Application of Cryogenic Treatment to Enhance the Sealing and Assembling Performance of the SUS316 Tube Fitting

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Abstract

The stainless steel SUS316 tube fitting is widely used in the fluid industries, and the carburizing process is often used to improve the sealing and assembling performance. This study evaluates the feasibility of applying a cryogenic treatment to replace the carburizing process. After cryogenic treatment, the assembling torque value, the contact surface roughness, and the grain size number of the tube fitting are reduced. Further, leaking resistance pressure and hardness are improved, and pipe engaging is smoother and drag marks are reduced. These results demonstrate that cryogenic treatment can effectively enhance the tube fitting sealing and assembling performance and may replace the carburizing process.

1. Introduction

The stainless steel (SUS316) tube fitting is a mechanical engaging part with good sealing performance and excellent corrosion resistance, and is often used in the chemical and semiconductor industries. A full set of tube fitting contains four parts: body, nut, back ferrule, and front ferrule, as shown in Figure 1. The function of the nut is to lock with the body, and to provide an axial force to force the front ferrule and the back ferrule into the pipe when it is locked. When the front ferrule and the back ferrule are pushed by the axial force, the front ferrule will engage both the pipe and the outside bevel of the front ferrule, and the inside diameter of the body bevel will form a seal to avoid fluid leakage. At the same time, the back ferrule will also engage the tube and fix the front ferrule to secure the sealing and shock resistance performance of the tube fitting. To further increase the engaging and assembling performance without sacrificing the corrosion resistance of the back ferrule, tube fitting manufacturers often perform a low-temperature carburizing process on the inside edge of the back ferrule (as shown in Figure 2) to ensure that the back ferrule has sufficient hardness and strength to engage the pipe. However, carburizing SUS316 stainless steel is not an easy task and is patented ^[1]. The carburizing process for the inside edge of the back ferrule of the SUS316 has some challenges, such as achieving an accurate, consistent carburizing layer and cost.

Cryogenic treatment is an add-on process to the conventional heat-treatment process that improves the material properties by decreasing the residual stress, stabilizing dimensional accuracy, and increasing the hardness, fatigue resistance, wear resistance, and life of the tool.

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Many studies have demonstrated that cryogenic treatment can effectively improve the mechanical properties and it has been widely used in the tool, cutting tool, and mold industries^[2-9].

To the best of our knowledge, there have been no reports on the cryogenic treatment of the SUS316 tube fitting in the literature. Thus, the purpose of this research is to examine the feasibility of applying cryogenic treatment to improve the sealing and assembling performance, and to replace the carburizing process. In this study, torsion test, leaking test, contact surface roughness measurement, hardness measurement, engaging mark observation, and metallography observation were used to clarify the impact of cryogenic treatment on the SUS316 tube fitting through different tests.



Figure 1. A full set of the tube fitting.



Figure 2. Metallography graphic of back ferrule $(50 \times)$.

2. Experimental methods

2.1 Specimen preparation

The specimens are prepared to measure the hardness, and to observe the microstructure before/after cryogenic treatment. The specimens need to be ground and polished in accordance with ASTM E112-12, ASTM E3-11(2017), and ASTM E384-17 standards ^[10-12]. Specimens (SUS316 front ferrules and back ferrules) can be divided into four types: (1) with carburization/without cryogenic treatment, (2) with carburization/with cryogenic treatment, and (4) without carburization/with cryogenic treatment.

2.2 Cryogenic treatment

Specimens were put into the cryogenic treatment processor (Applied Cryogenics CP-200vi). Liquid nitrogen was used as a coolant in the cryogenic treatment process. The process profile of the cryogenic treatment is shown in Table 1.

Step	Process				
Cool down	-196°C (11H)				
Soaking	-196°C (12H)				
Return to room temperature	27°C (15H)				

Table 1. Process detail of cryogenic treatment

2.3 Torsion test

To measure the influence of the front and back ferrule after the cryogenic treatment on the locking torque, a torque wrench (Kanon DTC-N100REX) was used. The measuring method is shown in Figure 3. Each tube fitting is locked by one and a quarter turns and each process is tested in five samples. The pipe used for the torsion test is SUS304 pipe conforming to ASTM A213 standard.



Figure 3. Locking torque measurement method.

2.4 Leaking test

Leaking test equipment is used to measure the limit pressure resistance of the tube fitting before/after cryogenic treatment. This test can be divided into an air and oil pressure test. The air pressure test can measure the leakage resistance ability of the tube fitting, and the oil pressure test can test the maximum pressure resistance of the tube fitting. The test conditions and specification are shown in Table 2^[13].

	Gas pressure (air)	Oil Pressure (Hydraulic oil)						
Equipment limit	415 Bar	650 Bar						
Test standard:								
ASTM F1387-19 Standard Specification for Performance of								
Piping and Tubing Mechanically Attached Fittings								

Table 2. Leaking test conditions and specifications.

2.5 Surface roughness measurement

To observe the change in the surface roughness of each contact surface of the tube fitting before/after the cryogenic treatment, a Surfcom 130A roughness measurement instrument

was used to measure the contact surfaces of each contact point of the tube fitting. The measured surfaces were numbered as shown in Figure 4. The measurements were carried out according to ISO 4287 and ISO 16610-21 specifications ^[14-15].



Figure 4. Roughness measurement surface numbers of the tube fitting.

2.6 Hardness test

A Vickers hardness tester (Future-Tech FM-300) was used to measure the hardness of the specimens before/after cryogenic treatment in conformance with ASTM E384-11 ^[12]. The load was 300 g for the base material (SUS316) and 100 g for the carburization layer. The loading time was 15 seconds for both.

2.7 Engaging mark observation

A macroscope (SMART-3000M) was used to observe and compare the difference of the engaging mark of the pipe locking by different processes of tube fitting.

2.8 Metallography observation

In addition to the macroscopic investigations, an optical microscope (ZEISS AXIO Imager. A1) was used to compare the differences in specimens before/after cryogenic treatment. In order to reveal the microstructure, the specimens were ground, polished, and etched by ferric chloride solution ^[11].

3. Results and discussion

3.1 Torsion test

The results of the torsion test are shown in Table 3. These results show that the locking torque of the tube fitting reduced significantly after cryogenic treatment, regardless of whether the tube fitting was treated with low temperature carburization treatment or not. Lower locking torque of the tube fitting not only effectively reduced the assembling difficulty, but also increased the assembling efficiency.

Process	1	2	3	4	5	Avg. (N-m)	
(1) with carburization /without cryogenic treatment	44.5	44.2	43.8	44.6	44.7	44.36	
(2) with carburization /with cryogenic treatment	34.4	33.9	32.9	31.3	32.6	33.02	
(3) without carburization /without cryogenic treatment	53.9	52.6	52.8	53.1	53.2	53.12	
(4) without carburization /with cryogenic treatment	35.8	33.9	32.3	35.5	35.2	34.54	

Table 3. Torsion test results

3.2 Leaking test

Leaking test results are shown in Table 4. The leakage resistance of the tube fitting was quite vital. Therefore, if the tube fitting has any leak in the air pressure test, it can be directly judged as unqualified and there is no need for an oil pressure test. These results show that the air and oil pressure resistance of the tube fitting increased significantly after the cryogenic treatment.

Table 4. Leaking test results

Process	Air (Bar)	Oil (Bar)
(1) with carburization /without cryogenic treatment	413	637
(2) with carburization /with cryogenic treatment	415	650
(3) without carburization /without cryogenic treatment	170	-
(4) without carburization /with cryogenic treatment	414	650

3.3 Surface roughness measurement

The results of each contact point surface roughness of the tube fitting with different processes are shown in Table 5. The results show that the contact point surface roughness of the tube fitting reduced after cryogenic treatment. The lower surface roughness can reduce the coefficient of friction, resulting in the improvement of the sealing ability and the assembling performance of the tube fitting. These results corresponded to the results of the torsion and leak test.

							U	nit: µm
Process Point	1	2	3	4	5	6	7	8
(1) with carburization /without cryogenic treatment	0.294	0.381	0.400	0.448	0.678	- 0.149	0.407	0.262
(2) with carburization /with cryogenic treatment		0.331	0.338	0.425	0.641		0.385	0.247
(3) without carburization /without cryogenic treatment	0.231	0.388	0.484	0.363	0.855	0.397	0.195	0.467
(4) without carburization /with cryogenic treatment		0.325	0.403	0.183	0.301		0.126	0.408

Table 5. Roughness measurement results.

3.4 Hardness test

The hardness test results of the SUS316 tube fitting specimens before/after cryogenic treatment are shown in Table 6. The results show increases in hardness of the specimens after cryogenic treatment. A higher hardness not only increases the assembling performance to decrease the locking torque, but also increases the engaging performance to enhance the leaking resistance performance of the tube fitting.

Process	1	2	3	4	5	AVG (HV)
(1) with carburization/without cryogenic treatment (Base)	259.17	258.34	253.64	257.76	254.24	256.63
(2) with carburization/with cryogenic treatment (Base)	291.66	292.48	296.54	295.84	292.22	293.74
(1) with carburization/without Cryogenic treatment (Coating)	812.00	812.10	816.99	812.22	814.67	813.59
(2) with carburization/with cryogenic treatment (Coating)	845.84	835.54	840.25	843.22	841.53	841.27
(3) without carburization/without cryogenic treatment	259.76	261.85	260.77	259.63	258.72	264.79
(4) without carburization/with cryogenic treatment	294.51	299.55	308.81	294.64	297.45	298.99

Table 6. Hardness measurement results.

3.5 Engaging mark observation

The engaging mark of the pipe after the torsion test most directly reflects the quality of the locking situation of the tube fitting, and the results of engaging mark observation are shown in Figures 5 to 8. The results show that the pipe with engaging marks locking by cryogenic-treated tube fitting are smoother and the drag marks are significantly reduced. These phenomena can further increase the sealing and shock resistance of the tube fitting, and the results correspond with the results of the torsion and leaking tests.



Figure 5. Engaging mark of tube fitting with carburization and without cryogenic treatment.



Figure 6. Engaging mark of tube fitting with carburization/with cryogenic treatment.



Figure 7. Engaging mark of tube fitting without carburization/cryogenic treatment.



Figure 8. Engaging mark of tube fitting without carburization/with cryogenic treatment.

3.6 Metallography observation

Metallography observation was conducted by following the ASTM E112-12 and ASTM E384-11 ^[10,12] at $100 \times$ magnification. The results are shown in Figures 9 to 10. The results indicated that the average grain size reduced from 4.0 to 3.0 (a larger number means a smaller grain size and the numbers are delimited by comparing the standard graphics) after cryogenic treatment. Much research also indicates that a smaller grain size of a material not only increases the hardness and wear resistance but can also decrease the surface roughness and friction coefficient ^[16-19]. The torsion test, hardness, and surface roughness measurement results were also confirmed by the increase in the number of smaller-sized grains.



Figure 9. Metallography graphic of tube fitting without cryogenic treatment $(100 \times)$.



Figure 10. Metallography graphic of tube fitting with cryogenic treatment $(100 \times)$.

4. Conclusions

In this study, the results of the torsion test, leaking test, surface roughness measurement, hardness measurement, engaging mark, and metallography observation confirmed that cryogenic treatment provided a significant improvement in the assembling and sealing performance of the SUS316 tube fitting. This is a valuable and original result that indicates that the carburizing process can be replaced by cryogenic treatment. Further research is needed to explore the optimal cryogenic process and to elucidate the mechanism and the relationships among cryogenic treatment, grain size, and surface roughness.

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