

## **Workshop on Quality Issues in Current and Future Silicon Detectors**

# **Cooling: Materials & Joining Technologies**

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**Presented by Tim Jones**

- KNOW WHAT YOU ARE DOING, IF NOT GET SOMEONE WHO DOES
- MINIMISE THE AMOUNT OF JOINTS IN YOUR SYSTEM
- PROTOTYPE AS MUCH AS POSSIBLE
- MAKE EVERYTHING FULLY TRACEABLE FROM SOURCE
- KEEP ALL TEST DATA
- DON'T REMOVE IDENTIFYING LABELS
- IT'S ALL ABOUT PRESSURE HANDLING = TEST WITH PRESSURE!
- My personal preference is to weld as many joints as possible removing all connectors.

# Material Design Requirements<sup>3</sup>

- General Detector Requirements
  - Pressure
  - Leak Rate
  - Operation of low temperature
  - Pressure drop (holistic system)
- Lifetime environmental compliance
  - Radiation damage
  - Radiation strain
  - Activation
  - Non magnetic
  - Galvanic compatibility (system)
- Temperature
  - Manufacturing & assembly
  - Ambient inc storage & transport
  - CTE (must be understood in design)
- Humidity
  - Corrosion resistant
  - Strain

- Very obvious detector performance requirements:

- High Strength
- Low Mass [XO]
- Thermal Conductivity
- Robustness for handling & assembly

- Very obvious design mistakes

- Such low mass = grain size too thin
- Lack of prototyping
- Lack of testing
- Lack of standardisation
- Geometric non-compatibility

*[use of non-standard sizes – please take note of this as dramatically increases procurement, tooling manufacture & testing time & costs]*

- Biggest mistakes

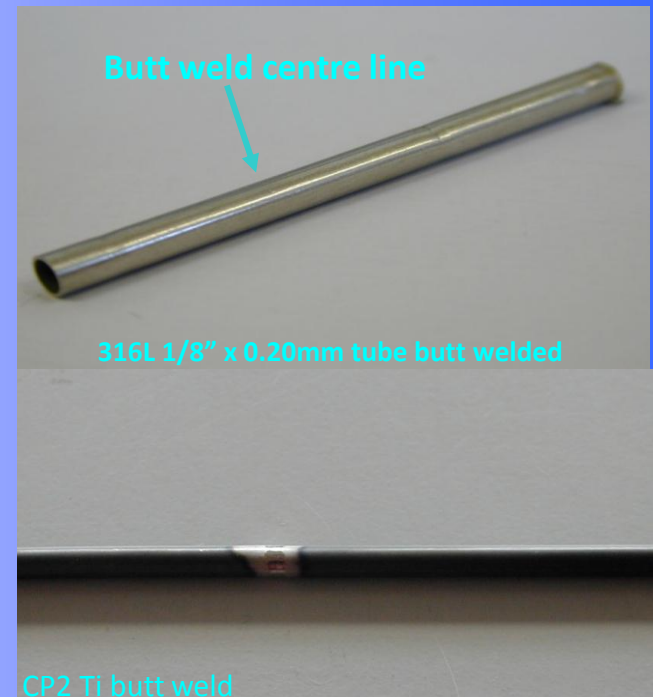
- Corrosion – usually due to:
- Joining metallurgy totally misunderstood or ignored
- Connectors inc torque (twisting or cantilever) damage to other joints
- Change of plan without time for significant testing

# Cooling transfer line material selection

ATU R&D in the UK from 2007 to present has refined thinking to 2 clear candidates for on-detector & cryostat services tubing:

## *CP2 Titanium & 316L Stainless Steel*

- 316L does not match well with on detector component CTE
- Titanium grades CTE excellent match.
- Corrosion, both galvanically more stable than components likely to be joined to detector services.
- Bending requirements: specify the temper of the tube = ¼ hard for both 316L & Ti.
- CP2 Titanium shows most promise for meeting all requirements.
- 316L stainless significantly easier to make welded joints
- Knowledge of Ti welding now catching up fast,



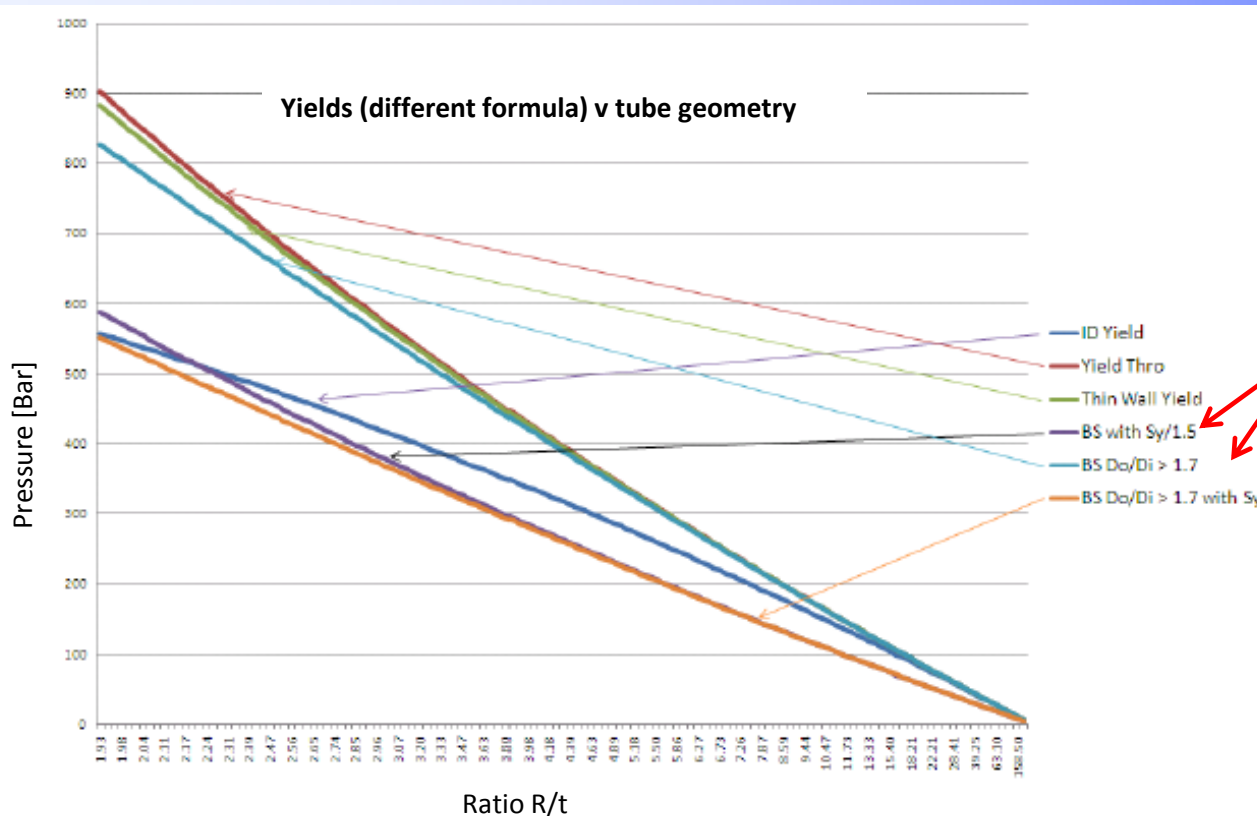
- Most grades of aluminium tube samples procured by UK (c2008/9) now indicate signs of corrosion.
- CuNi too weak for CO<sub>2</sub> vs material in wall. Welds are also prone to porosity



# Tube wall selection by calculation/standards?

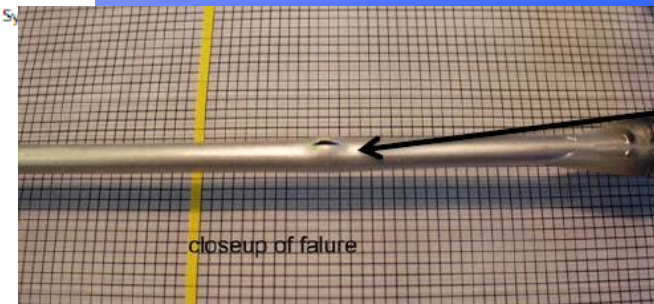
Still working to factor of safety [ $S_y$ ] of 1.5 100Bar working / 150Bar Fault RF:2011

**Design by formula =DBF** Roark's thin wall standardised in BS13480



**Cross check  $S_y$  with  
TDL103-TR-0001**

DBF burst pressure of 316L  
tube was est at 120Bar  
= Tube burst at 120Bar



closeup of failure

(J.Tarrant 2009)

Standard above is good in principle – however standards explicitly apply to tube diameters of 6mm and above!

# Application of industrial standard: Tubes under 6mm?

- GOOD LUCK! Nothing works well under 6mm OD in terms of calculation
- DBF safety factor .2x pressure + as defined by standards 1/1.5 of 0.2% proof stress based on principle stress.
- If a tube is significantly rigidised into any structure then DBA should be taken (design by analysis) = FEA using Von Mises Yield criterion to establish safety factor [tube behaviour same,  $S_y$  alters]

Proof stress in 316L from BS EN 12392

Material	Min. stress (N/mm <sup>2</sup> )	Strength type *	OD (mm)	Pressure (bar)	Pressure (N/mm <sup>2</sup> )	Raw Wall 't' no safety using 13480-0.085	Wall 't' (mm) @ 60%PS	Final spec OD	Final min. chosen wall 't' (mm)	Final ID (mm)	Final wall t to ID ratio 1/x	Final Safety Factor	Recalc'd Pallow (bar)	Do/Di check (< / = 1.7)
Stainless Steel 316L (annealed)	1.70E+08	0.2%PS	2	150	1.50E+07	0.085	0.137	2.00	0.14	1.72	14.29	1.67	250	1.16
Stainless Steel 316L (annealed)	1.70E+08	0.2%PS	3.175	150	1.50E+07	0.134	0.217	3.18	0.22	2.74	14.45	1.67	250	1.16
Stainless Steel 316L (annealed)	1.70E+08	0.2%PS	4.7625	25	2.50E+06	0.035	0.058	4.76	0.19	4.38	25.05	1.67	42	1.09
Stainless Steel 316Ti (annealed)	2.05E+08	0.2%PS	2	150	1.50E+07	0.071	0.115	2.00	0.11	1.78	18.18	1.67	250	1.12
Stainless Steel 316Ti (annealed)	2.05E+08	0.2%PS	3.175	150	1.50E+07	0.112	0.182	3.18	0.18	2.82	17.67	1.67	250	1.13
Stainless Steel 316Ti (annealed)	2.05E+08	0.2%PS	4.7625	25	2.50E+06	0.029	0.048	4.76	0.19	4.38	25.05	1.67	42	1.09

When using Roark's the ID to OD ratio drifts below 6mm OD and starts to produce unusual results. Rough explanation = Inner wall of tube starts to come close to permanent deformation (wrt yield) hence the formula no longer works effectively.

- PD 5500:2006+A3:2008 Specification for unfired fusion welded pressure vessels = Pressure test information
- BS EN 378-2:2008 Refrigerating systems and heat pumps. Safety and environmental requirements. Design, construction, testing, marking and documentation
- BS EN 14276-1:2006 Pressure equipment for refrigerating systems and heat pumps. Vessels. General requirements
- BS EN 13480 Series: Metallic industrial piping. General
- BS EN 10216-5:2004 Seamless steel tubes for pressure purposes. Technical delivery conditions. Stainless steel tubes
- BS EN 4180:2002 Aerospace series. Circular tubes, for fluids in titanium and titanium alloys. Wide tolerances. Diameter 4 mm ≤ D ≤ 40 mm. Dimensions
- BS EN ISO 1127:1997 Stainless steel tubes. Dimensions, tolerances and conventional masses per unit length



# Joining by connector



AN Fitting (Aluminium anodised used in aerospace – not ideal)

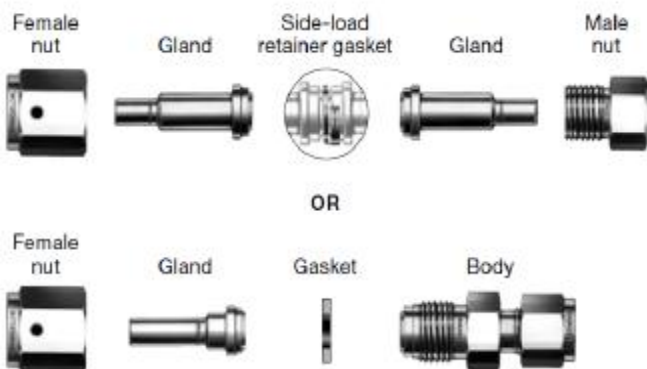
Ferrule type fittings are not suitable to detector applications, thin tube needs internal support altering pressure drop, more leak paths than VCR type fittings.

- Most connectors in order to achieve a seal need high torque applying to the fitting. With a restraining bulkhead, the mass (XO) increases dramatically.
- Space for tooling (spanner) to tighten the renders most unusable!

Swagelok VCR welded to 316L 1.8" OD x 0.20mm wall tube



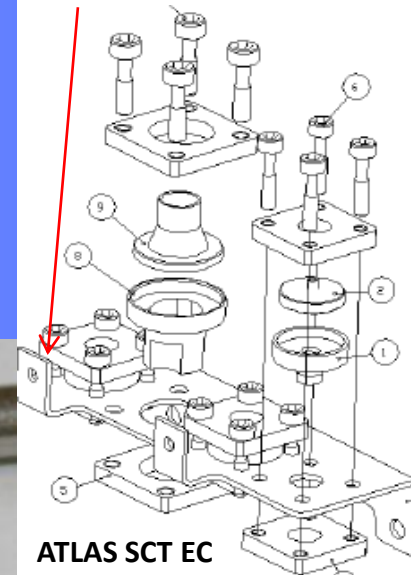
NB: Temp test connector for UK Stave replaced with fully welded joint



- 6LV 316L Stainless VCR fitting vacuum brazed to Titanium tube for temporary test fitting, again used on UK Stave concept.
- Best experience of a fitting so far other than the End-Cap Exhaust fitting.
- Dissimilar material joints are only as good as the Ni or Cu plating of the 316L!

NB: Bad plating = leaking fittings (testing underway)

Minimised torque load

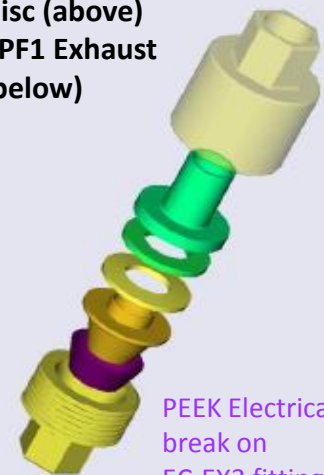


ATLAS SCT EC

Disc (above)

PPF1 Exhaust

(below)



PEEK Electrical  
break on  
EC-EX3 fitting

# Permanent Joining technologies<sup>8</sup>

- TIG orbital welding:

Portable automated equipment, no filler material (autogenous process) or sleeve joint required for welding. Highly repeatable, low cost, low XO when compared with connectors. - Electrical arc investigations are ongoing to validate on detector welding process

- EB welding

Non portable, highly repeatable, requires sleeve joint (XO+). If misunderstood fluxes can cause corrosion on Titanium

- Vacuum Brazing

Useful for dissimilar materials using a plating of usually Ni or Cu for stainless steel to titanium. Need to understand very carefully tolerances and plating used. Non-portable.

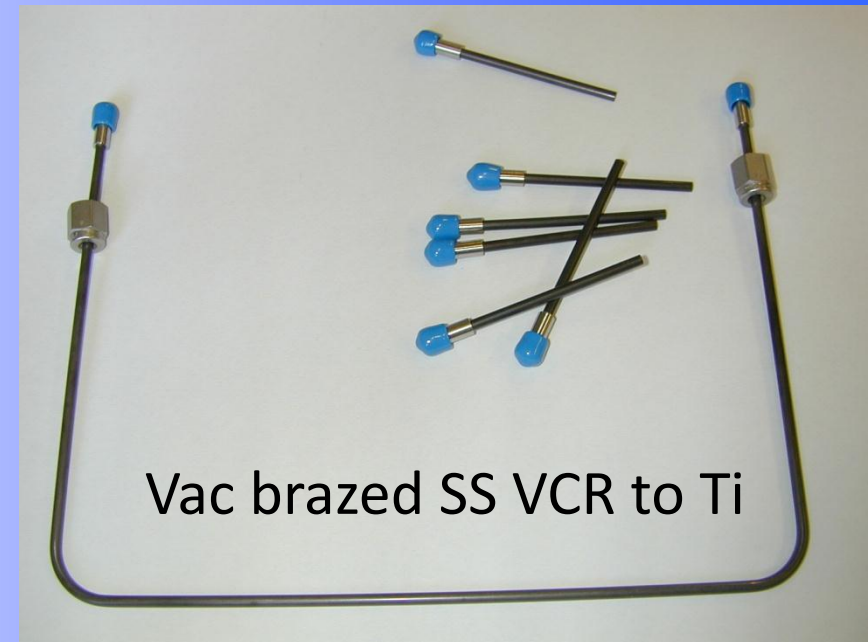
- Laser welding –

Excellent welds, high repeatability, high cost, non portable, limited availability

- Hard soldering / Brazing

A “get out of jail” method. Fluxes can contribute to accelerated corrosion. Non-uniform process unless carried out with a induction coil for heating.

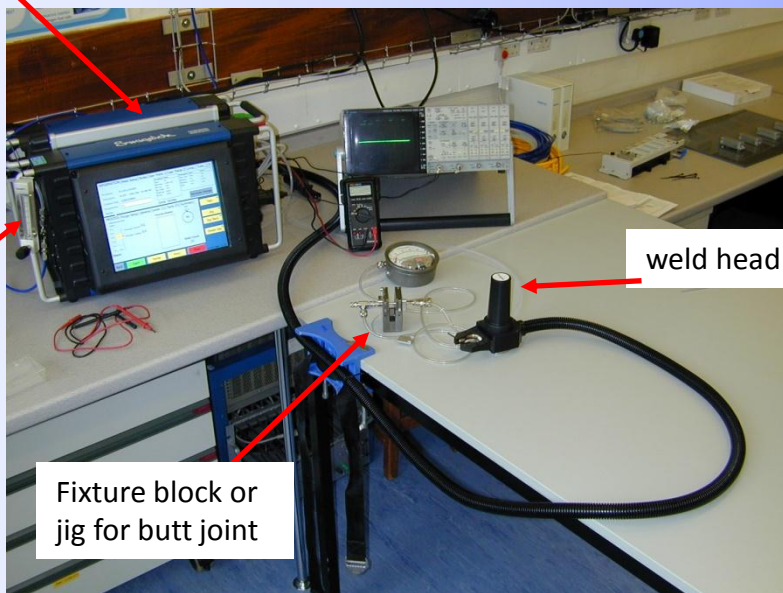
- *Many other forms of joining available but the above are most applicable to detector technology*



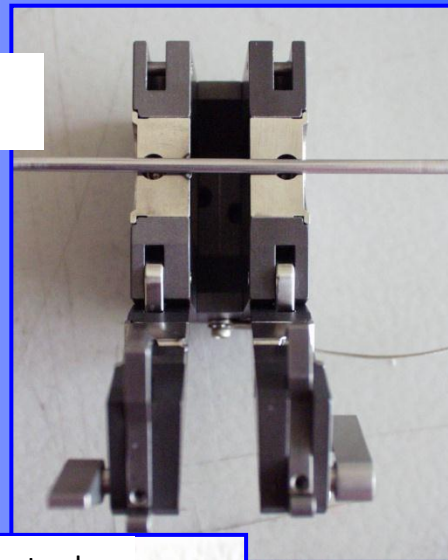


# TIG Orbital welding of 316L & CP2<sup>9</sup>Ti

## Orbital welder setup



Tube in  
block



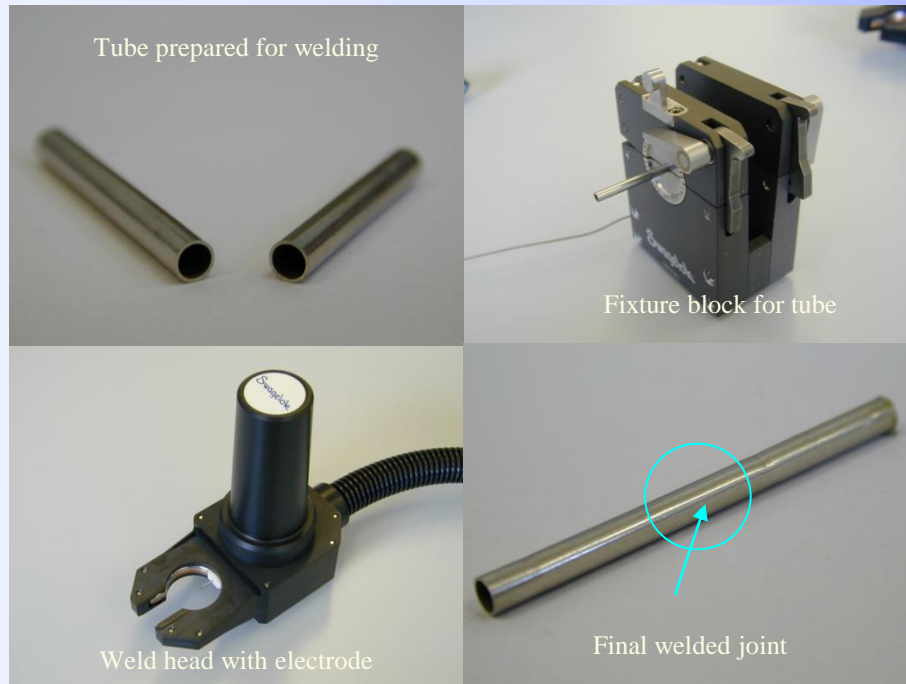
Rotary tungsten electrode



2 parts together for weld

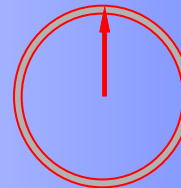


# The System and existing technique.



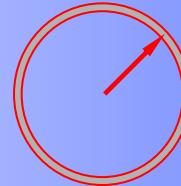
**Accumulation of significant R&D into thin wall joining of 316L Stainless Steel by TIG Micro-Orbital Process**

## Multi-pass Orbital Welding



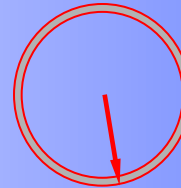
Ramp up and start 1st pass

360° Split into multiple sections with power and voltage parameters!



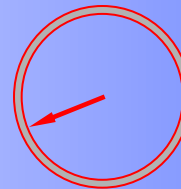
2nd pass with more power

Power ramps up close to start point and continues for pass



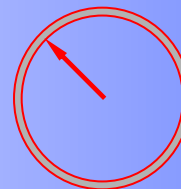
**3rd pass at full power**

**Max set A&V reached. Full weld penetration achieved.**



**4th Final full power pass**

**Weld reaching completion with uniform fusion**

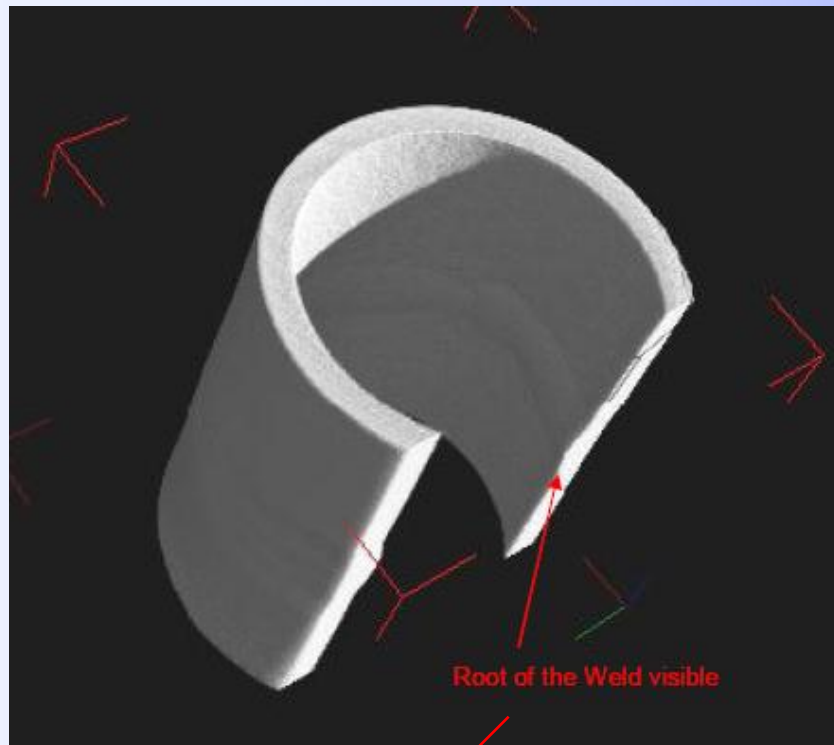


**Final ramp down and tail off**

Very gentle reduction in power with tail off to avoid puddling of weld

# Micro focus C.T X-ray scan with TWI

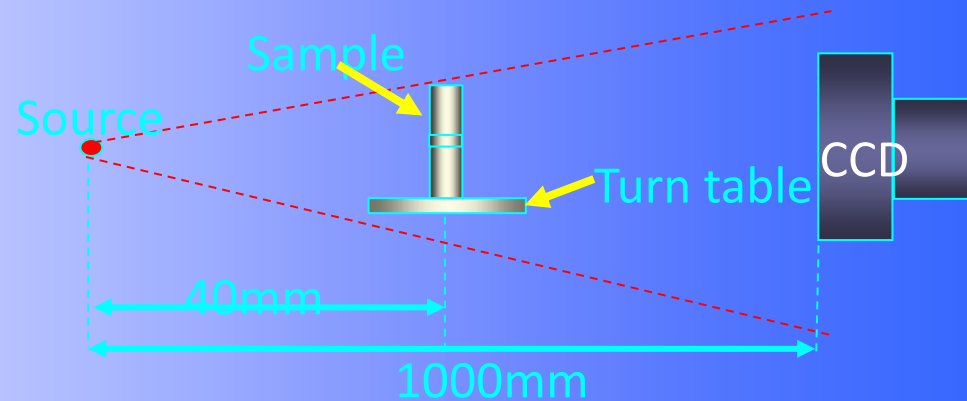
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**First 316L sample set 2006**

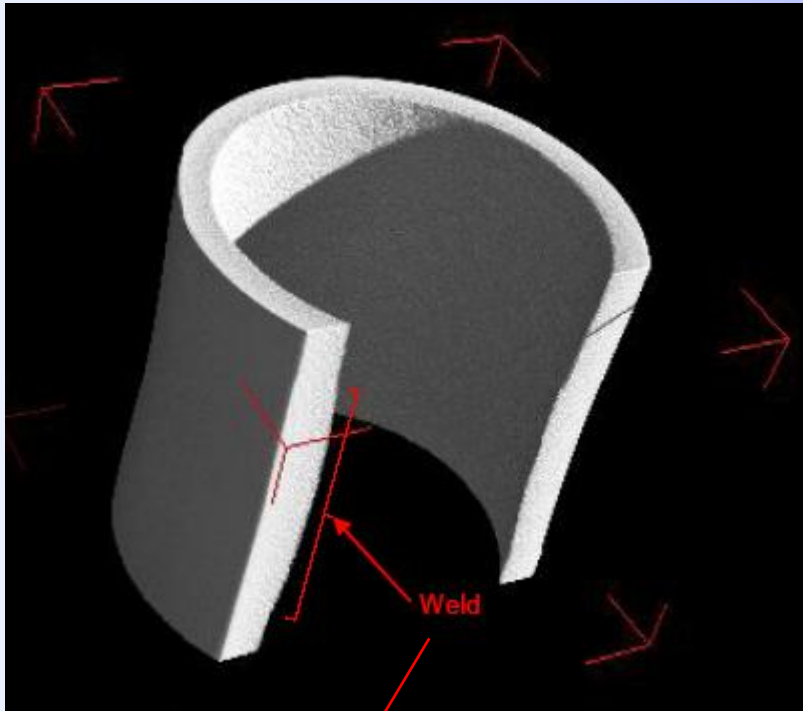
**3.18mm OD x 0.22mm wall thickness.**

04/11/2011



- Reconstruction at a level of 1 pixel.
  - Can view image from any section
- Grain growth is reasonable
  - Crystalline structure of material is altered but not within the realms of concern wrt weld standards of larger pipe sections.
- Severe excess weld penetration reducing ID and wall
  - The visual inspection is indicative of the CT results. Far too much heat = distortion.
- Tube concentricity is deformed. Severe bowing and damage.

# Micro focus C.T X-ray scan with TWI



316L Stainless Steel 3.18x0.22mm

Same CT imaging as before

- Comparison to previous 316L SS sample results

The reconstruction tells us:

- Less deformation to the crystalline structure
- HAZ is negligible – visual is first indication
- Weld is porosity free and virtually perfect.

## Room for improvement!

- Slight bowing (concave) to the wall due to slightly lower internal gas purge. Remedied but bows (convex)
- Gas regulation equipment not sensitive enough
- Internal pressure varies over length.
- Methods now understood and practiced for < 25m tube length using restrictors creating overpressure.

**Second sample set '08 & '09 made in Sheffield on new machine**



# Ti tube joining – TIG welding

Titanium welding work has progressed: Highly repeatable in 180µm wall Ti, now concentrating on 120µm wall Ti with some success. There are two main problem areas.



## *Electrode burn-out.*

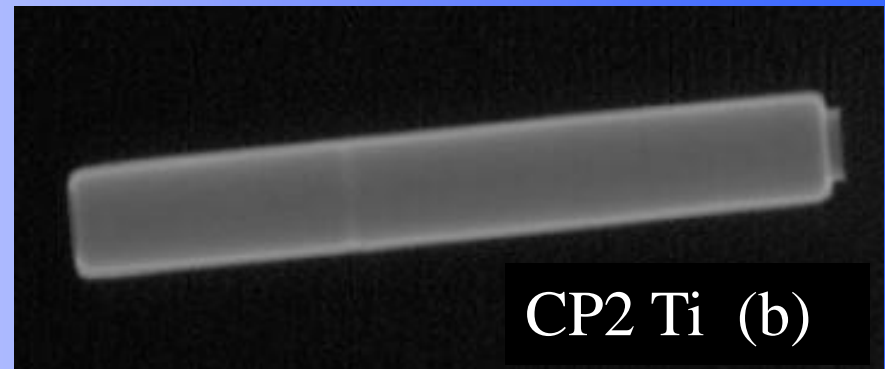
High arc temperature required for Ti welds is causing burn-out of Ciriated Tungsten electrodes. Changed to Thoriated electrodes that we make in house at Sheffield with the correct length and tip. These tungsten electrode contains 2% Thoria which is slightly radioactive. Processing of these electrodes should not be done without extraction.

The new electrodes and allow for a cooler arc during welding. This improved weld quality without depositing tungsten into the weld. Lower power could be used and weld and HAZ reduced.

## *Argon purge gas:*

Ag used is BOC Pureshield. From variable welds on thin wall tube, the transfer lines were holding moisture varying arc temp. Changing to BIP Ag from Airproducts with all 316L gas transfer lines gave immediate improvement.

Now investigating automatic gas purge control to allow for perfectly flat tubes and welding of multiple manifolded structures.



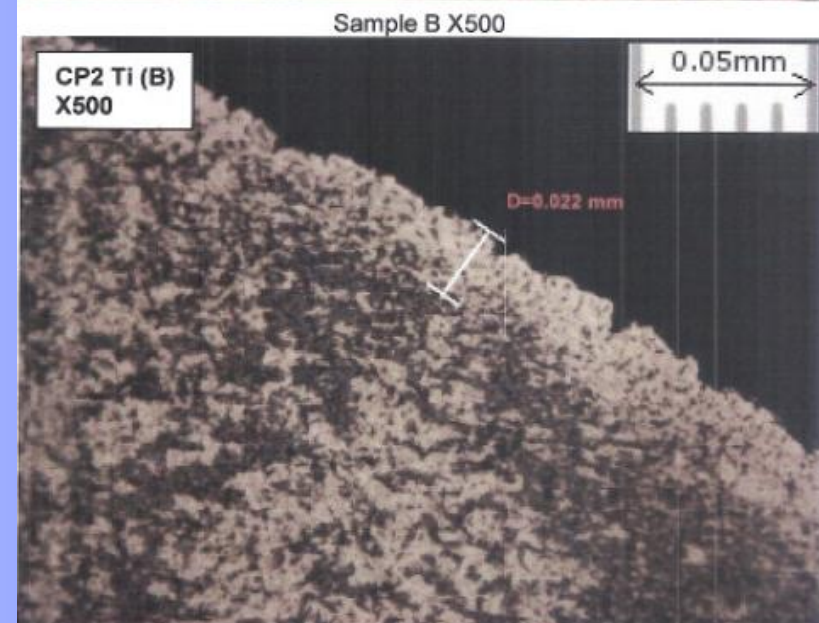
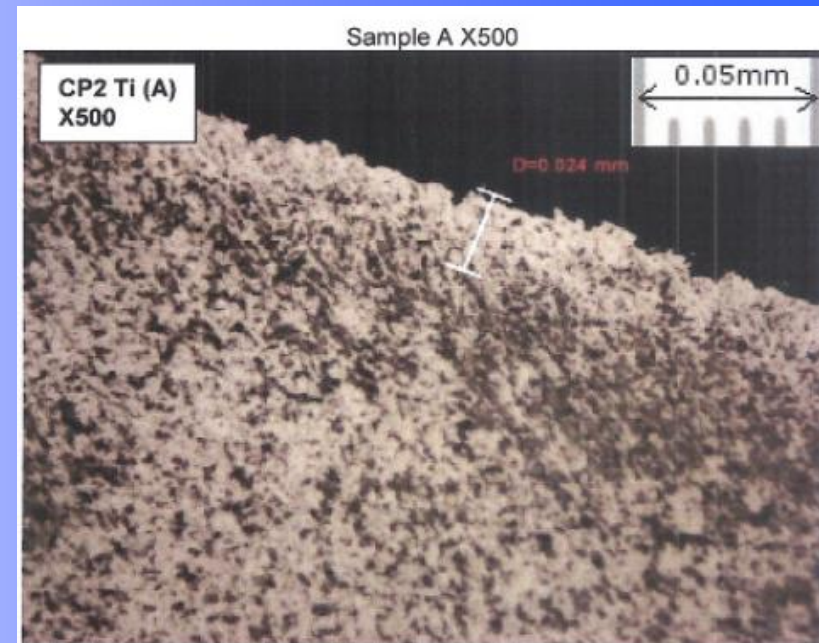


If time allows, some Titanium metallurgical studies in oxide removal.

Mass manufactured oxide free tube available in UK .

A&B = CP2 Titanium oxidisation outer wall thickness SEM image on 1/8" OD x0.18mm wall R&D tube used in UK.

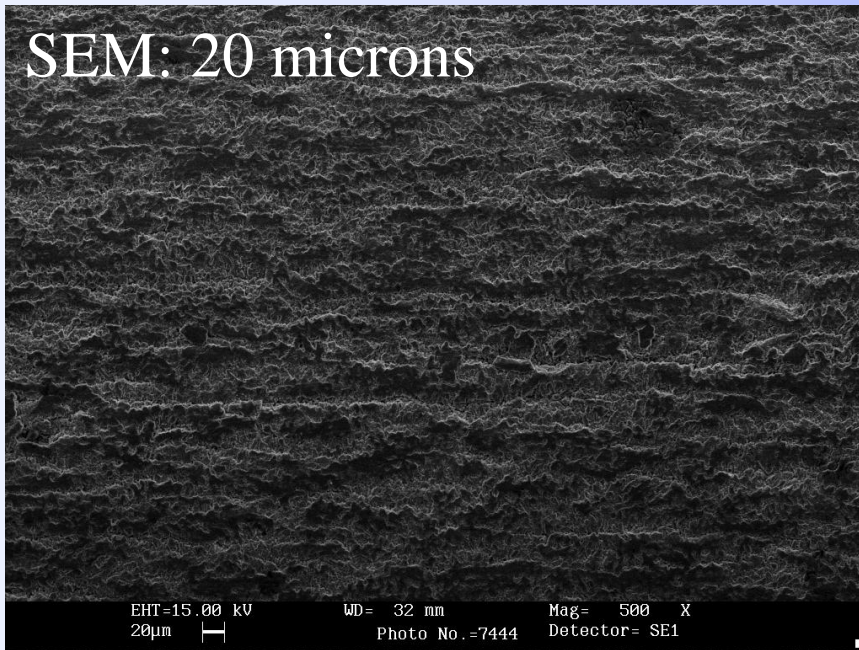
- Long term testing from 2009 still showing no problems in standard lab environment.
  - Sample kept on window of lab with half length immersed in salt water still ok.
    - [Like watering a house plant with seasoning]
- What happens if we remove this and why would we want to?
  - Welded joints with or without the use of a filler material are theoretically easier to make if the oxide is removed.
  - = Red herring for orbital TIG welding, made no noticeable difference to welds when the process was eventually understood.
  - Maybe thinner wall <120µm is a problem?



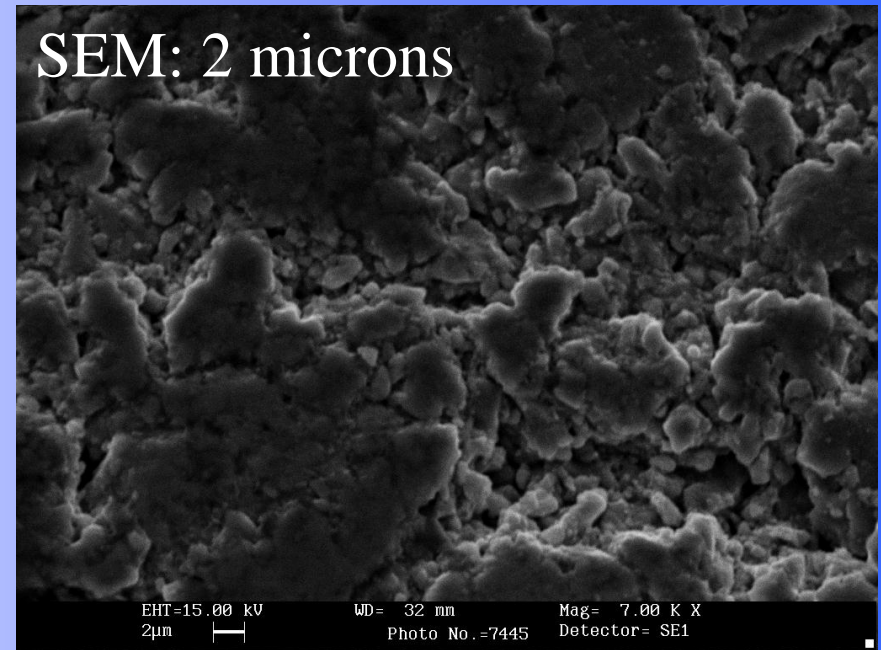


# Ti tube oxide images

SEM: 20 microns



SEM: 2 microns



Optical @ max magnification







# HF Etching

## Summary

Initial attempts at etching a titanium dioxide coating on titanium tubes resulted in removal of the coating but in most cases significant attack in the form of pitting and thinning of the titanium tubes. Diluting the etchant acids did not reduce attack of the titanium. It was noticed that the etchants used here did not actually dissolve the oxide but instead, caused the oxide to break up and flake off in pieces.

Best results were obtained with very highly concentrated etchant (samples 8 and 9). In these samples it appeared that the titanium in the oxide was being attacked. This caused the oxide to crumble and powder off rather than flake off. If left too long, the highly concentrated etchant would attack the titanium tubes. This necessitated the etching being stopped early. In samples 8 and 9 the tube wall thickness was preserved and these were the only samples where wall thickness was preserved. It was not clear if the inner oxide had been fully removed.

All samples are available for inspection and welding trials. (Further etchant details are available on request).

Titanium oxide wet etching in Nitric/Hydrofluoric acid



Unetched sample



Sample 3: HNO3 : HF (diluted); 8:1; 10mins



Sample 1 and 2: HNO3 : HF; 6:1; 6mins



Sample 4: HNO3 (diluted) : HF (diluted); 20:1; 20mins



Sample 5: HNO3 : HF (v.dilute); 5:2; 17mins



Sample 8: HNO3 (conc): HF (conc); ~20:1; 3mins



Sample 6: Repeat sample 5; 6mins



Sample 9: HNO3 (conc): HF (conc); ~10:1; 1mins



Sample 7: HNO3 : HF (dilute); 4:1; 6mins