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TECHNICAL REPORT 4498

THE LOCATION OF ADHESIVE BOND FAILURE  
BY  
USING RADIOACTIVE TRACERS



C. A. L. WESTERDAHL  
J. R. HALL

MARCH 1973

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PICATINNY ARSENAL  
DOVER, NEW JERSEY

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## OBJECT

The object of this program is to develop a technique for accurately determining the location of failure in an adhesive bonded joint by application of radioactive tracer methods.

## SUMMARY

The adsorption of several radioactive ions on aluminum surfaces during the surface treatment of coupons for adhesive bonding was studied. These were  $^{51}\text{Cr}^{+7}$  in the FPL chromic-sulfuric acid etch, and  $^{45}\text{Ca}^{+2}$ ,  $^{54}\text{Mn}^{+2}$ ,  $^{59}\text{Fe}^{+3}$  ions in the rinse water. Calcium was the most strongly adsorbed ion; manganous and ferric ions showed less than 1% monolayer coverage.

$^{14}\text{CH}_3$ -Bisphenol A diglycidyl ether was synthesized from labeled bisphenol A and epichlorohydrin, and used in a room temperature curing adhesive to bond 2024 T-3 aluminum lap shear specimens. Autoradiography was performed on coupons with very thin layers of adhesive remaining after tensile failure of the bonded specimens.

## INTRODUCTION

The objective of this work was twofold. The first was to explore methods for determining the location of bond failure in an adhesive joint tested to failure, e.g. cohesive in the adhesive, cohesive in the substrate, or at the adhesive/substrate interface (adhesive failure).

The second objective was to determine whether the effect of certain ions present in the etching bath or rinse water in the surface preparation of aluminum could be correlated with adsorption on the treated metal surface. This question is of interest because of the demonstrated effectiveness of dichromate in the etching bath, and the effect of divalent cations such as calcium in preventing the formation of weak oxide layers when an etched coupon is soaked in hot rinse water.

The location of adhesive bond failure is of interest to both theoreticians and engineers, engineers in order to find ways of improving bond strength or durability, and theoreticians for the value of such information for understanding of the phenomenon of adhesion and the nature of interfaces.

Bond failure may occur in either the substrate or adhesive, or may appear to be at the interface. For example, poor surface preparation may result in a weak metal oxide layer or permit a film of contaminants to which the adhesive cannot bond. Poor surface preparation is one aspect of the problem. Of perhaps greater significance is the question of locus of failure in an adhesive joint which has been prepared in accordance with good engineering practice.

The most obvious way of finding where a bond failure has occurred is visual examination. Adhesives of a contrasting color or high opacity make thin layers or patches of adhesive easier to detect. Many adhesives are naturally fluorescent or can be made so by addition of a phosphor; examination under ultraviolet light is very effective for these.

Optical microscopy may be used to extend the useful range of this method; however, a very thin layer approaching a monolayer in thickness would escape detection by this method.

A straight-forward method of detecting low concentrations of a substance is by use of radioactive tracers. By the proper choice of an isotope, e.g., an alpha emitter, single atoms may be detected with high efficiencies. For epoxy adhesives, the most logical choice was  $^{14}\text{C}$ , a beta emitter. The detection method was autoradiography using X-ray film. Although autoradiography of  $^{14}\text{C}$  is not as efficient as for higher energy emitters, long exposure time may be used to detect very small amounts of labeled material. Quantitative measurements may be made by use of a densitometer on exposed film. The location of the activity is, of course, easily seen.

The fate of elements present as ionic species in etching baths and rinses used in the surface treatment of aluminum can also be followed with radioactive tracers. Suitable radioactive isotopes of elements of interest, as the designed ionic species, were introduced into the treatment solutions or rinse solutions. Isotopes used were calcium ( $^{45}\text{Ca}$ ), iron ( $^{59}\text{Fe}$ ), manganese ( $^{54}\text{Mn}$ ), and chromium ( $^{51}\text{Cr}$ ). Only one isotope was used in any single experiment. Since the determination of the quantity of tagged substance present, not its distribution, was the primary object, scintillation or Geiger counters were used for detection because of their rapidity of measurement compared to autoradiography.

## RESULTS

The introduction of radioactive isotopes of elements found in surface-treating solutions into these solutions provided a method of following the fate of these elements. It was found that although  $^{51}\text{Cr}$ , present in solution in the form  $\text{Cr}_2\text{O}_7^{=}$ , could be detected on the aluminum surface after the dichromate etching solution had been washed off, the method was not particularly accurate. The results indicated at least 1% monomolecular coverage calculated as  $\text{Cr}_2\text{O}_3$  (Table II). Iron, calcium and manganese, elements present in the tap water rinse, remained on the aluminum surface after a deionized water rinse and were easily detected. Carbon-14 tagged epoxy provided a means of detecting amounts of adhesive too small to be seen with the unaided eye. X-ray films exposed to the halves of the broken joints clearly showed the locations of small amounts of adhesive.

## DISCUSSION

This work can be divided into two parts: the use of metal ion tracers in the surface preparation solutions and the use of a radioactive adhesive to detect very thin layers of adhesive.

The first part grew out of previous work done in the laboratory.<sup>2</sup> That work showed that the composition of the water used in the hot water rinse of aluminum coupons after chromic-sulfuric acid treatment affected the bond strength of adhesive joints made with the treated coupons. The acid treatment consists of the following steps:

(1) vapor degrease or solvent wipe the aluminum coupons (2) etch in dichromate solution (3) rinse in running water to remove the acid (4) soak in deionized warm water to complete the rinse. It was found that soaking the aluminum in hot deionized water for 5 minutes or longer would cause the formation of a weak oxide layer on the aluminum, resulting in low strength of bonded joints. If tap water instead of deionized water was used, a strong bond could be formed. An obvious difference between the two types of water was the presence of salts in the tap water. Addition of calcium salts or salts of other divalent ions to deionized water gave high bond strength also. An analysis of Picatinny tap water showed the presence of calcium, iron and manganese ions (Table I). Since suitable isotopes of these elements,  $^{45}\text{Ca}$ ,  $^{59}\text{Fe}$  and  $^{54}\text{Mn}$ , were available, they were used in the rinse water of the test specimens to see if these elements were incorporated into the surface layer of the aluminum. Experiments were made using only one isotope at a time. The panels were treated in the standard way with FPL etch, allowed to stand in warm "tagged" tap water, and rinsed with room temperature deionized water to remove excess radioactive solution. After the panels were dried they were examined for radioactivity. A substantial amount of the radioactivity which was introduced to the aluminum surface by the tap water rinse remained after the deionized water rinse. The activity of  $^{54}\text{Mn}$ -treated specimens was well above the background and in one case implied about 1% of a monolayer coverage assuming a cubic structure for  $\text{MnO}$ . The  $^{59}\text{Fe}$  readings

also correspond to 1% coverage. Because of experimental difficulties these figures are only approximate.

The radioactive panels were then bonded to other panels which had been treated in an identical manner using non-active solutions. The tensile test specimens so formed were then broken and the two halves examined, particularly for the presence of radioactivity on the coupon which had not been treated with tagged solutions. The presence of activity implied that some of the surface layer on one side had broken from the adherend and had transferred with the adhesive to the other side. The  $^{54}\text{Mn}$  and  $^{59}\text{Fe}$  readings were well above background with up to 10% of the activity transferred to the "inactive" coupon. The  $^{45}\text{Ca}$  readings by liquid scintillation corresponded to coverage by one to seven monolayers. This transfer of activity implies that at least part of the bond failure occurred within the surface layer of the aluminum or between it and the body of the aluminum.

Radioactive  $^{51}\text{Cr}$  in the form of  $\text{Na}_2\text{Cr}_2\text{O}_7$  was used in the dichromate etch. This was done to elucidate the role of the  $\text{Cr}_2\text{O}_7^{=}$  ion in the preparation of the aluminum surface. One theory<sup>3</sup> holds that the presence of unfilled d shells in chromium gives it a unique role as an intermediate between the aluminum surface and the adhesive. This role would require the presence of a substantial portion of a monolayer of chromium on the aluminum. Other work, including X-ray diffraction studies done for this group, has failed to detect the presence of large amounts of chromium on the treated surface. Since

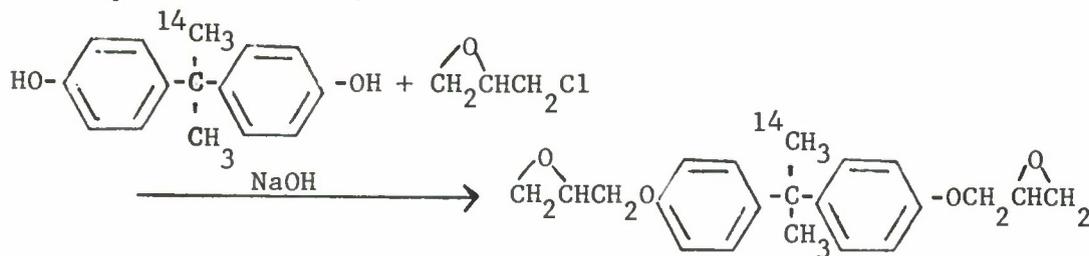
radioactive tracers provide a particularly sensitive method of detection, it was felt that a tracer could be used to define the role of chromium in aluminum surface treatment. Unfortunately the results were inconclusive. A straightforward calculation from the counting data shows about 1% coverage. However this figure involves large multiplicative factors and a counting rate only slightly above background. Thus this figure is not very reliable.

The second part of this work used a radioactive epoxy synthesized from bisphenol A containing  $^{14}\text{C}$  in the methyl group. Among the isotopes of carbon and hydrogen,  $^{14}\text{C}$  has the most convenient half life (5730 years) and energy. It is not really ideal for autoradiography since it decays with the emission of a rather low energy (0.156 Mev) beta minus. Beta particles from a nuclear decay are not mono-energetic and their detection efficiencies are lower than those of alpha particles. However much autoradiography has been done using  $^{14}\text{C}$ , particularly with biological samples and many experimental techniques are well developed.

#### $^{14}\text{CH}_3$ Labeled Bisphenol A Diglycidyl Ether

Bisphenol A diglycidyl ether, labeled with  $^{14}\text{C}$  at the methyl group was prepared from labeled bisphenol A and epichlorohydrin by the procedure listed by Sorensen and Campbell<sup>4</sup>, except that 40% sodium hydroxide solution was used<sup>5</sup> for convenience instead of the solid

pellets specified. The procedure



was similar to the usual macro scale laboratory synthesis of epoxy resin. However, modification and several dry runs were necessary to adapt the method to the small scale required.

The adhesive made from epichlorohydrin and the labelled bisphenol A was used to bond aluminum coupons. These coupons were divided into two groups prior to bonding. The first group, after the acid etch, was soaked in warm tap water followed by a quick deionized water rinse; the other group was soaked in warm deionized water. The specimens formed by bonding the two types together were broken in a tensile test and the distribution of adhesive was investigated by autoradiography. On some of the coupons an amount of adhesive too thin to be seen with the naked eye (Fig. 1) could be easily detected on the aluminum by a two-hour exposure of the X-ray film (Fig. 2). These results show that some failures which appear to be interfacial are actually cohesive in the adhesive. This method is quite sensitive as the exposure can be very long, the length being limited only by the background fogging of the photographic film. The image can be examined with a microscope to determine the location of small amounts of adhesive exactly. Here the limitation is the graininess of the film. This method can be very precise when used with care.

## EXPERIMENTAL

### Materials

Coupons for adhesive bonding were 4" x 1" x 1/8" 2024 T3 bare or Alclad alloy cut from 1/8" sheet. Also used were 2024 T3 bare "finger" panels 1/16" thick; these consist of four 1/2"-wide parallel "fingers" joined at one end and near the middle by small necks of metal. These may be conveniently bonded in jigs designed for lap shear tensile specimens with 1/2" overlap; the finished panel is snapped apart into individual specimens with cutters.

Forest Products Laboratory (FPL) etch was prepared from 3000 ml. of deionized water, 545 ml of conc. reagent grade sulfuric acid, and 100 g. of technical grade sodium dichromate.

The radioactive tracers used were purchased from New England Nuclear Corporation in the following forms:

$^{51}\text{Cr}$  as sodium dichromate in saline solution, 247 mCi/mg., 1 mCi/ml, 99% radiometric purity (28 d half-life,  $\gamma$  emitter).

$^{59}\text{Fe}$  as  $\text{FeCl}_3$  in 1 N HCl, 18.6 mCi/mg, 2.37 mCi/ml, 99% radiometric purity (45 d half-life,  $\beta$  and  $\gamma$  emitter).

$^{45}\text{Ca}$  as  $\text{CaCl}_2$  in 0.5 N HCl, 14.0 mCi/mg, 10 mCi/ml,  $\approx$  99% radiometric purity (165 d half-life,  $\gamma$  emitter).

<sup>54</sup>Mn as MnCl<sub>2</sub> in 0.5N HCl, carrier free, 2 mCi/ml, 99% radio-metric purity, total solid ~ 0.2 mg/ml (291 d halflife, γ emitter).

<sup>14</sup>C labeled bisphenol A was purchased from Mallinckrodt Nuclear and is described in a later section of the experimental (5770 y halflife, soft β emitter).

#### Surface Treatment of Aluminum, General Procedure

The aluminum sheet was first wiped with acetone to remove grease and dirt. It was then immersed for ten minutes in FPL etching solution at 60°C. The aluminum was removed to a container of water at 40°C. When no activity was used in the treating solutions this container was placed under the running tap; otherwise there was a quick rinse with inactive tap water from a squeeze bottle and the aluminum was then placed in the container of water. It was left in this water for about 5 minutes, then removed and given a quick rinse with deionized water from a squeeze bottle. It was placed in a holder to dry and left there for about 24 hours with a cover over it which allowed air circulation but protected the aluminum surface from airborne contaminants.

The coupons were initially checked for the presence of activity with the survey meter. This meter was also used for safety purposes such as checking hands and any other object which might have come into contact with radioactivity. The isotopes used in the surface treatment were monitored with a NaI(Tl) crystal attached to a 1000 channel analyzer. With this analyzer it is possible to discriminate among the activity associated with several isotopes. The coupons were placed in a polyethylene envelope and laid on top of the crystal. The gamma rays of

the  $^{51}\text{Cr}$ ,  $^{59}\text{Fe}$  and  $^{54}\text{Mn}$  can easily penetrate the plastic. The 0.26 Mev beta minus of the  $^{45}\text{Ca}$  was not too badly attenuated by the envelope, although better results were obtained by using a thin window counter.

#### Treatment of Alclad Coupons with $^{51}\text{Cr}$ -labeled Dichromate-Sulfuric Acid Etch

Seven 4 x 1 x 1/8" 2024 T3 Alclad coupons were treated at 60 - 70°C for 10 minutes in approximately 80 mL of FPL etch containing 100  $\mu\text{l}$  of  $^{51}\text{Cr}$  labeled sodium dichromate (total activity approximately 0.1 mCi); the treatment was carried out by immersing one end of the coupons to a depth of about one inch in the radioactive etch in a 250-ml. beaker. The coupons were removed, placed in a beaker containing tap water at 40°, and then rinsed with tap water at 40° from a wash bottle (this procedure was followed to minimize splashing of highly active etch solution). The coupons were then rinsed in a second beaker of tap water at 40° (no yellow dichromate color was visible), rinsed with deionized water from a wash bottle and placed in a rack to dry overnight.

Radioactivity remaining on the coupons was counted the following day using a "jelly" detector at a distance of approximately 1/2 inch. The counting rate was about 6 counts/minute, only slightly above background.

In a second experiment 2 Alclad coupons were treated by the same method, using 15 ml. FPL etch containing 200  $\mu\text{l}$  of  $\text{Na}_2^{51}\text{Cr}_2\text{O}_7$  solution (total activity, 0.2 mCi). After the final rinse, the coupons were dried overnight in air. The counting rate was still very low even with this level of activity in the solution.

Two 4" x 1" x 1/8" 2024 T3 Alclad coupons were treated in 15 ml FPL etch containing 100  $\mu\text{l}$   $\text{Na}_2^{51}\text{Cr}_2\text{O}_7$  solution, at 65° for 10 minutes. The excess active FPL etch was removed from one coupon by draining and carefully blotting the liquid at the bottom edge of the coupon. The other coupon was immersed in a tap water rinse (75 ml) at 40° for four minutes and air dried.

An additional 50  $\mu\text{l}$  of  $\text{Na}_2^{51}\text{Cr}_2\text{O}_7$  solution was added to the FPL etch from above and two 2024 T3 bare coupons were etched at 65° for 10 minutes. One coupon was drained and allowed to dry without rinsing. The second was immersed for 5 minutes in a beaker containing tap water at 40°.

The unrinsed coupon was counted with a 4-inch NaI crystal; no correction was made for background radiation. The activity was 3000 c/m. The rinsed coupon showed no activity above background.

#### Surface Treatment of Bare Aluminum Coupons - $^{59}\text{Fe}$ Rinse

Two 2024 T3 bare coupons were treated in FPL etch for 10 minutes at 60°, rinsed with 40° tap water from a wash bottle, and immersed for 5 minutes in tap water (30 ml) containing 10  $\mu\text{l}$   $^{59}\text{FeCl}_3$  solution (total activity  $\sim$  0.024 mCi). One coupon was allowed to drain, and dry; the second was rinsed with deionized water from a wash bottle, and allowed to dry.

The rinsed specimen gave 25,600 c/m ( $\gamma$ ).

Surface Treatment of Bare Aluminum Coupons - <sup>45</sup>Ca and <sup>54</sup>Mn Rinse

Four 4 x 1 x 1/8" 2024 T3 bare coupons were treated by immersion of one end in FPL etch at 65° for 10 minutes. The dichromate solution was rinsed off with tap water at 40° from a wash bottle. The coupons were then soaked in tap water containing radioactive tracers as follows:

Two coupons were placed in tap water containing 10 μl of <sup>45</sup>Ca labeled CaCl<sub>2</sub> (total activity ~ 0.1 mc) at 40° for 5 minutes. One was rinsed with deionized water and allowed to dry; the other was allowed to drain and was then blotted to remove the "active" water. The unrinsed coupon gave 3000 c/m, rinsed 570 c/m.

A similar procedure was followed using 10 μl of <sup>54</sup>MnCl<sub>2</sub>. The rinsed coupon gave 75,000 c/m.

Bonding of Finger Panels - Radioactive Tracers in Rinse

Bonding experiments were performed, using finger panels which had been treated with FPL etch and immersed in warm rinse water containing various radioactive metal cations. Bare 2024 T3 finger panels were cut in half to give pairs of 3" x 1/2" x 1/16" coupons. A total of 16 pairs were treated in FPL etch at 60° for 10 minutes and the dichromate solution was rinsed off with warm tap water from a wash bottle.

Two half-panels were immersed in tap water containing <sup>45</sup>Ca (solution from previous experiment, total activity 0.1 mCi; ~15 ml) at 40° for 5 minutes. One pair was rinsed with deionized water from a wash bottle and allowed to dry; the other pair was allowed to drain onto a Kimwipe and dry.

Two half-panels were exposed to the  $^{54}\text{Mn}$  solution used in the previous experiment (0.02 mCi total activity,  $\sim$ 15 ml) following the same procedure.

Panels were bonded with 70/30 pbw Epon 828 epoxy resin and Versamid 140 polyamide curing agent with no filler. Specimens were assembled using a radioactive panel together with an inactive panel, weighted lightly and allowed to cure at room temperature for 1 week, cut into individual specimens with tin snips, and tested at 600 lb/min loading rate (1" in grips). Bond strengths varied between about 1700 and 2200 psi, and are shown in Table III.

A second group of specimens were prepared in the same manner. Bond strengths were in the range of 1700 to 2300 psi.

The "active" coupons from the failed joints showed very high activity. The "inactive" coupons showed much less radioactivity but significantly above background.

## <sup>14</sup>C-Labeled Epoxy Adhesive

The preparation of <sup>14</sup>CH<sub>3</sub>-bisphenol A diglycidyl ether was carried out in a radiochemical hood with a good draft, using standard radiochemical safety precautions. A Victoreen Thyac II survey meter (Model 489), equipped with a thin window Geiger Müller detector was used to monitor spills.

The reaction was carried out in a 3-necked 20-ml round-bottomed flask equipped with a stirrer, a 10-ml dropping funnel, and a Claisen adapter equipped with an Adjustatherm thermometer and a condenser protected with a drying tube filled with Drierite. The stirrer bearing was lubricated with Celloseal stopcock grease, all other ground glass joints were fitted with Teflon sleeves to avoid introduction of grease.

<sup>14</sup>CH<sub>3</sub>-Labeled bisphenol A (Mallinckrodt Nuclear, lot #3062), 2.0 mCi, 1.94 mCi/mM, 0.2357 g and unlabeled bisphenol A (recrystallized twice from 90:10 benzene-dioxane), 2.0475 g., combined weight 2.2832 g., 10.001 mM, were placed in the reaction flask and washed in with 8.0 ml (9.43 g. 102 mM) of redistilled epichlorohydrin. Sodium hydroxide solution, 40%, 2.05 g., 20.5 mM, was weighed into the dropping funnel and 2 additional drops were added to compensate for solution remaining on the funnel walls.

The initial portion of 0.3 - 0.4 ml of NaOH solution was added, the mixture was heated to 95<sup>o</sup>, and the remainder of the NaOH was added gradually. The reaction temperature was kept at 80 - 95<sup>o</sup> during the addition, and then raised to refluxing temperature. The reaction mixture was allowed to cool, and the condenser was replaced by a 105<sup>o</sup> angle connecting tube fitted with a vacuum adapter and a 25 ml

receiving flask. The receiver was cooled in ice, and evacuated with a water aspirator connected through a dry ice trap. The excess epichlorohydrin was removed by distillation at approximately 50 mm pressure, and the residue was heated to 150° until no more volatile material distilled.

Five ml of toluene was added to the viscous reaction mixture, and the white precipitate of NaCl was removed by filtration through a medium fritted glass funnel; the walls of the flask were rinsed with small amounts of solvent. The precipitate was washed with small portions of toluene and the combined filtrate was transferred to a small Claisen distillation apparatus equipped with a 25 ml. pear-shaped distillation flask and a glass capillary. Two Teflon boiling chips were added and toluene was removed by distillation, first at atmospheric pressure until the pot reached about 125 to 130°; distillation was continued by cautiously applying aspirator vacuum until no more volatile material came over.

The light-colored viscous residue was transferred to a tared vial while still warm, the flask was clamped and warmed occasionally to allow as complete drainage as possible. The <sup>14</sup>C - labeled epoxy resin weighed 3.2165 g., corresponding to 95% recovered yield of product.

Several films were used in the autoradiography work: DuPont NDT 75, Kodak RD single side, and Kodak AAO. X-ray film is characterized by a rather large silver grain size and by having emulsion coated on both sides of the backing film. Although both of these factors increase its sensitivity they tend to produce an indistinct image. This is true of the double coating because of the geometric factor. In order to get a good clear image it is necessary to get good contact between the active source and the film.

If better contact is necessary, as in the case of rough surfaces, it is possible to paint on a gel containing the photographic emulsion. This is then developed in place. Although this procedure was not deemed necessary in this case it was nevertheless necessary to remove any bead of adhesive remaining on the coupon which would make it difficult to get close contact between the coupon and the film. These beads also produced intense blackening in their vicinity and tended to mask other nearby weaker sources. After their removal much clearer images were obtained. It is possible to obtain a fairly good image on a fast film after a 2-hour exposure but it was decided that an overnight exposure with a single coated fine grain film would give the best overall results.

#### CONCLUSIONS

Radioisotopes offer definite promise for the study of adhesive bonding. By their use trace amounts of elements can be followed through a complicated sequence of events. The adsorption of less-than-monolayer quantities of divalent cations on aluminum coupons during the surface preparation was readily detected and measured; the fate of these "tagged" ions in the failed adhesive joint could be followed. Radioactive chromium ( $^{51}\text{Cr}$ ) as dichromate in the chromic acid etch showed little incorporation into the treated aluminum surface. Autoradiography of failed specimens bonded with  $^{14}\text{C}$  - labeled epoxy adhesive readily detected very thin layers of adhesive.

## RECOMMENDATIONS

It is recommended that application of radioactive tracer techniques to adhesion problems be continued and extended.

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TABLE I

## TYPICAL ANALYSIS OF PICATINNY TAP WATER (in ppm)

	<u>Well 129</u>	<u>Well 130</u>
Mn	0.71	0.69
Fe	0.4	0.18
Ca (hardness)	164	88
CO <sub>3</sub> <sup>=</sup>	6.0	2.0
NO <sub>3</sub> <sup>-</sup>	0.02	---
Alkalinity	165	105
Cl <sup>-</sup>	166	17
	<hr/>	<hr/>
Total Hardness	282	144

Water from well 129 and well 130 is sampled annually at the well head and sent for analysis to the U. S. Bureau of Mines at the University of Maryland, College Park, Maryland. Water from both wells is mixed in varying proportions and supplied to Arsenal mains. Actual hardness at the taps will therefore vary between 144 and 282 ppm.

TABLE II

## COUNTING RESULTS

<u>Isotope</u>	<u>Corrected<sup>a</sup> Counting Rate/in<sup>2</sup></u>	<u>No. of Active<sup>b</sup> Atoms on Specimen</u>	<u>No. of Inactive<sup>c</sup> Atoms on Specimen</u>	<u>% Monomolecular<sup>d</sup> Coverage</u>	
<sup>54</sup> Mn	<sup>e</sup> 82,500	5.35 x 10 <sup>10</sup>	4.28 x 10 <sup>14</sup>	4.2	
	<sup>f</sup> 1,560	1.01 x 10 <sup>9</sup>	8.11 x 10 <sup>12</sup>	7.9 x 10 <sup>-1</sup>	
	<sup>f</sup> 1,750	1.14 x 10 <sup>9</sup>	9.09 x 10 <sup>12</sup>	8.8 x 10 <sup>-1</sup>	
	<sup>g</sup> 176	1.14 x 10 <sup>8</sup>	9.13 x 10 <sup>11</sup>	8.9 x 10 <sup>-2</sup>	
	<sup>g</sup> 204	1.32 x 10 <sup>8</sup>	1.06 x 10 <sup>12</sup>	1.03 x 10 <sup>-1</sup>	
	<sup>g</sup> 68	4.4 x 10 <sup>7</sup>	3.5 x 10 <sup>11</sup>	3.4 x 10 <sup>-2</sup>	
	<sup>g</sup> 40	2.6 x 10 <sup>7</sup>	2.1 x 10 <sup>11</sup>	2.0 x 10 <sup>-2</sup>	
	<sup>g</sup> 32	2.1 x 10 <sup>7</sup>	1.7 x 10 <sup>11</sup>	1.6 x 10 <sup>-2</sup>	
	<sup>g</sup> 24	1.6 x 10 <sup>7</sup>	1.2 x 10 <sup>11</sup>	1.2 x 10 <sup>-2</sup>	
	<sup>g</sup> 52	3.4 x 10 <sup>7</sup>	2.7 x 10 <sup>11</sup>	2.6 x 10 <sup>-2</sup>	
	<sup>59</sup> Fe	<sup>e</sup> 51,200	4.79 x 10 <sup>9</sup>	8.72 x 10 <sup>13</sup>	8.2
		<sup>f</sup> 5,900	5.51 x 10 <sup>8</sup>	1.00 x 10 <sup>13</sup>	0.94
<sup>f</sup> 5,490		5.13 x 10 <sup>8</sup>	9.3 x 10 <sup>12</sup>	0.87	
<sup>g</sup> 180		1.7 x 10 <sup>7</sup>	3.1 x 10 <sup>11</sup>	2.9 x 10 <sup>-2</sup>	
<sup>g</sup> 620		5.8 x 10 <sup>7</sup>	1.05 x 10 <sup>12</sup>	9.8 x 10 <sup>-2</sup>	
<sup>g</sup> 850		8.0 x 10 <sup>7</sup>	1.5 x 10 <sup>12</sup>	0.14	
<sup>g</sup> 650		6.1 x 10 <sup>7</sup>	1.1 x 10 <sup>12</sup>	0.10	
<sup>g</sup> 690		6.4 x 10 <sup>7</sup>	1.2 x 10 <sup>12</sup>	0.11	
<sup>51</sup> Cr	<sup>f</sup> 72	4.2 x 10 <sup>6</sup>	2.1 x 10 <sup>14</sup>	6.1	

TABLE II (Cont'd.)

## COUNTING RESULTS

Isotope	Corrected <sup>a</sup> Counting Rate/in <sup>2</sup>	No. of Active <sup>b</sup> Atoms on Specimen	No. of Inactive <sup>c</sup> Atoms on Specimen	% Monomolecular <sup>d</sup> Coverage
<sup>45</sup> Ca	$2.459 \times 10^4$	$1.501 \times 10^{10}$	$4.56 \times 10^{15}$	528
	$3.346 \times 10^4$	$2.043 \times 10^{10}$	$6.19 \times 10^{15}$	703
	$2.564 \times 10^4$	$1.565 \times 10^{10}$	$4.75 \times 10^{15}$	540
	$2.944 \times 10^4$	$1.798 \times 10^{10}$	$5.45 \times 10^{15}$	620
	$1.971 \times 10^4$	$1.204 \times 10^{10}$	$3.65 \times 10^{15}$	414
	$1.051 \times 10^4$	$6.418 \times 10^9$	$1.94 \times 10^{15}$	220
	$4.79 \times 10^3$	$2.926 \times 10^9$	$8.8 \times 10^{14}$	100
	$1.436 \times 10^4$	$8.768 \times 10^9$	$2.66 \times 10^{15}$	302

a. Background subtracted and remainder corrected for decay to time of

shipment  $N = N_0 \exp\left(-\frac{t \ln 2}{t_{1/2}}\right)$  and  $\frac{dN}{dt} = \frac{\ln 2}{t_{1/2}} N$ , where  $\frac{dN}{dt}$

and  $\left(\frac{dN_0}{dt}\right)$  are the counting rate at time  $t$  and time of shipment ( $t_0$ )

respectively.

b.  $\frac{dN_0}{dt} = \text{Eff} \frac{\ln 2}{t_{1/2}} N_0$ , no correction for counting efficiency except

for <sup>45</sup>Ca where (Eff = efficiency for counting <sup>14</sup>C =  $\frac{27040}{48760}$ )

TABLE II (Cont'd.)

c. From fraction of tracer placed into solution

$$mCi = 3.7 \times 10^7 \frac{d}{sec} \times 60 \frac{sec}{min}; \quad \left( \frac{dN_o}{dt_o} \right) = \frac{\ln 2}{t_{\frac{1}{2}}} N_o \text{ times specific}$$

activity of isotope solution  $\left( \frac{mCi}{ml} \right)$ , which gives number of active atoms

in solution. The number of inactive atoms was calculated from Table I.

The ratio in solution was assumed to be the same as that on the specimen.

d.  $r(O^{\circ}) = 1.40\text{\AA}$

$r(Mn^{++}) = 0.80\text{\AA}$

$r(Fe^{++}) = 0.76\text{\AA}$

$r(Ca^{++}) = 0.99\text{\AA}$

$r(Cr^{+++}) = 0.69\text{\AA}$

The divalent cations and  $O^{\circ}$

assumed to be arranged in a square array.

The  $Cr_2O_3$  was assumed to be

arranged in a hexagon.

Linus Pauling "The Nature of the Chemical Bond" Cornell University Press,  
New York, N. Y., 1970

e. Not rinsed

f. Rinsed but not bonded

g. "Inactive" sides of adhesive bonded specimens.

TABLE III

RADIOACTIVITY AND BOND STRENGTH, FINGER PANEL COUPONS  
TREATED WITH LABELED TAP WATER

Isotope in Tap Water	Unrinsed		Rinsed with Deionized Water	
	Bond Strength psi	Activity* cps	Bond Strength psi	Activity* cps
None	2253 $\pm$ 140	--	--	--
$^{45}\text{Ca}$	2174 $\pm$ 58	--	2228 $\pm$ 140	--
$^{59}\text{Fe}$	1796 $\pm$ 92	3	1968 $\pm$ 124	10
$^{54}\text{Mn}$	2132 $\pm$ 244	43, 28	1696 $\pm$ 128	36, 41

\*"Inactive" coupon from failed joint

TABLE IV

BOND STRENGTH, FINGER PANEL COUPONS TREATED  
WITH LABELED TAP WATER

<u>Isotope in Tap Water</u>	<u>Bond Strength, psi</u>	
	<u>Unrinsed</u>	<u>Rinsed With Deionized Water</u>
None <sup>a</sup>	2253 $\pm$ 140	---
Ca <sup>b</sup>	2174 $\pm$ 58	2228 $\pm$ 140
Fe <sup>b</sup>	1796 $\pm$ 92	1968 $\pm$ 124
Mn <sup>b</sup>	2132 $\pm$ 244	1696 $\pm$ 128

<sup>a</sup> Average of 4 values

<sup>b</sup> Average of 2 values

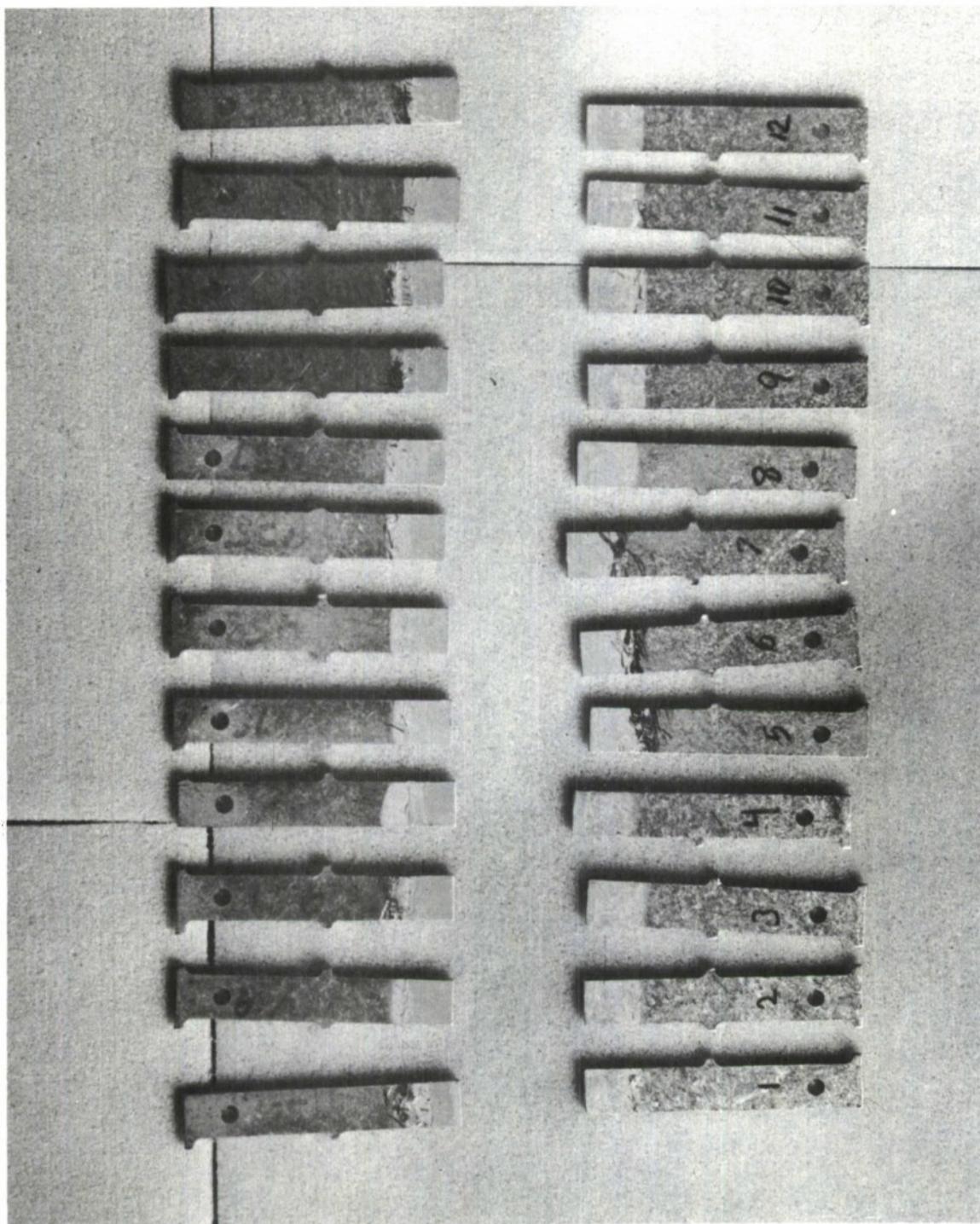


FIGURE 1

Finger panel specimens, bonded with  $^{14}\text{CH}_3$ -labeled epoxy adhesive, after testing to failure

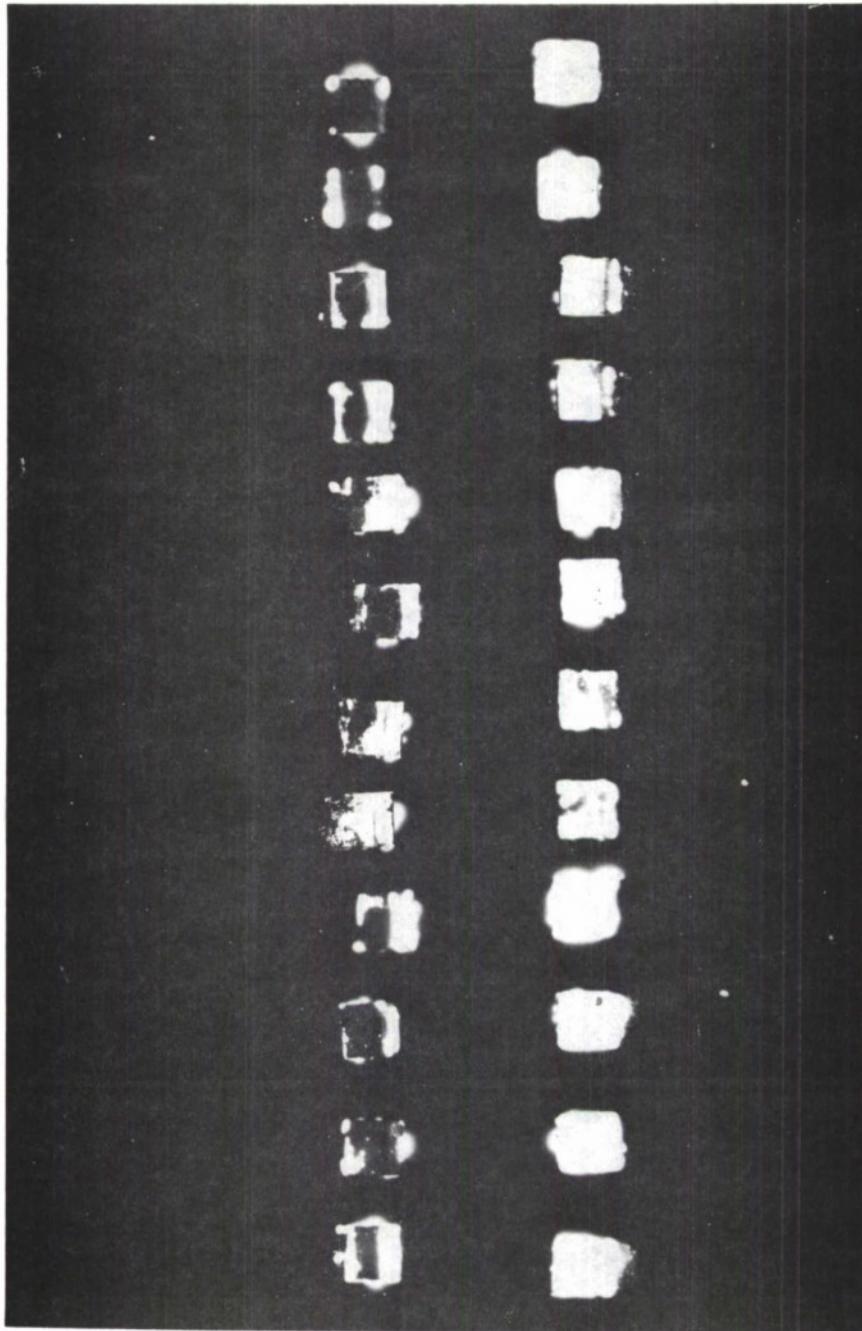


FIGURE 2

Autoradiograph of finger panel specimens, bonded  
with  $^{14}\text{CH}_3$ -labeled epoxy adhesive, after  
testing to failure (2 hour exposure)

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<p>The adsorption of several radioactive ions on aluminum surfaces during the surface treatment of coupons for adhesive bonding was studied. These were <math>^{51}_{Cr} +7</math> in the FPL chromic-sulfuric acid etch, and <math>^{45}_{Ca} +2</math>, <math>^{54}_{Mn} +2</math>, <math>^{50}_{Fe} +3</math> ions in the rinse water. Calcium was the most strongly adsorbed ion; manganous and ferric ions showed less than 1% monolayer coverage.</p> <p><math>^{14}_{CH_3}</math>-Bisphenol A diglycidyl ether was synthesized from labeled bisphenol A and epichlorohydrin, and used in a room temperature curing adhesive to bond 2024T-3 aluminum lap shear specimens. Autoradiography was performed on coupons with very thin layers of adhesive remaining after tensile failure of the bonded specimens.</p>			

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Adhesive bonding Surface preparation of aluminum Adhesive joints Autoradiography <sup>14</sup> C-Labeled epoxy, synthesis Radioactive tracer metal ions, adsorption on aluminum <sup>45</sup> Ca <sup>54</sup> Mn <sup>59</sup> Fe <sup>51</sup> Cr Chromic acid - sulfuric acid etch, <sup>51</sup> Cr-labeled Location of bond failure Adhesive failure Cohesive failure						