Back ferrule 의 내식성과 체결시 기계적특성을 만족시키는 최적의 플라즈마 침질탄화공정조건의 확립

Improvement of corrosion resistance and hardening the back ferrule surface by plasma treatment

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Abstract:

Back ferrule is a circular ring shaped metallic object which is used for fastening, joining or reinforcement during the tube fitting as well as to prevent leakage. Therefore, during tube fitting the leading edge of the back ferrule should be sufficiently hard enough to prevent leakage. In our research, we concentrated the improvement of two major factors. Firstly, to improve the surface hardness of the back ferrule made by AISI 316 Stainless Steel. Secondly, the enhancement of corrosion resistance of back ferrule after plasma treatment. Initially, the corrosion resistance and hardness of the back ferrule (both commercial and without treated) was not good enough for tube fitting but after applying plasma treatment with suitable conditions on ferrule, we improved the corrosion resistance and hardness of the back ferrule dramatically.

1. Introduction:

In a two-ferrule tube fitting system, the back ferrule pushes the front ferrule forward to spring load the fitting assembly and seal with the fitting body, and create the primary tubing seal (Fig. 1). The back ferrule also swages the tube to provide the grip needed to keep the fitting and tubing firmly in place (Fig. 2). To do this the back ferrule's leading edge must be sufficiently harder than the tube [1].

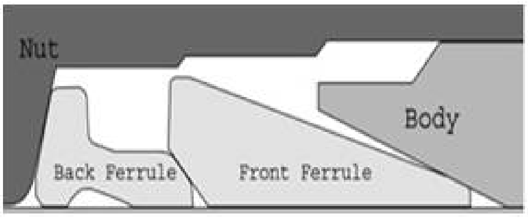

Fig.1. Tube Fitting with Advanced Geometry Back Ferrules Prior to Make-up

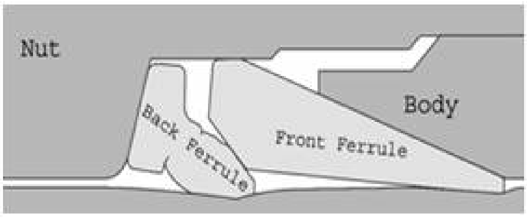

Fig.2. Tube fitting with advanced geometry Back Ferrule prior after make-up

2. Main Body:

Generally, untreated ferrule which is made by AISI 316 stainless steel has low hardness and less corrosion resistance which is not satisfactory for the tube fitting (Fig. 3(a)). In order to solve this problem, manufacturing company attempted to increase the hardness of the top side of back ferrule by applying commercial salt bath carburizing treatment. This procedure helped to increase the surface hardness of back ferrule but couldn't pass the corrosion test (Fig. 3(a)). Therefore, we tried to solve this problem by applying plasma treatment which was fulfilled both the requirements.

The samples (Ferrule) were cleaned carefully by ethyl alcohol (C_2H_5OH) before the plasma treatment by means of ultrasonic cleaner. Next, the samples were dried and then placed on the cathode table in the pulsed plasma ion nitriding system. The vacuum chamber was pumped down to 50mTorr for the pre sputtering operation. In this operation Ar and H_2 ion sputtering was performed at 300°C for at least 40 minutes for further surface cleaning (Voltage: 400 V, gas composition: Ar/ H_2=20%/80%). After this plasma nitrocarburizing process was immediately carried out with a pulsed D.C. potential for 15 hours in the glow discharge environment of a gas mixture of N_2, H_2 and CH_4. After treatment, the samples were cooled in the vacuum chamber to room temperature. The plasma treated samples were sectioned for the microstructure studies and were first etched with a special chemical solution which contains 50% HCl, 30% HNO_3 and 20% H_2O. The microstructure of the cross sectioned surfaces of all the samples were observed by using an optical microscope. Also, the corrosion test was performed by submerging the treated ferrule in NaClO solution for 24 hours.

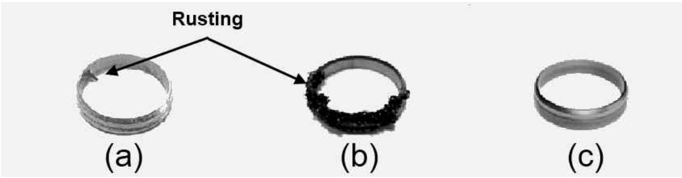

Fig.3. (a) Untreated (b) commercial salt bath carburized & (c) Plasma treated ferrule after 24 hrs corrosion test.

The low temperature plasma nitro carburizing process results in the formation of a dual-layer structure (also known as S-phase) consists of a N-enriched layer (γN) with a high nitrogen content on top of a C-enriched layer (γC) with a high carbon content, leading to significant increases in the surface hardness and the corrosion resistance of ferrule [2]. The average thickness of the dual layer is about 20-22 μm. The top layer of the nitrocarburized (HK0.05is1000-1100) layer is very hard and corrosion resistance. Therefore, by applying plasma treatment with suitable conditions we achieved not only satisfactory hardness but also good corrosion resistance which is necessary requirements for tube fitting. Fig.3 shows the corrosion test result of untreated ferrule which was submerged in NaClO solution for 24 hours. In the picture it is clear that the top part of the ferrule was corroded slightly and during the tube fitting that part will be very important for sealing. To overcome this situation the sample was nitrocarburized for 15 hours and then submerged in NaClO solution for 24 hours showed good corrosion (no rusting and no chemical attack) resistance than the untreated sample which is showed in the fig.4.

And the Fig. 4 given below exhibits the cross sectional views of the BMT BA ¾ inch ferrule after the plasma nitro carburizing treatment. It is clear from the Fig. 4 that the white layer was formed after the plasma nitro carburizing treatment. This layer helps the ferrule in two main ways. First of all, this layer is a highly corrosion protective layer which is the prior requirement of the ferrule. Secondly, this layer has very high hardness than the untreated ferrule surface. Both this conditions will fulfill the characteristics of good ferrule.Normally, the surface hardness of untreated sample is around 300-400(HK0.05).But after the plasma nitrocarburizing the hardness of the ferrule improved 3 times better than the untreated sample (650-1100 HK0.05).

It should be mentioned that, in many cases it was very hard to get minimum thickness of the s-phase on the ferrule surface especially in the bottom part. And if the bottom part of the ferrule was not covered sufficiently with the S-phase then the sample will not pass the corrosion test (NaClO test). Additionally the minimum thickness of the S-phase should be 18-20 μm which will prevent the surface from corrosion .

3. Conclusion:

The low temperature plasma nitro carburizing process the surface of back ferrule is covered by a duplex layer ($\gamma N+\gamma C$) which is free from Cr_2N precipitation and highly corrosion protective. For tube fitting the most important factor is the surface hardness of the leading edge of the back ferrule. If the leading edge of the back ferrule is not sufficiently hard enough there is a possibility of leakage in the tube during the fitting operation which is not desirable. Also, if the corrosion resistance of the ferrule is not very good then it will not be used in the corrosive environment like chemical processing industry as well as semiconductor industry. Therefore, plasma nitrocarburizing of ferrule is the perfect surface treatment to overcome these problems.

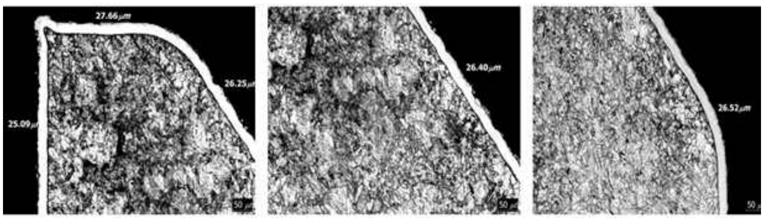

Fig.4. Cross sectional view of BMT BA ¾ Inch Ferrule by Optical Microscope (OM)

References:

1. 316 Stainless Steel Swagelok Tube Fittings with Advanced Geometry Back Ferrules; Swagelok Company October 2003, R3MS-06-16-E.
2. Influence of Temperature and Time on Low-Temperature Plasma Nitrocarburizing of AISI 304L Austenitic Stainless Steel; Journal of the Korean Physical Society, Vol. 54, No. 3, March 2009, pp. 1131-1135.