

# **Metallographic Sample Preparation and Examination of NOCOLOK<sup>®</sup> Flux Brazed Heat Exchangers**

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## **ABSTRACT**

This paper/presentation describes the metallographic preparation and examination of a typical brazed heat exchanger. The information contained herein can be used by the metallographer to standardize the method of sample preparation and evaluation and to help the brazing engineer interpret the results. The report includes a few definitions and metallurgical concepts. With the aid of photomicrographs, the report shows how to characterize such parameters as fillet size, extent of erosion and residual cladding in tube-to-fin joints and tube-to-header joints. The actual mounting, polishing and grinding technique is described in a separate document.

## **1 SAMPLE PREPARATION**

### **1.1 Orientation**

The heat exchanger chosen for the purpose of this report is a NOCOLOK<sup>®</sup> Flux brazed radiator. To provide some orientation as to where the metallographic sections will be taken from, Figure 1 shows the water-side header area (top) and part of the fin-pack, side-support and header area.

In most metallographic investigations of brazed heat exchangers, the critical joints to examine are the tube-to-header joints and the fin-to-tube joints. For instance, a leak in a tube-to-header joint constitutes a failure. The fin-to-tube joint on the other hand, although not as critical as the tube-to-header joint, is the key area where heat transfer takes place. It is therefore necessary that the fin-to-tube joints are metallurgically bonded (i.e. brazed) for maximum heat transfer efficiency.

### **1.2 Sectioning**

With a band saw, the radiator can be cut down through the center of the tubes (see Figure 2, top). This will keep the fins intact. If necessary, one can saw through the fins if the blade is kept as close to the outer tube wall as possible. That is the outer tube wall can be used to guide the saw blade. Otherwise, it is better to saw through the center of the tube where the inner walls will act as the guide.

Once the above sections have been obtained, the samples can be sawn longitudinally through the center of the tube and header as shown in Figure 2, bottom. The tube-to-header and tube-to-fin sections can then be cut. The size of the cut samples must fit inside a 30 mm or 40 mm mount. Note that it is the cut face that will be grinded and polished.

## 2 GRINDING AND POLISHING

Appendix 1 describes the actual mounting, grinding and polishing procedure with respect to technique. The following section shows what the sample actually looks like after each grinding and polishing step. The intention is to help the metallographer track the progress of grinding and polishing with the help of visual aids.

Figure 3 shows what a section of "un-brazed" brazing sheet looks like under the microscope after wet grinding with 220, 500 and 1000 grit SiC paper. In each case, the sample is ground until all the grinding lines appear in the same direction, across the entire grinding face. It also helps that the grinding lines go in the direction of, or perpendicular to the area of interest. In this case, the grinding lines all run perpendicular to the braze sheet after 220 grit paper. After 500 grit paper (rotating the sample 90°), the grinding lines all run parallel to the tube and after 1000 grit, once more perpendicular to the braze sheet.

Figure 4 shows what the braze sheet looks like after each successive polishing step. After the 6 µm diamond suspension, the microstructure of the braze sheet becomes visible (more on microstructure later). At this stage, there are still a number of scratches. After the 3 µm diamond suspension, the microstructure is clearer and there are less scratches. Only after the colloidal silica are all scratches removed. The 64x magnification in Figure 4 is too small to reveal fine micro-structural details after the colloidal silica polish, but what is evident is that the braze sheet is now highly polished and scratch free.

## 3 METALLURGY AND MICROSTRUCTURE

### 3.1 Microstructure – Before Brazing

Figure 5 shows the same cross-section of the un-brazed braze sheet shown at the bottom of Figure 4, but at a higher magnification. To someone not skilled in the art, one sees only a number of black dots on a white background. These black dots are actually constituents or intermetallic phases which make up that particular alloy. In this case, the core alloy is AA3003 or Al-1.25% Mn. The Mn addition provides certain mechanical properties to Al. During processing, the constituents which are not soluble in Al coalesce and precipitate out of solution (the black dots) to give, in this case, intermetallic phases such as  $MnAl_8$ ,  $\alpha Al(FeMn)Si$  or Mn-Al dispersoids:

Metallography may then be defined as the "science concerning the constitution and structure of metals as revealed by the microscope."

A full treatise on the metallography of Al is beyond the scope of this paper and we will concentrate on the brazing aspects, as revealed under the microscope.

Returning to Figure 5, one can see a distinct layer at the top surface which appears different than the core. This top layer is the brazing clad and consists of, in this case an Al-10% Si alloy, designated AA4045. It is this thin layer which melts during brazing and flows to the joints, drawn there by the capillary forces. The constituents in the clad layer are thus Si particles uniformly distributed in the Al matrix. The term braze sheet is then defined as a core alloy clad with a lower melting point cladding alloy.

### **3.2 Microstructure – After Brazing**

As described above, during brazing the clad layer melts and flows to all joints to be formed. Upon cooling, or more correctly upon solidification, the microstructure of the original clad looks very different under the microscope. Figure 8 shows an example of what a fin-to-tube joint looks like after brazing. Inside the dotted line in Figure 8 (bottom) is a typical Al-Si alloy microstructure upon re-crystallization and solidification. The Si is now present as a needle like structure. Another characteristic feature upon solidification is the presence of dendritic cells surrounded by the Al-Si eutectic network.

What is noteworthy here is that the filler metal (clad) formed a metallurgical bond between the fin and the tube. If one looks closely at Figure 8, there is a continuous metal path between the fin and the tube. This is the essence of brazing:

Brazing is a process that produces coalescence of materials by heating them to the brazing temperature in the presence of a filler metal having a liquidus above 450°C and below the solidus of the base metal. The filler metal is distributed between the faying surfaces of the joint by capillary action.”

## **4 BRAZED JOINTS**

### **4.1 Fin-to-Tube Joints**

Figure 8 (top) shows a typical well brazed fin-to-tube joint. The fillet is neither too large nor too small. Note also that there is no longer any evidence of a clad layer on the outer tube surface to the left and right of the joint. This means that most or all of the filler metal has flowed to the joint. If there was still evidence of filler metal on the tube surface, it might indicate that the brazing temperature was not high enough.

The joint is also shown at a higher magnification (bottom). This Figure was described in detail earlier from a metallurgical point of view. From the brazing point of view, what one should note here is the extent to which the original fin and tube thickness was maintained. In this case there is minimal "erosion" of the fin or the tube by the filler metal. This will be discussed in more detail below.

### **4.2 Tube-to-Header Joint**

There are 2 sides to a tube-to-header joint, namely the air-side and water-side. In service, the air-side is the side exposed to the atmosphere while the water side is exposed to the coolant. Installed on a vehicle, one would not be able to see the water-side tube-to-header joint since it would be enclosed inside the plastic tank where the coolant flows, hence the name water-side. Most information about brazing can be obtained by examining the air-side tube-to-header joint.

Figure 9 shows a typical, well brazed tube-to-header joint on the air-side. Note that at the same magnification of the tube-to-fin joint shown in Figure 9 bottom, the fillet size in the tube-to-header joint is much greater. There is much more filler metal available for joining since both the tube and the header are clad. This is necessary to have enough filler metal to braze the entire

length of the joint, from the air-side through to the water-side. Furthermore, these joints are deemed to be "critical joints". In production, every single radiator is leak tested to ensure that there are no failures in the tube-to-header joints. A failure here means that the radiator would not hold the engine coolant. In production, a failure at the tube-to-header joint is called a "leaker".

One of the features to look for here is again the amount of tube erosion. In this case, most of the original tube thickness has been maintained. Erosion in the header is not as critical since the header is much thicker than the tube. One will also note that there is some residual filler metal on the header. This is not unusual in the case of the header for a couple of reasons. One is that the header cladding alloy is often different than the tube cladding alloy. Heat exchanger manufacturers often chose an alloy that is less fluid than the cladding alloy used on the tube. This prevents pooling of the filler metal and is less affected by gravity, which tends to pull the filler metal to the downside of the radiator during brazing.

Another reason that one often sees some residual filler metal on the header is that the filler metal tends to be less fluid on surfaces that have been severely deformed. When the header was produced, a stamping tool is used to punch the holes in the header where the tube is inserted. This stamping operation causes severe localized deformation. For metallurgical reasons, the filler metal fluidity is reduced in these deformed areas.

## 5 EROSION

A metallographic examination of a brazed heat exchanger is not complete without looking at the extent of erosion. Erosion occurs when Si from the cladding alloy diffuses into the core or base metal, to form more filler metal. The result is dissolution of the base metal, which reduces its effective cross-sectional thickness. The intention here is to teach the metallographer how to identify and measure the extent of erosion.

### 5.1 Measuring Extent of Erosion

Figure 10 shows 2 examples of fin-to-tube joints where severe erosion has taken place. The dashed lines in Figure 10 shows how much of the fin and tube was consumed by erosion. By measuring the original thickness of the tube and the thickness of the tube where maximum erosion occurred, one can calculate the extent of erosion, expressed as a percentage:

$$(19 \text{ mm} - 14 \text{ mm} / 19 \text{ mm}) \times 100 = 26\% \text{ erosion}$$

A similar calculation can be made with the tube-to-header joint shown in Figure 11:

$$(19 \text{ mm} - 7 \text{ mm} / 19 \text{ mm}) \times 100 = 63\% \text{ erosion}$$

A certain amount of erosion is always expected and based on our current knowledge, the heat exchanger industry tolerates about 10% to 20% erosion.

## 6 ETCHING

Etching the metallographic mounts after the final polish is often done to reveal fine micro-structural details that are not otherwise evident. Figure 6 shows what a section of "un-brazed" brazing sheet looks like under the microscope after HF-etching (precipitation structure). Figure 7 shows a section of the same "un-brazed" brazing sheet after  $\text{HBF}_4$ -etching (grain structure).

### 6.1 Etching Applied for Examination of the Precipitation Structure

The most common etchant for Al metallography is a 0.5% HF solution (v/v) with an etching time of about 15 seconds. Etching works by preferentially attacking some of the constituents and intermetallic phases, essentially changing their color and appearance when viewed under the microscope. Often times, the purpose of etching is for the identification of phases present in the microstructure. To a skilled metallographer, this can provide clues as to the material's thermal history, processing route etc.

Compare the 2 photomicrographs in Figure 12 where the sample at top is as-polished and the sample at bottom was etched for 15 seconds in 0.5% HF. The constituents in the etched sample are darker, more pronounced. For our purposes however, the etched sample does not provide any other information about the size of the fillet, the amount of erosion, residual filler metal on the tube etc. In other words, for the purpose of examining the quality of the braze, etching is not absolutely necessary.

### 6.2 Etching Applied for Examination of the Grain Structure

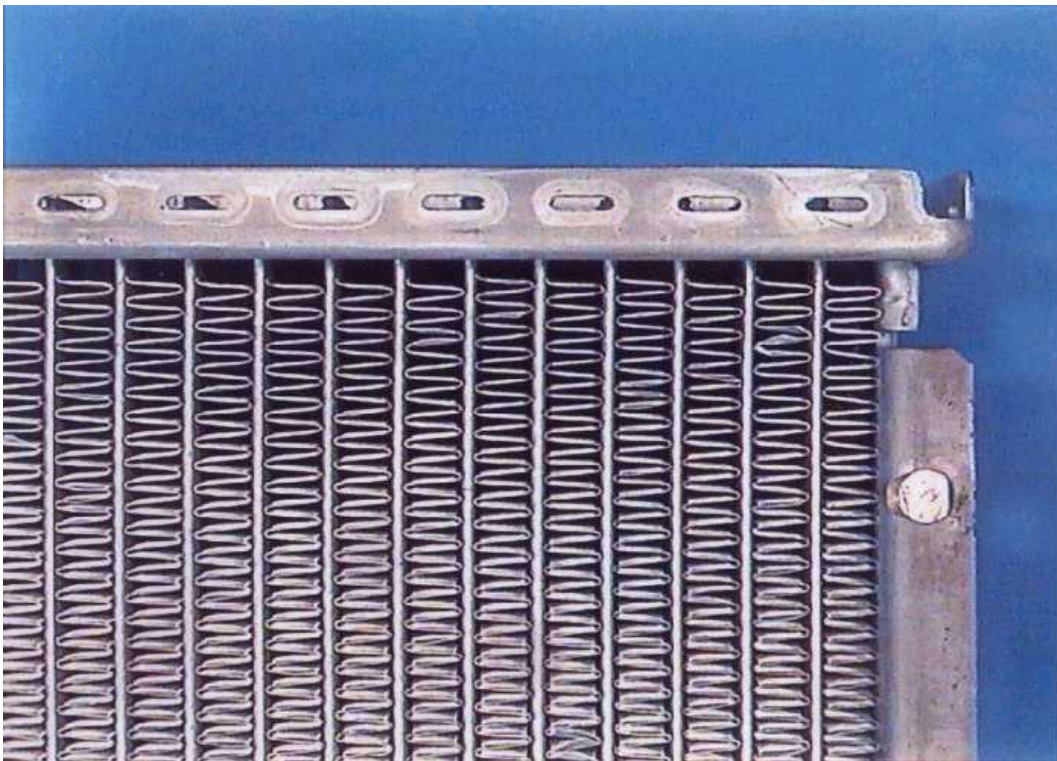
To reveal the grain structure of the sample, electrochemical etching is used. The procedure is based on Barker's etch (aqueous hydrofluoroboric acid solution with 3.2% (wt/wt)  $\text{HBF}_4$ ). Etching is performed in an electrolysis-cell where the polished specimen (anode) is wired through a drill-hole from the back side and a steel cathode. Electrolysis time is about 2 minutes. The voltage is 20 V. For visualization of the grain structure optical microscopy with crossed polarizing filters is used.

## CONCLUSION

The topics discussed in this report dealt only with a typical NOCOLOK<sup>®</sup> flux brazed radiator. The same principles apply however to all brazed heat exchangers whether it is a condenser, evaporator, heater core etc. The key areas of interest are always the fin-to-tube joints, tube-to-header joints, residual filler metal, size of the fillet and erosion.

**Figure 1**

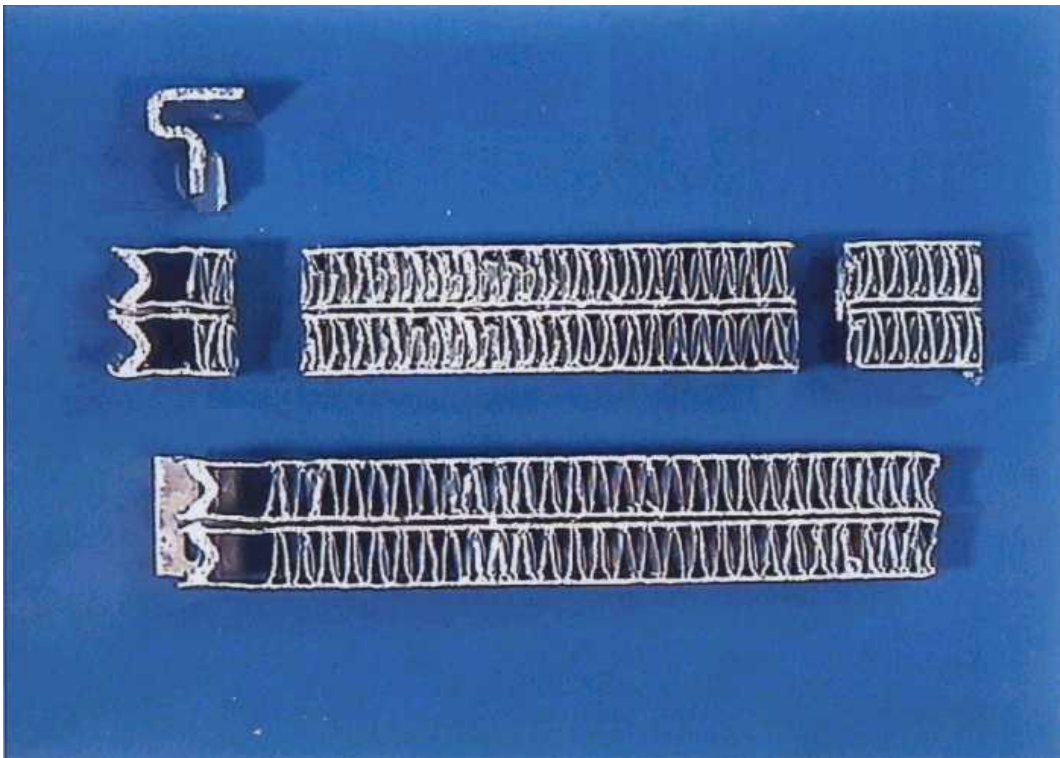
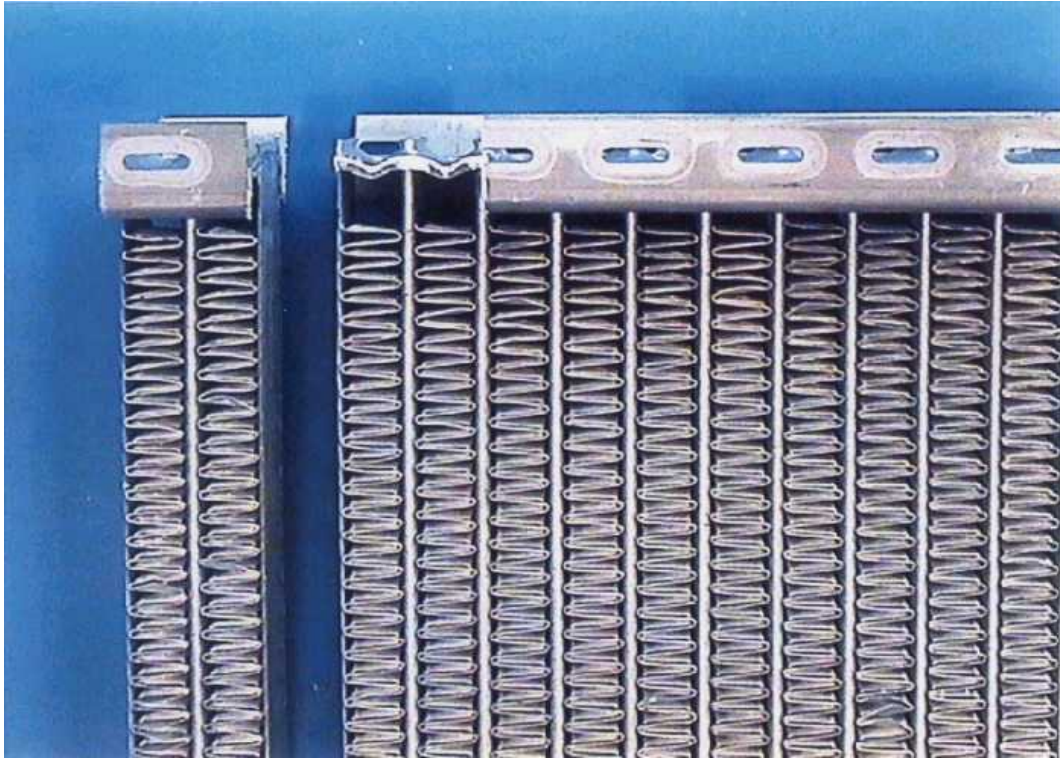
**NOCOLOK Flux Brazed Radiator – Water-Side Header View (top)  
and Fin-Pack with Side-Support and Header (bottom)**



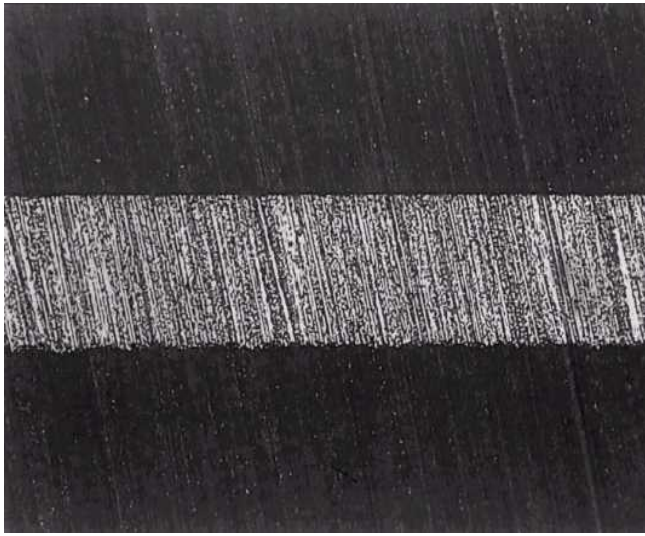


**Figure 2**

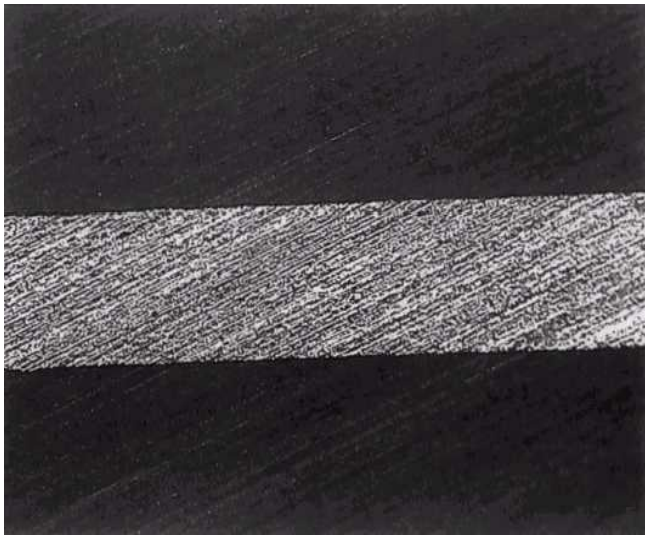
**Radiator Section Sawn Trough Center of Tube (top)  
and Sawn in Half Longitudinally (bottom)**



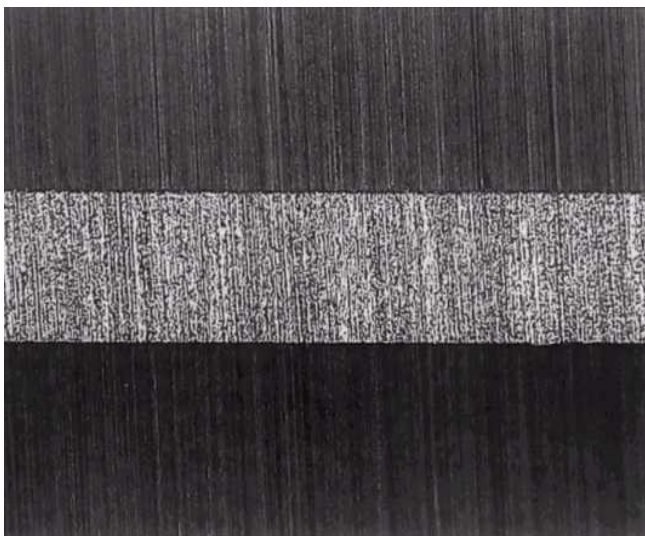
**Figure 3**  
**Appearance After Wet Grinding with 220, 500, and 1000 Grit SiC Paper**  
**(64 X magnification)**



220 grit  
SiC paper



500 grit  
SiC paper

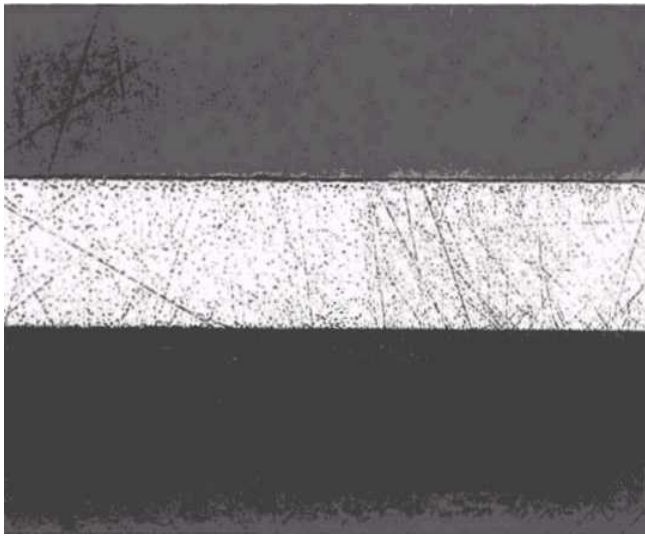


1000 grit  
SiC paper

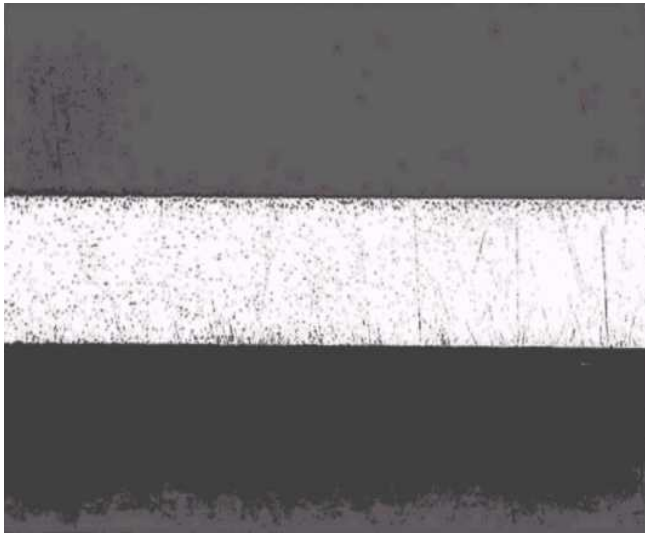


**Figure 4**

**Appearance After Polishing with 6 and 3 $\mu$ m Diamond Suspension and Final Polish with Colloidal Silica (64 X magnification)**



6 $\mu$ m  
diamond  
suspension



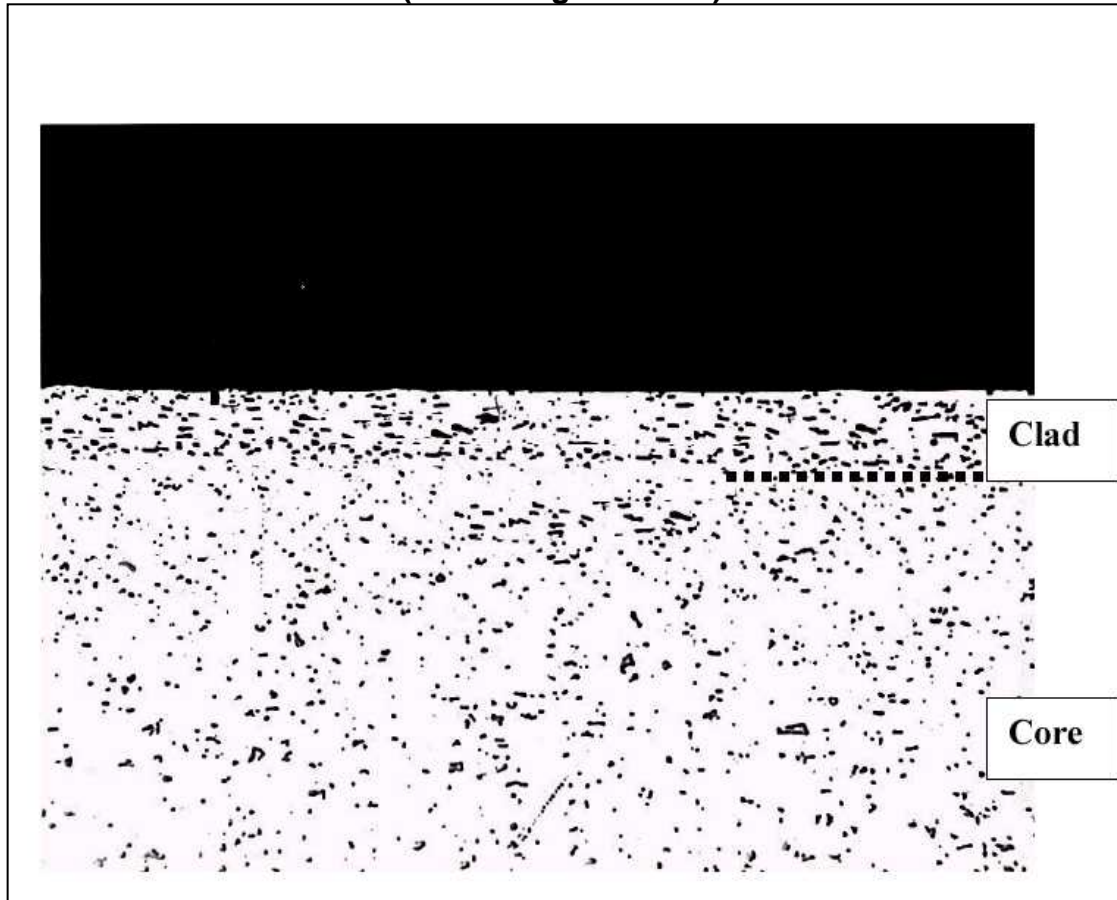
3 $\mu$ m  
diamond  
suspension



Colloidal  
Silica

**Figure 5**

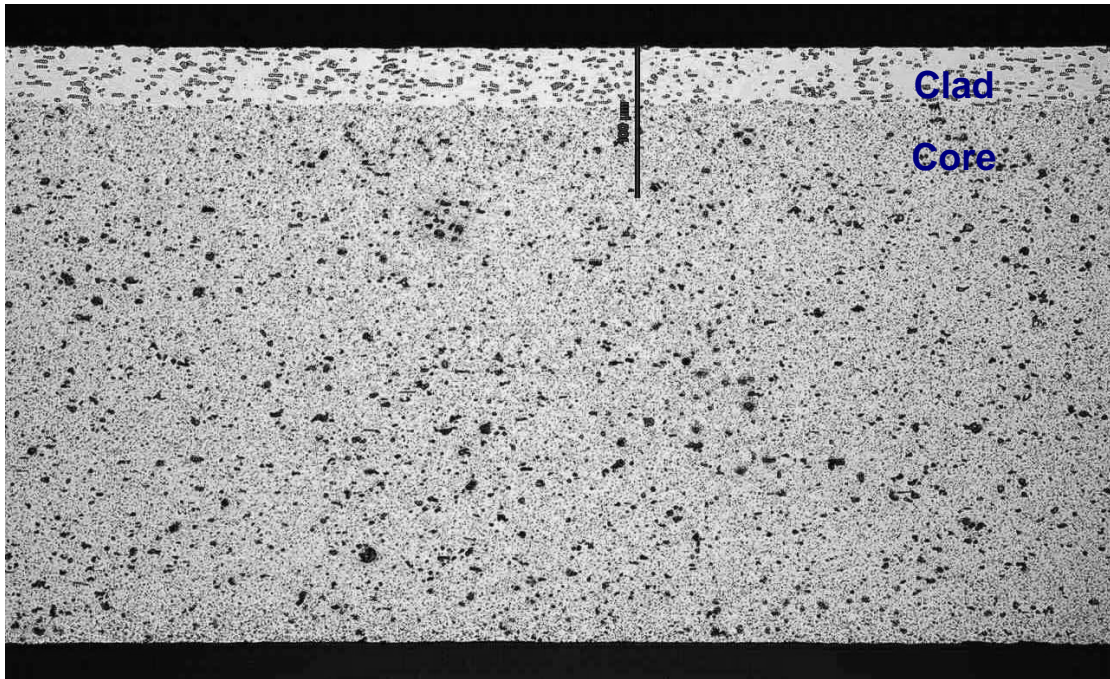
**Higher Magnification View of Cross Section Shown in Figure 4.  
Note Clad – Core Interface  
(200 X magnification)**





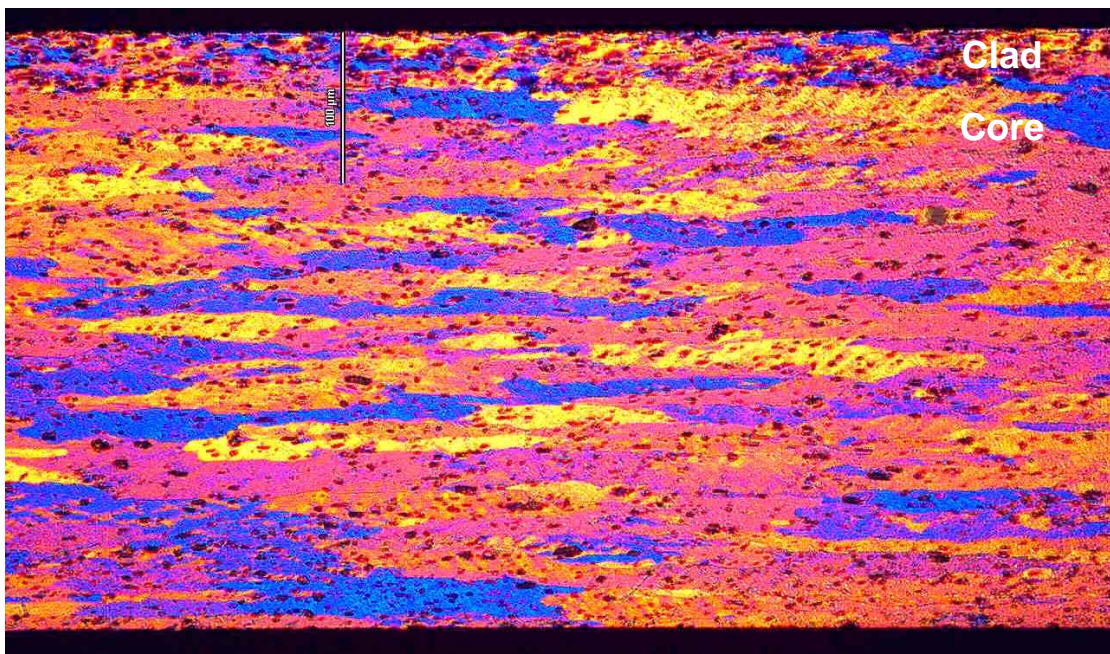
**Figure 6**

**View of Cross Section of Brazing Sheet (HF-etched).  
Clad AA4343 (7.5%Si) – Core AA3003 (200 X magnification)**



**Figure 7**

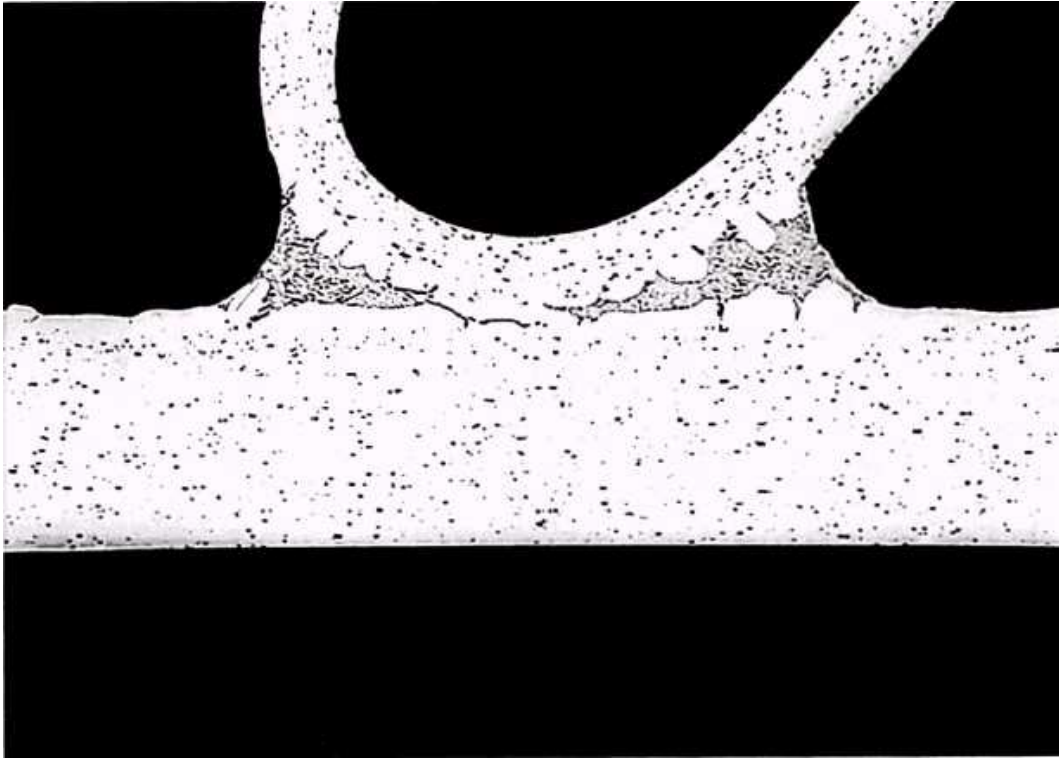
**View of Cross Section of Brazing Sheet (HBF<sub>4</sub>-etched).  
Clad AA4343 (7.5%Si) – Core AA3003 (200 X magnification)**



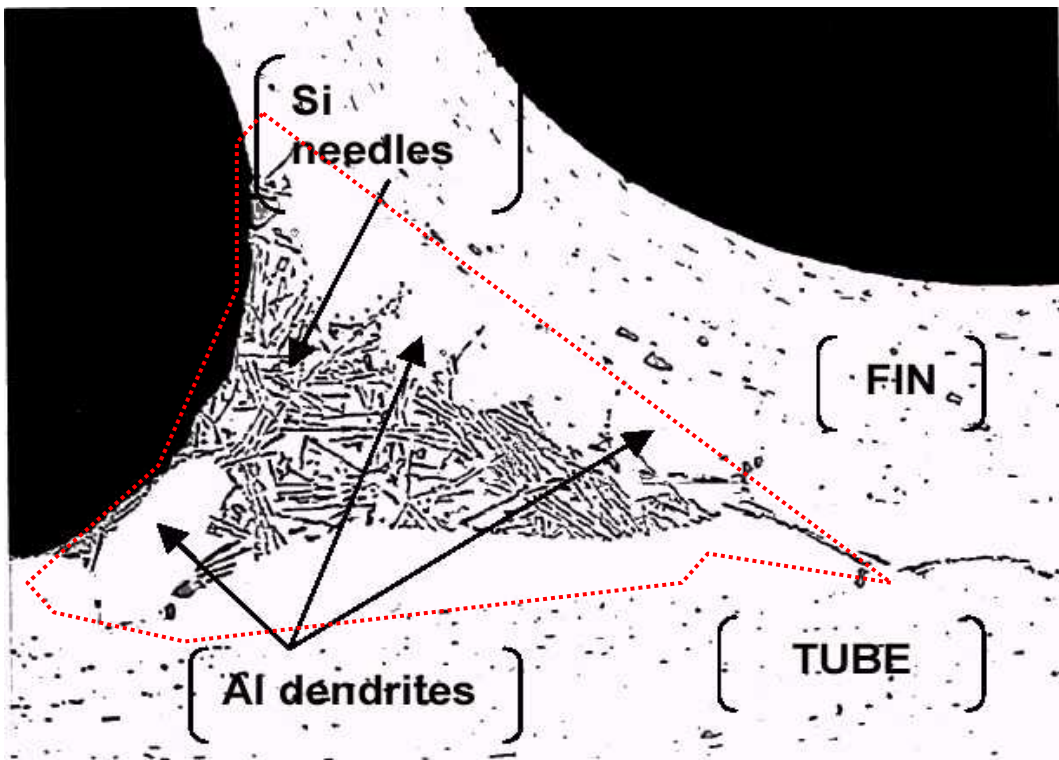


**Figure 8**

**Fin-to-Tube Joint (top)  
and Same at Higher Magnification (bottom)**



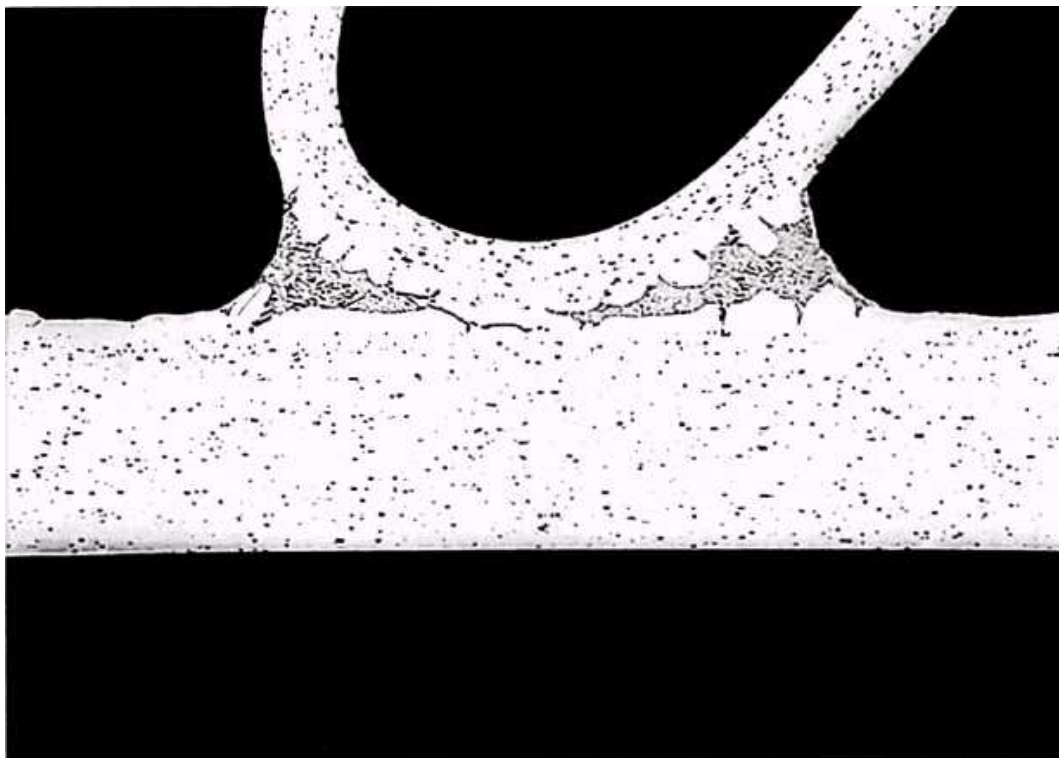
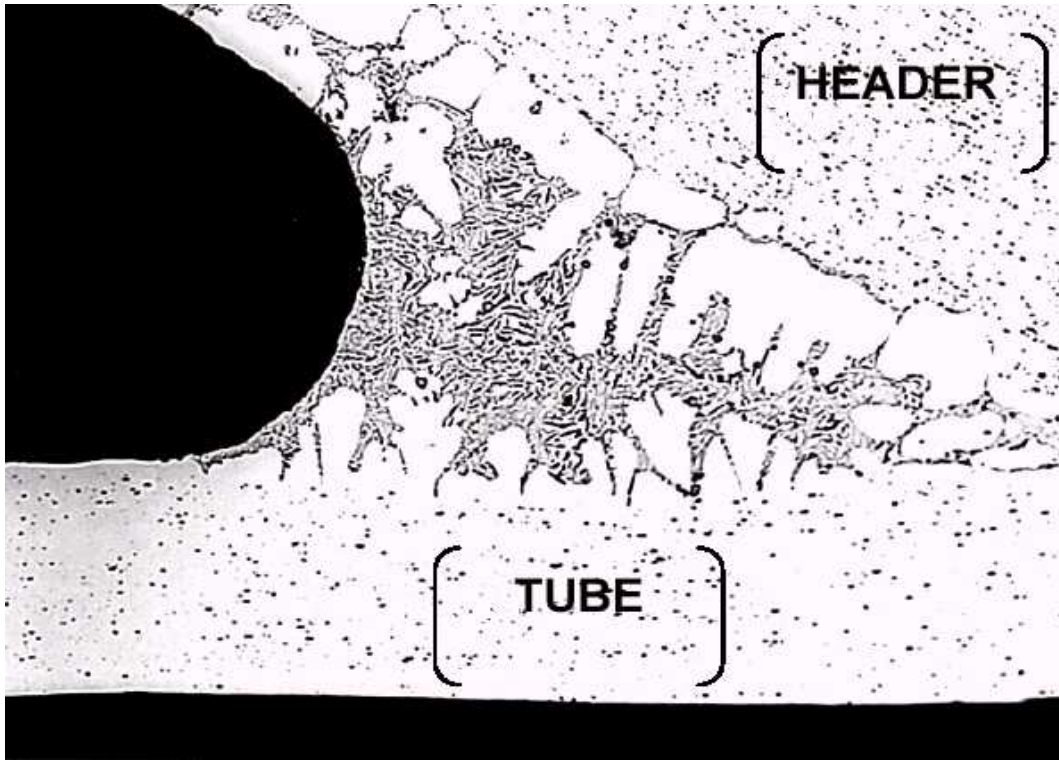
64 X



200 X

**Figure 9**

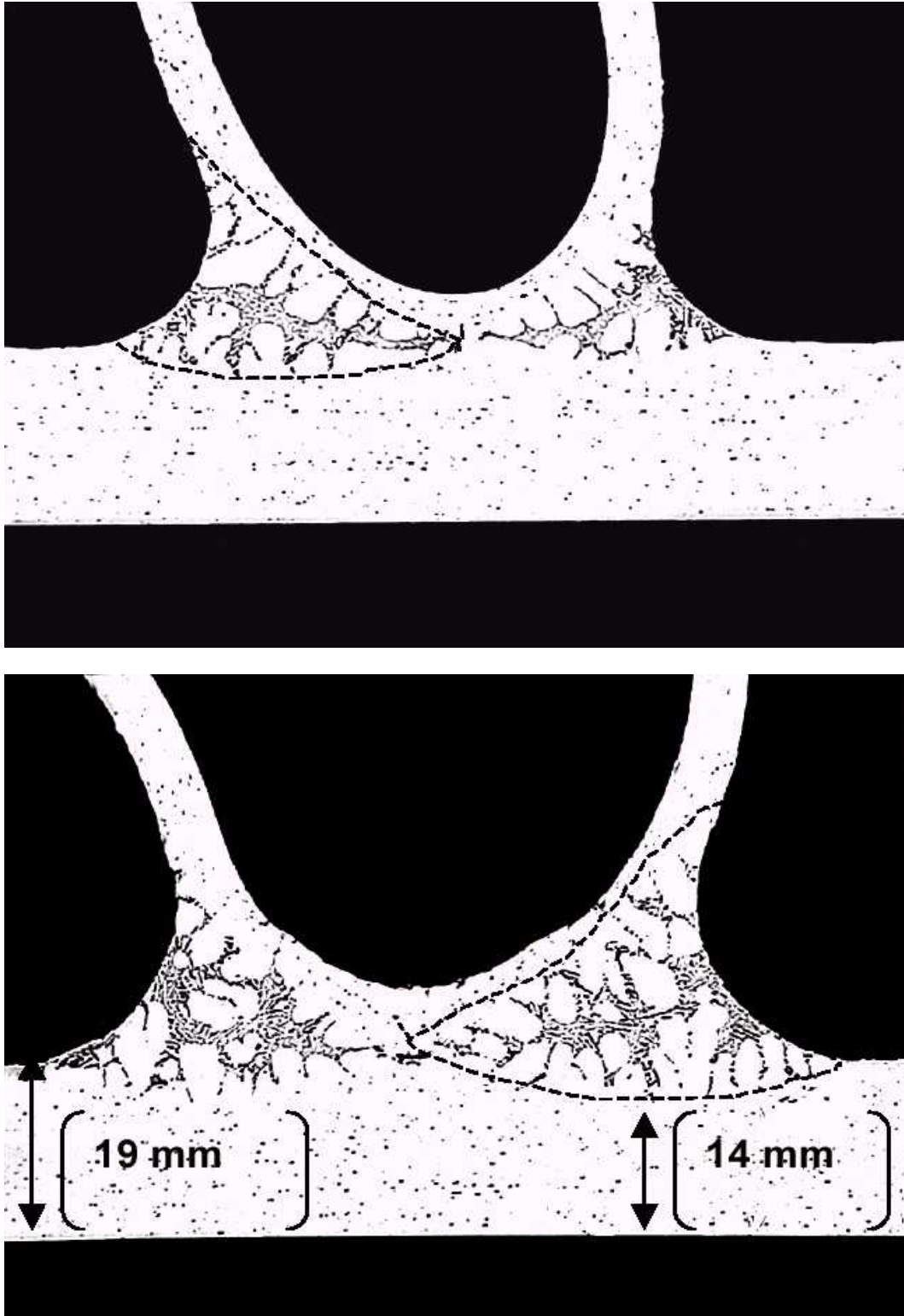
**Tube-to-Header Joint (top) Note Large Fillet Size in Comparison  
with Same Magnification Tube-to-Fin Joint (bottom)  
(64 X Magnification)**





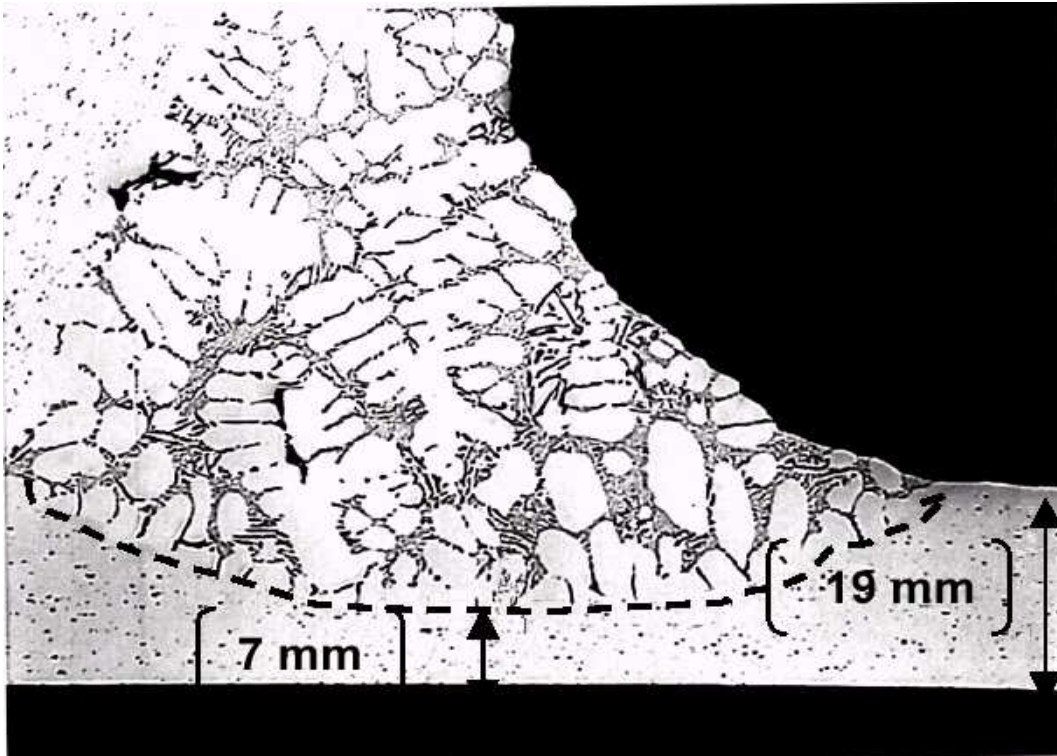
**Figure 10**

**Examples of Tube-to-Fin Joints Showing Severe Erosion.  
Dashed Line Shows Extent of Penetration into the Fin and Tube  
(64 X Magnification)**



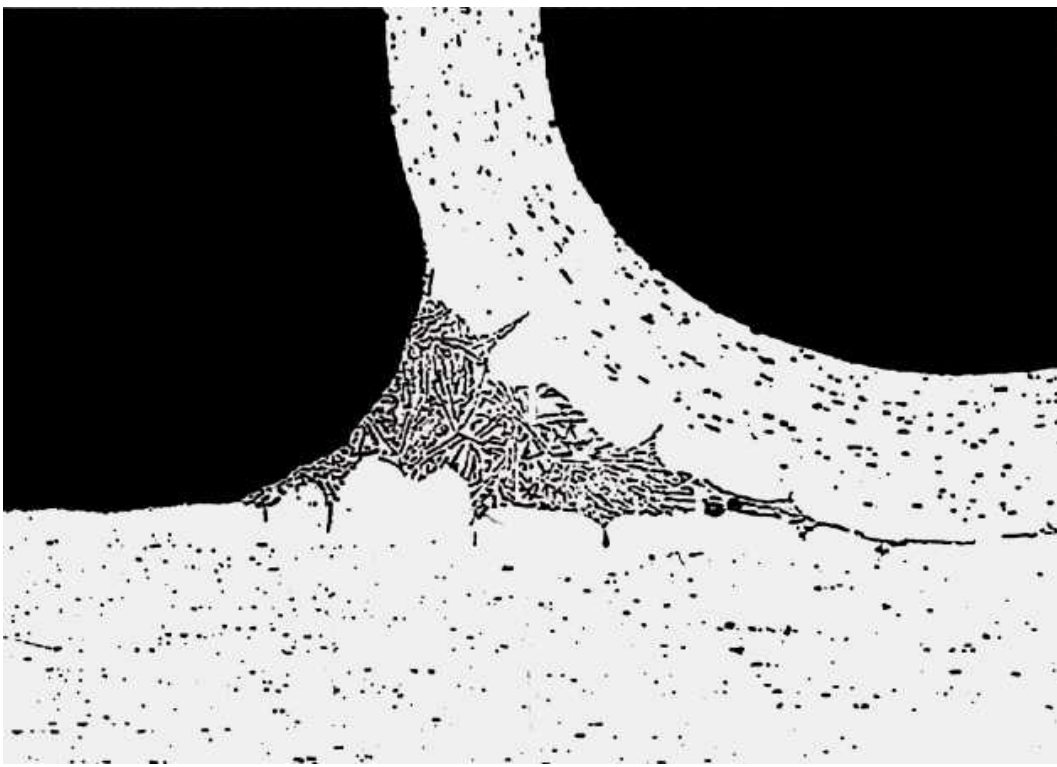
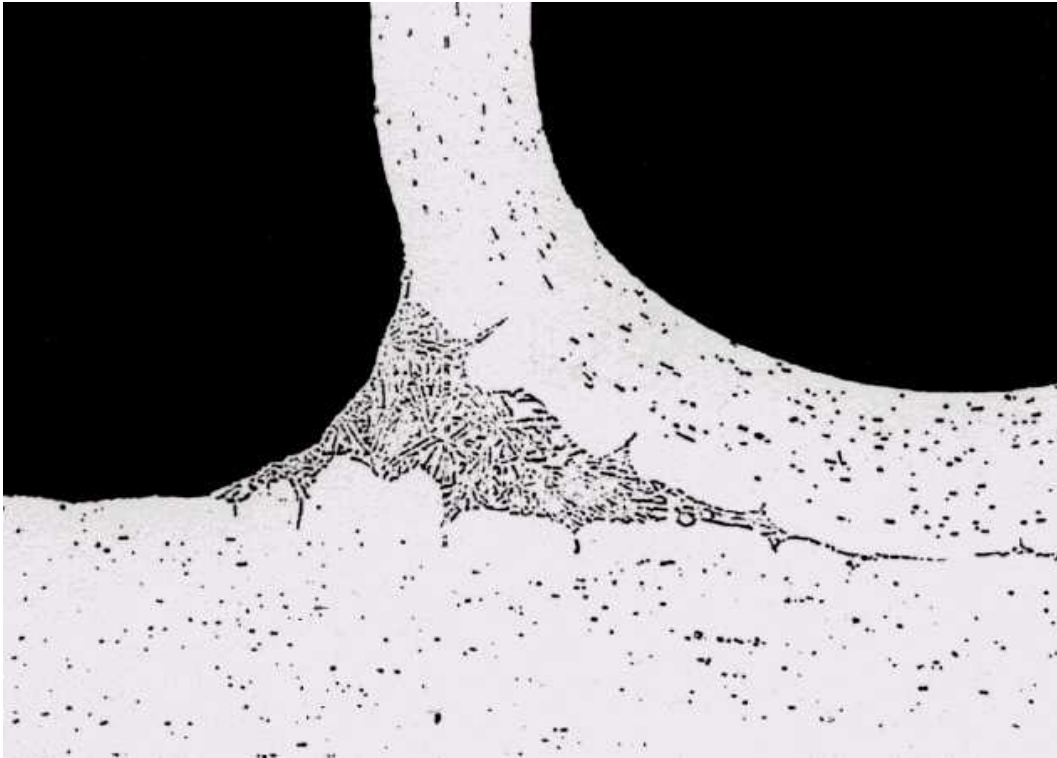
**Figure 11**

**Tube-to-Header Joint Showing Severe Erosion.  
Dashed Line Shows Extent of Penetration into the Tube  
(64 X Magnification)**



**Figure 12**

**Section of Fin-to-Tube Joint. As Polished (top) and Etched for 15 Seconds in 0.5% HF (bottom)  
(200 X Magnification)**



## **APPENDIX 1**

### **Metallographic Sample Preparation**

#### **Introduction**

The following description for metallographic mount preparation, grinding and polishing is certainly not the only method that works. However, it is one technique that is tried and true and if followed, should provide the user with satisfactory results every time.

As all of Solvay's equipment and consumables are supplied by Struers, the Struers tradename is used throughout the appendix.

#### **Mounting**

It is preferable to use a cold curing epoxy resin as a mounting media such as Epofix. These are slow, cold curing epoxy resins with virtually no shrinkage. Acrylic and polyester resins may also be used and the advantage of these are the relatively short curing times. However, the disadvantage with any fast curing resin is that they are susceptible to shrinkage. The problem with shrinkage is that during curing, a gap between the mounting media and the aluminum component may form. During polishing and grinding, dirt may accumulate in the gap and be carried over to subsequent grinding and polishing stages. This dirt can contaminate the cloth and cause undesired scratches on the mount and the area of interest. Furthermore, a gap will also cause the edges of the part being polished to be rounded, thereby losing detail during microscopic viewing. If one must chose a fast curing mounting medium, it is essential that shrinkage be kept to a minimum.

It is preferable to work with cylindrical mounts, either 30 mm or 40 mm diameter. Larger mounts or rectangular mounts are avoided since these are more difficult to handle for grinding and polishing. Furthermore, larger mounts require more resin and more hardener. Since the curing process is exothermic, larger mounts generate more heat during curing and therefore more susceptible to shrinkage and deformation, even with slow curing epoxies.

After the epoxy resin and harder have been mixed and poured into the mount, it should then be placed in vacuum to remove trapped air bubbles. A glass desiccators attached to a house vacuum line of about 200 torr is sufficient.

#### **Grinding**

Prior to grinding, the edges of the mount should be rounded, both on the grinding face and the top side. This will prevent the mount from catching the polishing cloth during polishing and, without sharp edges, is more comfortable to hold during manual grinding.

Grinding is often performed manually, especially when there are specific areas of interest to be examined and many times when the metallographer is skilled. Otherwise, automatic grinding can also be performed, but the metallographer has no way of knowing if all the scratches from previous grinding stages have been removed, as will be explained in more detail below.

The metallographic mounts are first wet ground with SiC paper in the following sequence at a rotational speed of 300 rpm: 220 grit, 500 grit and 1000 grit. Note that it is not wise to reuse grinding paper over and over again. Each paper is good for 1 or 2 mounts and should then be discarded.

#### Initial Grinding – 120 or 220 grit

After the edges have been rounded, the first grinding step begins. This is one of the most important steps and the procedure should be observed carefully. These steps do not apply if the grinding is done automatically, but the principles remain the same. First, the mount should be held flat on the paper preferably with 2 hands to stabilize the mount. Using a slight side to side motion (to use more of the paper's surface area), count slowly to 10 and lift the mount from the paper and examine the ground area. Without rotating the mount, return it to the paper and count to 10 again. This is continued in this fashion until the scratch marks, going all in the same direction, cover the entire grinding face.

When this point has been reached there are 2 choices. If the area of interest has been reached, then it is time to switch to the next finer grinding paper. If the area of interest has not been reached and more grinding is required, then the mount is to be rotated 90° and grinding is continued as described above until once again all the scratch marks appear in the same direction. This procedure should be followed until the area of interest is reached. In any case, before switching to the next finer paper, all the scratch marks should appear in the same direction.

#### 500 and 1000 Grit

Any time the paper is switched, the mount should be rinsed clean to avoid any SiC particle carry-over onto the next finer paper. The mount should again be rotated 90° and then ground using the same procedure as above. As soon as all the scratch marks appear all in the same direction, it is time to switch to the next finer paper. After the 1000 grit paper, it is time for polishing.

### **Polishing**

Polishing is carried out in 3 stages with an automatic sample holder at 150 rpm. Each of the 3 polishing stages uses a special combination of polishing cloth and polishing medium:

1. DP-Dac cloth with 6 micron diamond suspension – 3 minute polishing time
2. DP-Mol cloth with 3 micron diamond suspension – 5 minute polishing time
3. OP-Chem cloth with colloidal silica – 45 second polishing time

In between each polishing stage, the mount is cleaned with a mild detergent and cotton batting. This is again important to avoid carry-over of contaminants and polishing media.

These cloths are intended to be used over and over again and should only be changed when they become heavily contaminated. The level of contamination is difficult to gauge. As the metallographers gain experience, they will be able to better gauge when it is appropriate to change the cloths. Furthermore, these cloths perform well when they are well conditioned (i.e., after a number



of samples have been polished). Brand new cloths may require slightly longer polishing times than described above until they become well conditioned.

Special care should be exercised with the final polishing step. Colloidal silica dries quickly, both on the cloth and on the mount. If it dries on the cloth, very hard particles will form which will cause more harm than good. Care should be taken to avoid any drying of the cloth. After use, the cloth should be rinsed under running water for a period of time to remove as much of the colloidal silica as possible. Also, after polishing with colloidal silica, the mount should be rinsed and cleaned as quickly as possible.

### **Etching**

The best known etchant for Al heat exchangers and for most Al alloys is 0.5% (v/v) HF with an etching time of about 15 seconds. This etchant will reveal most features of interest without masking fine micro structural details.

To reveal the grain structure of the sample, electrochemical etching is used. The procedure is based on Barker's etch (aqueous hydrofluoroboric acid solution with 3.2% (wt/wt)  $\text{HBF}_4$ ). Etching is performed in an electrolysis-cell where the polished specimen (anode) is wired through a drill-hole from the back side and a steel cathode. Electrolysis time is about 2 minutes. The voltage is 20 V. For visualization of the grain structure optical microscopy with crossed polarizing filters is used.

In any event, the sample should be viewed under the microscope briefly prior to etching. This practice will teach the metallographer which features are being highlighted by etching.