Metallurgical Laboratory Report

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I. Stainless Steel 409 (1.4512)

1. General:

The photographs below illustrate the “as-received” 2-off seam welded tubes, one double-skinned, submitted for metallurgical evaluation of Flowdrilled holes. Each tube contained Flowdrilled holes identified as follows:

**SAMPLE A:** Stainless Steel 409 – 50.8mm OD * 1.5mm wall thickness

- **2-off Flowdrilled holes “A”**
  - 17.2mm dia; Pilot hole 4mm dia; CNC Flowdrilled @ 450mm/min

**SAMPLE B:** Double-skinned Stainless Steel 409 – Inner tube= 42mm OD * 1mm wall thickness; Outer tube = 45mm OD * 1.0 mm wall thickness.

- **1-off Flowdrilled Hole “B”**
  - 17.2mm dia; Flowtap M18 * 1.5; Pilot hole 4mm dia; Flowdrilled @ 3000rpm; Feed 450mm/min

- **1-off Flowdrilled Hole “C”**
  - As “B” but with manual feed and no pilot hole

AISI 409 stainless steel is a “ferritic” class of stainless steel containing 12 % chromium, 0.08% carbon max, with an addition of a minimum of 6 * % carbon content of titanium whose purpose is to combine with the carbon to form stable titanium carbo-nitride inclusions (TiCN) which ties up the carbon preventing it from migrating to grain boundaries during thermal processing where it can locally denude the chromium and adversely effect the material’s corrosion resistance. It is therefore ideally suited to the Flowdrilling process which without the titanium addition could otherwise be susceptible to this metallurgical phenomenon.
2. Examination:

2.1 Metallographic:

All 3 holes were sectioned diametrically using a water-cooled abrasive cutting wheel, mounted in phenolic resin and polished to a 1 micron finish using standard metallographic grinding & polishing procedures.

After polishing the sections were etched electrolytically for up to 30 secs in a 10% solution of HCl in methanol at 6 volts DC to reveal the microstructure in preparation for examination on the metallurgical optical microscope.

The microstructure of the base tube material remote from the Flowdrilled holes comprised polyhedral grains of ferrite with a grain size around 5-6 ASTM. This is typical of cold worked and annealed material.
Base material microstructure remote from the Flowdrilled holes- Polyhedral Ferrite grains (size: ASTM 5-6)

Examination at higher magnification also revealed the presence of TiCN inclusions:

Photomicrograph showing a typical TiCN cuboid inclusion
Photomicrographs – Flowdrilled Hole “A”
Enlarged view showing "Cold-Worked" / "Flowed" material at the Flowdrilled surface (See hardness profile below)

Hardness Profile - Flowdrilled Hole "A"

- Distance From Flowdrilled Surface (mm)
- Vickers Hardness Value (Hv 0.5)
Hardness Profile Comparison – Flowdrilled Holes “A”, “B” & “C”
3. Remarks:

The metallographic evidence reveals that all three Flowdrilled holes have responded metallurgically similarly to each other in that they exhibit a shallow, “work-hardened” zone approximately 0.5mm deep from the Flowdrilled surface. The microstructures in all cases have remained totally ferritic with no evidence of transformation to martensite or other intermediate phases. This is indicative of the low carbon content of the base material and a maximum localised temperature attained during the Flowdrill process which is below the critical transformation temperature (around 800 deg C).

The indications from this examination are that the Flowdrill process has produced no deleterious metallurgical effects. However, since corrosion resistance is of prime importance to this product and type of material we would recommend that separate corrosion testing is undertaken on Flowdrilled material. Whilst the use of a “Stabilised” grade of stainless steel (titanium addition) will guard against inter crystalline corrosion there may be a possibility that the work-hardening at the surface of the Flowdrilled holes could act like dissimilar metals and produce a galvanic cell effect, producing corrosion around the work-hardened areas.

Corrosion testing is unfortunately outside the scope of our laboratory facilities.
II. Stainless Steel 18% Chromium/8% nickel

1. General:
Several samples of the “Flowdrilled” holes. Flowdrill required a general metallurgical assessment carrying out, detailing what metallurgical effects the process had on the base material microstructure & properties. No details of the base material were provided but initial cursory examinations showed that the material was not ferromagnetic and therefore from the onset we suspected that the material was an austenitic stainless steel. (18% Chromium/8% nickel type; e.g. AISI 304)

Fig 1. General photograph showing the range of Flowdrilled samples submitted for Examination

Dimensions of the samples were as follows:-

- A) Plate - 100 * 54 * 2 mm thick
- B) Plate - 100 * 53 * 3mm thick
- C) Small Box Section - 40 * 40 * 80mm long *1mm thick
- D) Large Box Section - 50 * 50 * 133mm long * 2mm thick

The Flowdrilled holes were approx 10mm diameter with an approx 5mm long tapered extruded tail on the exit side of the Flowdrilled holes. Detailed dimensional examination was not carried out.
2. Examination:

2.1 Metallographic:

Selected holes were sectioned diametrically using a water-cooled abrasive cutting wheel, mounted in phenolic resin and polished to a 1 micron finish using standard metallographic grinding & polishing procedures.

Fig. 3. Photograph of typical "as cut" section prior to metallographic preparation.

After polishing the sections were electrolytically etched for 10 secs in a 10% aqueous solution of oxalic acid at 6 volts DC and examined on the metallurgical microscope. The base material remote from the Flowdrilled holes & their Heat-Affected–Zones revealed a typical austenitic stainless steel single phase microstructure of pure, polyhedral, twinned austenite grains. This is typical of cold worked material that has been subsequently solution annealed. (Typically water quenched from approx 1050 deg C.) Some of the samples contained small amounts (<1%) of delta ferrite stringers which is normal for this material. The grain boundaries were only sparsely revealed by the etchant indicating very low carbon content in the steel and insignificant grain boundary carbide precipitation in the H.A.Z as a result of the thermal affects of the Flowdrill process. (see Fig 5)

The inside surface of the hole exhibited a 50 micron deep zone of refined microstructure; beyond this a layer of deformed/ "cold worked" grains. Both these zones exhibited higher hardness than the base material due to the “cold working” or “work-hardening” effect of the process. (See Figs 6 & 7 and separate plot of microhardness values on the following pages)
Photomicrographs illustrating microstructure around the Flowdrilled Hole- Diametrical section

Fig 4.
Fig 5. Photomicrograph illustrating base material microstructure comprising polyhedral grains of austenite with a small amount of delta ferrite (stringers). Etched: electrolytic 10% oxalic acid, 6V
Microhardness = approx 200HV0.5

Fig 6. Photomicrograph illustrating refined microstructure at the hole surface.
Microhardness approx 300HV0.5 Etchant: as above
Fig 7. Photomicrograph illustrating "Cold-Worked" zone between the hole surface & the base material. Note the distorted, elongated grains. Hardness = approx 250HV0.5
2.2. Hardness:
Vickers microhardness tests were carried out across the hole profile extending from the hole surface, through the HAZ and into the base material using a 500g load. Results are presented graphically below:-

![Vickers Hardness Profile- Flowdrilled Stainless Steel Holes](image)

Microhardness Profile illustrating the increasing hardness towards the hole surface.

3. Remarks:
The subject base material is thought to be an austenitic stainless steel of the type AISI 304 (18% Cr, 8% Ni) and as such these steels are non-hardenable by heat-treatment, like standard engineering or plain carbon steels. The latter materials would produce a hard and brittle martensitic structure in the Heat-Affected-Zones around the Flowdrilled holes as a result of the phase transformation following cooling from the high temperatures generated during processing. Since austenitic stainless steels do not undergo phase transformation on heating the problem of generation of a hard, brittle microstructure does not exist. There are, however, other metallurgical effects that austenitic stainless steels incur during thermal exposure. These relate to the precipitation of chromium carbides at the grain boundaries which may cause severe degradation of the material’s corrosion resistance.

The Flowdrill process has affected a refinement and distortion of the base material microstructure by “work-hardening” of the austenite grains up to a depth of 3mm around the hole surface. Thermal effects on the microstructure appear to be minimal and there does not appear to be any grain boundary carbide precipitation in the Heat-Affected-Zone that could adversely affect the corrosion properties of this type of material.

The main metallurgical concern during thermal metallurgical processing of this type of material is that of chromium carbide precipitation at grain boundaries. This takes place at intermediate temperatures between 650 deg C and 900 deg C, and is commonly encountered, for example, in the HAZ of fusion welds that are subjected to this temperature range. The formation of grain boundary carbide phases locally denudes the grain boundary of chromium which renders the material susceptible to “inter-granular corrosion”. The problem can be controlled by three methods:-

1. By solution annealing after thermal processing.
   This redissolves the grain boundary chromium carbides and holds them in “solid solution” where they are harmless.

2. By stabilising the carbon.
Precipitation of chromium carbides can be prevented by small additions of either titanium (grade AISI 321 stainless steel) or by Niobium (grade AISI 347 stainless steel) which “fix” the carbon as stable titanium or niobium carbides, thus preventing the formation of chromium carbides.

3. By limitation of carbon content.
   By ensuring that the carbon content is kept below 0.030% any chromium carbide precipitation may be avoided. (Grade AISI 304L stainless steel)

The microstructure of the subject material does not contain any titanium or niobium carbides, neither has it been solution annealed after Flowdrilling as evidenced by the presence of cold-worked grains which would have recrystallised to a polyhedral morphology if annealing had been performed.

It is also unlikely that the material is of the ultra low carbon variety (AISI304L) therefore the absence of grain boundary chromium carbides in the HAZ’s can only be attributed to the rapid heating & cooling times that the Flowdrill process produces which minimises the carbide precipitation.

Whilst our examinations have found no significant deleterious metallurgical effects of the Flowdrill process to the base material the greatest concern with the material is loss of corrosion resistance in and around the Flowdrilled holes. In this respect we would recommend that specific corrosion tests be carried out in order to confirm our metallographic findings.

Suitable corrosion tests for austenitic stainless steels of the type here are detailed in USA standard AISI A262, but these are beyond the scope of our laboratory facilities here.

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