The changes of $\Sigma 3^n$ grain boundary ratio are mainly due to the release of strain energy storage during annealing, and the amount of strain energy storage has an important influence on the formation of $\Sigma 3^n$ grain boundary ratio [18].

Numerous studies [23–25] indicate that special large angle grain boundaries can effectively improve the intergranular corrosion resistance of alloy, while the corrosion rate and pitting initiation rate optimized by grain boundary engineering are reduced obviously. The grain boundary restructuring image shown in figure 4(b) clearly reflects that the low $\Sigma$ CSL grain boundaries ($\Sigma \leq 29$) such as $\Sigma 3$, $\Sigma 9$ and $\Sigma 27$ distribution on the grain boundary network are composed of the general large angle grain boundaries. It can effectively prevent grain boundary corrosion cracking or other failure modes. Therefore, it plays a decisive role in improving the corrosion resistance of nickel-based alloy 825. Because the diffusion rate of carbon to the grain boundary is greater than that of chromium, the content of chromium in the grain boundary and its adjacent areas is greatly reduced due to the precipitation of $\text{Cr}_2\text{C}_6$ carbide, and the chromium in the grain boundary is too late to diffuse to the grain boundary, thus forming the chromium poor area. For nickel-based alloys with high chromium content, the content of chromium in the low $\Sigma$ CSL grain boundary is much higher than that in the general large Angle grain boundary, which reduces the intergranular corrosion. In addition, the annealed twins produced during the annealing process can reduce the tendency of carbide precipitation, improve the surface stability of the matrix and effectively reduce the corrosion tendency.

3. Effect of grain boundary on corrosion resistance of alloy

The corrosion morphology of nickel-based alloy 825 before and after grain boundary engineering treatment at 90 °C for 30 days in different sulfur conditions are shown in figure 5. Under the blank conditions, pitting...
corrosion occurred in the sample, the pitting density of the metal matrix surface before treatment was larger and the corrosion was more serious. Under the suspended sulfur conditions, the surface of the untreated specimen produced obvious grain peeling and intergranular corrosion. After the treatment, the corrosion degree of the alloy was reduced. Although the grain was loose, the grain boundary protruded obviously and no grain peeling occurred. Under the deposited sulfur conditions, the intergranular corrosion cracking occurred on the surface of the alloy substrate before treatment, and the intergranular corrosion cracking was also produced [18]. Although the surface of the treated alloy continued to produce grain peel off, there was no phenomenon of corrosion cracking, which shows that the untreated alloy is more seriously eroded. Based on the analysis of matrix morphology before and after the treatment, it is found that the deposited sulfur state has a serious effect on the corrosion of the alloy, and gradually changes from pitting to intergranular corrosion. As the corrosion time is prolonged, the corrosion is aggravated; corrosion cracking occurs before the treatment while the alloy after treatment has a strong resistance to sulfur corrosion, which is related to the increase of grain boundaries at special angles [25].

Figure 6(a) demonstrates the trigeminal grain boundary of the $\Sigma3-\Sigma3-\Sigma9$ grain cluster. The $\Sigma3-\Sigma3-\Sigma9$ ternary grain boundary is the basic form of intergranular corrosion. When the corrosion is generated from $\Sigma9$ and extends to the two associated grain boundaries such as $\Sigma3$, the $\Sigma3$ energy which is low and has a good corrosion resistance can prevent the further expansion of corrosion, effectively reducing the degree of intergranular corrosion of the alloy. When corrosion occurs in the grain clusters, as shown in figure 6(b), the corrosion first appears at $\Sigma27$ and then extends to $\Sigma9$ grain boundary and stops at the trigeminal grain cluster $\Sigma3-\Sigma3-\Sigma9$. Therefore, in figure 6(c), no matter whether intergranular corrosion begins at $\Sigma27$, $\Sigma9$, or $\Sigma3^n$ ($n > 3$), the triangular grain boundary cluster such as $\Sigma3-\Sigma3-\Sigma9$ will prevent propagation to achieve the effect of blocking corrosion [26]. Single-step processing comprising small amount of deformation (typically 5 to 10 pct) followed by annealing at 1273 K for 1 h has resulted in a large fraction of $\Sigma3^n$ boundaries and a significant disruption in random high-angle grain boundaries (RHAGBs) connectivity [27]. The untreated samples have low $\Sigma$CSL content, so the formation of the triple grain clusters is also less and the ability to inhibit corrosion is weak. On the contrary, the treated sample has increased $\Sigma$CSL content and therefore has better resistance to intergranular corrosion [24]. Taken as a whole, the low $\Sigma$CSL grain boundary is not easy to corrode, so the higher proportion of the low $\Sigma$CSL grain boundary occupancy is, and the smaller, the tendency of corrosion is. And less corrosion will be caused by intergranular corrosion [25]. The corrosion pit is more and more less, which also explains the phenomenon that the enhanced corrosion resistance of the treated nickel-based alloy 825.

The passivation range of the alloy after GBE treatment becomes wider and the bluff breaking point increases from 0.322 v to 0.423 v (figure 7), indicating that the corrosion tendency of the alloy surface is reduced. In
addition, after heat treatment, Incoloy 825 has lower dimensional current density and passivation current density, which indicates that GBE treated alloy is easier to enter passivation state with good surface stability and lower pitting sensitivity. It is mainly due to the low energy and strong binding ability of low $\Sigma_{CSL}$ grain boundary. So, the corrosion tendency is very low. When the ratio of $\Sigma_{CSL}$ to grain boundary is low, the electrochemical corrosion performance is improved significantly.

Scanning electrochemical microscope (SECM) can be used to obtain the surface micro-information and the electrochemical activity distribution related to the metal corrosion morphology during the corrosion process to obtain the corrosion morphology and current distribution information of the metal surface in the corrosion solution. Figure 8 shows the SECM diagram of Incoloy 825 nickel-based alloy corroded at 90 °C for 30 days before and after GBE. When Pt probe scans the surface of nickel-based alloy 825, if the product film on the substrate surface breaks and dissolves locally, the oxidation current on the probe will increase and make the surface active point.

The size of SECM probe current $i$ reflects the electrochemical activity of the substrate electrode surface. The matrix has to be reduced before each experiment, the previously generated passivation film is reduced, and the new passivation film has not yet been formed, leading to a strong drop in the current value at the tip along the x direction of panel b in the first scan. From figure 8, we can observe that three sharp current peaks appear on the
surface of the alloy before GBE. The peak values are very large, the maximum beyond $1.2 \times 10^{-7}$ A, and other small peaks are so small. Although there are nonuniform small peaks on the alloy surface, the peak values are so small that the maximum is below $5 \times 10^{-10}$ A, reduced by 3 orders of magnitude after GBE. The activity of the basic surface before and after GBE is different. From the surface current peak value, we can infer that as there are less special angle grain boundaries before GBE, which can effectively prevent the corrosion of grain clusters, it can not effectively inhibit the initiation or expansion of pitting corrosion. Meanwhile, the increase of the proportion of special angle grain boundaries after GBE effectively controls the propagation of pitting points, resulting in less and lower peak frequency. In addition, after GBE treatment, the crystal faces are more inclined to $\{101\}$ and $\{111\}$ planes, and the free energy of the alloy surface decreases. Moreover, the pitting corrosion sensitivity of nickel-based alloy decreases. This result is consistent with the change of corrosion morphology of nickel-based alloy 825 before and after GBE treatment.

4. Conclusions

(1) After 1000 °C solution treatment for 1 h, water cooling 8 h and 700 °C annealing treatment, the substructure of Incoloy825 develops into a complete grain boundary, while the grain sizes become uniform and regular; the proportion of low $\Sigma$ CSL grain boundaries increases from 47.1% to 65.5%.

(2) Compared with the untreated alloy, the grain orientation of the treated nickel-based alloy 825 becomes easier to determine, the crystal grains tend to be $\{101\}$ and $\{111\}$ planes, which can form a complete grain boundary and decrease the pitting sensitivity of the alloy surface. The grain boundary ratio of the nickel-based alloy 825 after treated is changed. The small angle grain boundaries are reduced, while the large are increased. The increase of the large grain boundary is mainly due to the increase of the special large grain boundaries, especially the $\Sigma 3$, from 1.6% to 48.8%. So, the surface energy of the alloy can be reduced, and the resistance to intergranular corrosion increased. From the corrosion morphology at 90 °C under different sulfur conditions, breaking of the grain of the alloy before treatment is serious, and the phenomenon of intergranular corrosion cracking is also observed. Although the grain is broken after treatment, the degree of corrosion is obviously reduced.

(3) After GBE treatment, the passivation range and the break down potential of Inconel 825 increases. What’s more, the corrosion tendency decreases. The peak value of surface current of the alloy after GBE treatment decreases several orders of magnitude, the pitting sensitivity decreases while the surface stability of the alloy increases.

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