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TiN High Temperature Diffusion Barrier for Copper-Gasketed Stainless-Steel Flanges*

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Klystrons manufactured at the Stanford Linear Accelerator Center are typically baked at temperatures of 550°C for times as long as 200 hours. During these long bakeouts the copper-gasketed type 304 stainless steel flange joints (7 in all) diffusion bond so intimately that the flanges can be separated only with a jacking fixture, and copper is left on the sealing surfaces. Removal of this copper necessitates the use of abrasive materials, which can result in contamination of the klystron body and compromise its re-use. We report on the use of 50Å and 150Å TiN thin films as a diffusion barrier between the Cu gasket and the ~~sta~~inless-steel flange.

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Among the couples separated by a TiN diffusion barrier have been: Pt-Ti[1], Cr-Ni[2], Al-Si[3,7], Pt-Al[4], Au-InP[5], Pd-Si[6,7], Cu-Si[6,7], Au-Si[6,7], and Si-Ag[7]. We have also recently shown a large improvement in the diffusion barrier properties of amorphous TiN films containing Sm[8]. The TiN films used here were deposited on Cu gaskets at room temperature by reactive magnetron sputtering from a Ti target using N_2 + Ar mixture (20% N_2 - 80% Ar). Three sets of flanges (two types of seal, "Conflat"[9] and "crushed seal" of SLAC design[10]) were tested. The cross-sections of all three are shown in Figure 1. The gasket coated with 150Å of TiN could not be sealed in the klystron window flanges (Figure 1a). This was attributed to the hardness of the TiN and the shallow steps on this design. Flanges 1b and 1c were sealed with 150Å of TiN using normal torque for these flanges (i.e. 15 ft-lb). Subsequent flanges and gaskets were coated with a 50Å film of TiN and all flanges (1a,1b and 1c) were sealed at the same torque as that for flanges with uncoated Cu gaskets. The flanges were then baked at 550°C in a vacuum of $\sim$1x 10^{-5}torr for up to 200 hours. The flanges were checked after the bakeout and found to be He leak tight to <2 x 10^{-10} std cc/sec. The flanges were then disengaged and the gaskets were removed without sticking.

Two four-inch-diameter, TiN-coated, crush-seal copper gaskets were removed from a klystron tube following installation and bakeout. These easily removed gaskets were examined in a scanning electron microscope. Using energy dispersive x-ray analysis, we examined the areas in the region of the seal for Ti concentration. All regions, except the steep shoulder in Figure 1b (regions A-1,A-2) created by the cutting stainless-steel edge, showed significant Ti (nitrogen is not detectable by our unit) remaining. Therefore, we have demonstrated that the 50Å thick TiN layer is an effective inter-diffusion barrier for stainless-steel-Cu couples and will be adopted for regular klystron production.

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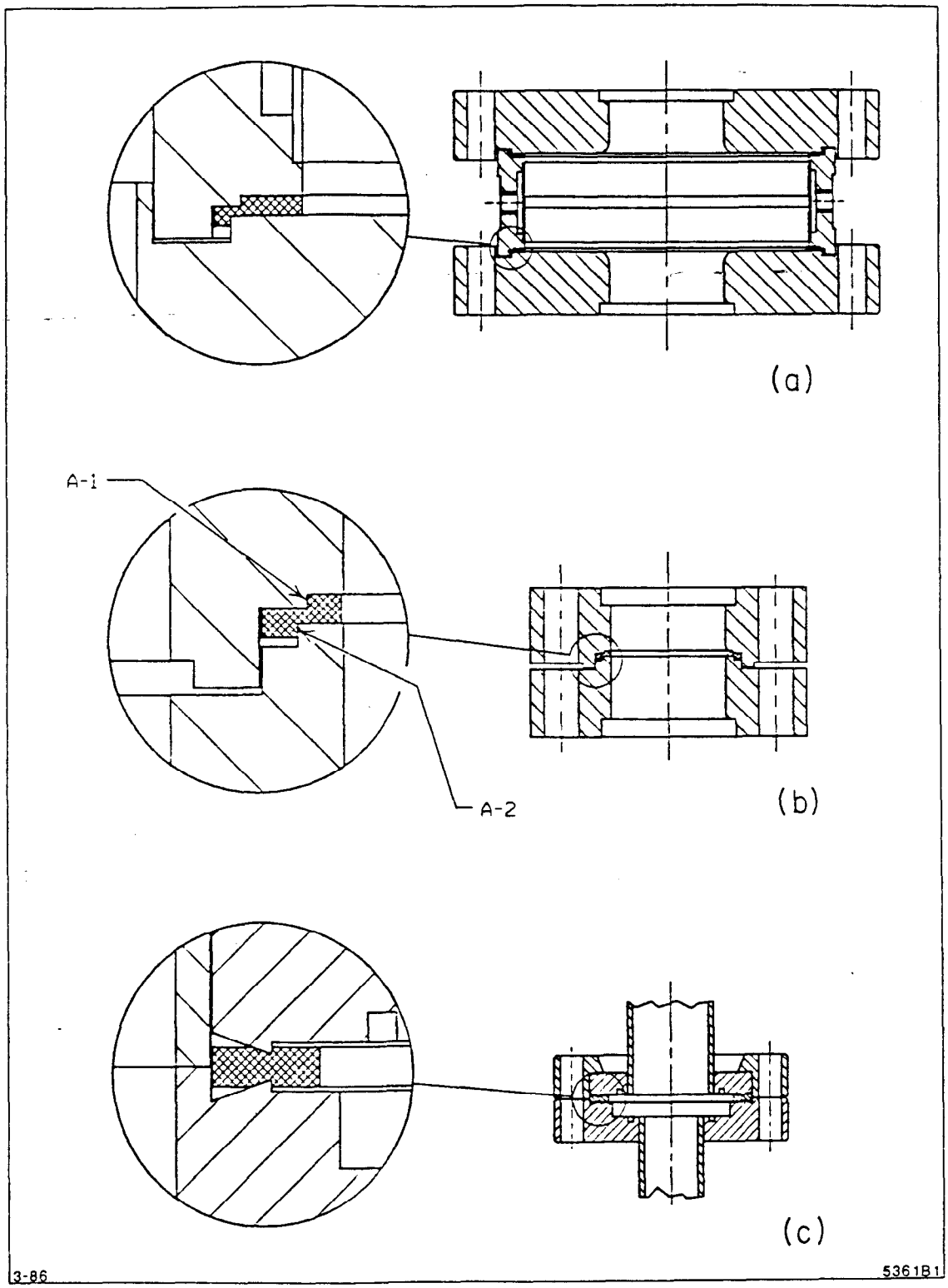

Figure 1. Cross sections of three types of copper-gasketed seals, showing gasket deformation details: 1a) Circular klystron window crush seal flange (10 cm O.D., 8.4 cm I.D. and 3.1 cm thick), 1b) Rectangular waveguide crushed seal flange (12 x 8.2 outside, 7.2 x 3.5 cm inside and 2 cm thick) and, 1c) Conflat[9] sexless flange seal (6.9 cm O.D., 3.5 cm I.D. and 1.25 cm thick).