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## **Heat Treatment of Zirconium Equipment**

### **Stress Relief Heat Treatment**

Stress relief annealing consists of heat-treating the material at 550°C +/- 50 °C for 1 hr per inch of thickness. This treatment is employed to reduce the residual stresses caused during severe forming and for weldments of heavy to light sections where shrinkage stresses are present. This heat treatment is also performed on zirconium equipment, which will be exposed to environments where zirconium is susceptible to stress corrosion cracking.

Stress relief heat treatment is required for all weldments of Zr 705 grade no matter what application it is to be placed in. This is to reduce the susceptibility for delayed hydride cracking in Zr705 weldments. All Zr705 vessels fabricated for ASME pressure vessel uses must be stress relief annealed according to ASME Code Section VIII, Div 1 UNF-56 which states “Within 14 days after welding, all products of zirconium Grade 705 shall be heat treated at 1000 °F - 1100°F for a minimum of 1 hr for thicknesses above 1 in. plus ½ hr for each additional inch of thickness. Above 800°F (427°C), cooling shall be done in a closed furnace or cooling chamber at a rate not greater than 500°F/hr (278°C/hr) divided by the maximum metal thickness of the shell or head plate in inches but in not case more than 500°F/hr (278°C/hr). From 800°F (426°C), the vessel may be cooled in still air.”

### **Oxide Thickening Heat Treatment**

Oxide thickening is performed at the same temperatures as stress relief, i.e. 550°C, except for a longer time (4-6 hrs @ temperature). This heat treatment is used to produce a thick oxide coating, which resists erosion, galling and seizing. It is generally used on rotating or sliding contact parts such as pumps, shafts, valve parts, bolts and nuts. This treatment is also used on sieve trays and those parts where erosion / abrasion is considered a problem.

**Full Anneal Heat Treatment** (for weldments exposed to the higher concentrations of sulfuric acid)

In the higher concentrations of sulfuric acid >55% and at temperatures approaching boiling, zirconium weld and heat affected zones are susceptible to preferential attack. (See attached isocorrosion curve and reference to weld limit line) During welding, a relatively continuous network of second phase particles (agglomerations) form in the alpha grain boundaries in the weld and heat affected zones during solidification and

cooling of the weld. A heat treatment performed at temperatures of 625-788°C for 0.5 – 4.0 hrs will disperse and homogenize the particles so that there no longer presents a continuous network in which corrosion could occur. The optimum heat treatment is 760-788°C for 1 hr at temperature.

Furnace parameters, such as heat up and cool down rates, for zirconium equipment and components will depend on the design and maximum thickness of the vessel to be heat treated. It is recommended that the temperature of the furnace not exceed 427°C prior to placing the vessel or component in it. Typical heat up rates for zirconium equipment is in the range of 100-260°C per hour, but it should not exceed 260°C per hour. Cooling rates for zirconium equipment at temperature above 400°C should be done at a rate not greater than 260°C divided by the maximum metal thickness in inches, but should never exceed 260°C per hour.

Localized heat treatment can be employed where it may be undesirable to heat treat a full vessel due to the thermal expansion differences in the material used for construction. If the configuration of the vessel allows the use of localized heat treatment, this method should be considered. In heat exchangers, for example, the tube to tubesheet seal welds may be heat treated locally for improved weld and heat affected zone corrosion resistance. Localized heat treatment is generally accomplished using resistance heaters.

