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RELATIVE EFFECTS OF A GRAPHITE FIBER REINFORCED  
EPOXY COMPOSITE AND NICKEL-ALUMINUM BRONZE ON  
A CATHODIC PROTECTION SYSTEM IN SEAWATER

by

Harvey P. Hack  
Aleksander B. Macander

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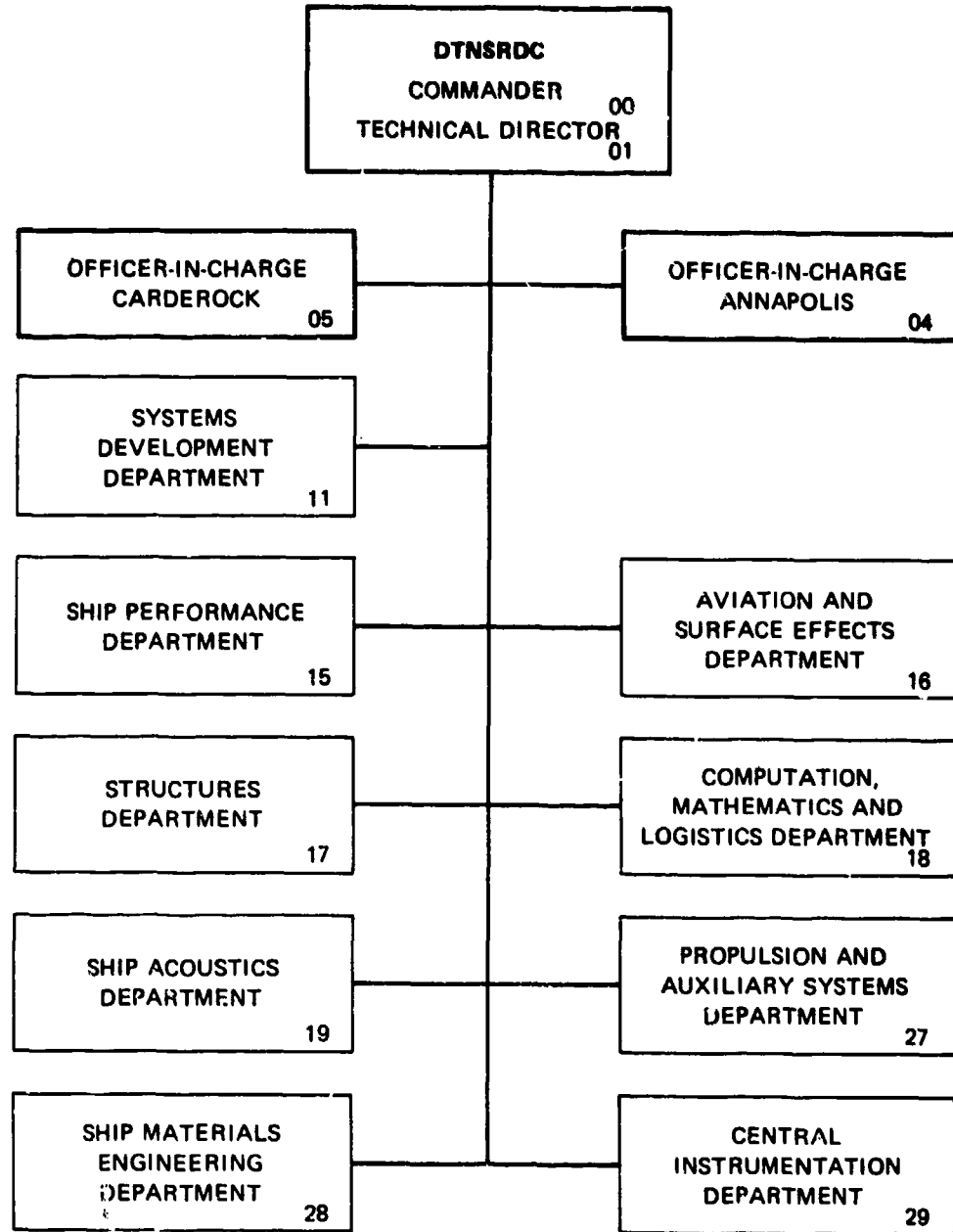
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## ABSTRACT

This study was designed to quantify the cathodic efficiency of a graphite fiber reinforced epoxy laminated composite material by comparing the amount of zinc consumption from a sacrificial-anode cathodic protection system on the composite with the consumption due to nickel-aluminum bronze in seawater. Specimens of the composite or of bronze were electrically coupled to anode zinc in seawater at flow velocities of 0 and 10 meters per second for up to 270 days. The composite did not cause a significantly different current demand than bronze on the zinc sacrificial cathodic protection system. It was noted that this represents a worst-case condition for the composite, since the specimens were deliberately prepared with the graphite-fiber ends exposed on the test surface.

## ADMINISTRATIVE INFORMATION

This work was conducted as part of an independent exploratory development program of the David W. Taylor Naval Ship Research and Development Center. It was conducted under Program Element 62766N, Task Area ZF 664 12001, Work Unit 1-2823-516.

## INTRODUCTION

Continuous graphite fiber reinforced epoxy laminated composites are one of several advanced composite materials which hold promise for a variety of applications in the marine environment. Noteworthy advantages that are inherent in these materials and not readily identifiable with marine grade alloys include corrosion resistance, high strength, high stiffness, low weight, and ease of fabrication into complex structures. With the exception of the graphite-fiber epoxy composite materials, most other fiber-reinforced composites are considered to be electrically nonconductive. Bulk graphite is, however, a material which has a noble potential and extremely high polarization resistance in seawater, making it an effective cathode. Thus, coupling bulk graphite electrically to any common metallic structural material in seawater would cause accelerated corrosion of that material. This severe condition is somewhat mitigated in the graphite-fiber organic matrix composite in that the matrix provides electrical insulation to those composite surfaces which do not have exposed graphite-fiber ends, thus decreasing the effective wetted area of the graphite. This study is designed to quantify the cathodic

efficiency of the laminated composite material by comparing the amount of zinc consumption from a sacrificial-anode cathodic protection system on the composite with the consumption on a material such as nickel-aluminum bronze which is commonly used in a seawater environment.

#### MATERIALS

The graphite-epoxy composite specimens used in this evaluation were prepared from a 6.4-mm thick by 300-mm wide by 460-mm long laminate which was fabricated at the Center. The specimens were cut to size with a diamond wet wheel. The specimen face exposed to seawater was ground with a belt sander until graphite fibers were exposed, thus removing the insulating epoxy surface layer. The composite laminate itself consisted of a lay-up construction having 43 plies. The fiber direction of each ply was either  $0^\circ$  or  $\pm 45^\circ$  as shown in the following laminate formula

$$(0_3/\pm 45)_4/\bar{0}_3/(\pm 45/0_3)_4 \text{ Total}$$

where  $(0_3/\pm 45)_4$  = three plies in  $0^\circ$  direction followed by one ply in the  $+45^\circ$  direction and one ply in  $-45^\circ$  direction; sequence is repeated four times,

$\bar{0}_3$  = three plies in  $0^\circ$  direction located at mid-thickness of laminate and being common to the mirror image of laminate,

$(\pm 45/0_3)_4$  = mirror image of  $(0_3/\pm 45)_4$ ,

Total = full number of plies shown in laminate formula, 43 in this case.

This particular construction represents certain structural requirements wherein the  $0^\circ$ -oriented fibers provide axial-load transfer capability in the principal loading direction while the  $\pm 45^\circ$  fibers provide torsional stiffness. A low modulus HTS\* graphite continuous fiber reinforcement, supplied as a prepregged 300-mm wide tape was used to construct the laminate. The prepregged matrix was a  $177^\circ\text{C}$  curing epoxy

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\*HTS = high tensile strength graphite fiber; tensile strength  $\approx 2240$  MPa; tensile modulus  $\approx 2.34 \times 10^5$  MPa.



system commonly found in the aerospace industry. A vacuum bag autoclaving procedure, including the cure schedule, is given in Figure 1 and illustrates the fabrication process used.

The nickel-aluminum bronze used in this study was purchased to Military Specification MIL-B-21230. This material had a nominal composition of 4-5.5 Ni, 8.5-11.0 Al, 4-5 Fe, 3.5-max Mn, with the balance being copper. The zinc was purchased as a nominal 23-lb sacrificial anode and is the standard anode grade used in U.S. Navy shipboard cathodic protection systems. Specimens were rough-cut from blocks and machined to final dimensions. Holes were drilled partially through the dry sides of specimens of both materials to facilitate electrical connection.

#### EXPERIMENTAL PROCEDURE

Specimens of either the graphite-epoxy composite or the nickel-aluminum bronze ( $19.4 \text{ cm}^2$ ) were electrically coupled to one-fourth size specimens of zinc ( $4.8 \text{ cm}^2$ ) and exposed to natural seawater flowing at about 0 m/s or at 10 m/s for 31, 120, or 270-days duration. Uncoupled control specimens of all materials were also exposed at similar velocities and durations. These exposures were conducted in the Basil Flow Cells (Polarization Cells) at the LaQue Center for Corrosion Technology in Wrightsville Beach, NC. This apparatus is designed to mount specimens 30-mm long by 73-mm wide by 6-mm thick, with one surface of each specimen facing a second specimen across a 6-mm-wide water channel. For this test the zinc specimens were 30-mm long by 18-mm wide by 6-mm thick, and nonmetallic spacers were used to fill the remaining space for flow continuity. Electrical connection was made to the specimens on the back surfaces, which remained dry. Each cell had a salt-bridge arrangement to allow monitoring of corrosion potentials with a silver/silver chloride (Ag/AgCl) reference half-cell. Galvanic current between the coupled specimens could be monitored using a zero-resistance ammeter. Couple currents and potentials and potentials of control specimens were monitored daily, except weekends, during the exposure. Seawater temperature was recorded nine times daily. At the conclusion of the exposures, all specimens were towel-dried, weight loss determined gravimetrically, and specimen appearance recorded. In addition, periodic reweighings of all graphite-epoxy specimens were performed to determine the amount of moisture absorbed in the epoxy and the rate of its subsequent evaporation.

## RESULTS AND DISCUSSION

### EXPERIMENTAL PROBLEMS

Several difficulties were encountered during the exposures. Twice the zinc specimens coupled to graphite epoxy were corroded sufficiently to require replacement. The first of these replacements took place after 32 days of the 120-day exposure at 10 m/s. During replacement, the first zinc was lost as it was being removed. Thus, the total zinc weight loss for this couple could not be accurately determined. The other replacement occurred after 83 days of the 270-day exposure at 0 m/s and occurred without incident. At this time it was discovered that the electrical contact between the zinc and the nickel-aluminum bronze specimen in the 270-day exposure at 0 m/s had not been made. Although this was then corrected the "couple" was actually not electrically connected for the first 83 days of the test. Potential and current data for this couple are therefore not reported during this period.

### SPECIMEN APPEARANCE

After 30-days exposure the wetted surface of the coupled graphite-epoxy specimens were covered with a thin whitish deposit, probably a calcareous deposit. Uncoupled specimens of this same material did not have this deposit, nor did any specimens exposed for longer durations. Otherwise, the graphite-epoxy specimens appeared unaffected by the exposure.

General attack on the nickel-aluminum bronze specimens was present only on the uncoupled control specimens exposed at 10 m/s. Localized corrosion at the fixturing points was experienced on uncoupled specimens exposed for 120 and 270 days. Minor localized corrosion also occurred on the specimen "coupled" for 270 days, but this corrosion likely occurred during the first 83 days of exposure when the specimen was inadvertently uncoupled. It was obvious from specimen appearance that the nickel-aluminum bronze experienced less corrosion when cathodically protected by coupling to zinc.

The appearance of the zinc control specimens is shown in Figure 2 and the coupled specimens in Figure 3. The amount of corrosion appears to increase with increasing exposure duration or velocity. Coupling of the specimens also increased visible corrosion, as would be expected if the zinc were cathodically protecting

the other materials. The morphology of the zinc corrosion was varied, ranging from general attack to localized pitting.

#### WEIGHT LOSS

Results of the weight-loss measurements on the zinc specimens are presented in the first part of Table 1. The weight loss of zinc increases with increasing velocity or exposure duration. Weight loss also increases upon coupling, indicating the sacrificial nature of the zinc in the couple. The difference in weight loss between zincs coupled to graphite-epoxy and those coupled to nickel-aluminum bronze are small compared to the effect of coupling in general, and the bronze tended to cause slightly more weight loss than the composite. Thus, both cathode materials have similar effects on the zinc corrosion. In the 120-day exposure at 10 m/s the weight loss of one specimen was not available and had to be estimated from the galvanic current. Given this fact, agreement is good. In the 270-day exposure, the nickel-aluminum bronze specimen was uncoupled for the early part of the exposure, thus invalidating the data from this specimen. In this case no comparison is possible.

Table 1 also presents weight-loss data for nickel-aluminum bronze. Weight loss of uncoupled specimens increased with increasing exposure, velocity, and duration. With the exception of the "coupled" 270-day exposure, coupling of the specimens to zinc reduced the weight loss to a fairly uniform value which was independent of exposure duration or velocity. Thus, the majority of the small amount of corrosion of the bronze occurred during the first 31 days of exposure. The nickel-aluminum bronze was therefore being cathodically protected by the sacrificial corrosion of the zinc.

The graphite-epoxy specimens gained weight during testing due to water absorption and buildup of calcareous deposits. This will be discussed in detail later.

#### POTENTIALS

Potential data for all couples and control specimens is summarized statistically in Table 2 and plotted in Figures 4 through 7. Figures 4 and 5 are for uncoupled control specimens at flow velocities of 0 and 10 m/s, respectively.

TABLE 1 - CORROSION OF METAL SPECIMENS

Exposure Duration (days)	Velocity (m/s)	Weight Loss of Zinc (g)			Weight Loss of Nickel-Aluminum Bronze (g)	
		Uncoupled	Coupled to Nickel-Aluminum Bronze	Coupled to Graphite-Epoxy	Uncoupled	Coupled to Zinc
31	~0	0.137	0.348	0.296	0.087	0.044
	10	1.362	12.861	10.918	0.600	0.045
120	~0	0.513	1.540	1.417	0.184	0.054
	10	2.082	7.882	4.123* + 1.409	0.561	0.039
270	~0	1.732	2.016**	0.624*** + 1.600	0.273	0.130**

\*Specimen lost during replacement after 32 days. Value reported was calculated by integrating the galvanic current over the first 32 days of test and applying Faraday's Law.

\*\*Specimen uncoupled for first 83 days.

\*\*\*Specimen replaced after 83 days.

Values for anode zinc stabilized quickly to around -1000 mV versus Ag/AgCl at 0 m/s and to around -1050 mV at 10 m/s. Potentials for the nickel-aluminum bronze were quite erratic for the first 30 to 60 days but eventually stabilized to around -100 mV at either velocity. The potentials for graphite-epoxy were very noble, stabilizing to about +300 mV after 20-days exposure at either velocity. A slight electropositive drift in the graphite-epoxy potential continued throughout the 270-day exposure period. All of the control specimen potentials were within the range reported elsewhere\* on similar materials.

Potentials of the couples at 0 and 10 m/s flow are plotted in Figures 6 and 7, respectively. The couple potentials under low flow conditions stabilized immediately whereas, under high flow conditions, 30 days were required for stabilization. Regardless of the material to which the zinc was coupled or the flow velocity, the

\*LaQue, F.L. and G.L. Cox, "Some Observations of the Potentials of Metals and Alloys in Seawater," Proc American Society Testing Materials, Vol. 40, p. 670 (1940).

TABLE 2 - POTENTIALS AND CURRENTS OF  
COUPLES AND CONTROLS

	Graphite-Epoxy Potential*	Nickel-Aluminum Bronze Potential*	Zinc Potential*	Zinc/Graphite-Epoxy		Zn/Ni-Al-Br	
				Potential*	Current, mA	Potential*	Current, mA
0 m/s							
<u>31-Day Test</u>							
1-31 Mean	266	-228	-1035	-1017	0.29	-1007	0.30
Dev	38.2	27.1	7.0	13.6	0.07	26.5	0.16
<u>120-Day Test</u>							
1-31 Mean	274	-228	-1024	-984	0.93	-985	0.96
Dev	54.6	35.7	19.1	21.8	0.61	25.4	0.70
32-120 Mean	334	-96	-991	-995	0.23	-983	0.26
Dev	20.8	11.4	8.2	18.3	0.10	16.8	0.10
1-120 Mean	296	-176	-1012	-989	0.66	-985	0.68
Dev	52.9	71.2	22.1	20.8	0.59	22.6	0.64
<u>270-Day Test</u>							
1-31 Mean	286	-187	-1028	-1006	1.47	-	-
Dev	62.8	42.6	12.2	24.8	4.00	-	-
32-120 Mean	298	-170	-1013	-998	0.28	-1004	0.31
Dev	24.2	67.9	15.2	19.8	0.08	36.6	0.27
121-270 Mean	355	-92	-1001	-994	0.08	-982	0.10
Dev	16.0	12.4	14.0	9.3	0.03	14.2	0.02
1-120 Mean	293	-177	-1019	-1001	0.69	-1005	1.78
Dev	41.3	60.6	16.0	21.9	2.38	35.8	6.04
1-270 Mean	306	-158	-1016	-999	0.56	-992	0.94
Dev	44.7	64.2	16.0	26.9	2.11	30.5	4.29
10 m/s							
<u>31-Day Test</u>							
1-31 Mean	291	-288	-1043	-893	13.62	-915	13.65
Dev	24.1	13.4	8.2	85.6	5.52	83.3	7.08
<u>120-Day Test</u>							
1-31 Mean	225	-262	-1033	-1001	4.54	-960	7.71
Dev	84.0	29.3	15.5	44.7	4.87	78.3	9.59
32-120 Mean	324	-90	-1047	-1031	0.78	-1039	0.92
Dev	20.5	6.3	8.3	7.9	0.52	8.7	0.49
1-120 Mean	262	-197	-1038	-1013	3.05	-990	5.00
Dev	81.6	86.6	14.7	38.2	4.19	72.2	8.09
*mV versus silver/s chloride.							

couple potentials remained very close to the potentials for uncoupled zinc. This indicates that the polarization resistance of the zinc was much lower than that of the cathodic materials and, thus, the galvanic corrosion occurring on the zinc in all couples was cathode limited; i.e. the controlling reaction kinetics were those at the cathodic material.

#### GALVANIC CURRENTS

Galvanic current data for all couples is summarized statistically in Table 2 and plotted in Figures 8 and 9 for flows of 0 and 10 m/s, respectively. Large variations in current occurred between couples of the same material at the same velocity. Differences in currents between couples containing nickel-aluminum bronze and those containing graphite-epoxy were minimal by comparison. This is in agreement with the conclusions from the weight-loss data presented earlier. Galvanic currents were approximately five times greater at 10 m/s than at 0 m/s. This too agrees with the weight-loss data. Galvanic currents were steadily decreasing over at least the first 150 days of test and possibly throughout the entire 270-day run. Thus, it is likely that exposures longer than 270 days in duration would yield average corrosion rates lower than those in these exposures.

Galvanic current from each exposure was computer-integrated to obtain the total charge passed during the test. This was converted to weight loss by using Faraday's law:

$$W = QE$$

where W = weight loss, grams,

Q = total charge passed, coulombs, and

E - electrochemical equivalent, grams/coulomb.

A comparison of values of weight loss calculated in this manner with actual measured weight loss is tabulated in Table 3 and plotted in Figure 10. The correlation coefficient for this data is 0.991 and a least-square straight-line best-fit curve has a slope of 0.968, indicating that the zinc was operating at close to 100% electrochemical efficiency.

TABLE 3 - COMPARISON OF MEASURED AND  
CALCULATED WEIGHT LOSSES

Days	Flow Velocity (m/s)	Weight Loss of Zinc Coupled to	Calculated from Current (g)	Actual (g)
31	~ 0	Graphite-Epoxy Nickel-Aluminum Bronze	0.258 0.246	0.296 0.348
31	10	Graphite-Epoxy Nickel-Aluminum Bronze	12.288 12.477	10.918 12.871
120	~ 0	Graphite-Epoxy Nickel-Aluminum Bronze	1.417 1.401	1.417 1.540
120	10	Graphite-Epoxy Nickel-Aluminum Bronze	1.387* 8.167	1.409** 7.882
270	~ 0	Graphite-Epoxy Nickel-Aluminum Bronze	3.608 2.927	2.224 2.016
*First 83 days of test excluded.				
**Value for specimen exposed on day 84.				

#### WATER PICKUP OF GRAPHITE-EPOXY

The graphite-epoxy specimens were towel-dried after exposure, and their weight determined. The weight gains thus measured were due to water pickup of the epoxy resin and buildup of calcareous deposits on the exposed surface. Weight gains of 15 to 70 mg (0.1 to 0.4% of the total specimen weight) were measured. The exact value was neither a function of exposure duration nor of the presence or absence of cathodic protection. The amount of water pickup was determined by allowing the absorbed water to evaporate. This was done by allowing the specimens to air-dry for several thousand hours and monitoring loss of weight as a function of time. A plot of weight decay versus time after removal from exposure is presented as Figure 11. The total weight loss over the period of measurement, which should be equal to the water absorbed during exposure, ranged from 17 to 28 mg and was not a function of exposure duration or cathodic protection (coupling to zinc). However, the rate of the decay was interesting. With one exception, the rate of weight decay was greatest over the first 100 hours for specimens with the shortest exposure and was least

for specimens with the longest exposure. This could be due to an increase of depth of penetration of water into the specimen with increasing exposure duration.

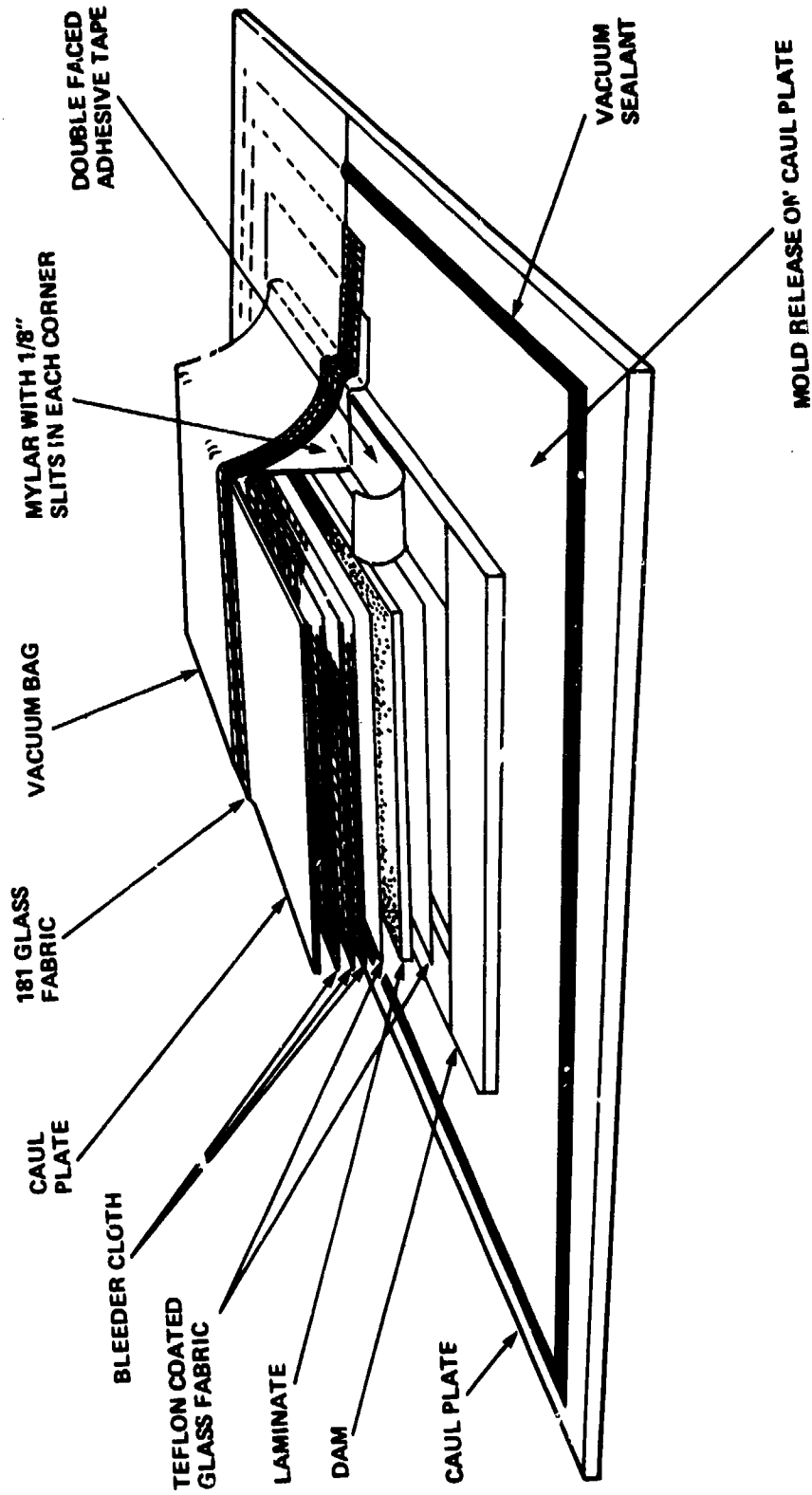
#### SUMMARY AND CONCLUSIONS

1. The graphite-epoxy specimens did not cause a significantly different current demand on a zinc sacrificial cathodic protection system in seawater than did nickel-aluminum bronze. This was true for exposure velocities of 0 and 10 m/s and for durations up to 270 days and was verified by weight loss and current measurements. Similar behavior was exhibited in short-term stepped-potential tests conducted previously on similar materials.

2. It is probable that nickel-aluminum bronze components of a cathodically protected structure in seawater could be replaced with graphite-epoxy without the necessity for redesign of the cathodic protection system.

3. It should be noted that the graphite-epoxy composite presents a worst-case condition in that the surfaces were made electrically uninsulated by grinding off the insulating epoxy surface layer. Normally, the epoxy coating on these surfaces would prevent the graphite fibers from exerting any significant demand on the cathodic protection system.





**Fabrication Cycle Note:**  
 Debulk - Vacuum and heat (121°C) (250°F) 1 hour; cure - raise pressure (655 KN/m<sup>2</sup>) (95 psi) and vent vacuum to atmosphere; maintain pressure (655 KN/m<sup>2</sup>) (95 psi); raise and maintain heat (177°C) (350°F) 2 hours; cool to room temperature at pressure.

Figure 1 - Vacuum Bag/Autoclave Procedure for Laminate Fabrication

~0 m/s

10 m/s

31 Days

120 Days

270 Days

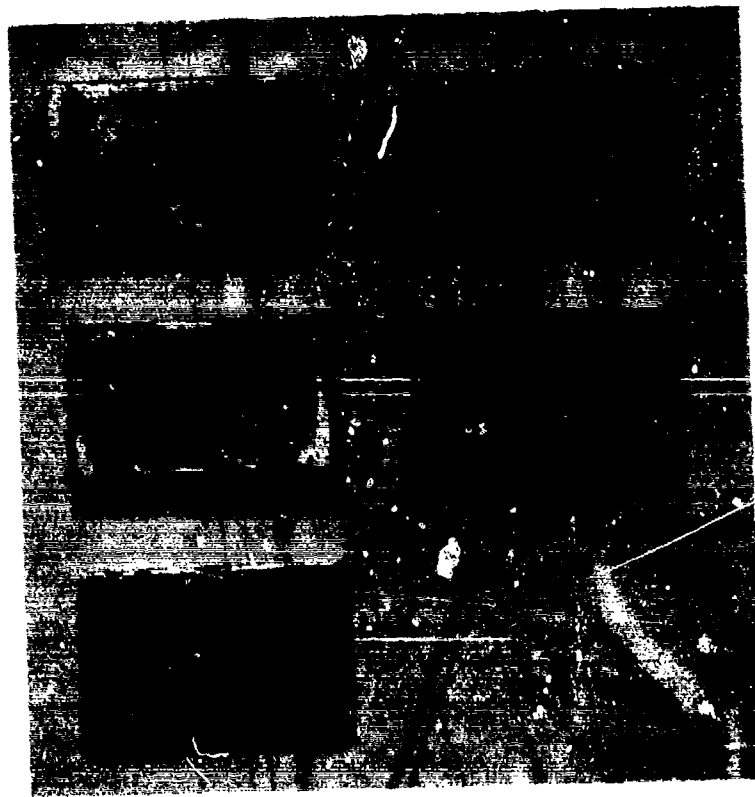


Figure 2 - Zinc Control Specimens


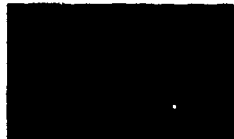





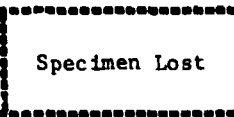



Duration	Coupled to	~0 m/s	10 m/s
31 Days	Ni-Al-Bronze		
	Gr-Epoxy		
120 Days	Ni-Al-Bronze		
	Gr-Epoxy		<p>Days 1-32 </p> <p>Days 33-120 </p>
270 Days	Ni-Al-Bronze	 Not Connected During Days 1-83	
	Gr-Epoxy	 Days 1-83  Days 84-120	

Figure 3 - Coupled Zinc Specimens

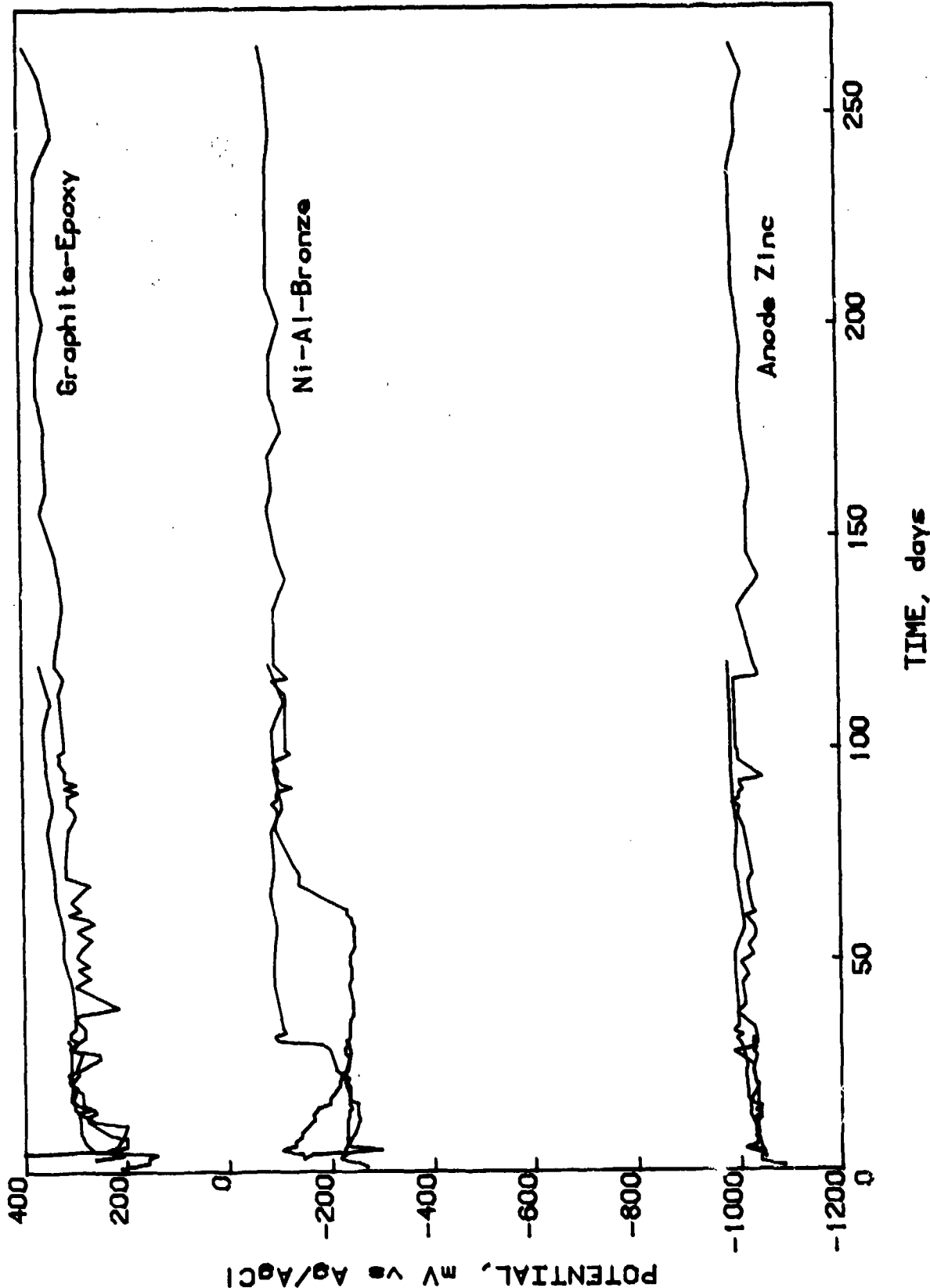


Figure 4 - Potential Data for Uncoupled Control Specimens at Flow Velocity of 0 m/s

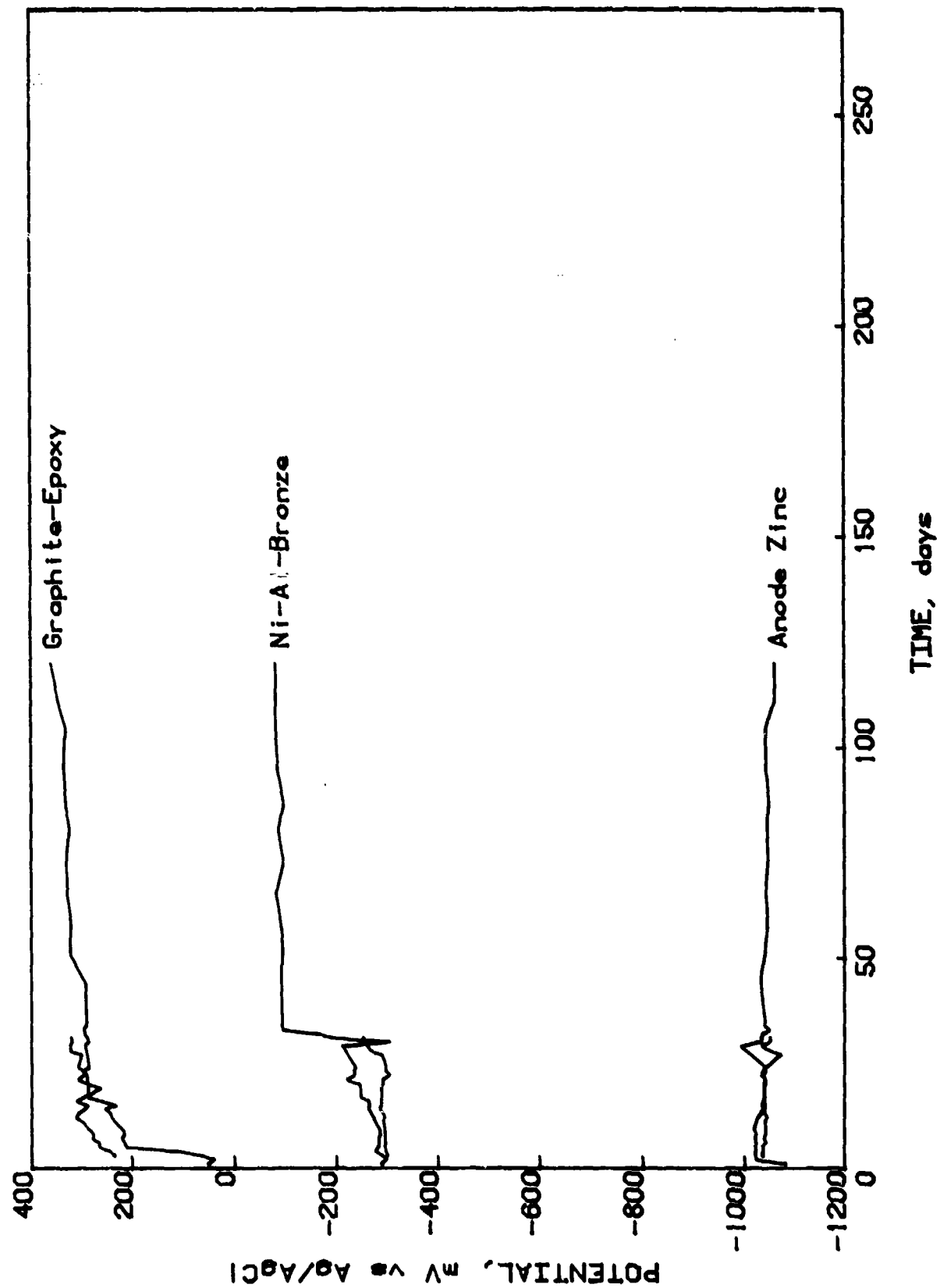


Figure 5 - Potential Data for Uncoupled Control Specimens at Flow Velocity of 10 m/s

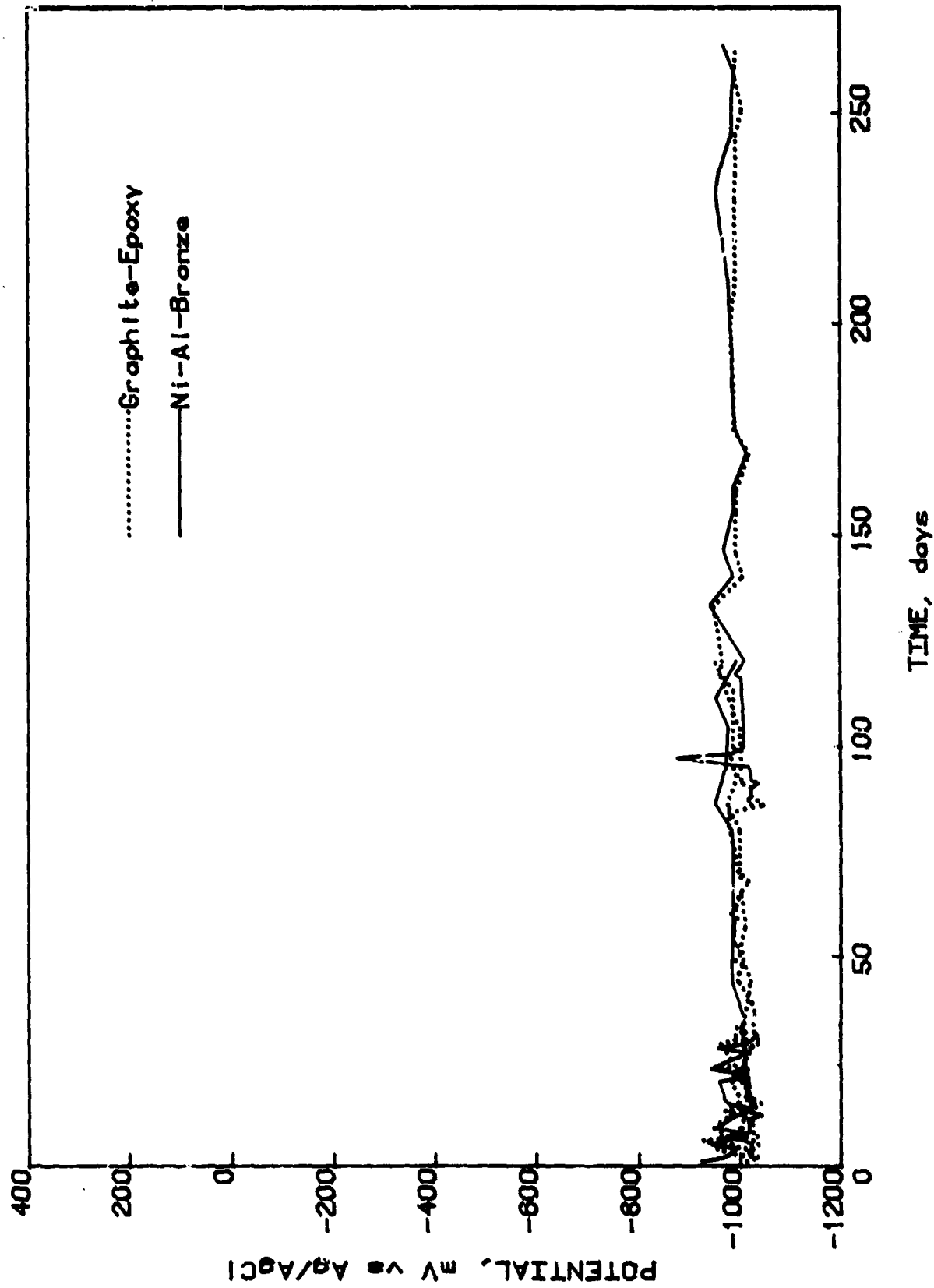


Figure 6 - Potential Data of Coupled Specimens at Flow Velocity of 0 m/s

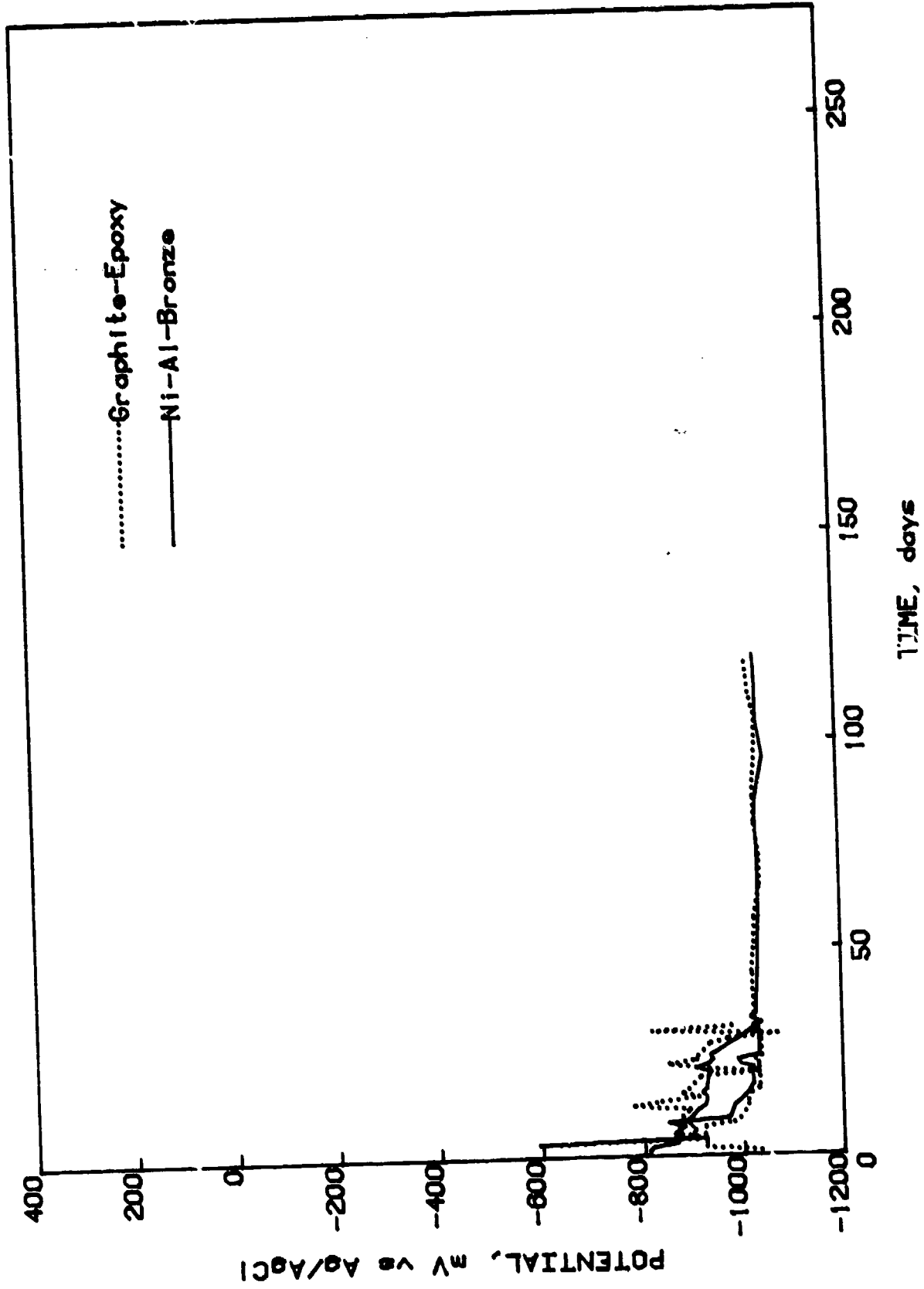


Figure 7 - Potential Data of Coupled Specimens at Flow Velocity of 10 m/s

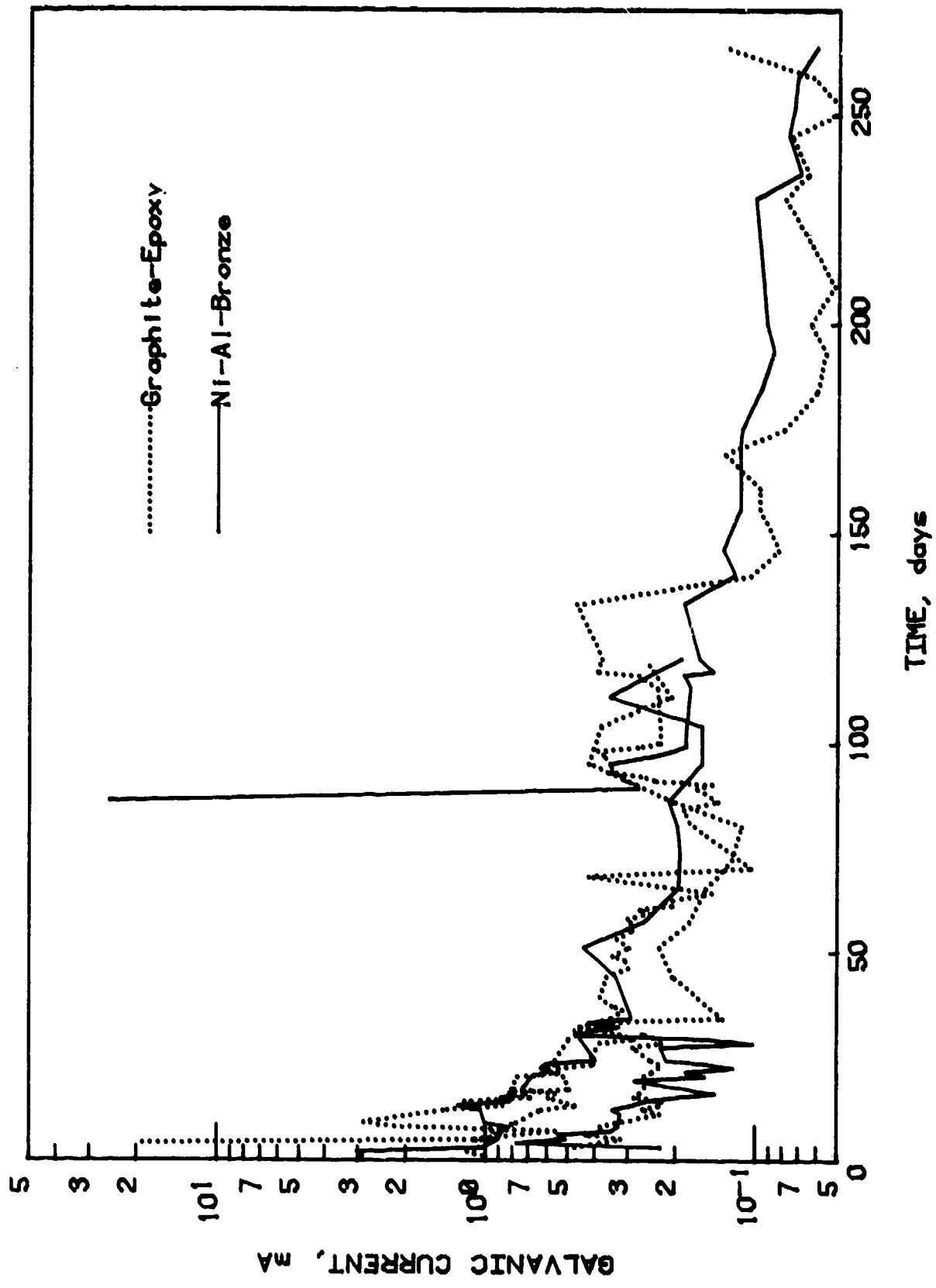


Figure 8 - Galvanic Current Data for Coupled Specimens at Flow Velocity of 0 m/s



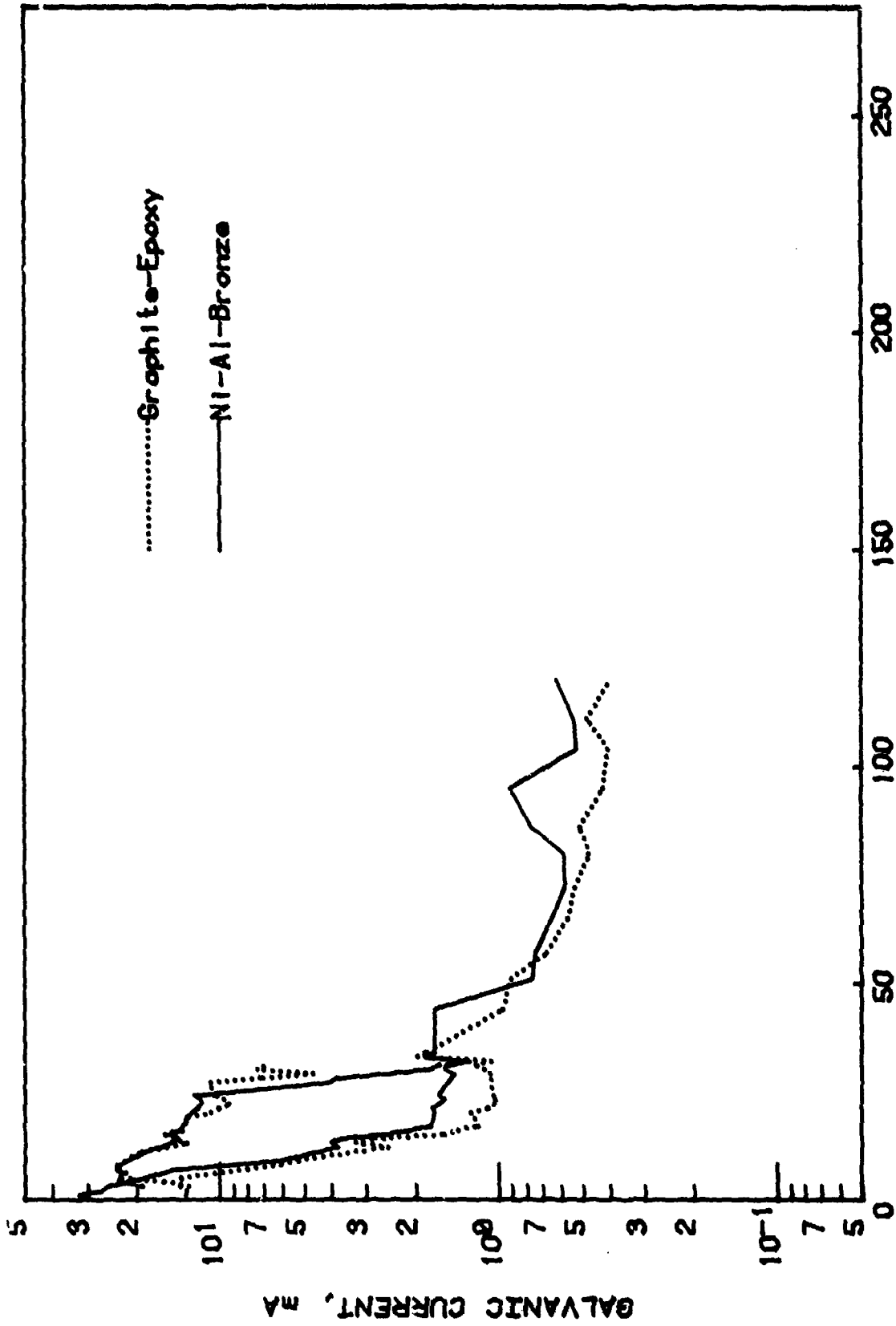


Figure 9 - Galvanic Current Data for Coupled Specimens at Flow Velocity of 10 m/s

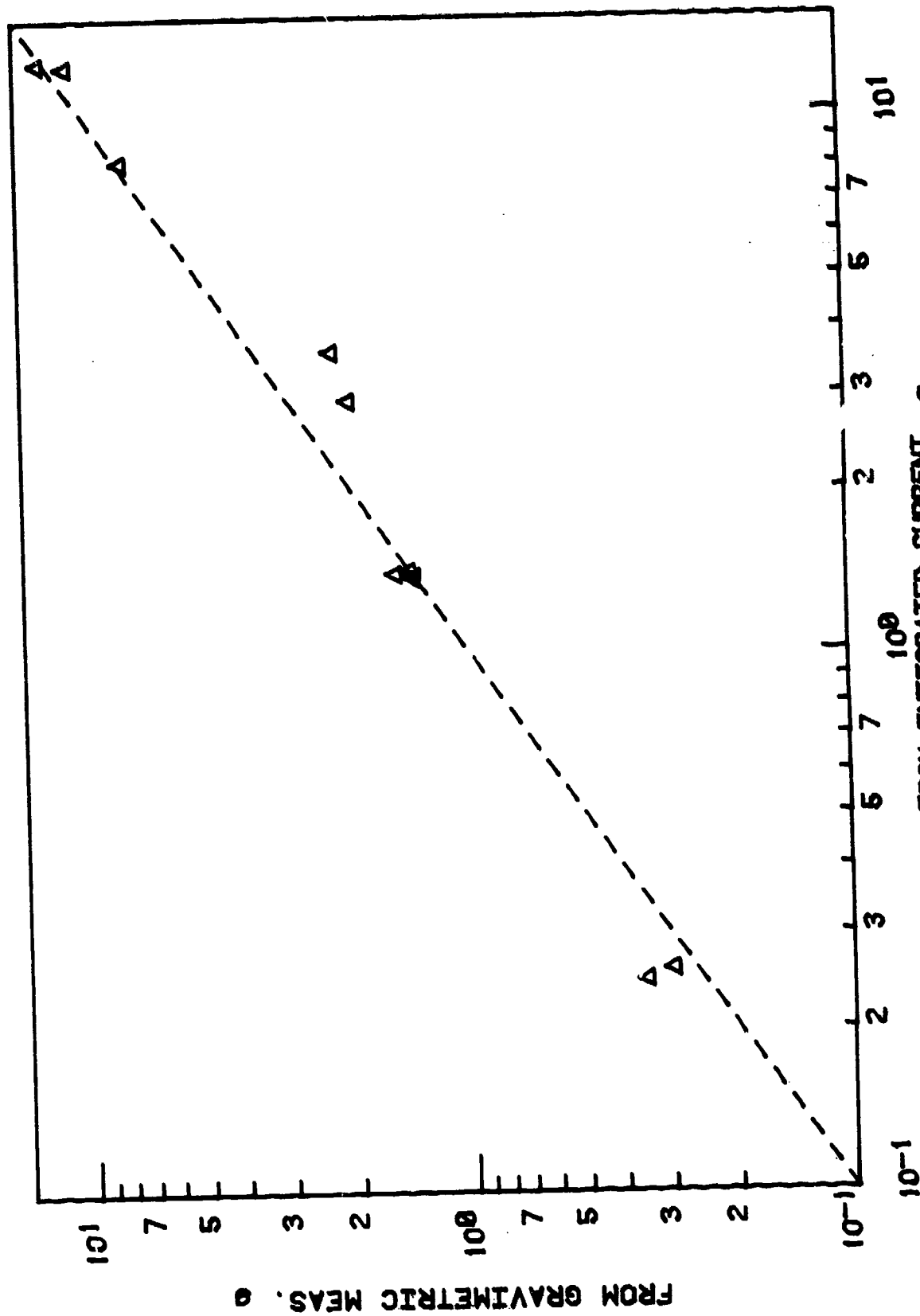


Figure 10 - Comparison of Weight-Loss Techniques for Zinc Anode Materials

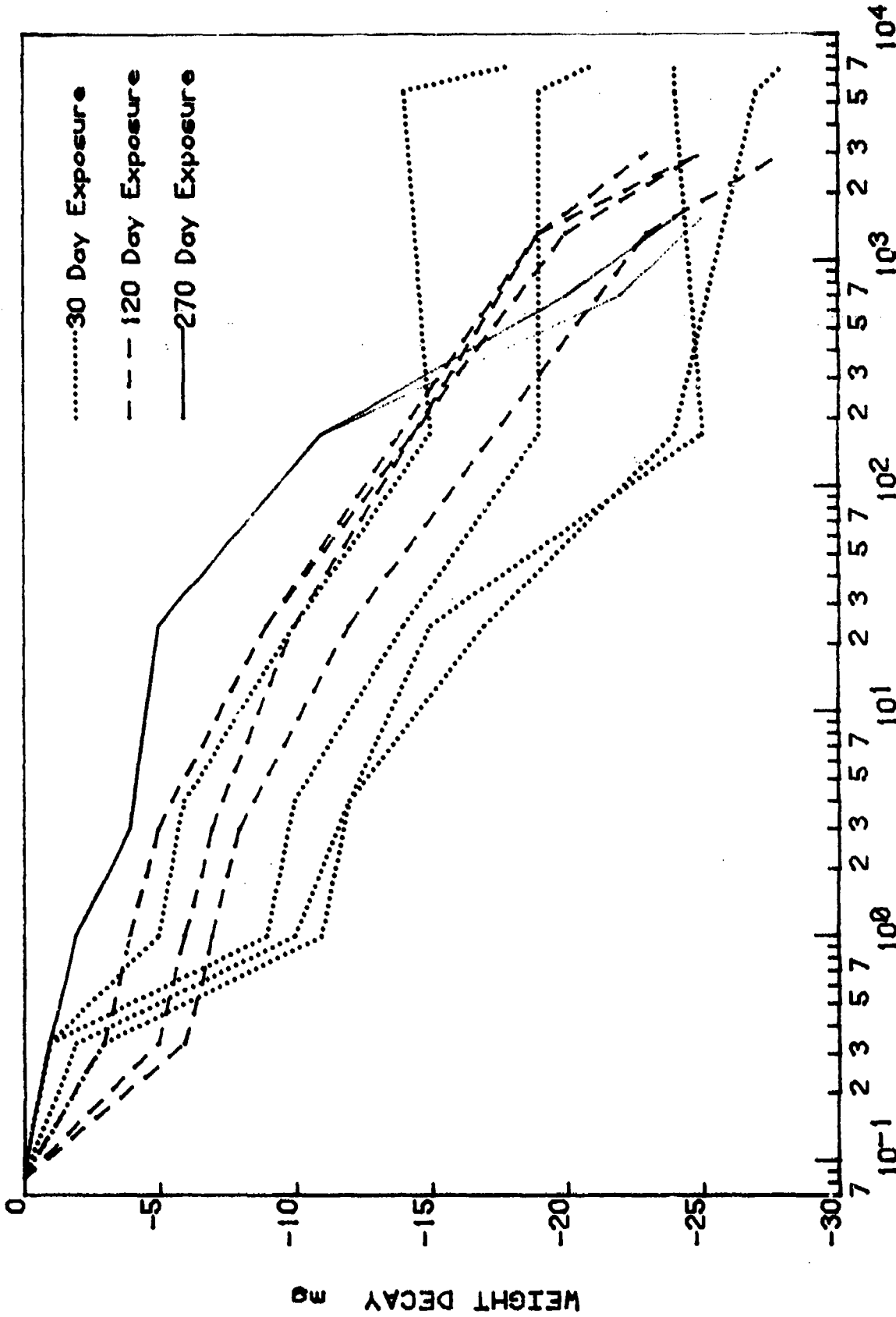


Figure 11 - Weight Decay of Graphite-Epoxy Specimens  
Removal from Water Exposure

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