Stress corrosion cracking of X-70 pipeline steels by eletropulsing treatment in near-neutral pH solution

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Published online: 5 October 2005

Slow strain rate tests (SSRT) and environmental scanning electron microscopy (ESEM) were utilized to investigate stress corrosion cracking (SCC) behavior of electropulsed POSCO and Bao Steel X-70 pipeline steels specimens in XJ solution purged with 5% CO_2 + 95% N_2 at the strain rate of 2E-6/s. The results showed that the ultimate tensile strengths (UTS) were raised considerably for the electropulsed specimens. The UTS for electropulsed POSCO X-70 pipeline steel was enhanced much more compared to electropulsed Bao Steel X-70 pipeline steel. In addition, the relationship among UTS, the original grain size and high voltage in electropulsing treatment was revealed. SCC sensitivity increased with the decreasing electrochemical potential, especially at high cathodic potential.

1. Introduction

Stress corrosion cracking (SCC) of buried pipelines from external surfaces has been recognized as a possible cause of a failure since 1965 [1–3]. In fact, no matter how well gas or oil transmission pipelines are designed, constructed and protected, once in place they are subjected to environmental abuse, external damage, coating disbondments, inherent mill defects, soil movements/instability and third party damage. Hence, SCC may occur if the combination of appropriate environment, stresses that may be absolute hoop and/or tensile, fluctuating stress, and material (including steel type, amount of inclusions, surface roughness) is present in order. If any of the three conditions can be eliminated or reduced to a point where cracking will not occur, then SCC can be prevented. Over the past several decades, it has been recognized that there are two types of SCC normally found on pipelines, and designated as high pH SCC with pH of around 9.5 and near-neutral pH SCC with pH in the vicinity of 6.5 [4–6]. High pH SCC caused numerous failures in USA in the early 1960's and 1970's [7, 8], whereas near-neutral pH SCC failures were recorded in Canada during the mid 1980's to early 1990's [2, 9]. The SCC-related failures of pipelines have occurred throughout the world including Australia [10], Russia, Saudi Arabia, Pakistan [1], South America, Iran [11], and Netherlands [12]. High pH SCC of pipeline steel is characterized by multiple, branched, intergranular cracks, whilst near-neutral pH SCC is transgranular in nature. Orientations of both types of cracks, typically parallel to the pipe axis, are perpendicular to maximum tensile stress. Near-neutral pH SCC is often associated with the formation of relatively large amounts of white iron carbonate between the pipe surface and the coating, whereas there is a quantity of black deposition, primary magnetite, in which sometimes very small amount of iron carbonate is incorporated, covering the crack surface. Up to the present time, there have been lots of papers about near-neutral pH SCC of pipeline steels, many of which are concerned with the environmental factor [1, 13–15], mechanical influence [16–20] and metallurgical effect [21–23]. Among these investigations, it is found that environmental [4] and mechanical [20] factors have strong influence on SCC, and there is a strong correlation between residual stress and the presence of near-neutral pH SCC colonies [23]. However, the study about metallurgical aspect is very insufficient. No statistically significant correlation is found between the occurrence of SCC on the pipeline steels and the other factors, such as, chemical composition, inclusion properties and local galvanic behavior [23].

It has been reported that the electropulsed samples of Cu-Zn alloy and common carbon steel are free of porosity, contamination and large microstrain. The appropriate eletropulsing treatment can affect the microstructure and mechanical properties [24, 25]

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0022-2461
DOI: 10.1007/s10853-005-1813-2

TABLE I The chemical compositions of Bao X-70 and POSCO X-70 pipeline steels (wt%)

Material	C	Mn	Si	S	P	Cr	Ni	Ca	Al	Cu	Ti	Nb	V	Mo	N
Bao X-70	0.046	1.58	0.23	0.0015	0.012	0.025	0.17	0.003	0.032	0.25	0.010	0.066	0.028	0.23	0.0033
POSCO X-70	0.060	1.59	0.18	0.003	0.007	0.007	0.24	0.001	0.025	0.013	0.020	0.060	0.052	0.23	0.005

since eletropulsing treatment refined the grains and the ultra-fine grains formed. Sometimes, electropulsing treatment can heal the pre-cracks in the specimen [26]. Many investigators have studied the effect of electric current on the behaviors of materials, such as the electromigration influence [27], the electroplastic effect [28], the solidification of metals and alloys [29], structural relaxation in amorphous solids and amorphous nanocrystallization [30]. However, no scientific papers have reported that the effect of eletropusling treatment on SCC of materials, so it is necessary to study whether the refined grain or similar results can be obtained in X-70 pipeline steels, and the influence of eletropulsing treatment on SCC of pipeline steel in near-neutral pH solution.

2. Experimental procedure

2.1. Specimen

Samples were from X-70 pipeline steel manufactured by Baoshan Iron and Steel Co. Ltd (Bao Steel) in China and Pohang Iron & Steel Co. (POSCO) in Korea respectively. Both of the steels were used in West-East natural gas transmission project in China. The weld was spiral around the axis of the Bao Steel line pipe, the angle between which was 30°. Chemical compositions of both steels were listed in Table I. The carbon equivalent in chemical composition on sensitivity of welding crack for Bao Steel and POSCO steel were 0.39 and 0.40%, respectively.

The cylindrical, waisted tensile specimens with a 12.50-mm gauge length and 2.50-mm gauge diameter were machined with gauge length along pipe longitudinal direction (L) for Bao Steel X-70 pipeline steel pipe, and with gauge parallel to the rolling direction (R) for POSCO X-70 steel plate. Specimens were finished longitudinally to 1200 grit with aluminum oxide waterproof abrasive paper, degreased in acetone, and masked (except for the gauge length) before testing.

2.2. Solution

The solution employed was the actual soil solution. The soil was from Xinjiang Uygur Autonomous Region, where West-East natural gas transmission project started. The composition of this soil was 0.0011% NO_3^-, 0.0145% Cl^-, 0.1172% SO_4^{2-}, 0.0064% HCO_3^-, 0.0422% Ca^{2+}, 0.0013% Mg^{2+}, 0.0016% K^+, 0.0195% Na^+, 19.2% water, 0.05% organic matter, 0.002% nitrogen in total, with 0.2038% calculated salt in total, collected from 1150 mm below the surface of earth in Xinjiang Uygur Autonomous Region. The conductance of lixivium was 0.562 mΩ^{-1}/cm 25°C, while the conductance of the mud was 1.33 mΩ^{-1}/cm 25°C.The soil was placed in a nylon/polyester bag and suspended in the container. The weight ratio of soil to distilled water was 1 to 5. The soil and distilled water were allowed to equilibrate at room temperature for a week prior to test. The soil solution was designated as XJ soil solution.

2.3. Electropulsing treatment

High voltage used in electropulsing treatment was 9.6 kV and 8.4 kV for POSCO X-70 pipeline steel, and 9.0, 9.2 and 8.4 kV for Bao X-70 steel, respectively. Fig. 1 showed an example of the waveform of electropulse, which was detected in situ by a Rogowski coil and a TDS3012 digital storage oscilloscope (Tektronix Inc., Beaverton, OR). In this study, the waveforms of different electropulses were similar.

2.4. SCC test

SCC tests were performed using slow strain rate tests (SSRT). The specimens were contained in airtight cells that were closed with rubber stoppers through which the ends of the specimens protruded for gripping. The test cell contained a probe to an external saturated calomel electrode (SCE), inlet and outlet through which the solution can be circulated via conduct and a small pump. Another container contained facilities for bubbling mixture gas, 5% CO_2 + 95% N_2 through solution. All of the circulated system was anaerobic. Specimens were subjected to conventional, monotonic SSRT in air as an inert environment and XJ soil solution at the strain rate of 2E-6/s and at room temperature. All electrochemical potentials were controlled by potentiostat and quoted with respect to the SCE. Upon the completion of the tests, the specimens were examined by XL30 environmental scanning electron microscopy (ESEM) from FEI Company and optical microcopy (OM) after appropriate preparation. Thereafter, metallographic longitudinal cross-sections were examined after finished and polished properly, and

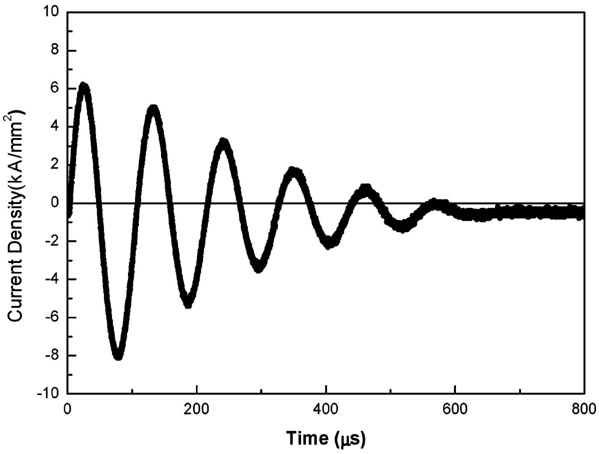

Figure 1 Typical waveform of electropulse used.

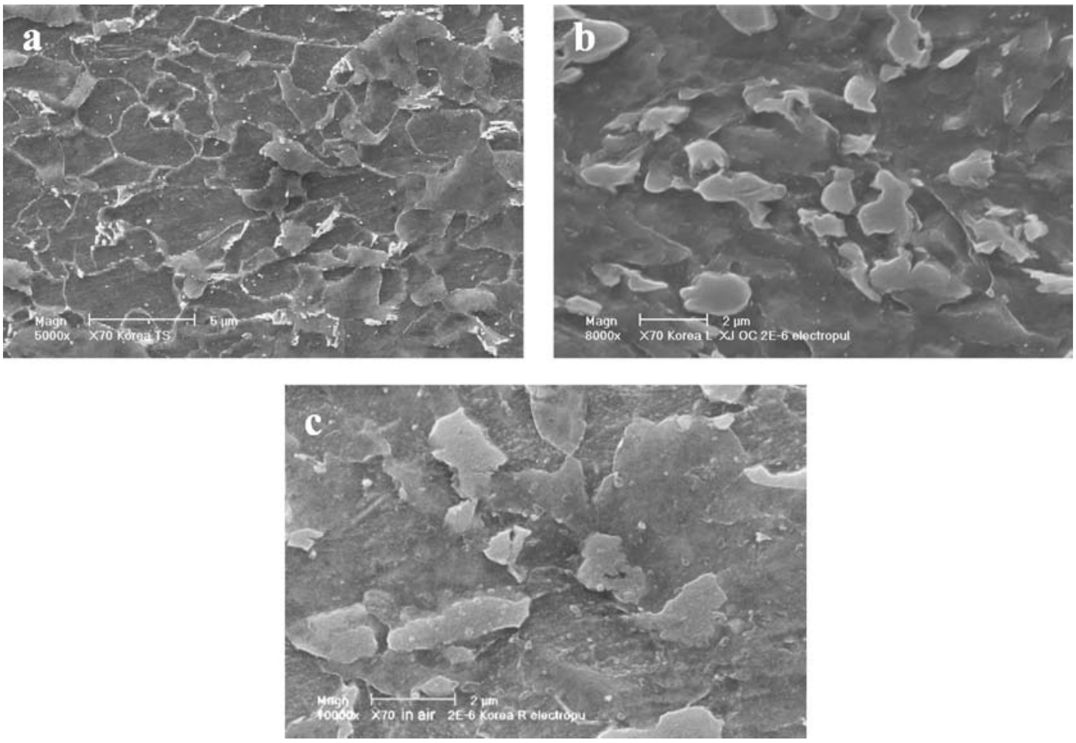

Figure 2 Microstructure of the POSCO X-70 specimen (a) before the electropulsing treatment; (b) electropulsing treated at 8.4 kV; (c) electropulsing treated at 9.6 kV.

then etched using 5% nital before cracking path was observed.

3. Results

3.1. Microstructure before and after electropulsing treatment

Fig. 2 showed the microstructure of POSCO X-70 line pipe steel before and after electropulsing treatment. Before electropulsing it was mainly equi-axial ferrite (Fig. 2a), and the average grain size was about 4 μm. When treated by a high voltage electropulse, the microstructure would change, engendered by Joule heating during the treatment, which means that the phase transformation occurred during the electropulsing treatment. In this case, the grain would be refined in the electropulsing treatment. As illustrated in Fig. 2b, the microstructure of sample treated at 8.4 kV electropulse was fine ferrite. The grain size was about 1.8 μm at some location, and the largest grain was about 2 μm. If the sample was treated at 9.6 kV electropulse, the microstructure was almost the same and the grain size was also refined, as shown in Fig. 2c.

The microstructure of Bao X-70 pipeline specimen before electropulsing treatment was shown in Fig. 3a, and the average grain size was about 7 μm. After treated at 8.4 kV electropulse, the main microstructure did not change, but the grain was refined a lot. At some location, the grain size was about 2.5 μm (Fig. 3b), but at other location, it was about 5 μm. For the specimens electropulsing treated at 9.0 kV and 9.2 kV, the microstructures were similar to that treated at 8.4 kV, and the grain sizes were almost similar too. These results mean that electropulsing treatment could produce the grain refinement and morphology change if the parameters during the electropulsing treatment were proper.

3.2. SCC of electropulsed specimens of POSCO X-70 pipeline steel

The ultimate tensile strength (UTS) of samples electropulsing treated at 9.6 kV in air at 2E-6/s increased by

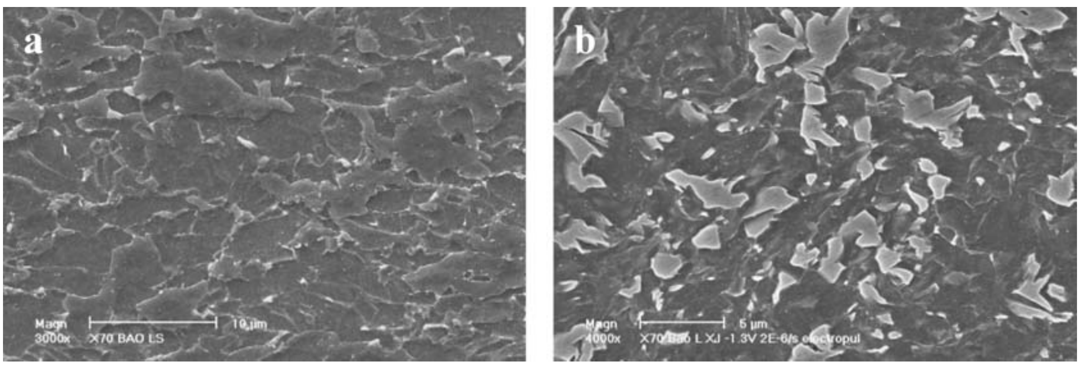

Figure 3 Microstructure of Bao X-70 pipeline steel (a) before and (b) after electropulsing treatment at 8.4 kV.

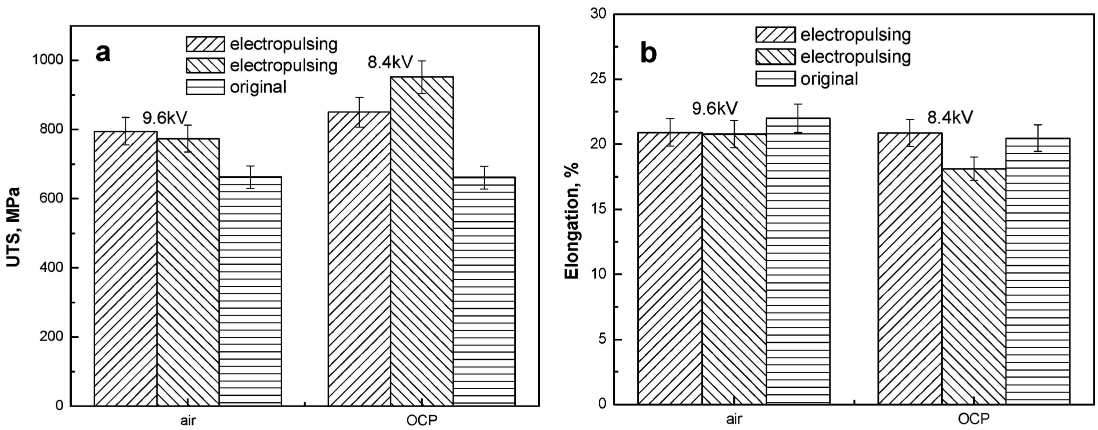

Figure 4 The results of (a) ultimate tensile strength and (b) elongation from SSRT for POSCO X-70 pipeline steel specimens with and without electropulsing treatment strained in air and solution at open circuit potential.

20 and 16.8% respectively (Fig. 4a, and the elongation only deceased by 4.9 and 5.5% respectively (Fig. 4b). The UTS in XJ solution purged with 5% CO_2 + 95% N_2 at open circuit potential (OCP) and strain rate of 2E-6/s after electropulsing treatment at 8.4 kV increased by 28.5 and 43.9% respectively, whilst the elongation increased by 2% and decreased by 11.5% respectively. Therefore, the UTS of the electropulsed specimen was enhanced without losing much ductility at the same time. For POSCO X-70 pipeline steel, the electropulsing treatment at a proper lower voltage could lead to the increase of the UTS appreciably.

Fig. 5a showed the surface morphology of the specimen elctropulsing treated at 8.4 kV with big loss of ductility in Fig. 4b. There were some deep and long second cracks perpendicular to the loading axis, however, some cracks also appeared to incline to the applied stress axis at about 45°. This implied that SCC did occur at this condition for the elctropulsed specimen. In the case of the specimen electropulsing treated at 8.4 kV with a little increase of elongation, there were fewer cracks appeared on the surface. On the specimen surface corresponding to the lower elongation in XJ solution, there were some features of steps as shown in Fig. 5b.

3.3. SCC of electropulsing treated specimens of Bao Steel X-70 pipeline steel

After the Bao Steel X-70 pipeline steel specimens were electropulsing treated, it was also found that the UTS was enhanced to some degree, as shown in Fig. 6a. The increase in UTS of the specimen strained at 2E-6/s in air after electropulsing treated at 8.4 kV twice was 5%, while the increase of the elongation was 2% (Fig. 6b). If the electropulse voltage during treatment was raised to 9.0 kV and the specimen was tested at –0.68 V_{SCE} in XJ solution purged with 5% CO_2 + 95% N_2, the UTS was enhanced by 13% and the elongation was lowered by 9.8% compared with the sample without electropulsing treatment. On the specimen surface, there were some cracks at some locations, as presented in Fig. 7a. If the parameters in electropulsing treatment were unchanged and the electropulsed sample was strained in XJ solution 5% CO_2 + 95% N_2 at OCP, the increases of the UTS and elongation compared to the samples without electropulsing treatment were 11 and 2% respectively. It was clear that crack initiated at the surface and then propagated toward the center of the specimen, as shown in Fig. 7b. Moreover, there were quasi-cleavage features near the crack initiation sites and the crack growth areas. At –0.75 V_{SCE} in XJ solution purged with 5% CO_2 + 95% N_2 after electropulsing treatment at 9.2 kV, the UTS of sample was also very high, 709.4 MPa. There were cracks on the specimen surface Fig. 7c). When the specimen was strained in XJ solution purged with 5% CO_2 + 95% N_2 at –0.8 V_{SCE} after electropulsing treatment at 9.0 kV, the UTS was raised by 11% while the elongation was lowered by 13%. There was quasi-cleavage on the fracture surface. However, if the specimen was strained in XJ solution at –1.3 V_{SCE} after electropulsing treated

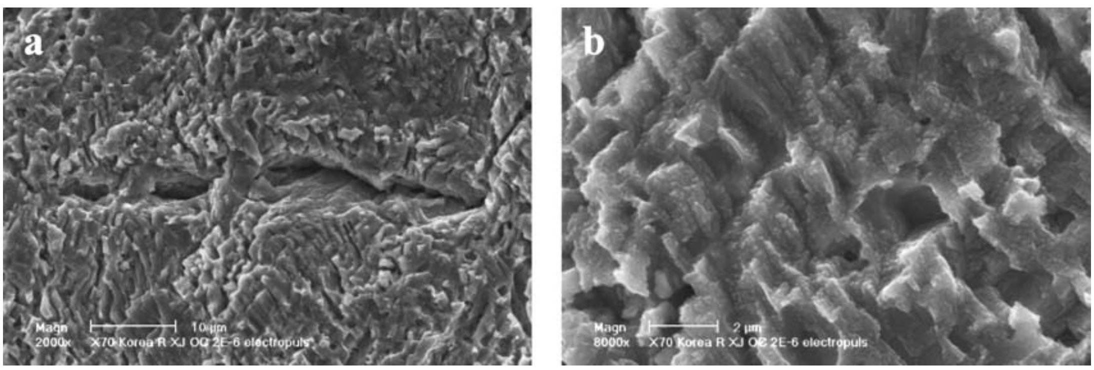

Figure 5 The surface morphology of the specimen strained at 2E-6/s in XJ solution purged with 5% CO_2 + 95% N_2 at OCP after electropulsing treated at 8.4 kV.

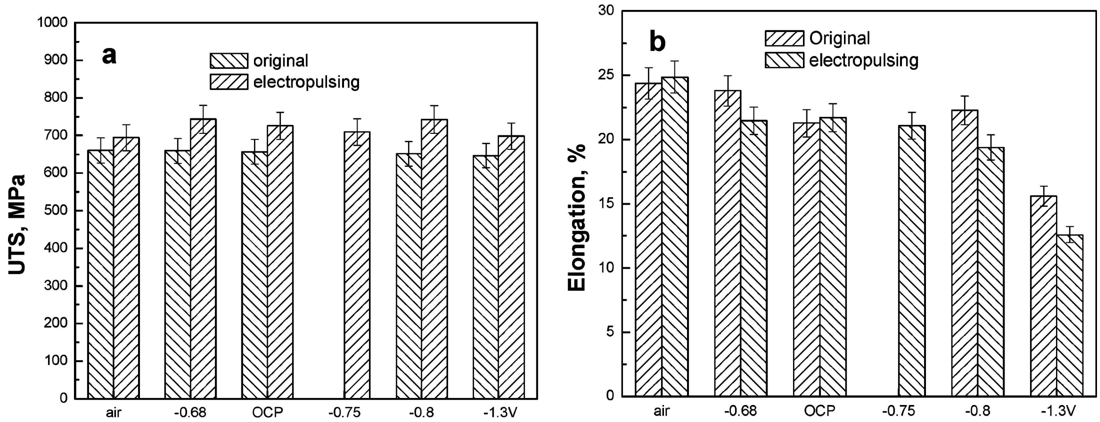

Figure 6 The results of (a) ultimate tensile strength (UTS) and (b) elongation from SSRT for Bao Steel X-70 pipeline steel specimens with and without electropulsing treatment.

at 8.4 kV, the UTS was still improved by 8% with the reduction of elongation of 19%. On the specimen surface, it was observed that there were many cracks (Fig. 7e), some of which were associated with pitting (Fig. 7d). It was also clearly observed that crack coalescence occurred at this condition. Individual cracks jointing together to form longer cracks, which caused crack growth sometimes (Fig. 7e). Fractographic ex-

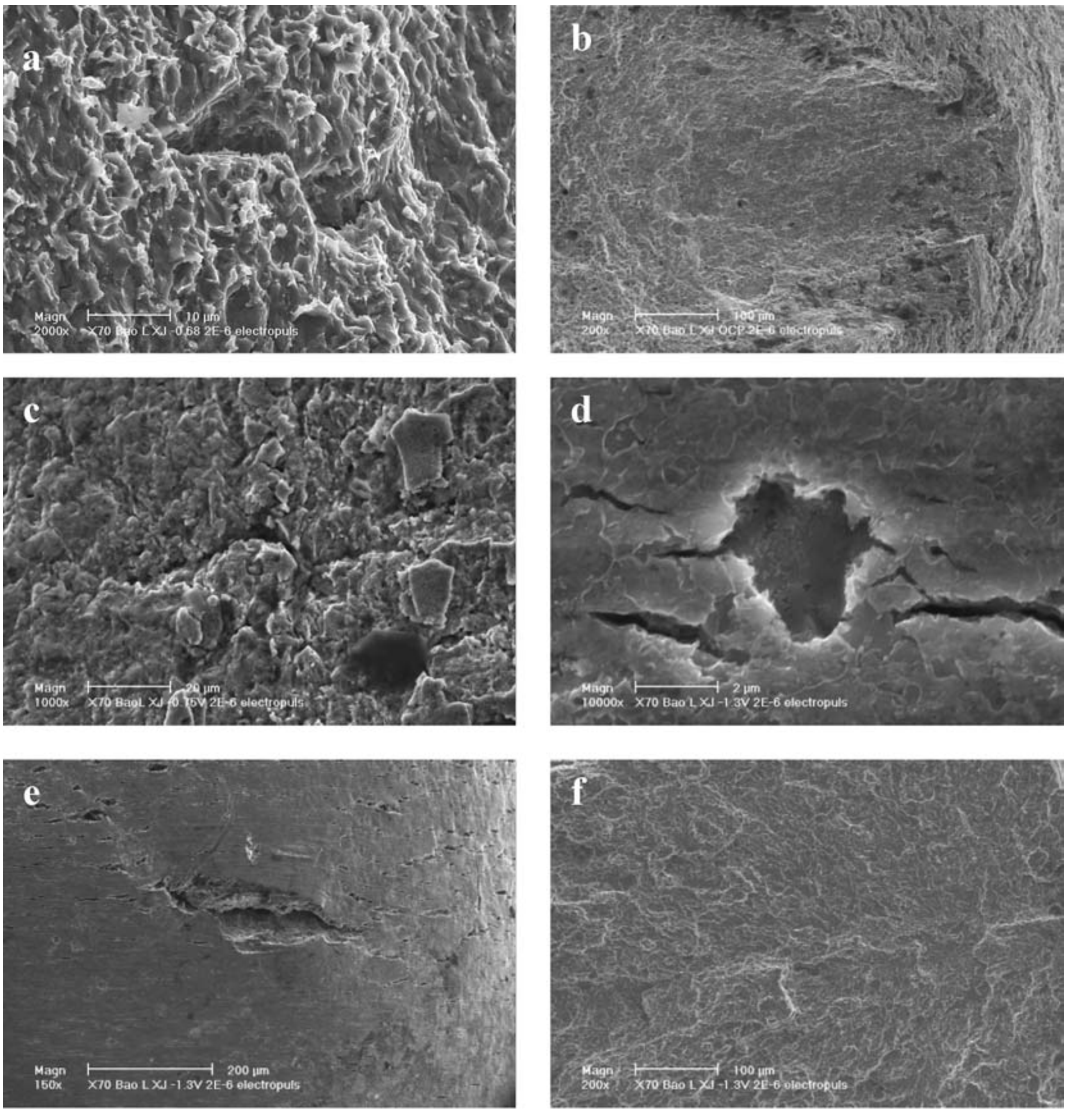

Figure 7 (a) The electropulsed specimen surface strained at 2E-6/s in XJ solution purged with 5% CO_2 + 95% N_2 at –0.68 V_{SCE} after treated at 9.0 kV; (b) the fractographic examination of electropulsed specimen strained at 2E-6/s in XJ solution purged with 5% CO_2 + 95% N_2 at OCP after treated at 9.0 kV; (c) the electropulsed specimen surface strained at 2E-6/s in XJ solution purged with 5% CO_2 + 95% N_2 at −0.75 V_{SCE} after treated at 9.2 kV; (d) and (e) the elctropulsed specimen surface tested at 2E-6/s in XJ solution purged with 5% CO_2 + 95% N_2 at −1.3 V_{SCE} after treated at 8.4 kV; (f) a fan-shaped feature on fracture surface of the electropulsed specimen in XJ solution purged with 5% CO2 + 95% N2 at −1.3V_{SCE} and at the strain rate of 2E-6/s after treated at 8.4 kV.

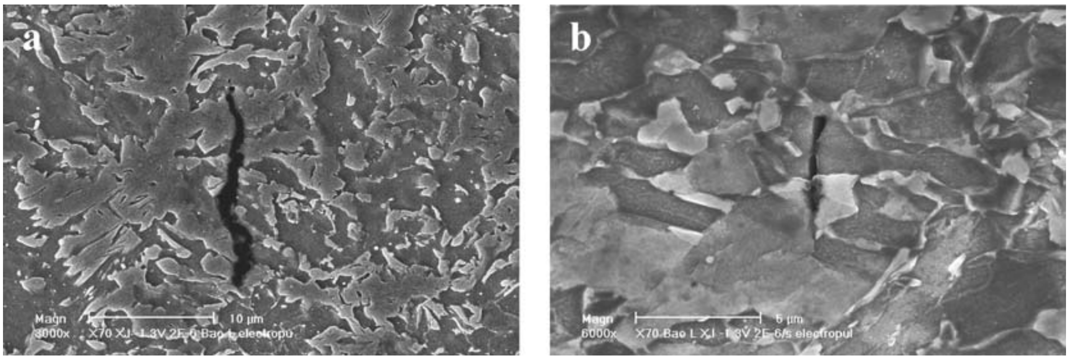

Figure 8 ESEM observation indicating (a) transgranular cracking, some ramiform feature also indicated; (b) transgranular cracking.

amination of the specimen showed the presence of fan-shaped transgranular cracks, as shown in Fig. 7f. When the applied electrochemical potential was lowered, the number cracks appeared on the specimen surface increased and the areas of quasi-cleavage also increased. In addition, the elongation also decreased with the decrease of electrochemical potential (Fig. 6b). The UTS of electropusled specimens was higher than the counterpart without electropulsing treatment.

Moreover, the cracking mode of the electropulsed specimens at various conditions was invariably transgranular, as shown in Figs 8(a) and (b). In Fig. 8a, it was clear that there were some ramiform-shaped features.

4. Discussion

The increase of the UTS of the elctropulsed specimens was correlated to the change of microstructure, i.e., the grain refinement during the electropulsing treatment in which the specimens could undergo the ferrite↔austenite solid-state phase transformation during cooling and heating of electropulsing treatment. For pure iron, the temperature of the transformation of ferrite into austenite was 1185 K, and the A_{c1} and A_{c3} for X-70 pipeline steels were lower than that value. In the electropulsing treatment, the maximum increase of temperature could be calculated according to the following equation:

$$\Delta T_{\max} = \rho j_m^2 t_p (c_p d)^{-1} \tag{1}$$

where ρ was the resistivity, j_m was the maximum current density, t_p was the width of elctropulsing, c_p was the specific heat capacity and d was the density of the specimen. As for the electropulsing voltage of 8.4 kV, the duration of an electropulse was about 800 μs, the width of electropulse was 100 μs, and the maximum current density was 8.3 kA/mm^2. So the maximum increase of the temperature was 960 K according to the Equation 1. Since the test was conducted at room termperature, about 298 K, then the temperature of the specimen was raised to about 1258 K, which was above A_{c3} and almost consistent with the reported ideal temperature for grain refinement in the experiment was about 1273 K [25].

During the electropulsing treatment, high heating rate could occur, so a large overheating would be achieved during the phase transformation $\alpha \leftrightarrow \gamma$, and the γ-phase could be produced at higher nucleation rate and smaller critical size of nuclei. Furthermore, the higher nucleation rate of γ-phase resulted from the electropulsing. Generally, γ-phase nucleus with spherical shape was formed in the original α phase. The change of free energy of the system, ΔW, including the change of free energy in a current-free system, ΔW_0, and the one associated with the change of distribution of the current in the formation of a nucleus, ΔW_e, which could be expressed in the following formula [26, 31–33]:

$$\Delta W_e = \mu g \xi(\sigma_1, \sigma_2) \Delta V j^2 \tag{2}$$

where μ was the magnetic susceptibility (in this study, μ=μ_0, where μ_0 was the magnetic susceptibility in vacuum), g was the geometric factor; ΔV was the volume of a nucleus, and j was the current density. $\xi(\sigma_1, \sigma_2)$ was the factor related to the electrical properties of nucleus and medium, σ_1 & σ_2 were the conductivity of the γ-phase and α-phase, respectively. Zhou [25, 26] gave the sign of Equation 2 was negative, which reflected that electropulsing could accelerate the nucleation rate of the γ-phase by decreasing thermodynamic barrier during phase transformation. In addition, Zhou [25, 26] also formulated the grain size between the current-free and current-carrying system:

$$\frac{D_e}{D} = \exp\left(\frac{\Delta W_e}{3kT}\right) \tag{3}$$

where D_e and D were the grain size in the current-carrying and current-free system respectively, k was the Boltmann's constant, and T was the absolute temperature. Due to ΔW_e<0, D_e/D<1 according to Equation 3, the average grain size would decrease when the electropulsing treatment was applied. According to the Equations 2 and 3, the effect of electropulsing treatment on the grain size was very significant. The smaller grain would lead to the increase in the total area of grain boundary, and when the specimens were loaded, the resistance of dislocation mobility would increase, so the UTS of the electropulsed specimen was higher than that without electropulsing treatment whereas the elongation in air for the former was also higher, as shown in Figs 4 and 6.

When the voltage of the electropulsing treatment was higher, the maximum current density increased, so the maximum increase of temperature increased.

The maximum increase of the temperature for the electropulsing voltages of 9.6 and 9.0 kV were 1254 and 1102 K respectively both of which were above the A_{c3}. So when the specimen was treated under electropulsing, the α-phase transformation into γ-phase could occur. Since electropulsing treatment was very quick, the nuclei of the γ-phase did not have enough time to grow. Since the γ-phase grains were very fine, the volume fraction of grain boundaries was very large, which could offer more nucleation sites for α-phase, so the nucleation rate of α-phase was high. Therefore the fine grain could be gained. When the electropulsing voltage was higher, which corresponded to the larger increase of the temperature, there should be much more enthalpy change released when most of γ-phase transformed back to the α-phase after the passage of electropulsing. However, this large enthalpy did not have enough time to release due to the fast electropulsing treatment, so it could produce the residual stress in the electropulsed specimens at a higher voltage. This residual stress could influence the mechanical properties. The UTS in air under electropulsing treatment of 9.6 kV were lower than those in XJ solution purged with 5% CO_2 + 95% N_2 at OCP under electropulsing treatment of 8.4 kV, as shown in Fig. 4. The elongation of electropulsed sample at higher voltage, 9.6 kV, was also reduced. As a matter of fact, the electropulsed specimens treated at high voltage of 9.6 kV exhibited a little bending, which revealed that the phase transformation in the specimens was not uniform and there were residual stresses. In the near-neutral pH SCC of pipeline steels, there was a strong correlation between residual stress and the presence of near-neutral pH SCC colonies [23, 34, 35]. So the electropusling treatment at a higher voltage also influenced the SCC behaviour. The report [25] indicated that the ideal temperature for grain refinement was about 1273 K. Even if the sample electropulsed at the same voltage, the residual stress might be different, so SSRT results were different. The effect of residual stress in air was not so significant as in solution. So the results in Fig. 4 could be interpreted. In the case of Bao Steel and POSCO X-70 pipeline steels, the original grain of Bao Steel was larger than POSCO X-70 pipeline steel, so the grain obtained after electropulsing treatment for Bao Steel specimen was larger than POSCO specimen according to the Equation 3. So the increases of the mechanical strength for the Bao Steel samples were not as much as that for POSCO specimens Figs 4 and 6. In electropulsing treatment, sometimes, ramiform-shaped feature could be obtained because the composition overcooling was very large and cystiform feature could be transformed into ramiform shape. The existence of ramiform-shaped feature produced poor mechanical properties, which provided another reason for the fact that Bao Steel X-70 pipeline steel had lower properties than POSCO steel after electropulsing, as shown in Fig. 6c. The microstructure of electropulsed specimen of POSCO was uniform, compared with those of Bao Steel, as shown in Figs 2b, 3b, 8a and b. Local plastic deformation tended to be easier to occur in the materials with mixed structure at relatively lower stresses than that with uniform microstructures, which might be correlated with the fact that the fine grain by electropulsing treatment for POSCO was resistant to SCC since there were more areas of boundaries for the fine grains, which provided more barrier for dislocation to move.

As far as Bao Steel X-70 pipeline steel was concerned, the electropulsed specimen had dispersed small carbide and the number of the carbide would increase. This dispersed distributed small carbide could act as efficient cathode and accelerate the dissolution of ferrite. In anodic region, such as –0.68 V_{SCE}, the corrosion accelerated was very clear, so the loss of ductile for electropulsed sample was larger than that for that without electropulsing treatment. At cathodic potential, these carbides could facilitate the hydrogen discharge since it had the lower hydrogen overvoltage [37]. Hydrogen could be produced easily, adsorbed on specimen surface and then diffused into the material. Sometimes, the hydrogen was trapped into the defect, or enriched around the plastic zone ahead of the crack tip, which could enhance the crack growth. The fractographic examination in Fig. 7f and the surface morphology in Fig. 7e both provided some evidence that hydrogen was involved in SCC for the electropulsed specimen. It could be observed that cathodic potential could reduce the elongation for the electropulsed specimens compared to those without electropulsing treatment. More negative potential increased the SCC susceptibility of the specimen due to hydrogen ingress.

5. Conclusion

(1) The UTS could be improved considerably after electropulsing treatment for Bao Steel X-70 pipeline steel specimens and POSCO X-70 pipeline steel specimens, which was attributed to the grain refinement by electropulsing treatment. The degree of microstructure refinement was related to the original grain size, i.e., the larger original grain size would produce the larger average grain after electropulsing. The change of UTS was also associated with the high voltage used in electropulsing treatment. Excessive higher voltage could cause large residual stress after electropulsing, which led to the loss of elongation and small increase of UTS compared with the counterpart without electropulsing.

(2) SCC did occur for the electropulsed specimens in XJ solution and the cracking mode was transgranular in nature. The SCC sensitivity of electropulsed specimens almost did not vary in XJ solution purged with 5% CO_2 + 95% N_2 at OCP and strained at 2E-6/s, compared to the one without electropulsing. While the SCC susceptibility would be raised a little in the cathodic range compared with the counterpart without electropulsing treatment. The SCC became more severe as the potential decreased, which indicated that hydrogen might be involved in SCC.

Acknowledgements

This work is supported by The Special Funds For The Major State Basic Research Projects G19990650 and The Hundred Talents Program. The authors acknowledge the assistance.

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Received 16 November 2004
and accepted 2 May 2005