Effect of Annealing Process on the Mechanical Properties of X70 Pipeline Steel

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Abstract. The influence of the mechanical properties of X70 pipeline steel under different annealing temperature was studied. The corresponding microstructure was investigated by the Field Emission Scanning Electron Microscopy. The results showed that the yield strength and the tensile strength both experienced from rise to decline with the increase of annealing temperature. The grain sizes were coarse and a large amount of cementite precipitated due to preserving temperature above 550 ℃, which induced matrix fragmentation and deteriorate the -10 ℃ DWTT Toughness. There were little changes on the microstructure and mechanical properties when the annealing temperature was under 500 ℃.

1 Introduction

With the increasing gas pressure and more harsh service conditions, long-distance pipeline is gradually required to possess thick wall, large caliber, high strength and toughness [1, 2]. Generally, X70 and X80 are applied during most of the major projects all over the world with a thickness from 15mm to 22mm. They are efficient, economic and can last for several decades and have been recognized as the mostly utilized steels for pipeline construction in the petroleum industry [3]. It is reported that [4, 5], heat treatment is usually adopted to achieve better comprehensive performance during the pipe making majorly for high-grade. Annealing is influential on the mechanical properties especially for low temperature toughness [6]. Cleavage fracture splits in separations are usually detected on such material after tensile test, impact test and DWTT (Drop Weight Tear Test) [7]. Cleavage in separations is a typical kind of fracture for controlled rolled steels, the mechanism of which however lacks of research. In the present work, the effect of annealing process on the mechanical properties was studied and the formation mechanism of cleavage in separations after DWTT was investigated.

2. Experimental procedures

2.1 Materials and sample preparation

The material studied in this work is X70 pipeline steel with thickness 20mm. The composition is shown in Table 1.

Table 1. Chemical composition.

C/%	Si/%	Mn/%	Nb+V+Ti/%	Cr+Cu+Ni+Mo/%
0.06	0.2	1.70	≤1.50	≤0.60

The steel sample was taken from 1/4 position of the width and 45° to the rolling direction by flame cutting and was cut into rough samples with dimensions 420mm × 65mm × T used for tensile test, 45mm × 18mm × T for impact test and 300mm × 90mm × T for DWTT before heat treatment.

2.2 Experiment

Muffle furnace was applied for annealing treatment on prepared samples. The samples were heated in the furnace up to 400~650 °C. Preserved for 4 hours respectively before furnace cooling down to 150 °C and then taken out for air cooling. The annealing process is shown in Table 2.

Table 2. Annealing process.

Sample No.	Annealing process	
	Heating temperature	Preservation time
1#	Blank sample (Without heat treatment)	
2#	400 °C	4 hours
3#	500 °C	4 hours
4#	550 °C	4 hours
5#	600 °C	4 hours
6#	650 °C	4 hours

After annealing, the rough samples were machined into standard tensile samples (Gauge Length 50mm), impact test samples (10 × 10 × 55mm) and DWTT samples (Pressed Notch). Tensile test was conducted on Zwick100 tensile test machine. The impact test was performed on PSW750 full automatic impact test machine from Zwick at a speed of 20 mm/min. The linear velocity at the point of impact was around 5.5m/s. DWTT was performed on the JL40000 testing machine from China.

Using Metallography and Field Emission Scanning Microscope to investigate the microstructure after the samples were polished and eroded by 4% nital.

3. Results and discuss

3.1 Tensile test

The tensile properties with different annealing process are shown in Fig. **1**. Rt0.5 and Rm goes through three stages with the increase of annealing temperature: (1) When the temperature is below 500°C, Rt0.5 increases with the annealing temperature very slightly while Rm stays the same; (2) After preserved under 550 °C for 4 hours, both Rt0.5 and Rm increase remarkably. Besides, the increase of Rt0.5 excesses Rm and as a result, the yield ratio increase rapidly; (3) When the temperature is beyond 550 °C, both Rt0.5 and Rm decrease while the yield ratio changes little.

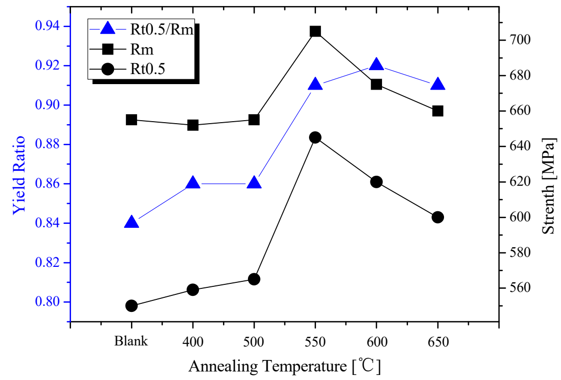


Fig. 1. Tensile test results of different annealing process.

The results of -20 ℃ impact test and -10 ℃ DWTT are shown in Fig. **2**. The impact absorbed energy decreased with the increase of annealing temperature but the general level maintained in a fine condition. When the annealing up to 650 ℃, the absorbed energy can still be around 300 J. The result of DWTT exhibited the same trend. When the annealing temperature was under 500 °C, the shear ratio experienced little changes and stay around 100%. However, the shear ratio of 4#, 5# and 6# sample showed an obvious decrease. That is to say, the DWTT properties deteriorated when the annealing temperature was above 550 °C. The fracture morphologies are shown in Fig. **3**. The fracture was mainly ductile when the annealing temperature is under 500 °C and became fragile with obvious separations when it was above 550 °C.

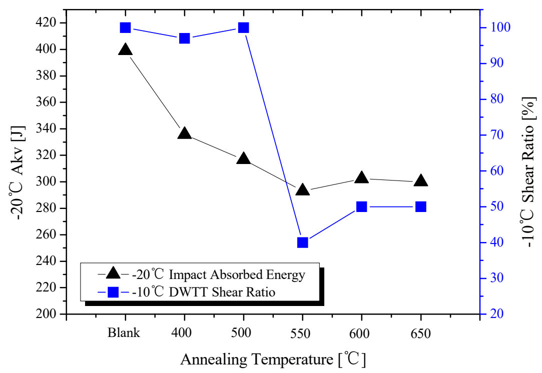


Fig. 2. Low temperature toughness with different annealing process.

1# Blank

2#400 ℃

3# 500 ℃

4# 550 ℃

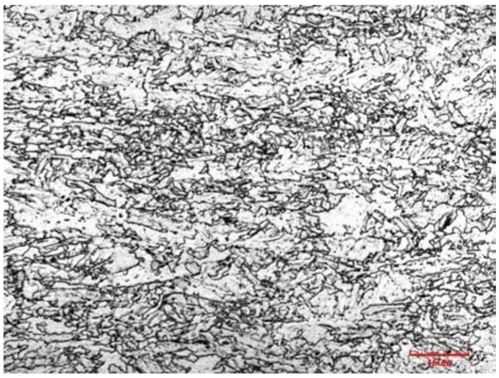
5# 600 ℃

6# 650 ℃

Fig. 3. DWTT fracture morphology.

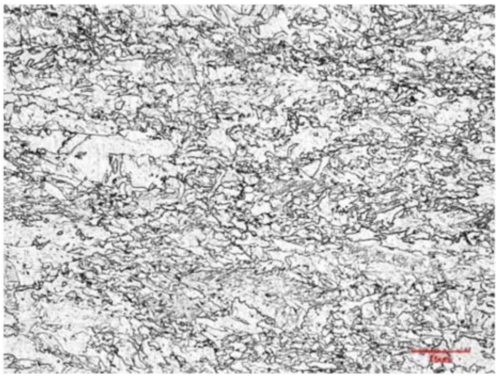
3.2 Microstructure observation

The microstructure was observed by optical microscope and the result is shown in Fig. **4**. The blank sample was dominated by acicular ferrite. There has been no significant change of microstructure when the annealing temperature was under 500 ℃. However, the grain size increased from 5 μm to more than 10 μm when it was above 550 ℃. The increase of grain size can become one of the main reasons of the decrease of low temperature impact properties.



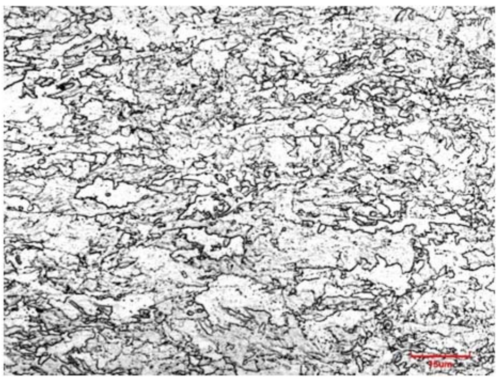
1# Blank

2# 400 ℃

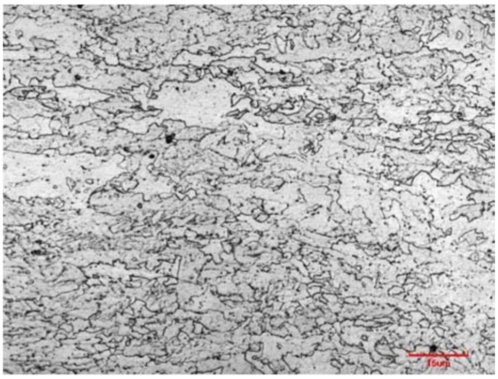


3# 500 ℃

4# 550 ℃



5# 600 ℃

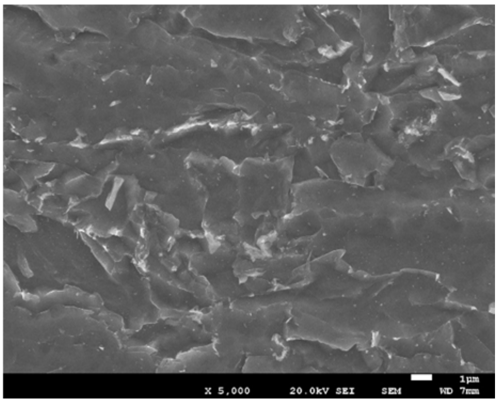


6# 650 ℃

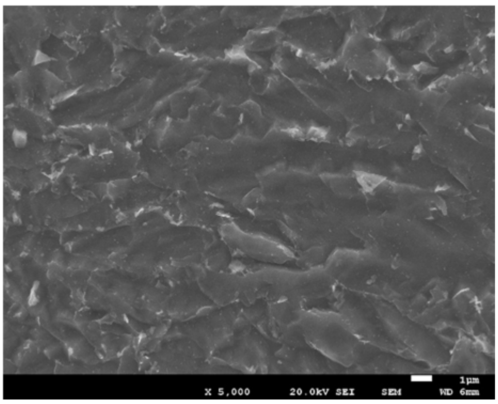
Fig. 4. Microstructure morphologies under different annealing process.

3.3 Scanning Electron Microscopy Analysis

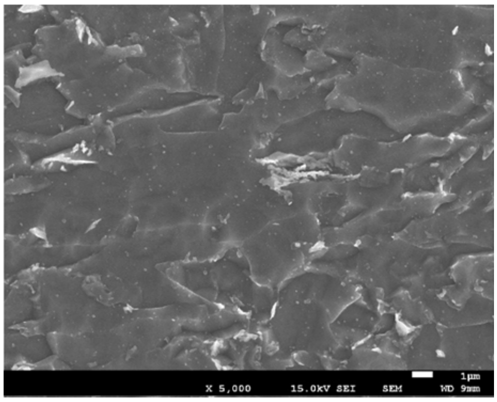
The SEM microstructure morphologies are shown in Fig. **5**. Mass precipitation particles were detected in 4#, 5# and 6# sample. The particles were mainly distributed in grain boundaries judged from 4# and 5# while even precipitated inside the grain of 6#. That is to say, with the increase of annealing temperature, the precipitation particles increased both with their number and size. No obvious precipitation particles were observed when under 500 ℃.



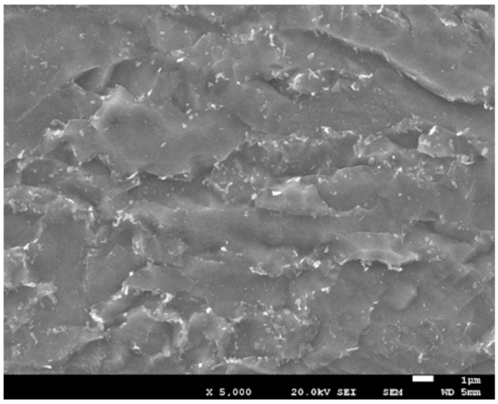
1# Blank



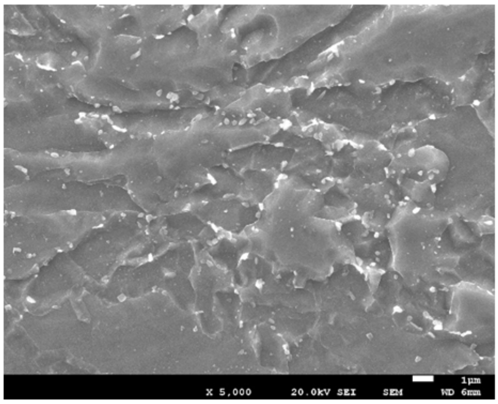
2# 400 ℃



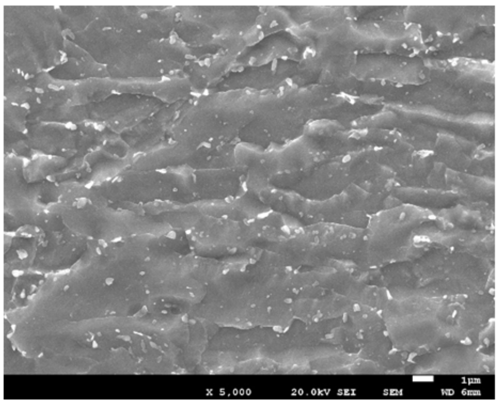
3# 500 ℃



4# 550 ℃



5# 600 ℃



6# 650 ℃

Fig. 5. SEM morphologies under different annealing process.

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In order to identify the precipitation particles, the energy spectrum analysis was conducted; the result is shown as Fig. **6** and Table 3. The particles are mainly cementite. The cementite precipitation particles can destroy the uniformity of the matrix [8, 9]. When the material is faced to impact, the stress will concentrate on the junction of the precipitation particles and the matrix and finally lead to fracture.

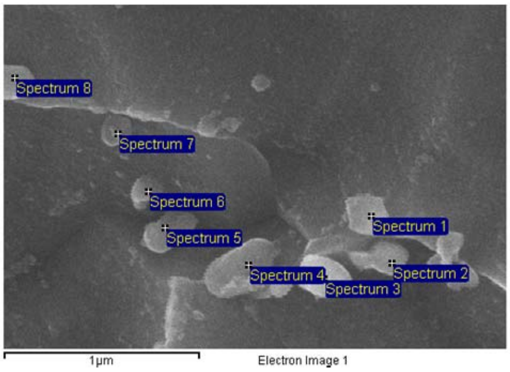

Fig. 6. Precipitation particles Energy Spectrum Analysis.

Table 3. Spectrum Results.

Spectrum	In stats.	C	Cr	Mn	Fe	Total
Spectrum 1	Yes	11.37	1.08	2.98	84.57	100.00
Spectrum 2	Yes	14.59		3.54	81.87	100.00
Spectrum 3	Yes	13.84	1.65	5.91	78.60	100.00
Spectrum 4	Yes	10.22		4.41	85.37	100.00
Spectrum 5	Yes	8.65		3.06	88.29	100.00
Spectrum 6	Yes	9.46		2.12	88.42	100.00
Spectrum 7	Yes	10.34		2.41	87.25	100.00
Spectrum 8	Yes	5.71			94.29	100.00

All results in weight [%]

The cleavage in separations morphology of DWTT fracture of 5# sample which preserved 4h under 600℃ is shown in Fig. **7**. Massive initial cracks were detected on the direction which the crack travels. Inside these small initial cracks were the cementite precipitation particles. It is obvious that, the stress concentrated around the cementite precipitation particles and emerge initial cracks, more than several cracks began to grow and therefore caused the cleavage in separations and worsened the Low Temperature DWTT Toughness.

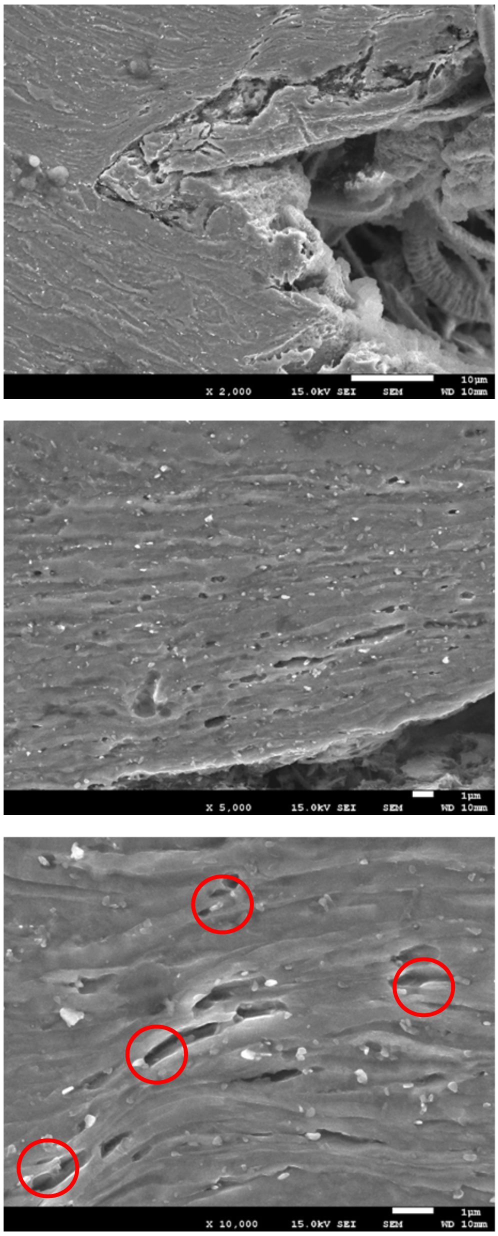

Fig. 7. Morphology of the cleavage in separations from DWTT fracture.

4 Summary

1) With the annealing temperature increases, the yield strength and the tensile strength of X70 pipeline steel both presented a trend from rise to decline. When the temperature reached 550 °C, the yield ratio experienced a remarkable increase.

2) With the annealing temperature increases, the -20 °C Impact Absorbed Energy decreases but the general level maintained in a fine condition. The -10 °C DWTT performance worsen when it is above 550 °C.

3）There is no significant change in microstructure when the annealing temperature is under 500 ℃. When it is above 550 ℃, massive cementite precipitation particles appear and the grain size grows up obviously which worsen the DWTT performance and caused the cleavage separations.

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