

## Effect of Anneal Temperature on Microstructure and Mechanical Properties of 0Cr21Ni6Mn9N Cold Drawn Pipe

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**Abstract.** The microstructure and mechanical properties of 0Cr21Ni6Mn9N austenitic stainless steel cold drawn pipe were investigated at various annealing temperatures. It was found that the tensile strength and the yield strength increased slightly, and then decreased with the temperature increasing from 200 to 750 °C. The highest value of tensile strength and yield strength reached 1062 and 942MPa at 600 °C, respectively. However, the elongation has an opposite trend to the tensile strength and yield strength. The microstructure evolution of the cold drawn pipe annealed at different temperatures was observed. The results indicate that when annealed below 550 °C, recovery is dominant. At 550 °C, it is recovery and partial recrystallization. Over 550 °C, complete recrystallization has likely occurred. Moreover, the Cr<sub>2</sub>N particles near the grain boundaries were found, and the amount of precipitates along the grain boundary indexed as M<sub>23</sub>C<sub>6</sub> were increasing at 650-800 °C.

### Introduction

Austenitic stainless steel, 0Cr21Ni6Mn9N (21-6-9) has been widely used in the aerospace, chemical engineering and nuclear industry due to its outstanding integrity and excellent toughness at elevated temperature.<sup>[1-5]</sup> The 21-6-9 austenitic stainless steel is generally used as a pipe after cold drawn process at room temperature. However, work hardening from the cold drawn process deteriorates the ductility of the steel considerably. Therefore, a proper annealing is obligatory to diminish the effect of work hardening. Fang<sup>[6]</sup> reported that multipass cold drawing led to increasing cumulative strain, strain hardening and flow stress of the material. Without inter-pass annealing, the deformation imposed exceeded the limit, which resulted in fracture. Annealing is necessary to improve properties, which has been proven in other austenitic stainless steels in technical literature.<sup>[7-10]</sup> Daymond<sup>[7]</sup> reported that extensive annealing at high temperature of heavily cold worked beta III Ti alloy resulted in grain growth, which reduced the elongation due to the lack of work hardening. However, the effects of annealing treatment on the microstructure and mechanical properties of 21-6-9 austenitic stainless steel are seldom reported.<sup>[11-15]</sup> In this study, the microstructure and mechanical properties of 21-6-9 austenitic stainless steel were investigated carefully after annealing at different temperatures. The effect of anneal temperature on grains size and shape, second phases and mechanical properties was studied. The findings are expected to provide some aspects for the optimization of annealing for austenitic stainless steel 21-6-9 cold-drawn pipes.

### Experimental Procedure

The composition of the austenitic stainless steel 21-6-9 cold drawn pipe studied is given in Table 1. The cold-drawn pipes were cut to  $\Phi 10\text{mm} \times 20\text{mm}$ , and then were annealed at 200, 400, 550, 600,

650, 700, 750, 800, 850°C with a holding time of 60 min followed by air cooling, respectively.

Table 1 Chemical composition of the 0Cr21Ni6Mn9N stainless steel (mass fraction, %)

C	Mn	P	S	Si	Ni	Cr	N
≤0.04	8.00~10.	≤0.03	≤0.0	≤1.0	5.5~8.0	19~21.	0.2~0.36
5	0	5	5	0		5	

Standard tensile tests were conducted at room temperature on transverse specimens using WDS-100 electronic universal testing machine at a cross head speed of 2mm/min. Tensile tests were performed at least three times for each specimen, and averages and standard deviations of the 0.2% proof stress, ultimate tensile strength and elongation-to-failure were examined. For the purpose of investigating the microstructure of 21-6-9 pipes, the specimens were etched and observed in the axial direction. The grains were observed and measured after electrolytic etching in a 60% nitric acid solution by using metallographic microscope (XJP-6A). The specimens were etched in a solution(1g picric acid+5ml hydrochloric acid+100ml alcohol), and then the secondary phase particles were analysed by using scanning electron microscopy(Nova Nano SEM 450) equipped with an energy dispersive spectrometer system. Transmission electron microscopy (JEM-2100) was used to identify secondary phase particles and the samples were taken from the grip section of the specimens and mechanically polished down to 80 μm thin discs, which were then thinned by twin jet polishing with a 7% perchloric acid, 57% methyl alcohol and 36% butyl electrolyte solution.

## Results and Discussion

### Grain Size and Shape

Fig. 1 shows the grains of the 21-6-9 cold drawn pipe annealed at different temperatures. It can be seen from Fig. 1a, most grains of the unannealed part were deformed and inhomogeneous. When annealed at 200-400 °C, recovery is dominant. The grain size and shape changed little due to no migration of high angle grain boundaries, thus still some deformed grains remained unchanged (Fig. 1b), When annealed at 550 °C (Fig. 1c), there were a few equiaxed grains presented in the boundaries, which indicated that some recrystallization might have occurred for the reduction of residual stress caused by the weak of the thermal activation energy at low temperature. After annealing at 600-650 °C, some grains began to consume small ones because of further recrystallization(Fig. 1d). When annealed at 700 °C (Fig. 1e), more equiaxed grains were observed. When annealed at 750 °C (Fig. 1f), most grains turned into larger equiaxed grains, which implied recrystallization had occurred. In addition, some grains maintained small which maybe due to the increasing precipitates of secondary phase particles.

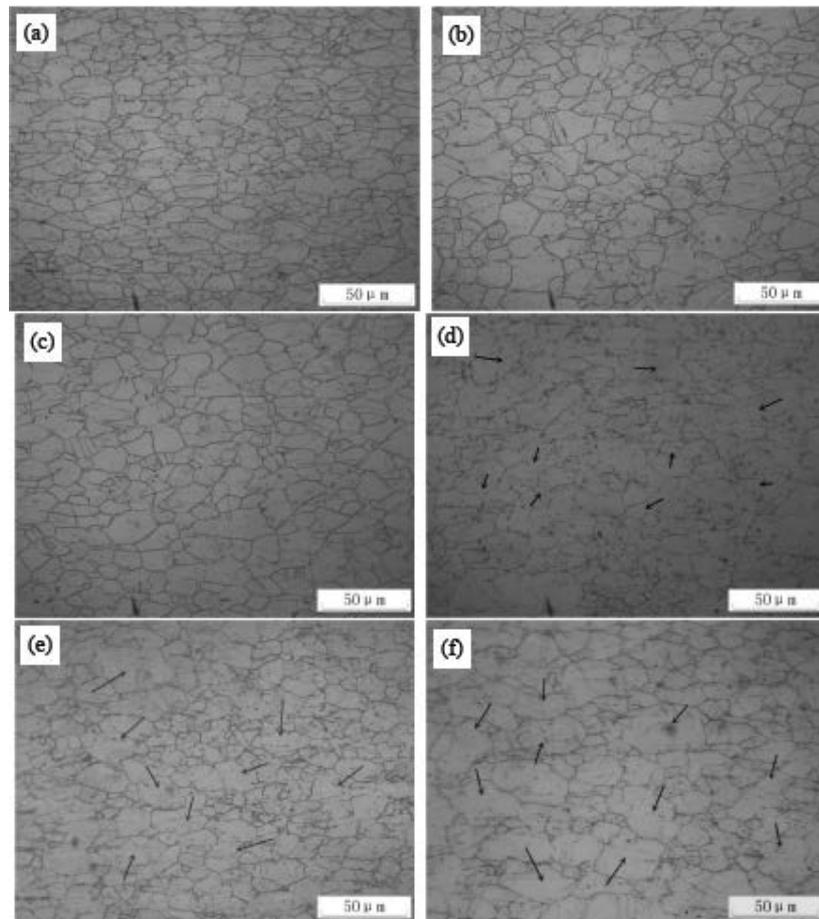


Figure 1. The distribution of grains at different temperatures: (a) untreated; (b) 400°C; (c) 550°C; (d) 650 °C; (e) 700 °C, (f) 750 °C.

### Secondary Phase Particles

Fig. 2 shows the secondary phase particles of the cold drawn pipe at different anneal temperatures. Viewed from Fig. 2a, it is worth emphasizing that the pipes already contain few intragranular precipitates, which might be nitrides formed during the cold drawing <sup>[16]</sup>. As reported in the literatures <sup>[17]</sup> and <sup>[18]</sup>, these precipitates near the grain boundaries might be Cr<sub>2</sub>N. Moreover, the precipitates, after annealing at 200, 400 (Fig. 2b), 550 and 600 °C (Fig. 2c), respectively, were similar to that of the untreated group. However, after annealing at 650 °C (Fig. 2d), the black spots began to precipitate along the grain boundaries. After annealing at 700 and 800 °C, respectively, the particles along the grain boundaries were gradually increasing and coarsening with the increase of annealing temperature (Figs. 2e-2g). This is because enhancing the anneal temperature accelerates the diffusion of alloy elements, which results in the forming and increasing of secondary phase particle <sup>[19, 20]</sup>. At 850 °C, there were nearly no precipitates of secondary phase particle, which maybe due to the dissolution of particles with the increasing anneal temperature. Combined with the thermodynamic calculation, these precipitates along the grain boundaries were considered as M<sub>23</sub>C<sub>6</sub>.

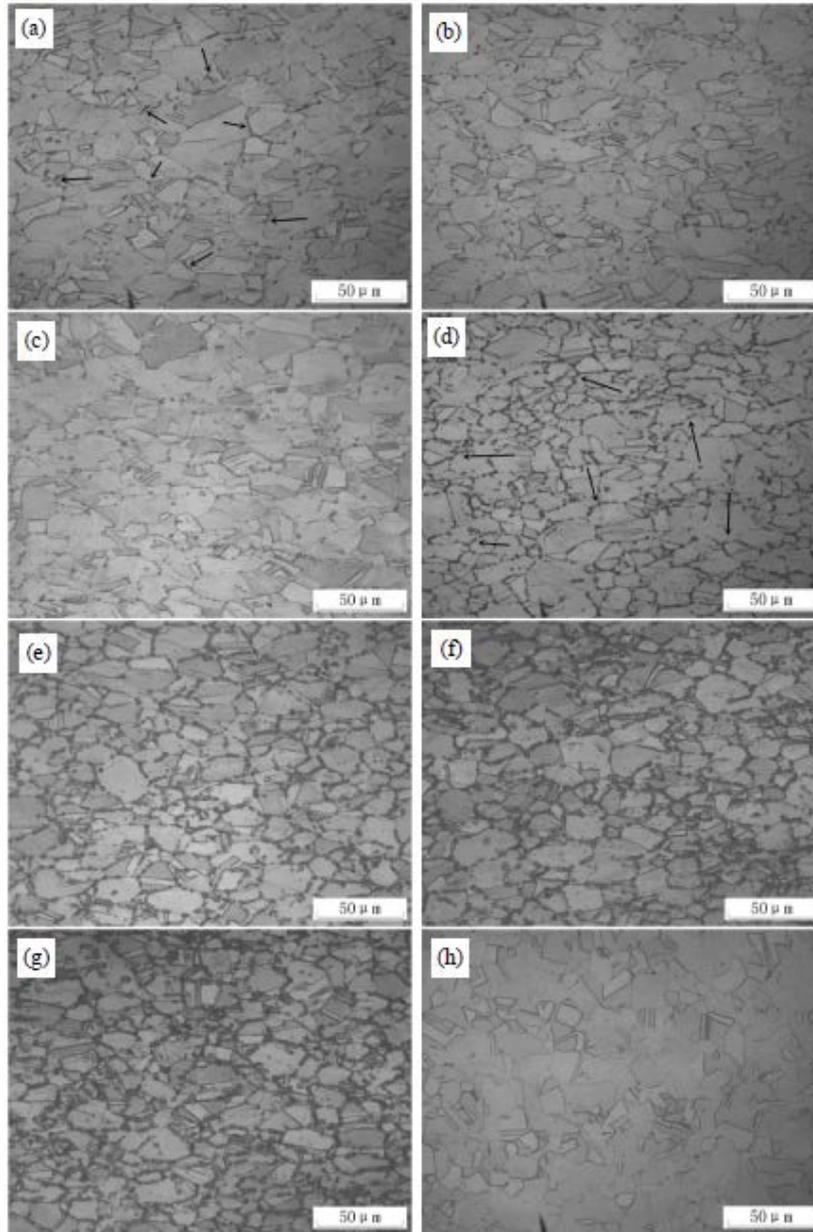


Figure 2. The second phase precipitation of the 21-6-9 cold drawn pipe annealed at different temperatures: (a) untreated; (b) 400 °C; (c) 600 °C; (d) 650 °C; (e) 700 °C; (f) 750 °C; (g) 800 °C; (h) 850 °C.

As exemplified by the SEM micrograph in Fig. 3, the precipitates along and near the grain boundary treated at 700 °C were seen clearly. These precipitates along the grain boundary were indexed as M23C6 due to the EDS analysis (show in Tab. 2), and the results also tallied with the thermodynamic calculation (Fig. 4). Using transmission electron microscopy, the black particles near the grain boundary in the bright field image were expected to be Cr2N (Fig. 5). The results were also found by some researchers [17, 18].

Few Cr2N particles precipitated in intragranular lattice defects of cold drawn pipes of the original state or annealed at 200-600 °C. Such precipitations block the dislocation movement in the annealing process, which slightly improves both tensile strength and yield strength. When the annealing temperature is over 650 °C, the M23C6 precipitations likely occurred. Annealing at 700-800 °C, the M23C6 precipitates were increasing in number and gradually coarsening. A detailed study is required to find out the mechanism and relationship of precipitation of these two secondary phase particles.

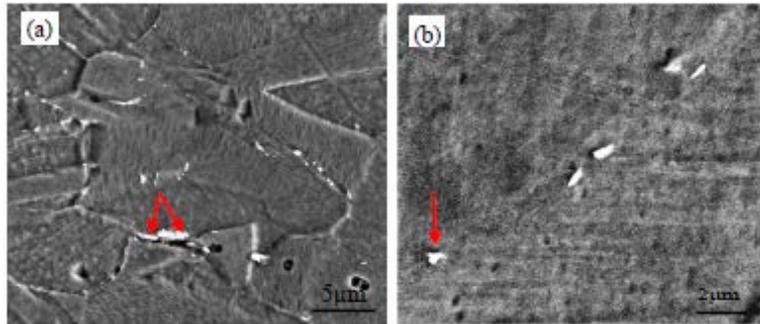


Figure3. Scanning electron microscopy analyses of the secondary phase particles of 21-6-9 cold drawn pipe at 700 °C.

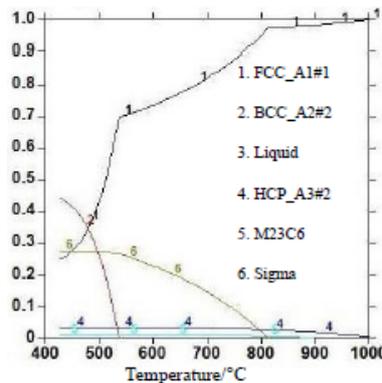


Figure4. The thermodynamic calculation of secondary phase particles of the 21-6-9 cold drawn pipe.

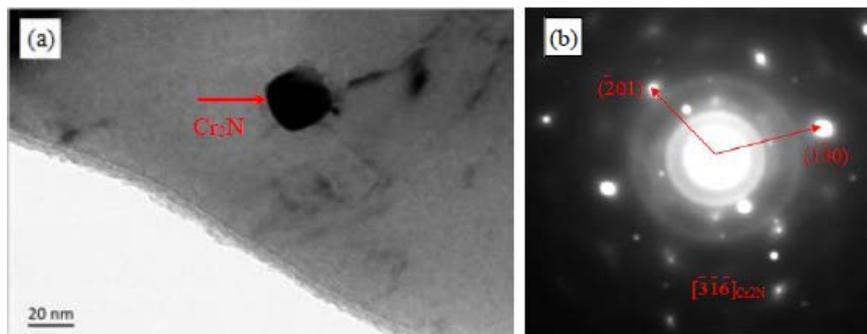


Figure5. Bright-field TEM image of the secondary phase particles and the corresponding SAED analysis of the 21-6-9 cold drawn pipe at 700 °C

Table 2 Chemical composition of the secondary phase particles marked in Fig. 3 (at. %)

zone	C	N	Cr	Mn	Fe	Ni
Fig. 3(a)	15.43	5.51	19.30	7.36	48.06	4.34
Fig. 3(b)	23.12	--	39.48	4.10	30.88	2.42

### Mechanical Properties

Tensile strength, yield strength and elongation of the austenitic stainless steel 21-6-9 cold drawn pipe annealed at different temperatures are presented in Fig. 6. It can be seen that before annealing the tensile strength, yield strength and elongation of cold drawn pipe were 1051 MPa, 923 MPa and 22.3%, respectively. The tensile strength and yield strength remained unchanged or moderately increased with increasing annealing temperature up to 550 °C. This behavior might have been attributed to recovery phenomenon, and related to the morphology change of Cr<sub>2</sub>N precipitates on

intragranular lattice defects in cold worked samples [18]. The highest value of the tensile strength and yield strength reached 1062 MPa and 942 MPa at 600 °C, respectively. When the annealing temperature was over 600 °C, the tensile strength and yield strength decreased with the increasing temperature, which may be due to the recrystallization and increasing precipitates of M23C6 particles along the grain boundaries. As a result, the austenitic stainless steel 21-6-9 cold drawn pipe has low tensile strength and yield strength. However, the elongation showed an opposite trend. The elongation initially decreased slightly and then increased extensively. The precipitations are coarsening and the defect concentration of the grain boundary increases with the increase of annealing temperature, which reduces the effect of grain boundary. Such phenomenon will induce the easiness of dislocation movement and plastic deformation of the 21-6-9 pipe. [21]

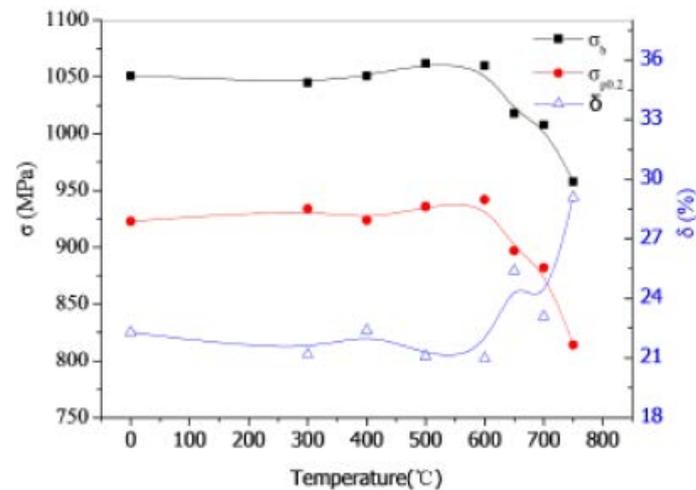


Figure 6 Effect of anneal temperature on strength and elongation of the 0Cr21Ni6Mn9N cold drawn pipe.

## Conclusions

In this study, the microstructure and mechanical properties of austenitic stainless steel 21-6-9 cold drawn pipe were investigated at anneal temperature ranging from 200 to 850 °C for 60min followed by air cooling. The main findings can be summarized as follows:

(1) When annealed below 550 °C, the tensile strength and yield strength of austenitic stainless steel 21-6-9 cold drawn pipe remained unchanged or slightly increased and then decreased over 550 °C, but elongation has the opposite trend. The tensile strength and yield strength reached 1062 MPa and 942 MPa at 600 °C, respectively.

(2) As cold drawn, grains were deformed along the drawing direction and inhomogeneous. When annealed below 550 °C, recovery is dominant. At 550 °C, it is recovery and recrystallization. Over 550 °C, complete recrystallization has likely occurred.

(3) Few Cr<sub>2</sub>N particles precipitated in intragranular lattice defects of cold drawn pipes, and remained rather unchanged below 650 °C. The M<sub>23</sub>C<sub>6</sub> precipitates were increasing in number and gradually coarsening at 700-800 °C. Then, the M<sub>23</sub>C<sub>6</sub> precipitates completely dissolved at 850 °C.

## Acknowledgments

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