

KOVAR[®] - GLASS SEALING PROCEDURE
(For Electron Tubes)

The recommendations given in the bulletin refer particularly to electron tube construction. For less critical applications the procedure may be altered to meet less exacting conditions.

I. Preparation of Metal Surface

- A. On edge type seals, the edge should be rounded by metal removal with a radius equal to approximately one-half of metal thickness.

Machine lubricants to be noncorrosive mineral oil base capable of being completely removed by trichlorethylene degreaser.

- B. Polish sealing surface with 100-Grit Aluminum Oxide (ALOXITE) abrasive cloth, followed by 180-Grit to remove all scratches, tool marks etc.

1. Emery cloth and other abrasives containing carbides must be avoided.
2. Centerless ground rod requires no additional polishing.
3. For best oxide adherence, avoid highly polished sealing surfaces.

- C. On Butt Type Seals, instead of cloth polishing, it is preferable to have a matte finish, as resulting from sand blasting using pure alumina (silica sand is to be avoided).

The metal surface which will be enveloped by glass should be clean and free from longitudinal die marks, scratches, and similar imperfections. Slight circular marks put in by grinding or polishing are not considered objectionable.

II. Preparation of Glass (Typical Sealing Glasses for Kovar-Corning
705-2 or 705-6)

- A. Remove dust by wiping with lint-free cloth.
- B. Rinse in 10% (by volume) Hydrofluoric Acid Solution
(with wetting agent).
- C. Rinse in running tap water.
- D. Rinse in distilled water.
- E. Dip in Menthanol and hot air dry.

III. Processing Kovar[®] Alloy Prior to Glass Sealing

- A. Vapor degrease in Trichlorethylene.
- B. Immerse for one to three minutes in concentrated hydrochloric acid without inhibitor heated to about 80° C. (Optionally followed by rinse in alkali solution).
- C. Rinse thoroughly in cold running water for five minutes, followed by immersion in distilled water.
- D. Dip in Menthanol and dry in hot air blast.
- E. Furnace heat treat in wet hydrogen atmosphere.
 - 1. Hydrogen to be saturated by passing thru water - bubbling bottle at room temperature.
 - 2. 1 - Hour at 900° C. or 30 minutes at 1000° C.
- F. Store in lint-free containers (Bell Jar or wrap in Polyethylene bags).
- F. Precautions:
 - 1. Sealing surface of cleaned parts not to be touched by bare hands.
 - 2. Processing of metal parts to be done as soon as practical before glass sealing.

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IV. Glass Sealing

A. Equipment:

1. Burner preferably using gas-oxygen flame (gas and air or hydrogen and air combinations may be used either of which are harder to adjust to required hard-sharp oxidizing flame than the preferred gas-oxygen flame).
2. Glass lathe.

- B. Heat metal and glass parts to approximately 850° C. (dull red heat) in air to develop oxidized surface and bring parts together by pressure.

Before glassing the oxide should be just thick enough to obscure all metallic reflection.

- C. Glass to be worked so that the meeting of the edge with the Kovar approaches a 90° angle (feather-edging of the glass and glass having a re-entrant angle both result in mechanically weak seals).

- D. Flame anneal seal.

- E. Furnace program anneal for large seals.

1. Advance to annealing temperature for 30 minutes.
2. Annealing temperature to 50° C. below strain point at approximately 1° C. per minute.
3. Strain point to room temperature at approximately 10° C. per minute.

(Note: Small seals may be flame annealed by smoky flame instead of program annealed.)

For high quantity production, the glass sealing operation may be done by automatic stem machine, induction heating or neutral atmosphere furnace. Pre-oxidized metal parts are generally used on the latter two methods. A typical cycle for pre-oxidation is heating for three minutes at 900° C. in an electric furnace with air atmosphere. The cycle must be varied according to furnace heating capacity and humidity conditions.

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V. Inspection

- A. Seal to have desired stress condition as determined by polariscope viewing or by other stress analysis method.
- B. The color of the seal depends upon the grade of glass and thickness of the oxide. A general color description is:
 - 1. Light gray for 7052 glass.
 - 2. Mouse brown for 7040 glass.
 - 3. Dark gray-black for 7056 glass.
- C. A shiny appearance indicates insufficient oxide and extremely black surface indicates excessive oxidation. The ideal condition is just sufficient oxide to obscure metallic appearance.
- D. Examination under 10 to 15 Power Magnification shall show freedom from a string or excessively large group of bubbles entrapped in the glass.
- E. Transformation Test:- Wrap seal in tissue paper and immerse in dry ice and Acetone mixture (-80° C.) for 30 minutes.
 - 1. Adequate paper wrapping is required to take care of difference in thermal conductivities of metal and glass.
 - 2. After the sample is allowed to cool to room temperature no cracks in the glass should be visible under 7 to 10-Power Magnification.

VI. Oxide Removal

- A. Vapor degrease in Trichlorethylene (if parts are greasy).
- B. Pickle
 - 1. 100% Hydrochloric Acid
 - 2. Heat to 80 to 85° C.
 - 3. Immerse parts for one minute (longer for more heavily oxidized seals).

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VI. Oxide Removal (Cont.)

C. Rinse

1. Running hot water - 30 seconds
2. Cold concentrated HCl - 5 to 10 seconds
3. Running cold water - 3 minutes
4. Rinse in distilled water
5. Dip in Acetone and air dry