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ABSTRACT

This report documents the tests conducted to find a reliable surface preparation method of treating the CRBRP dip seal blade (SA508 Class 2 steel) to insure its sodium wettability at 450F or less.

Two techniques were established which depressed the sodium wetting temperature of SA 508, Class 2 dip seal blade material to 375F. These techniques were:

1. Depositing an $\sim 60 \times 10^{-6}$ inch layer of tin on the blade surface by a "brush-on" plating process, and,
2. by cleaning the blade surface with ultrasonics while it is immersed in sodium.

The tin plating technique is recommended as the initial and primary surface preparation method and ultrasonics as a rewetting and backup technique.

This work was conducted in support of the Sodium Dip Seal Feature Test, DRS 32.05.

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I. INTRODUCTION

Gas attenuation tests in the sodium dip seal test article indicated that the dip seal and/or trough surfaces were not wetted by 400F sodium. Consequently the transport rate of gases across the dip seal was excessive. It was theorized that the gases were transported across the dip seal by diffusion through a porous oxide film formed on the unwetted metal surfaces.

Increasing the temperature of the sodium in the dip seal to 600F for 165 hours effected the wetting of the dip seal surfaces and resulted in adequate impedance of gas transport across the seal. For the CRBRP dip seal, temperatures greater than 450F are not achievable and therefore, conditions necessary to depress the wetting temperature are required.

II. OBJECTIVE

The primary objective of this study was to find a surface preparation method that would promote sodium wetting of the CRBRP dip seal blade (SA 508, Class 2 low alloy steel) at temperatures of 450F or less.

The purpose was to develop a reliable method of treating the surfaces of the dip seal blade and trough so as to ensure their sodium wettability.

III. TEST ARTICLE DESCRIPTION

Test specimens consisted of two series of 1/4" x 1" x 2-1/4" steel coupons. Actual wetting experiments were conducted on series A coupons which had been cut and serialized from a block of SA 508, Class 2 low alloy steel. Material verification of the Series A coupons was obtained from AI Quality Assurance Laboratory. Appendix A contains the ASTM chemical and tensile requirements for SA 508 Class 2 steel.

Series B coupons were made from 1020 steel and were used primarily for system setup and checkout.

IV. TEST PROCEDURE

The wetting study consisted of tests on SA 508 Class 2 coupons that had been treated in various manners. The study was conducted per AI Test Procedure N707DTP810012 (Ref. 1) with the following modifications.

1. Only pure sodium (< 2 ppm O_2) was used in wetting tests. Barium and calcium additive tests were deleted, as were oxygen saturated sodium tests.
2. Refluxing trichlorethylene was not used as a surface cleaning method.
3. The use of ultrasonic agitation with tin plated specimens was added to the test matrix.

A literature search of the wetting behavior of metals provided many of the surface preparation techniques incorporated in the wetting study (Refs. 2 thru 11).

The following coupon preparation techniques were included in the wetting tests:

Coupon Surface Finish

a. As received, approximately 125 rms
b. Mechanically polished, approximately 30 rms
c. Sandblasted, 220 grit Al_3O_4
d. Tin plated

Coupon Surface Cleaning

a. Wiped with acetone
b. Degreased in refluxing acetone
c. Scrubbed with trisodium phosphate solution
d. Etched in aqua regia
e. Immersed in hot 50% NaOH
f. Ultrasonic abrasion under sodium

Initial wettability tests were conducted using sodium at 450F. Combinations of surface finish and cleaning methods that wet with 450F sodium were repeated with sodium at lower temperatures.

V. TEST RESULTS AND RECOMMENDATIONS

Results of the sodium wetting study are presented below. Tests were conducted per N707DTP810012 (Ref. 1) with the exceptions noted in Section IV of this report. Details of testing are recorded in AI Lab Notebooks N06028, N17025 and N17043.

A. GENERAL DISCUSSION

Wetting of individual coupons was measured as a function of the contact angle between the coupon surface and the sodium. In the simplest form, wetting has occurred when the angle between the sodium and metal is $< 90^{\circ}$ as illustrated in Figure 1. Initial tests were conducted using both the sessile drop and the immersion techniques to determine the advantages and disadvantages of each method.

The sessile drop technique amounts to placing a drop of hot sodium on a test coupon that is held at a constant specified temperature (Fig. 2). The degree of wetting is then determined by observing the contact angle between the sodium droplet and the coupon. The sessile drop technique for measuring wetting has several disadvantages. The inability to maintain a uniform temperature between the sodium droplet and the large exposed specimen surface is one, and another is that the hot coupon is exposed, and thereby vulnerable to surface oxidation.

In the immersion method the degree of wetting is determined by observing the contact angle of the sodium adhering to the surface of the partially submerged coupon. Figure 3 shows such a coupon that has not wetted and the depressed meniscus that is indicative of an unwetted condition. A coupon that has wetted will exhibit a raised meniscus, as illustrated in Figure 4. This method of measuring wetting permits accurate temperature control and prevents surface oxidation of the coupon. In addition, the immersion method more closely duplicates actual use conditions of the CRBRP submerged dip seal than does the sessile drop method.

Comparison of the sessile drop and immersion techniques in the initial tests easily showed the immersion method to be superior. Consequently, it was the method used in the wetting study tests.

B. TIN PLATING

The technique used to tin plate specimens in this study was a special "brush-on" process, perfected by Brooktronics inc., North Hollywood, California.

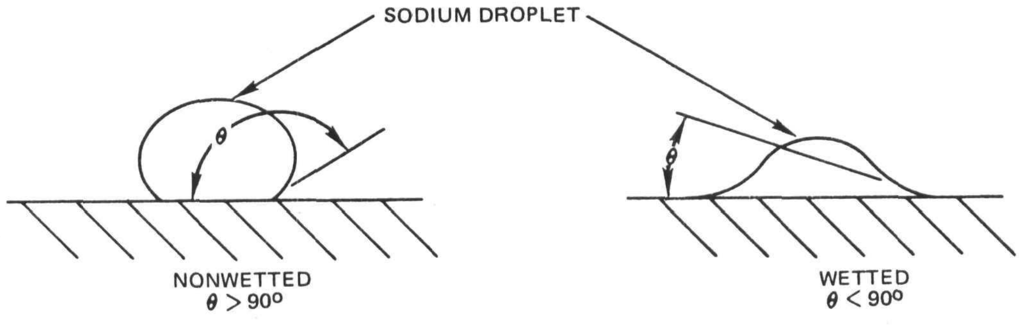

Figure 1. Contact Angles for Sodium Wetting

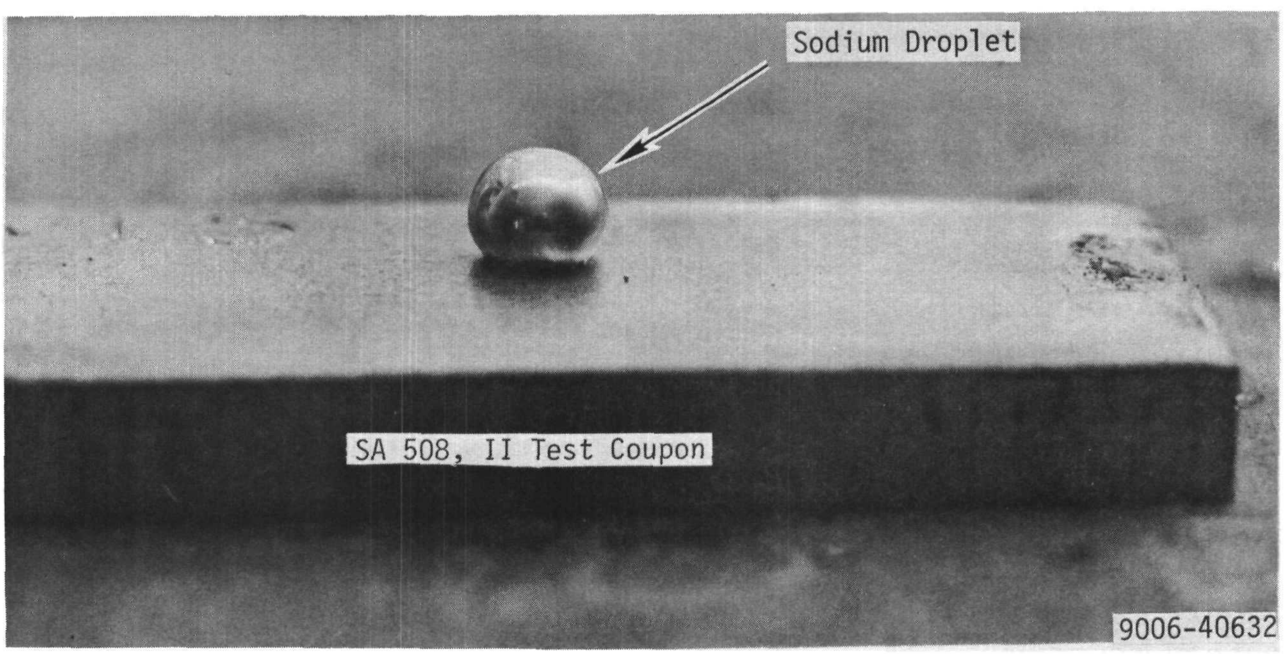

Figure 2. Unwetted Coupon, Sessile Drop Technique

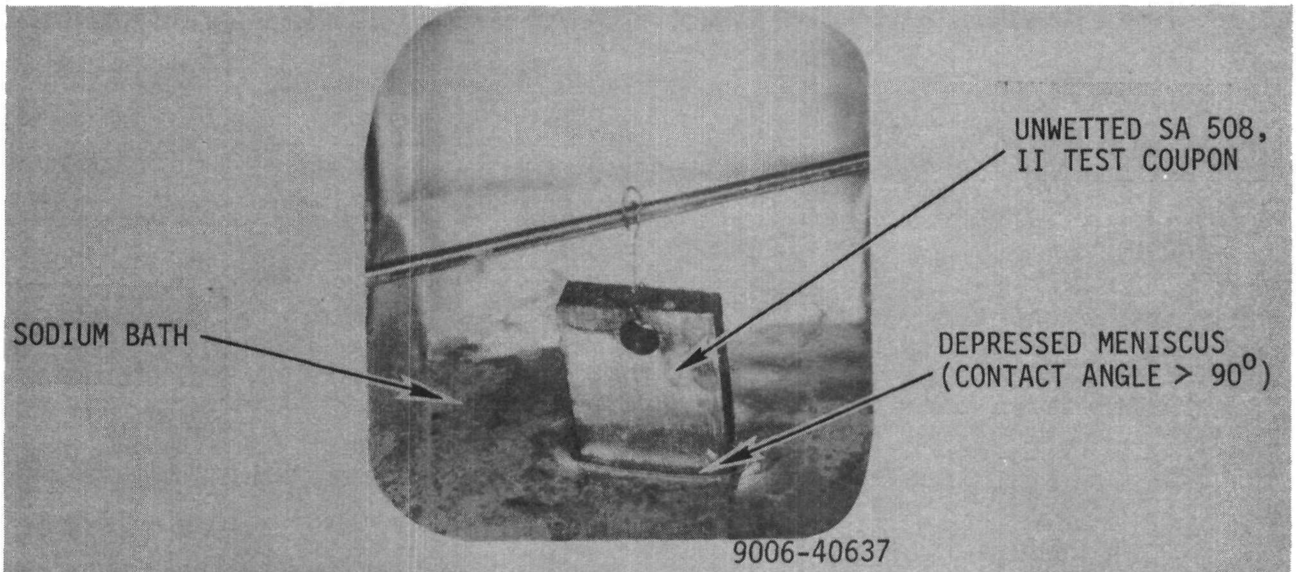

Figure 3. Unwetted Coupon, Immersion Method

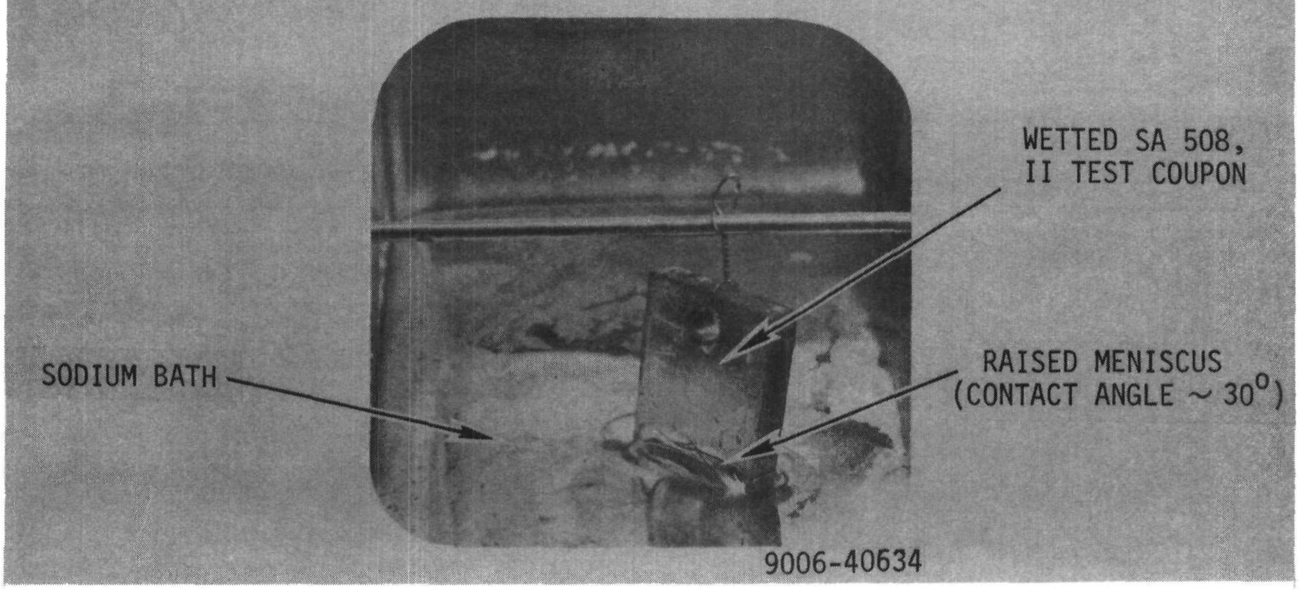

Figure 4. Wetted Coupon, Immersion Method

In this process the article to be plated is first cleaned by grit blasting its surface with 220 mesh Al_2O_2 . An electric circuit is made with a cotton swab applicator as the anode,and the article to be plated as the cathode. The swab is then immersed in a proprietary plating solution and the plating applied by rubbing the applicator across the surface of the part to be coated. Preset current density and application time determines the thickness of the plating.

A number of SA 508 Class 2 steel coupons were tin plated by this "brush-on" process in order to evaluate the effectiveness of tinning as a wetting aid. Coupons were plated with 30, 60, 100 and 200 x 10^{-6} inch of tin, and tested at temperatures of 325 to 375F.

Table I summarizes the results of a few representative tests conducted with tin plated coupons. These tests illustrate the importance of temperature on wetting. Whereas Coupon A18 fully wet within one hour in 390F sodium, Coupons A20 and A21 in 375F sodium were only 50% wet after an hour. It required another 19 hours to fully wet A20 and A21 at 375F. That temperature is critical, was further demonstrated by tests at 325F. Coupon A22, which was immersed in sodium at 325F did not fully wet, even after 100 hours of soaking.

A coupon that had only been partially immersed in sodium was found to have a dark gray band just above its sodium immersion line, shown in Figure 5. It is believed that this gray substance is an alloy of tin and sodium, the result of tin being slowly dissolved by sodium. The sodium tin phase diagram, illustrated in Figure 6, suggests that the alloy is α $Na\,Sn_2$.

A test was conducted to determine whether sodium would completely dissolve (strip) the tin plating from a coupon, and if so, what effect this would have on wetting. Total immersion of a tin plated coupon in 450F resulted in immediate wetting. After 2000 hours in the sodium the coupon showed no signs of dewetting. The coupon was then removed from the sodium and its surface analyzed for tin content. No tin was found. It was concluded that sodium at 450F will dissolve tin plating but that the stripping of tin does not result in dewetting.

Several test coupons that appeared to be fully wetted were found, upon close examination, to have small unwetted spots along their edges. It is believed that this resulted from marginal tin plating of the edges and indicates that care must be taken to insure complete plating.

Overall, the plating tests demonstrated that tinning significantly enhances the sodium wettability of SA 508 Class 2 steel. Wetting is insured at 390F or above, but that tinning is not fully effective and therefore, wetting is not guaranteed at temperatures less than 390F.

TABLE I

SODIUM WETTING TEST RESULTS
(Tin Plated Specimens)

Coupon I.D. (SA 508 Class 2 Steel)	Surface Treatment	Na Temperature	Time	Results
A13	Tin Plated (30×10^{-6} in)	340F	15 min	Fully Wet
A18	Tin Plated (Front-100 x 10^{-6} inch) (Back -200 x 10^{-6} inch)	390F	1 Hr	Fully Wet Both Sides
A20	Tin Plated (60×10^{-6} inch)	375	1 Hr 20 Hrs	50% Wet Fully Wet
A21	Tin Plated (60×10^{-6} inch)	375	1 Hr 20 Hrs	50% Wet Fully Wet
A22	Tin Plated (60×10^{-6} inch)	325	1 Hr 48 Hrs >100 Hrs	50% Wet 80% Wet 80% Wet

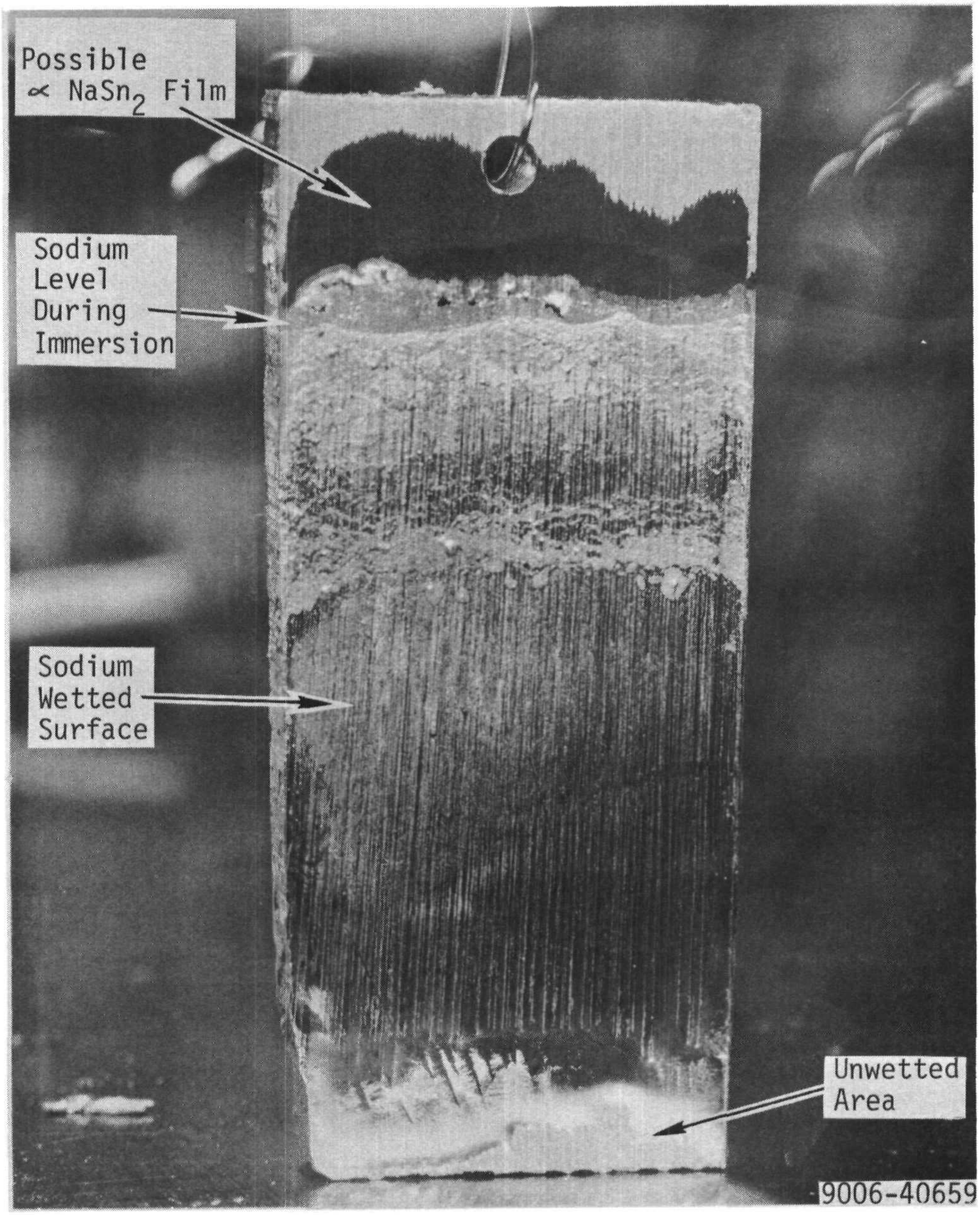

Figure 5. Partially Immersed Tin Plated Coupon

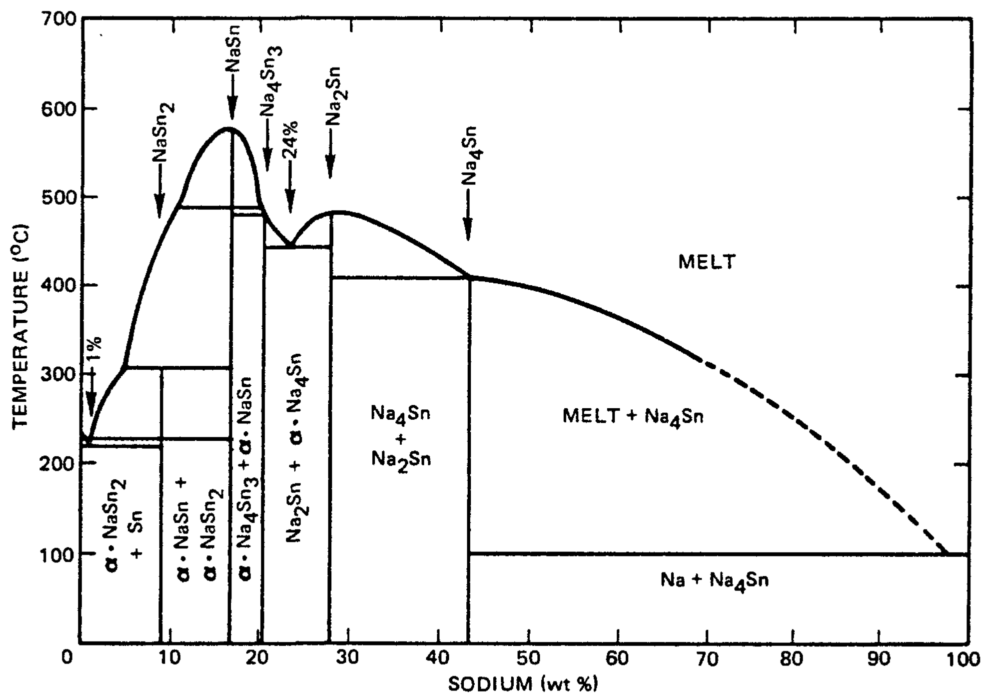

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Figure 6. Sodium-Tin Phase Diagram

C. ULTRASONIC ABRASION

Wetting with the aid of ultrasonics was studied at 413F, 375F and 325F. A 350-Watt ultrasonic unit was set up such that the ultrasonic horn was immersed about 1 inch in a temperature controlled sodium bath. Test coupons were suspended in a wire basket directly under the horn at a distance of ~ 1/8 inch. The coupons, completely submerged in sodium, were treated with ultrasonics in two-minute ON two-minute OFF cycles.

Twelve minutes of ultrasonic, plus a 16-hour soak were required to wet test coupons at 413F. Coupons were wet in 375F sodium with ~ 30 minutes of ultrasonics and a 16-hour soak. At 325F, 60 minutes of ultrasonic, plus a 72-hour soak did not produce wetting.

Figure 7 shows one of the coupons that wet at 375F, and illustrates how wetting occurs only on the surface area directly under the ultrasonic horn.

Initial ultrasonic wetting test utilized a 150-Watt ultrasonically agitated sodium bath that was restricted to a maximum of 270F. All attempts to wet the test specimens at this limited power-temperature level were unsuccessful.

These tests indicate that ultrasonic agitation can be used to induce wetting of SA 508 Class 2 steel if: 1) an ultrasonic unit with at least 350 Watts is employed, and 2) the sodium temperature is 375F or greater.

D. CHEMICAL CLEANING

Test coupons were cleaned in a variety of chemical manners to see if a chemical method could be found that would promote wetting in 450F sodium. SA 508 Class 2 coupons were cleaned by: 1) acetone rinse, 2) immersion in aqua regia, 3) immersion in hot sodium hydroxide and 4) scrubbing with trisodium phosphate. Coupons cleaned by each technique were then immersed at 450F to establish time-to-wet as a function of cleaning technique.

Representative wetting tests conducted with chemically cleaned coupons are listed in Table II. As is readily seen, none of the chemical treatments were capable of consistently promoting full wetting of the test coupons. In fact, only the acetone rinse technique was capable of inducing complete wetting and it did not accomplish this consistently.

The best aqua regia and sodium hydroxide treatments could achieve was 90% wetting after ~ 80 hours in 450F sodium. Whereas, coupons scrubbed with trisodium phosphate did not wet at all.

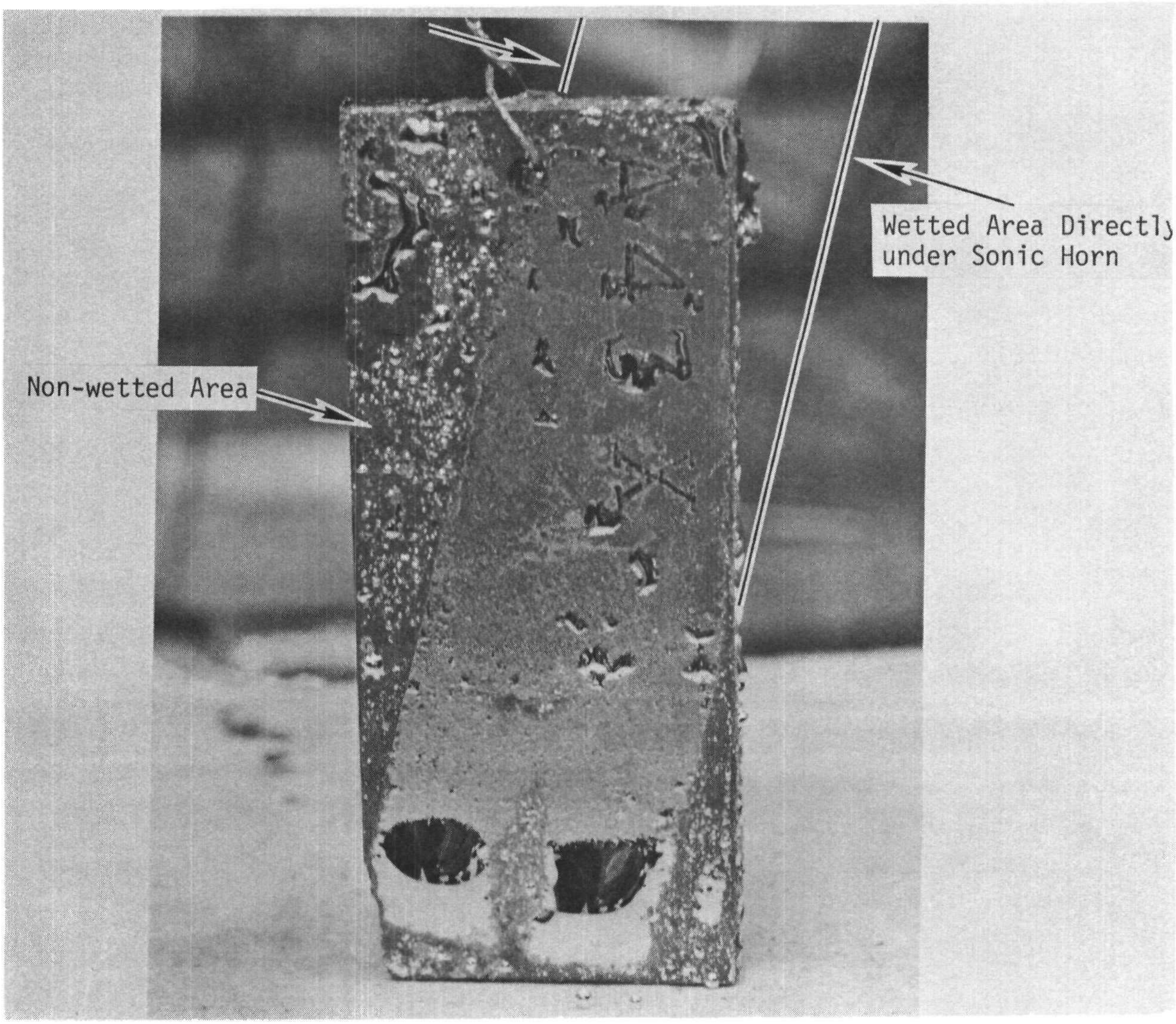

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Figure 7. Test Coupon Partially Wetted by Ultrasonics

The chemical cleaning tests show that of the techniques tried only the acetone rinse treatment was capable of promoting full wetting of SA 508 Class 2 steel at 450F and even it is not guaranteed to succeed every time.

TABLE II

SODIUM WETTING TEST RESULTS
(Chemically Cleaned Specimens)

Coupon I.D. (SA 508 Steel)	Surface Treatment	Na Temperature	Time	Results
A01	Acetone Rinse	450F	~ 3 Hrs	Not wet
A19	Acetone Rinse	450F	28 Hrs	Fully wet
A29	Acetone Rinse	450F	48 Hrs	Spotty wetting
A03	Aqua Regia Etch	450F	1 Hr	Not wet
A25	Aqua Regia Etch	450F	44 Hrs	Spotty wetting
A28	Aqua Regia Etch	450F	100 Hrs	90% wet
A26	NaOH Immersion	450F	44 Hrs	Spotty wetting
A27	NaOH Immersion	450F	80 Hrs	90% wet
A06	Trisodium Phosphate Scrub	450F	48 Hrs	Not wet

E. CONCLUSIONS AND RECOMMENDATIONS

Two techniques were established which depressed the sodium wetting temperature of the SA 508, Class II dip seal blade material to 375F. These techniques were: 1) depositing a $\sim 60 \times 10^{-6}$ inch layer of tin on the blade surface by a "brush-on" plating process by Brooktronics Inc., North Hollywood, California, and 2) ultrasonic cleaning of the blade surface while immersed in sodium.

The tin plating technique is recommended as the initial and primary surface preparation method. Using this technique, complete wetting of SA 508 Class 2 steel can be accomplished in 20 hours at 375F or one hour at 390F.

The ultrasonic technique is recommended as a backup method and for rewetting the blade, should it become necessary. With a 350-watt ultrasonic unit, ~ 30 minutes of ultrasonic cleaning in 375F sodium, followed by a 16-hour soak, will promote full wetting of SA 508 Class 2 steel.

Recent tests on the dip seal test article (Ref. 12) has shown that complete wetting can be accomplished with only 5 minutes of ultrasonics in 400F sodium, with no soak time required when a 750 watt ultrasonic unit is used. It appears, therefore, that ultrasonic wetting is highly dependent on generator power level, and that ~750 watts is the minimum acceptable level. Ultrasonic cleaning is not suggested as the primary wetting technique because it has the disadvantage that wetting of metal is effected only directly under the ultrasonic horn. Consequently, wetting of large surface areas, such as the blade of the CRBRP dip seal, would be very time-consuming.

The ultrasonic wetting method has the advantage that it can be used to rewet the dip seal blade in situ, if it became necessary. For this reason, and as a backup, it is suggested that the CRBRP dip seal incorporate an ultrasonic wetting unit.

VI. TEST EQUIPMENT AND INSTRUMENTATION

A detailed description of the sodium wetting study test equipment is presented in AI Test Procedure N707TP810012.

All experiments were performed in an inert atmosphere glovebox which had sodium piped into it from an external supply vessel. The glovebox sodium supply vessel and associated instrumentation are shown in Figure 8.

The glovebox was maintained at oxygen and water levels of less than 10 ppm by a Vacuum Atmosphere MO-20-2-H Purification System. Oxygen and water levels were monitored with a Teledyne Model 310 Trace Oxygen Analyzer and a Panametrics 2000 Hygrometer.

A 350-Watt Branson ultrasonic unit was set up so that ultrasonic abrasion of coupons could be performed within the inert atmosphere of the glovebox.

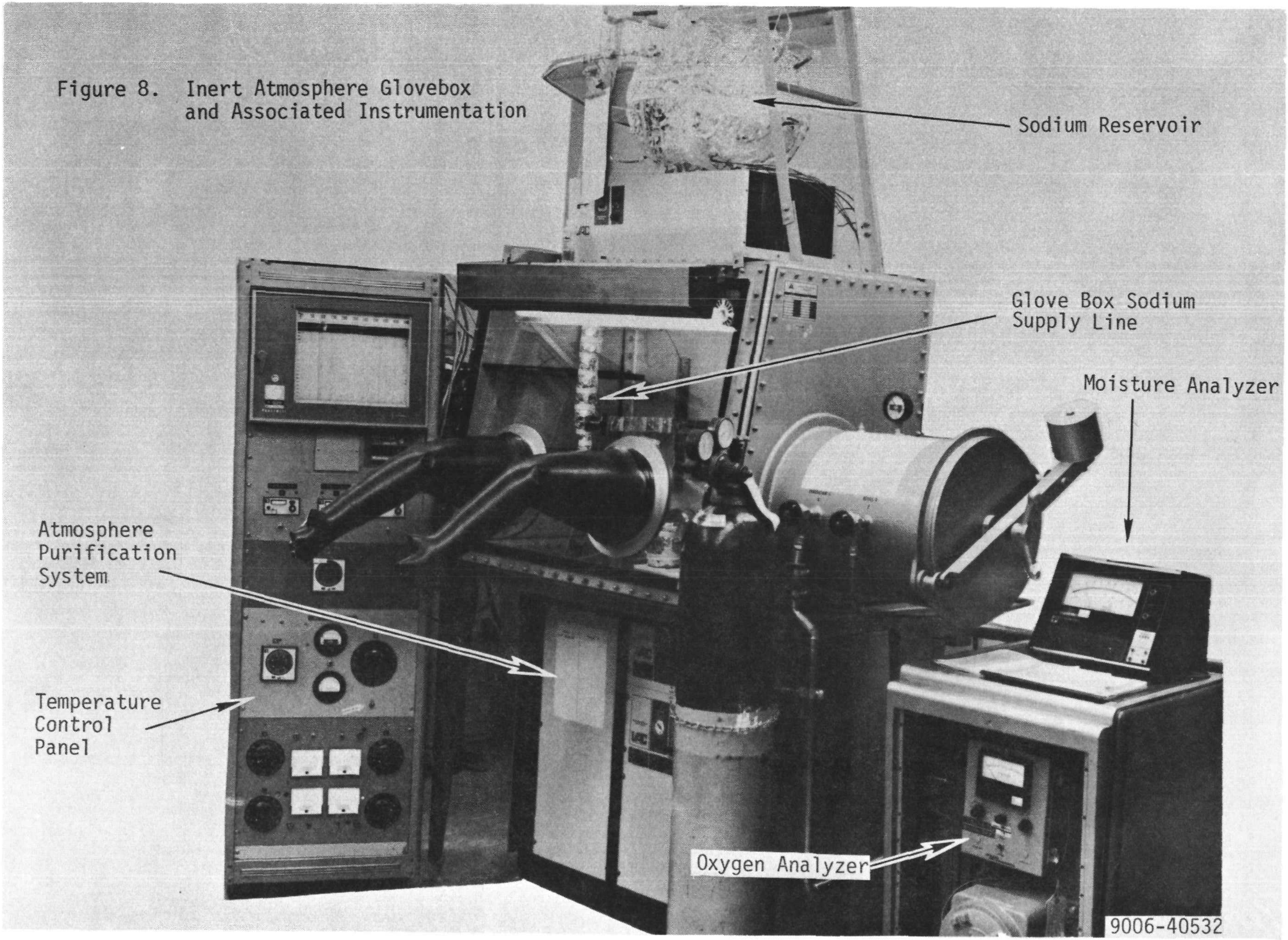

Figure 8. Inert Atmosphere Glovebox and Associated Instrumentation

VII. DISPOSITION OF TEST ARTICLES

All scrap sodium was disposed of according to approved safety procedures. Test coupons are stored in Bldg. 006, AI, Santa Susana and they will be retained for a minimum of one year.

VIII. REFERENCES

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APPENDIX A

ASTM SPECIFICATIONS FOR SA 508 CLASS 2 STEEL

Chemical Requirements

Carbon, per cent	0.27 max
Manganese, per cent	0.50 to 0.90
Phosphorus, per cent	0.025 max
Sulfur, per cent	0.025 max
Silicon, per cent	0.15 to 0.35
Nickel, per cent	0.50 to 0.90
Chromium, per cent	0.25 to 0.45
Molybdenum, per cent	0.55 to 0.70
Vanadium, per cent	0.05 max

Tensile Requirements

Tensile strength, min. psi	80,000
Yield strength, min (0.2 per cent offset), psi	50,000
Elongation in 2 in. min per cent	18
Reduction of area, min per cent	38