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Assessment of Chromate and Non-Chromate Conversion Coatings for Al Alloys Using Electrochemical Impedance Spectroscopy

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A study was conducted to evaluate whether a non-chromate conversion coating (Sanchem) for Al alloys (Al 2024 and 7075) could be a suitable replacement for the currently used chromate conversion coating (Alodine) without compromising corrosion resistance. Electrochemical impedance spectroscopy (EIS) and salt spray testing were employed to compare the corrosion behavior of coatings consisting of a) pretreatment only (Sanchem or Alodine), b) pretreatment (Sanchem or Alodine) plus primer (epoxy polyamide or waterborne epoxy), and c) pretreatment (Sanchem or Alodine) plus epoxy polyamide primer plus polyurethane topcoat. Results indicated that the experimental impedance values provided a reliable estimate of the film integrity and corrosion protective capability, that the Sanchem conversion coating generally compared favorably with the Alodine coating, and that higher impedance values attributed to the application of topcoat of both alloys which were treated with either Sanchem or Alodine and epoxy polyamide are characteristic of low conductivity, good barrier type coatings. However, additional tests are required before the Sanchem can be recommended as a reliable alternative to the Alodine coating.

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Assessment of Chromate and Non-Chromate Conversion Coatings for Al Alloys Using Electrochemical Impedance Spectroscopy

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Introduction

Environmental restrictions are demanding changes in common coatings such as chromium (Cr^{+6}) and cadmium as well as processing technologies including electroplating and immersion treatments that produce high levels of hazardous materials at Army Depots. Concomitantly there is a dramatic increase in cost and logistical problems associated with safe waste disposal. As a consequence new alternative environmentally acceptable solutions must be found.

Chromium and its salts that are used in the processing of conversion coatings for Al alloys are on the Environmental Protection Agency's list of 17 materials that the Government and industry are trying to reduce by 50% by 1995. This paper evaluates whether a non-chromate conversion coating for Al alloys could be a suitable replacement for the currently used Alodine chromate conversion coating without compromising corrosion resistance. Specifically, Electrochemical Impedance Spectroscopy (AC Impedance) and salt spray testing have been employed to compare the corrosion behavior of the non-chromate conversion coating against the Alodine treatment in combination with selected primers and a topcoat.

Experimental

Coated test panels, 7.62 cm x 12.7 cm x 0.1 cm, were supplied by the Naval Air Warfare Center (NAWC), Warminster, PA, as shown in Table 1.

Before AC Impedance testing each treated panel was removed from a desiccator and examined visually for the presence of defects. Each panel was positioned in the test cell shown in Figure 1 without any water rinsing or solvent cleaning. AC impedance was performed with a PAR 378 Electrochemical Impedance System consisting of a 5208 two-phase lock-in analyzer, a model 273 potentiostat/galvanostat, and a IBM PC XT computer and printer. Periodic measurements were made from the sample exposed to 0.5N NaCl solution at the corrosion potential (stabilized within 1 hour) over the frequency (f) range 5 mHz to 100 KHz during a 300 day period at room temperature. The single sine technique with an input sinusoidal voltage of 5 mV was used in the frequency range 100 KHz - 5 Hz. In the frequency range 10-0.005 Hz, the multisine technique was used with an input sinusoidal voltage of 10 mV. The data collected were plotted and evaluated in both Bode and Nyquist formats. Impedance values were extrapolated from the linear region of the Bode plot at low frequency to 1 mHz at the log |z| axis and plotted as a function of exposure time. Mansfeld and Kendig (1) have used similar experimental data for determining corrosion resistance of anodized Al alloys. Leidheiser (2) has reported that coating system impedance measured by AC impedance techniques degraded with time: at a lower limit of about 10^6 ohms cm^2 corrosion was found to occur underneath the coating. Salt spray testing by the NAWC was performed in accordance with ASTM B117, using a 5% NaCl solution at 95°F.

Results

Pre-Treatment Only Tests

Figure 2 contains plots of impedance (derived from Bode plots, log |z| vs. log f) as a function of exposure time for Al 2024-T3 samples treated with an aqueous solution of chemicals conforming to MIL-C-81706 (standard Alodine chromate conversion coating) and with the Sanchem Boehmite process (non-chromate conversion coating, processing steps shown in Figure 3). The Sanchem treated sample characteristically exhibits an order of magnitude higher impedance than the standard Alodine treated alloy (10^6 vs. 10^5 ohms cm^2). Also, the Sanchem treated material showed no evidence of degradation (decrease in impedance) over a 200 day period of exposure to the 0.5N NaCl solution. After 75 days of exposure the Alodine treated alloy showed a decreasing trend in impedance, thus increasing the difference in impedance between the two conversion coatings, indicating a reduction in its corrosion resistance. Figure 4 contains SEM photomacrographs showing the Sanchem and Alodine treated Al 2024 alloys before and after completion of the impedance test. The Sanchem treated alloy remained unaffected after the 200 hour exposure to 0.5N NaCl solution while the Alodine treated alloy

(1) F. Mansfeld and M.W. Kendig, Impedance Spectroscopy as Quality Control and Corrosion Test for Anodized Al Alloys, CORROSION, 41, (8), 490 (1985).
(2) H. Leidheiser, Jr., Review of Electrochemical and Electrical Measurement Methods for Predicting Corrosion at the Metal-Organic Coating Interface, CORROSION, 38, (7), 374 (1982).

showed the presence of pits and corrosion products. These observations are in accord with the impedance data. Nevertheless both treatments passed the 336 hour salt spray test (Table 1).

Plots of impedance as a function of exposure time for both treatments on the Al 7075-T6 alloy are shown in Figure 5. Impedance values for the intervals up to 40 hours of exposure time are fairly constant for both treatments, although the Alodine treatment exhibits somewhat higher values. For both treatments, as exposure time increased to 200 hours impedance values generally decreased but the Sanchem processed alloy displayed the lower impedance. The impedance data are in good agreement with the photomacrographs shown in Figure 6; the Alodine treatment provided better corrosion resistance than the Sanchem treated alloy. But both treatments provided the Al 7075-T6 alloy with 336 hours of acceptable salt spray resistance (Table 1).

The Alodine and Sanchem conversion coatings were analyzed by an ESCA/Auger and Scanning Auger Microprobe (SAM) before and after the impedance tests. The results of Auger analyses are contained in Figures 7 and 8. The Auger spectra for the Alodine treated Al 2024-T3 and Al 7075-T6 alloys (Figure 7 a -d) show that the conversion coating (thickness ~ 2000A°) contains chromate as the major constituent both before and after the test. Aluminum is neither present as a constituent of the coating nor exposed as the substrate. These data are in good agreement with the other reported test data. Comparable spectra for the Sanchem treated alloys (Figure 8 a - d) indicates the conversion coating (4000A° thick) contains an oxide of aluminum as the major constituent. This coating remains essentially intact after 200 hours of exposure to the 0.5N NaCl solution. These data also appear to be in accord with the other test data.

Pre-Treatment plus MIL-P-23377 Epoxy Polyamide Primer Tests

The effect of the epoxy polyamide primer (MIL-P-23377) on the impedance of Al 2024-T3 which was pretreated with either the Alodine or Sanchem process is shown in Figure 9. The impedance values are in the range 10^6 to 10^7 ohms cm^2. This primer significantly improves the performance of the Alodine treated alloy; impedance increased from 10^5 to 10^7 for the first 65 days of exposure. Beyond 65 days the impedance drops to 10^6 ohms which is comparable to the impedance of the Sanchem plus primer system. The epoxy primer had little effect on the performance of the Sanchem treated alloy; the impedance which was constant throughout the 200 days of the test remained at 10^6 ohms cm^2, which was surprisingly equivalent to the bare pretreatment values after exposure. Post test visual examination showed no evidence of corrosion for either protective scheme. However microscopic examination of the Alodine plus primer system revealed a small blister which appeared unbroken (Figure 10a). This defect however did not appear to affect the impedance. At times, impedance plots may be insensitive to certain paint coating failures such as the formation of non-perforated blisters. If the blister had been perforated the impedance would fall (3). Microscopic examination of the Sanchem plus primer system showed no evidence of corrosion (Figure 10b).

Impedance vs. time plots for the Al 7075-T6 alloy treated with either the Alodine or Sanchem process in combination with the MIL-P-23377 primer are contained in Figure 11. Impedance values for both systems were higher than the bare pretreated samples and remained quite stable during the entire test; the Alodine treated scheme impedance was 10^7 ohms cm^2, the Sanchem processed scheme was 10^6 ohms cm^2. Post-test microscopic examination (Figures 12a + b) showed the presence of small unperforated blisters in both protective schemes but they were undetected by the impedance plot.

Both alloys treated with Alodine-P-23377 primer scheme passed the 336 hour salt spray test and the Sanchem/MIL-P-23377 primer processed alloys completed 1000 hours of salt spray without failure.

Pre-Treatments Plus MIL-P-85582 Waterborne Epoxy Primer Tests

Figure 13 compares impedance vs. time plots for Al 2024-T3 given the Alodine and Sanchem pretreatments plus an overcoat of the waterborne epoxy primer. Impedance values are in the same range (10^6 - 10^7 ohms cm^2) as reported above for the MIL-P-23377 epoxy polyamide primer. The plot for the Alodine treated systems shows that impedance fluctuated with time but remained above 10^6 ohms cm^2. The Sanchem pretreatment exhibited relatively stable behavior during the course of the exposure (10^6 ohms cm^2). Post test microscopic examination showed the presence of several non-perforated blisters on the Alodine/primer scheme and a single non-perforated blister was observed on the Sanchem/primer scheme (Figure 14 a + b). Again, these blisters passed unnoticed in the impedance plot.

(3) S. Feliu, J.C. Galvan and M. Marcillo, "The Charge Transfers Reaction in Nyquist Diagrams of Painted Steel", Proceedings of the Symposium on Advances in Corrosion Protection by Organic Coatings, Volume 89-13, p. 281, Electrochemical Society, 1989.

Impedance vs. time plots for these protective schemes applied to the Al 7075-T6 alloy are contained in Figure 15. The very erratic impedance behavior of the Sanchem/MIL-C-85582 primer treated alloy (impedance below 10^5 ohms cm^2 after 200 hours of exposure) is reflected in the photomacrograph of Figure 16a which shows the presence of numerous blisters, both perforated and unperforated. The Alodine/primer treated alloy displays some fluctuation in impedance but generally remains in the range of 10^6 to 10^7 ohms cm^2. A single unperforated blister is revealed in the post test microscopic examination (Figure 16b). Nevertheless these protective schemes passed the 336 hour salt spray test.

Pre-Treatments Plus Primer Plus Topcoat Tests

Impedance values for this series of protective schemes (Figure 17) were higher than our other samples (10^7 - 10^9 ohms cm^2). These higher impedance values are characteristic of low conductivity, good barrier type coatings. Differences between the Alodine and Sanchem pretreatments are minimal, particularly in the case of the Al 7075-T6 alloy where the impedance is 10^9 ohms cm^2 in the time intervals between 200 and 320 days. Microscopic examination supports the impedance data although some staining was observed only on the Sanchem treated alloys (Figure 18 a + b).

Conclusions

1. The good agreement between the impedance data, microscopic observations, and salt spray results suggests that the experimental impedance values provide a reliable estimate of the film integrity and corrosion protective capability of the coatings/substrates studied. However, at times, impedance values appeared to be insensitive to the formation of non-perforated paint blisters; in the case where the ionic resistance of the paint film is much greater than the metal transfer resistance.

2. The non-chromate conversion coating generally compared favorably with the standard chromate conversion coating; used singly or in combination with a primer and topcoat. Additional testing is required before we can recommend this environmentally acceptable conversion coating as a reliable alternative to the currently used chromate conversion coating.

3. The higher impedance values attributed to the application of the topcoat to both alloys which were treated with either the non-chromate or

chromate conversion coating and the MIL-P-23377 epoxy polyamide primer are characteristic of low conductivity, good barrier type coatings.

Acknowledgement

The authors gratefully acknowledge the assistance of Steve Spadafora, NAWC, in providing the coated test panels and salt spray data.

Table 1. Evaluation of Alloys/Coating Systems Resistance to Salt Spray Testing

Panels Supplied by NAWC

PRIMER/TOPCOAT SYSTEM	ALLOY	PRETREATMENT SYSTEM: Deoxidize+Alodine (MIL-C-81706) Salt Fog Resistance (hrs)	PRETREATMENT SYSTEM: SANCHEM Boehmite Coating (SBC) Process Salt Fog Resistance (hrs)
Pretreatment Only	2024-T3	336 NC*	336 NC
	7075-T6	336 NC	336 NC
Pretreatment + MIL-P-23377 Epoxy Polyamide Primer	2024-T3	336 NC	1000 NC
	7075-T6	336 NC	1000 NC
Pretreatment + MIL-P-85582 Waterborne Epoxy Primer	2024-T3	336 NC	1000 NC
	7075-T6	336 NC	1000 NC
Pretreatment + MIL-P- 23377 + MIL-C-83286 Polyurethane Topcoat	2024-T3	336 NC	336 NC
	7075-T6	336 NC	336 NC

* NC - no corrosion

Note: Alodine panels were tested only to 336 hrs with no failure.

Sanchem panels were tested with no failures to 1000 hrs. to evaluate the new system

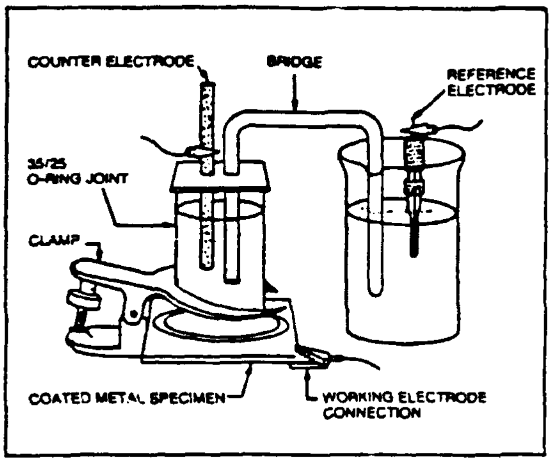


Fig. 1 Test Cell for Electrochemical Impedance Measurements

Fig. 2 Al 2024, Sanchem vs. Alodine

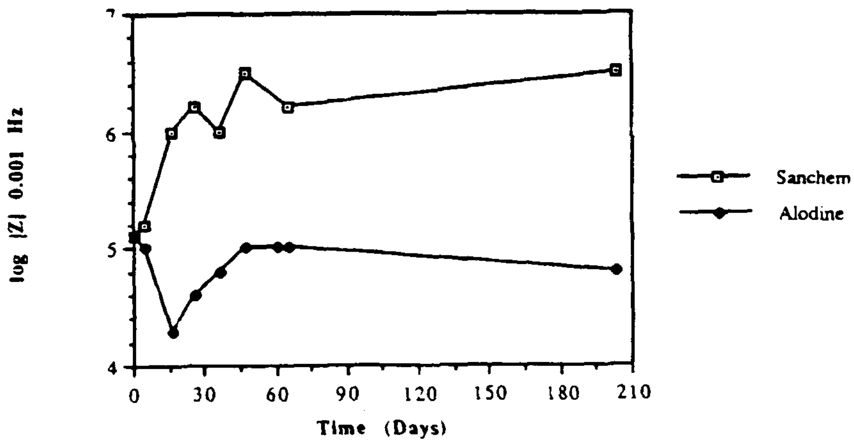


Fig. 3 Non Chromated Conversion Coatings

SANCHEM Boehmite Coating Process

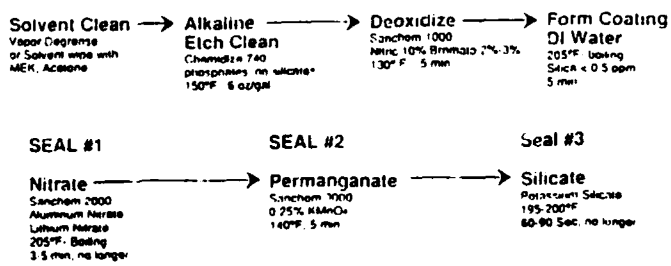
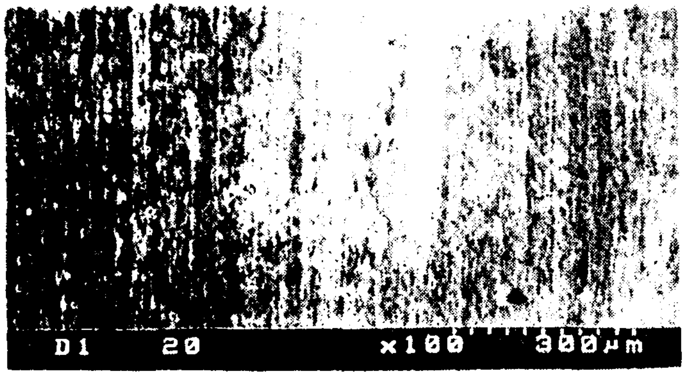


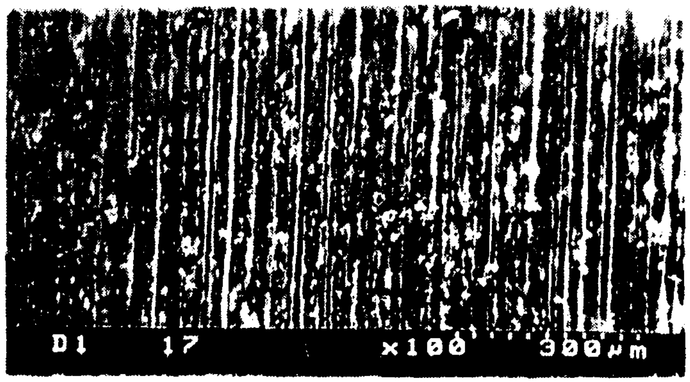
Fig. 4 Sanchem vs. Alodine on 2024 Al



A. Sanchem Before Testing at 100X

C. Alodine Before Testing at 100X

B. Sanchem After Testing at 100X



D. Alodine After Testing at 100X

Fig. 5 Al 7075, Sanchem vs. Alodine

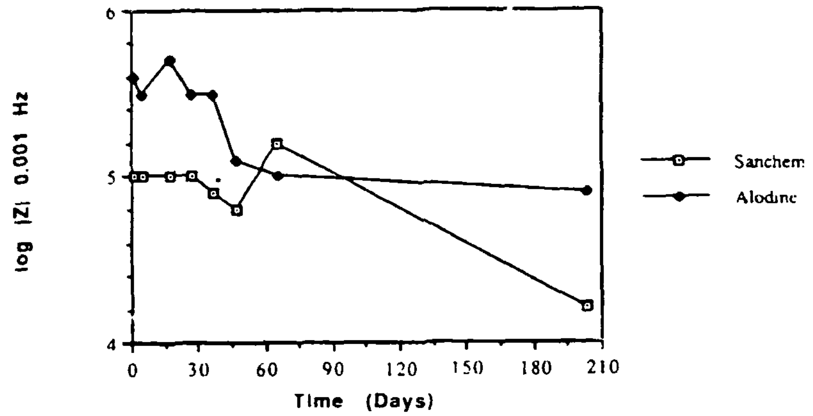
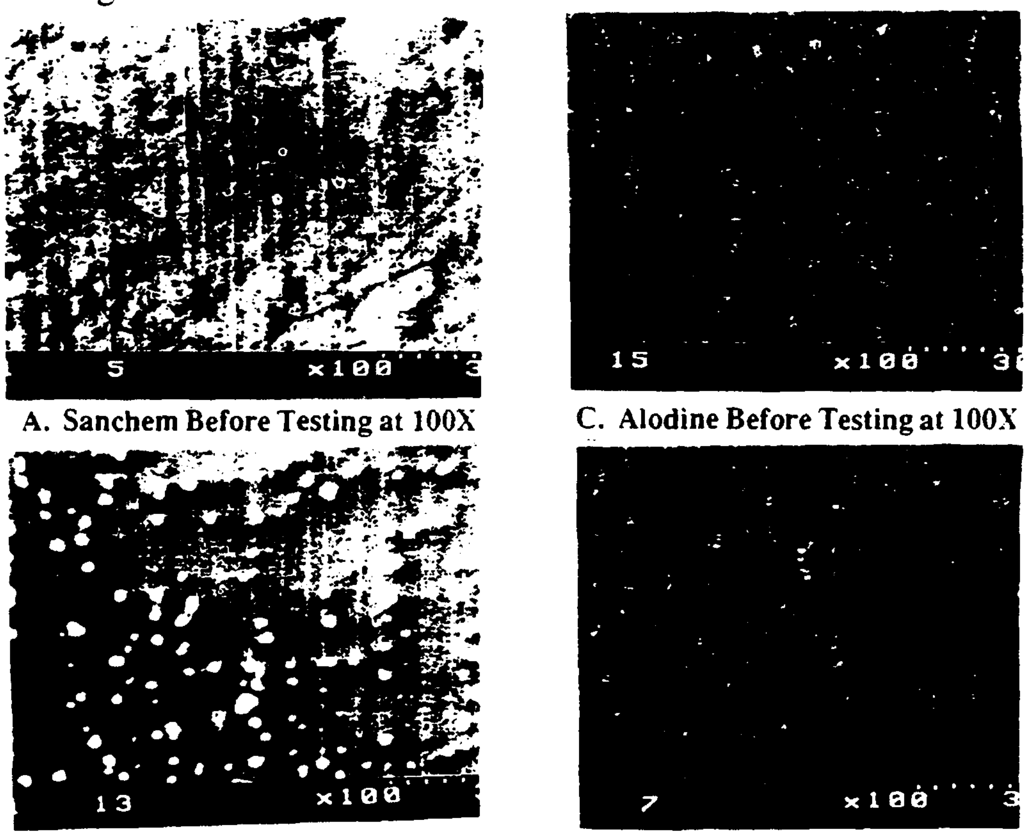


Fig. 6 Sanchem vs. Alodine on 7075 Al



A. Sanchem Before Testing at 100X

C. Alodine Before Testing at 100X

B. Sanchem After Testing at 100X

D. Alodine After Testing at 100X

Auger Spectra

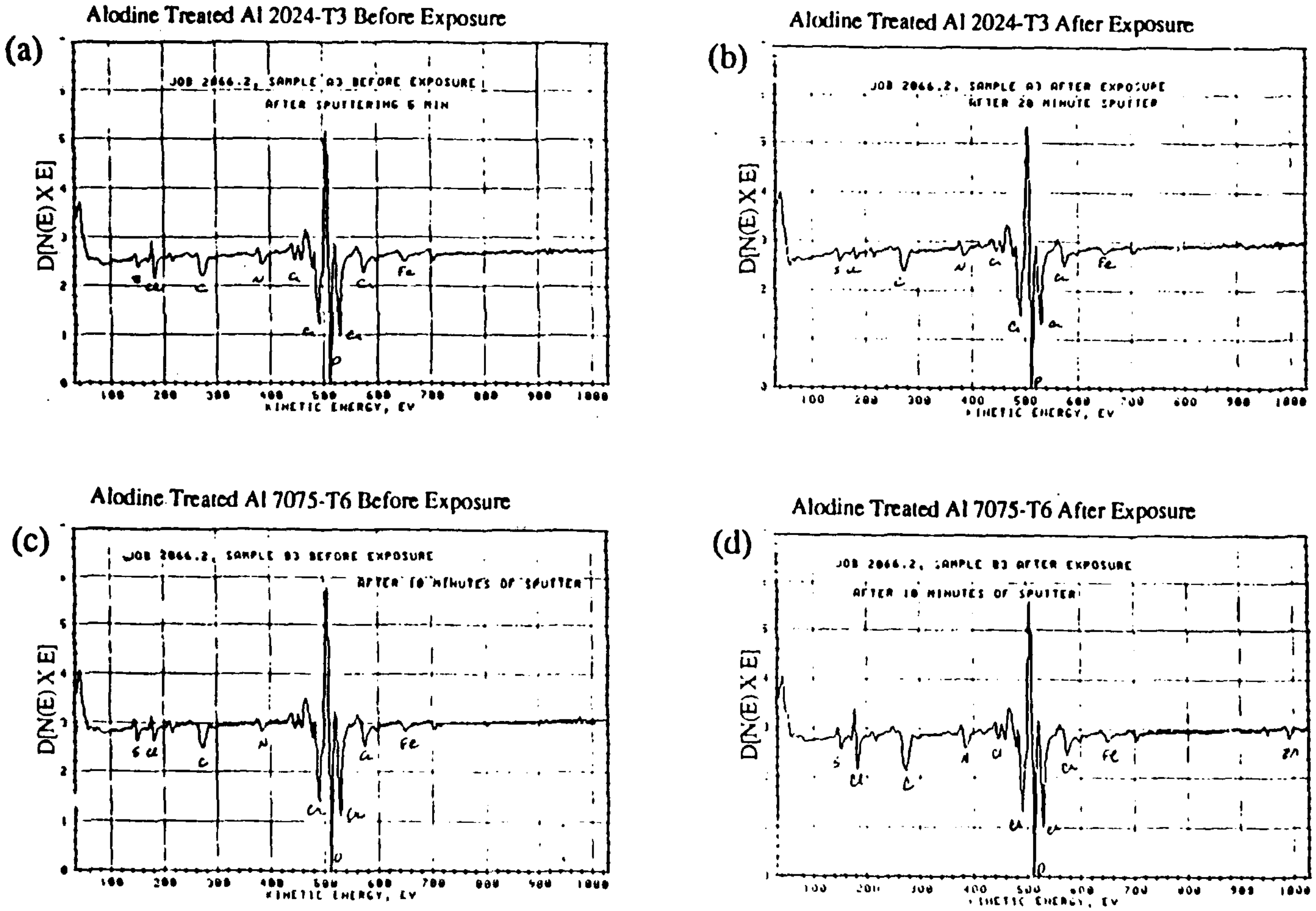


Figure 7

Auger Spectra

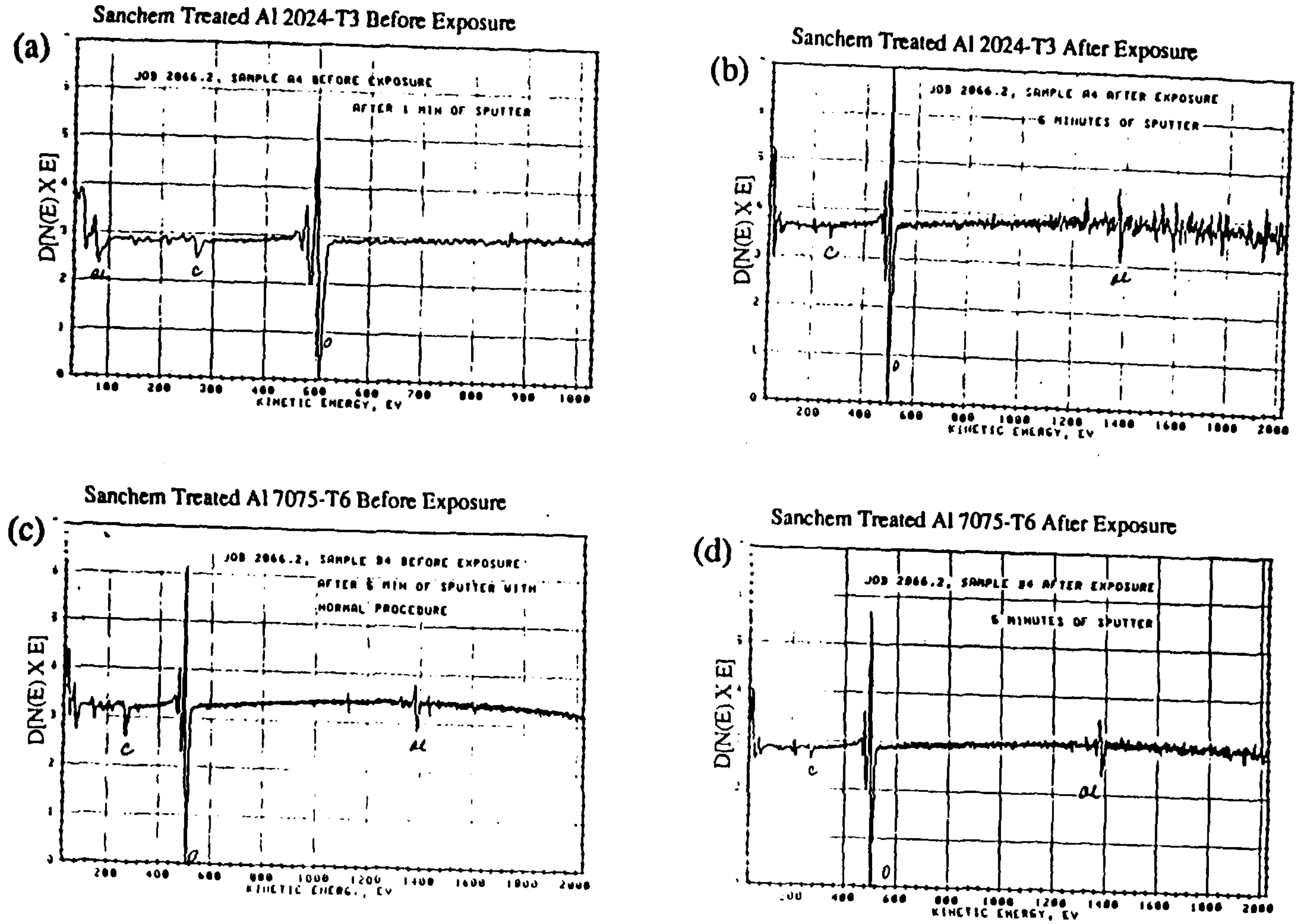
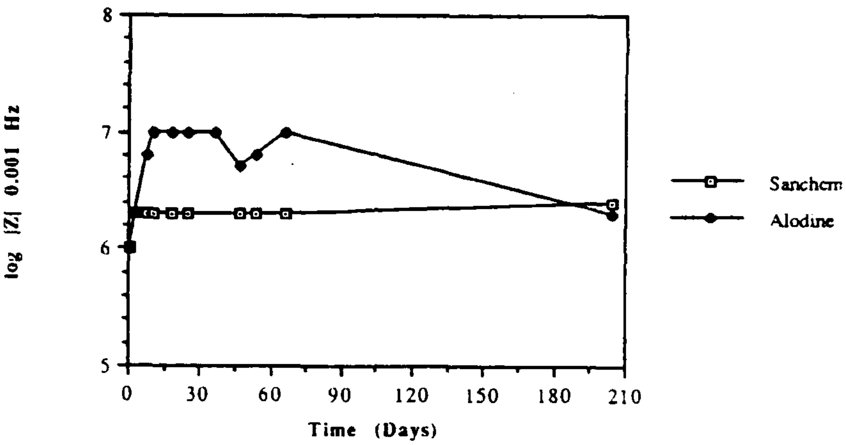
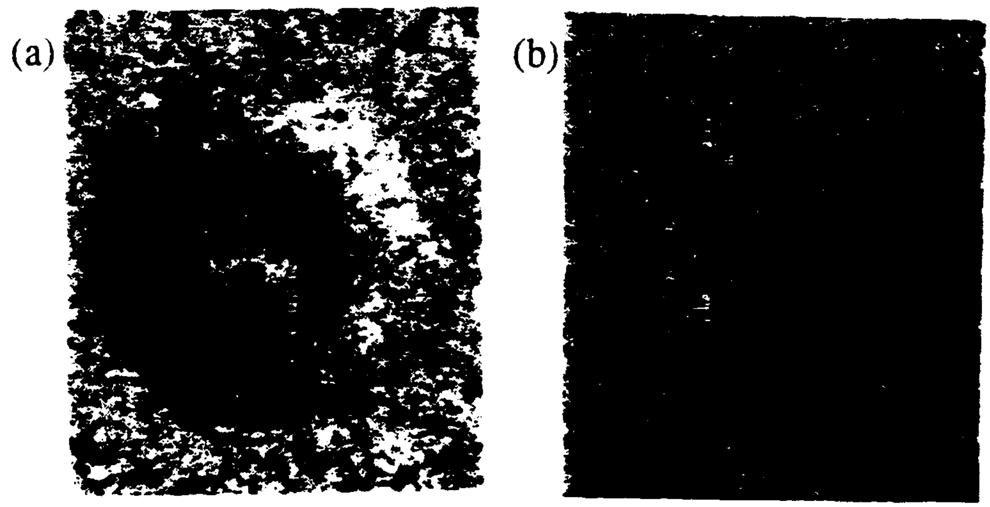


Figure 8

Fig. 9 **Al 2024 + P23377, Alodine vs. Sanchem**



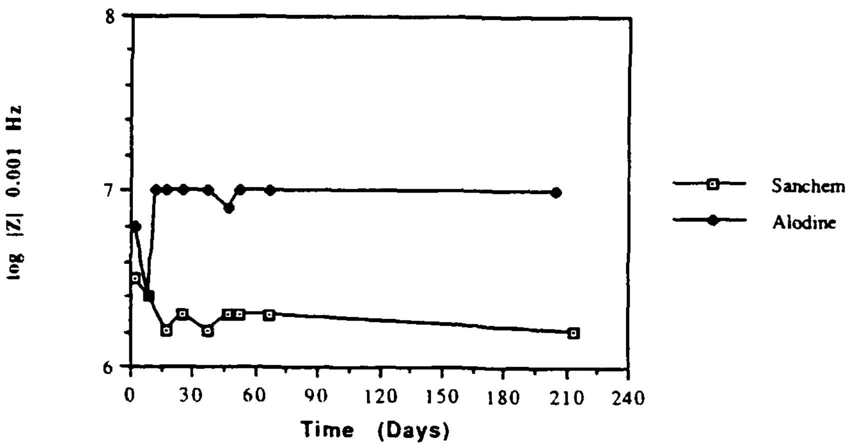


60X Macro of Al 2024 + P23377 with Alodine After 200 Days

60X Macro of Al 2024 + P23377 with Sanchem After 200 Days

Fig. 10

Fig. 11 **Al 7075 + P23377, Sanchem vs. Alodine**



(a)

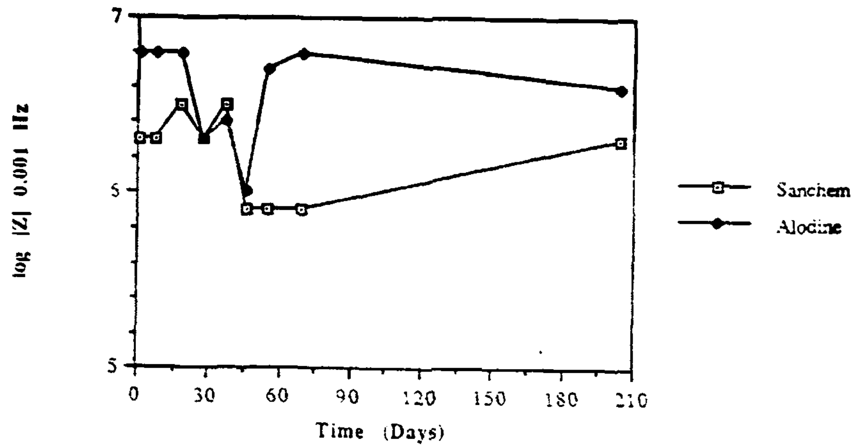
60X Macro of Al 7075 + P23377 with Alodine After 200 Days

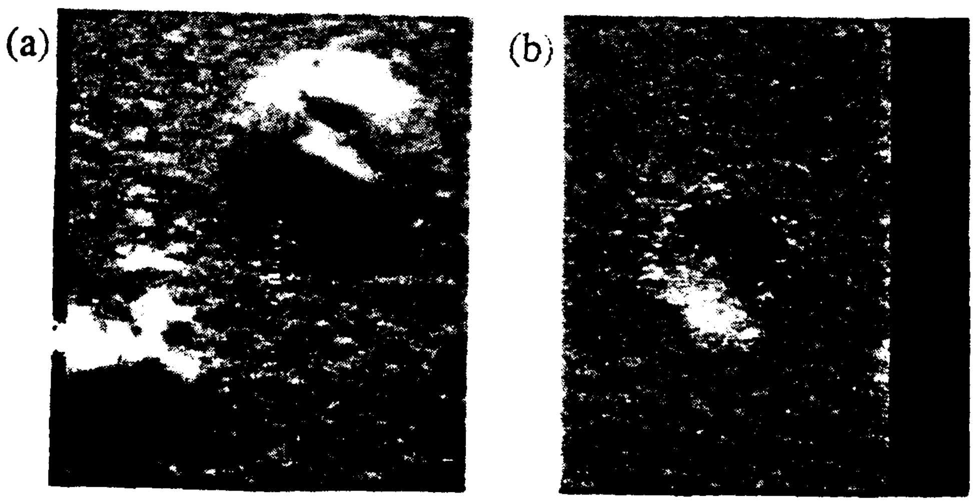
(b)

60X Macro of Al 7075 + P23377 with Sanchem After 200 Days

Fig. 12

Fig. 13 **Al 2024 + P85582, Sanchem vs. Alodine**



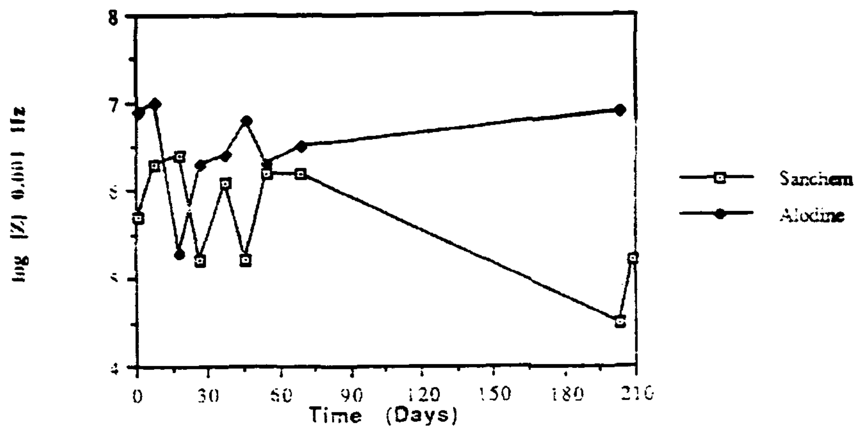


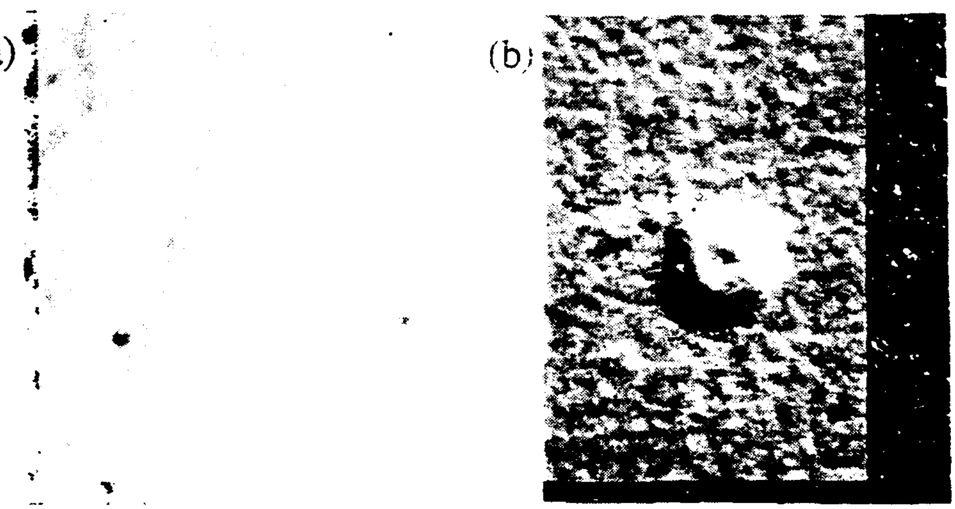
40X Macro of Al 2024 + P85582 with Alodine After 200 Days

40X Macro of Al 2024 + P85582 with Sanchem After 200 Days

Fig. 14

Fig. 15 **Al 7075 + P85582, Sanchem vs. Alodine**





a) 10X Macro of Al 7075 + P85582 with Sanchem After 200 Days

(b) 60X Macro of Al 7075 + P85582 with Alodine After 200 Days

Fig. 16

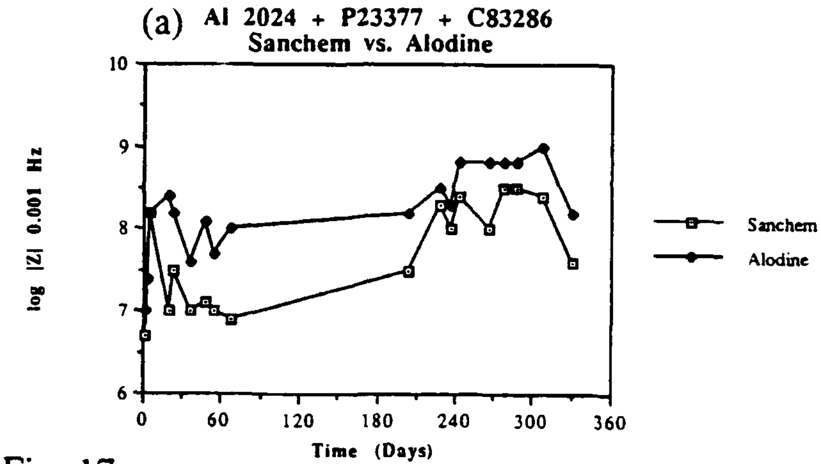
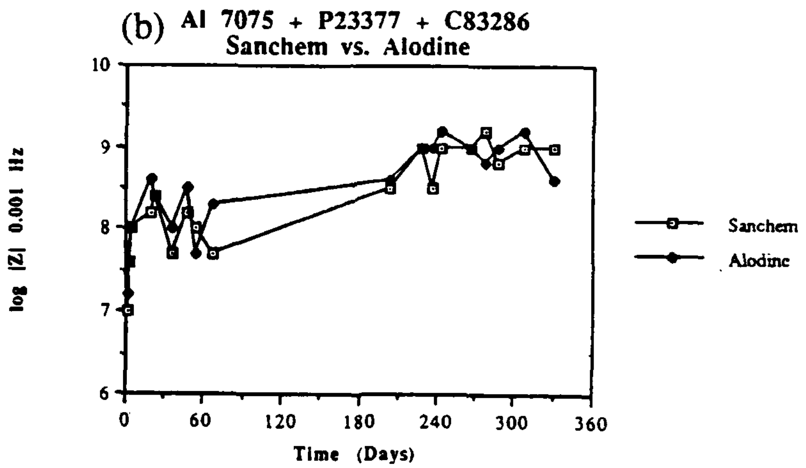
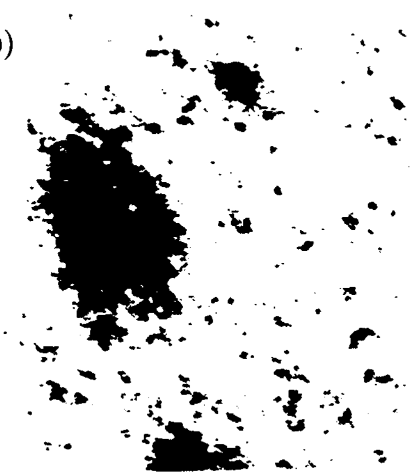


Fig. 17




(a)

60X Macro of Al 7075 + P23377 + C83286 with Alodine After 200 Days


(b)

60X Macro of Al 7075 + P23377 + C83286 with Sanchem After 200 Days

Fig. 18

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