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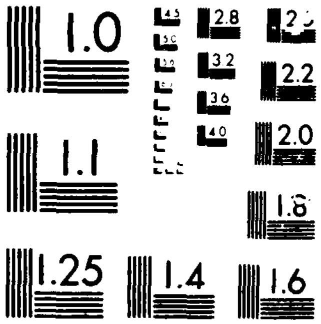
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**US Army Corps
of Engineers**
Construction Engineering
Research Laboratory

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USA-CERL TECHNICAL REPORT M-87/03
December 1986

Development of New Materials and Design Configurations to Improve Ceramic Anode Performance

by
J. Boy
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*This report documents research to improve per*formance of the CERANODE, a ceramic anode developed by the U.S. Army Construction Engineering Research Laboratory (USA-CERL) to provide cathodic protection of Civil Works structures. New materials and design configurations for constructing the CERANODE were investigated to find those that would provide the greatest protection from ice and debris impacts, have low material costs, and provide a long service life. Of the plasma-sprayed coatings in*vestigated, the undoped ferrite* (Fe_3O_4) *had the* longest dissolution rates. Mixed metal oxide coatings such as ruthenium oxide/titanium dioxide and iridium oxide/titanium dioxide have dissolution rates of less than 0.001 g per ampere-year and also show significant advantages as coatings for anodes. The new flat anode configurations developed minimize exposure to damaging ice and debris impacts and are currently being field tested.

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This report documents research to improve performance of the CERANODE, a ceramic anode developed by the U.S. Army Construction Engineering Research Laboratory (USA-CERL) to provide cathodic protection of Civil Works structures. New materials and design configurations for constructing the CERANODE were investigated to find those that would provide the greatest protection from ice and debris impacts, have low material costs, and provide a long service life. Of the plasma-sprayed coatings investigated, the undoped ferrite (Fe_3O_4) had the longest dissolution rates. Mixed metal oxide coatings such as ruthenium oxide titanium dioxide and iridium oxide/titanium dioxide have dissolution rates of less than 0.001 g per ampere-year and also show significant advantages as coatings for anodes. The new flat anode configurations developed minimize exposure to damaging ice and debris impacts and are currently being field tested.

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FOREWORD

This study was conducted for the Directorate of Civil Works, Headquarters, U.S. Army Corps of Engineers (HQUSACE), under CWIS 31204 (Corrosion Mitigation in Civil Works Projects). The research was conducted by the Engineering and Materials (EM) Division, U.S. Army Construction Engineering Research Laboratory (USA-CERL). The OCE Technical Monitor was Mr. Robert Kinsell (DAEN-CWE-E). Dr. R. Quattrone is Chief of USA-CERL-EM.

COL Norman C. Hintz is Commander and Director of USA-CERL, and Dr. L. R. Shaffer is Technical Director.

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DEVELOPMENT OF NEW MATERIALS AND DESIGN CONFIGURATIONS TO IMPROVE CERAMIC ANODE PERFORMANCE

1 INTRODUCTION

Background

The U.S. Army Corps of Engineers is responsible for maintaining many types of metallic structures. A commonly used corrosion prevention technique is cathodic protection—the application of a small electric current from an external source to the corroding structure. One ampere of current will stop the corrosion of 500 sq ft* of uncoated steel. The current is supplied through the anode and eventually consumes it. The anode is the positive terminal in the cathodic protection circuit, and the structure is the negative terminal.

For the past 30 years, two materials—silicon-iron and graphite—have been used as anodes for cathodic protection. However, these materials are brittle, cannot be machined or welded, and have consumption rates on the order of pounds per ampere-year (i.e., if 1 A of current is passed through the anode for 1 year, 1 lb of material will be consumed). Consequently, large anodes are required, making the anode vulnerable to debris and ice damage and also prone to field installation problems.

The ceramic anode (CERANODE)[1], developed and patented by the U.S. Army Construction Engineering Research Laboratory (USA-CERL), is entirely factory-assembled. The factory-fabricated, anode-to-wire connection minimizes field installation problems, such as those experienced with silicon-iron or graphite anodes, which can lead to failure. In addition, elimination of the field-made electrical connection allows installation of the ceramic anode by nonspecialized personnel.

The consumption rate of the CERANODE conducting material is 200 times less than that of silicon-iron or graphite anodes. As a result, the ceramic anode is much smaller than the silicon-iron anode (100 times by weight) and can be installed in areas prohibitively small for graphite and silicon-iron anodes. The CERANODE's small size also makes it less susceptible to ice and debris damage, so it is ideally suited for cathodic protection of lock and dam structures.

In previous investigations[2], an electrically conducting ceramic was plasma-sprayed onto a titanium or niobium substrate (Figure 1*). Plasma spraying provides excellent electrical and mechanical ceramic-to-metal interface properties. When the conducting ceramic is plasma-sprayed onto a valve metal substrate, it provides a path of electrical continuity, allowing anode current to pass easily. If the ceramic coating has interconnected porosity or is damaged, the valve metal substrate will passivate (i.e., cease passing current) and protect itself from further corrosion damage. Metal substrates are also easier to machine than ceramic ones, and thus lend themselves to more efficient designs.

A disadvantage of using valve metal substrates is their high cost. Niobium costs up to 18 times more than titanium, while tantalum can cost 30 times more. These metals are also typically more difficult to machine than most standard engineering metals. Thus, the need to reduce substrate material and machining costs is crucial.

Objective

The objective of this investigation was to improve the performance and lower the costs of the CERANODE through development of inexpensive and slowly consumed materials as well as new design and configurations that would further minimize exposure to ice and debris.

Approach

New materials were fabricated and their performance evaluated under anodic polarization in various environments to determine materials suited for impressed current anodes. New design configurations to lower material costs, facilitate manufacturing, and minimize exposure to debris and ice damage were developed and evaluated.

Mode of Technology Transfer

It is recommended that the information in this report be used to develop procurement specifications for

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*Metric conversion factors: 1 sq ft = .0929m^2; 1 lb = .4535 kg; 1 ft = .3048 m

[1] E. G. Segan and A. Kumar, *Preliminary Investigation of Ceramic Coated Anodes for Cathodic Protection*, Technical Report M-333 ADA133440 (U.S. Army Construction Engineering Research Laboratory [USA-CERL], 1983).

[2] USA-CERL Technical Report M-333; J. H. Boy, M. Olson, V. Hock, and A. Kumar, *Improved Ceramic Anodes for Corrosion Protection*, Technical Report M-85/02/ADA149492 (USA-CERL, 1984).

*Figures are located at the end of the report (see p 13).

incorporation in Corps of Engineers Draft Guide Specification 2310, *Cathodic Protection of Lock Gates*.

2 IMPROVING CERANODE PERFORMANCE

Previous attempts to decrease the material cost of the CERANODE's solid metal substrate by multi-layer plasma spraying investigated the electroplating of tantalum onto low-cost substrates.[3] Stainless steel, 316L, and copper, rather than tantalum or other valve metal substrates, had been chosen because of their lower cost and ease of machining. Hemispherical substrates were machined for the anode configuration shown in Figure 1. The substrates were polarized in a molten tantalum fluoride salt solution maintained at more than 800°C. A 0.125-mm coating of tantalum was deposited at a rate of 6.35 μm/hour under an argon atmosphere. The electroplated substrate was then plasma-sprayed with a 0.5-mm conducting lithium ferrite layer.

The plasma-sprayed ferrite on tantalum-coated substrate was subjected to anodic polarization testing by passing 40 mA of current through the anodes in 3.5 percent sodium chloride solution for 24 hours. The tests indicated that although the anodes with just the tantalum electrodeposited coatings passivated, indicating good quality of the valve metal coatings, the tantalum coatings that were subsequently plasma-sprayed failed. During electrochemical testing, some aspect of the plasma spray process abraded or otherwise damaged the thin electrodeposited tantalum coating, exposing the underlying base metal. When the base metal was exposed to the electrolyte, it corroded and caused catastrophic failure in the anode. Further investigation of this system was therefore discontinued.

In a continued effort to improve the performance of the CERANODE and lower its costs, the current research developed and evaluated several materials. The materials were tested in both fresh water and saltwater. In addition, new design configurations were developed that would minimize the anode's exposure to damaging impacts.

Materials

Plasma-Sprayed Ferrite

The original ceramic anode (CERANODE) consisted of plasma-sprayed lithium ferrite on a niobium hemispherical substrate. To improve the anode's properties, other ferrite compositions were investigated. These new ferrite coatings were plasma-sprayed on niobium substrates and tested at various current densities in both fresh water and saltwater to determine their anodic dissolution characteristics. Lithium-zinc ferrite, manganese ferrite, and iron oxide were plasma-sprayed onto niobium coupons. The dissolution rates were determined by weight loss of the test coupons. The pure iron oxide yielded the lowest anodic dissolution of 3.27 g/ampere-year in saltwater and 9.1 g/ampere-year in freshwater. The lithium-zinc ferrite had dissolution rates of 7.2 g/ampere-year in saltwater and 12 g/ampere-year in fresh water (Table 1). The plasma-sprayed manganese ferrite yielded very high dissolution rates of 120 g/ampere-year in fresh water (Table 2). The manganese ferrite tested in saltwater had an initial dissolution rate of 10 g/ampere-year, which decreased during the 14-day test to 3.5 g/ampere-year; its average dissolution rate in saltwater was 8.2 g/ampere-year.

Lithium-Doped Nickel Oxide

Samples of nickel oxide doped with 10 percent mole of lithium oxide were prepared using standard ceramic processing techniques and were sintered in air at 1300°C. The samples were tested in an electrochemical cell and proved to be electrically conducting. Although the samples continued to pass current, there was significant dissolution and flaking of the sample into the solution. Therefore, further testing of this system was terminated.

The same composition was also plasma-sprayed onto niobium sheet and tested in electrochemical cells containing fresh water and saltwater. The cells were energized at the end of a working day and by the next morning, the coatings that were in fresh water had totally dissolved, and no current was flowing in the circuit. Thus, nickel oxide doped with lithium was found to be unsuitable for anodic applications. Because of the poor performance in fresh water, saltwater tests were discontinued.

Mixed Metal Oxide Coatings

Metal oxides such as ruthenium and iridium oxides (RuO_2 and IrO_2) are known to exhibit metallic electrical conductivity over a wide range of temperatures[4].

---

[3] J. H. Boy, M. Olson, V. Hock, and A. Kumar.

[4] J. B. Goodenough, "Metallic Oxides," *Progress in Solid State Chemistry*, Vol 5, edited by H. Riess (Pergamon Press, 1971), p 363.

Table 1
Anodic Dissolution Rates of Plasma-Sprayed Ferrites

Material	Anode Current (A)	Current Density (A/m²)	Dissolution Rate (g/A-yr)	Electrolyte
Li-Zn Ferrite*	0.03	47	11-14	Fresh Water
Li-Zn Ferrite	0.12	186	11	Fresh Water
Li-Zn Ferrite	1.3	2015	4-5	3.5% NaCl Soln.
Li-Zn Ferrite	0.03	47	10	3.5% NaCl Soln.
Ferrite**	0.12	186	6.91	Fresh Water
Ferrite	0.12	186	10.04	Fresh Water
Ferrite	0.12	186	10.30	Fresh Water
Ferrite	0.12	186	2.95	3.5% NaCl Soln.
Ferrite	0.12	186	3.87	3.5% NaCl Soln.
Ferrite	0.12	186	3.00	3.5% NaCl Soln.

*$Li_{0.9}Zn_{0.1}Fe_2O_4$

**Fe_3O_4

Table 2
Dissolution Rates of Plasma-Sprayed Manganese Ferrite Coupons

Coupon* No.	Wear Rate For The Next 3 Days**	Wear Rate For The Next 5 Days	Wear Rate For The Next 6 Days	Wear Rate For The Entire 14.146 Days	Average
123	79.98 g/A-yr	136.51 g/A-yr	43.70 g/A-yr	123.15 g/A-yr	
124	74.01 g/A-yr	125.80 g/A-yr	58.85 g/A-yr	119.53 g/A-yr	120.01 g/A-yr
125	78.27 g/A-yr	123.61 g/A-yr	54.94 g/A-yr	117.34 g/A-yr	
126	11.56 g/A-yr	5.78 g/A-yr	3.61 g/A-yr	8.45 g/A-yr	
127	11.36 g/A-yr	6.27 g/A-yr	3.12 g/A-yr	8.33 g/A-yr	8.22 g/A-yr
128	9.63 g/A-yr	5.78 g/A-yr	3.61 g/A-yr	7.88 g/A-yr	

*Coupons No. 123-125 were in an electrolyte of fresh water (Dayton, Ohio tap water).
Coupons No. 126-128 were in an electrolyte of 3.5 percent NaCl in tap water.
Current density for all coupons was 186 A/m².
**Electrolyte was changed only after the first 3 days of operation.

The main advantages of fabricating anodes from these materials are their very low resistivity (<0.001 ohm-cm) and their very low dissolution rates (0.001 g per ampere-year at 10 A/sq ft).

Mixed metal oxide anodes can be fabricated by a variety of ceramic processing techniques.[5] IrO_2 or RuO_2 films a few microns thick can be deposited onto inert metallic substrates at comparatively low temperatures by the decomposition of metal chlorides such as $RuCl_3$ in air. In comparison to a solid material, the use of an oxide film on a metal substrate minimizes the material costs of using oxides derived from precious metals. Repeated painting and firing can be used to achieve the desired thickness of the coating.

Alloying RuO_2 or IrO_2 with TiO_2 is known to form a solid solution, increasing chemical stability, yet not significantly degrading the electrical conductivity[6]. This type of anode is commercially available under the registered trademark LIDA®, and has been widely used in the production of chlorine and in cathodic protection

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[5] H. B. Beer, U.S. Patent No. 2,840,443, October 1974.

[6] K. J. O'Leary, T. J. Navin, "Morphology of Dimensionally Stable Anodes," *Proceedings of Chlorine Bicentennial Symposium* (1974).

systems. The LIDA® anode consists of a titanium substrate with a precious metal oxide coating (Figure 2). The anodic dissolution of the LIDA® anode has been less than 0.001 g ampere-hour in industrial applications (Figure 3). The hardness of precious metal oxide coatings is about 6 on the Moh's hardness scale. Thus, the LIDA® anodes are far more resistant to abrasion than platinum-clad anodes. This is particularly important for cathodic protection applications where the anodes are exposed to impact or abrasion. The LIDA® anodes are available in a variety of geometries, including tubular and mesh-expanded configurations. These anodes have been demonstrated in cathodic protection systems in seawater and saline-mud applications (Tables 3 and 4). However, flat disk or button configurations that would be most suitable for water applications are not available.

Unfortunately, the working potentials that may be applied to LIDA® anodes are limited by the titanium substrate. Studies[7] have shown that the LIDA® substrate material (titanium) is susceptible to pitting when polarized to 10 V. Figure 4 shows a potentiodynamic scan of a bare titanium substrate. The protective oxide coating formed on the titanium breaks down when more than 10 V are applied. Therefore, if there are major bare areas in the coating, or if the coating is subsequently abraded or otherwise damaged during use, and the anode is then polarized with more than 10 V, the exposed titanium metal surface will pit. Pitting could lead to failure of the anode and catastrophic failure of the cathodic protection system. However, the LIDA® anode's manufacturer claims that small scratches and minor defects will not cause the electrical field distribution at the defect to be much different from that at the coated surface. The voltage drop across the interface will remain below the breakdown voltage of titanium.

To avoid possible failure of the precious-metal-coated anodes due to pitting of the titanium substrate, USA-CERL is investigating the use of other materials as substrates for precious-metal, oxide-coated anodes. Niobium and tantalum can withstand much higher polarization (>100 V) before pitting begins. However, these materials are susceptible to rapid oxidation during the thermal treatments required to convert the metal chloride solutions to oxide (i.e., the substrate is heated to 400°C to 500°C in an oxidizing atmosphere to convert the salt solution directly to the oxide). At this time, the painting and firing process cannot be applied to niobium substrates due to substrate oxidation. Research is being conducted to find a way to minimize substrate oxidation by controlling the atmosphere during the thermal treatment of the precious metal oxide coatings.

Design Configurations

Damage from ice and debris is a major cause of cathodic protection anode failure in water projects. To solve this problem, APS Materials, Inc., which has been licensed to manufacture the CERANODE, is working closely with USA-CERL on several new anode configurations that would minimize ice and debris damage as well as machining costs.

A flat anode has been developed which consists of a niobium plate with a plasma-sprayed ferrite or mixed metal oxide coating. Two types of flat anodes were developed for fresh water and saltwater applications. The ruthenium oxide/titanium dioxide coating, which reacts well with sodium, was chosen for saltwater applications. The iridium oxide/titanium dioxide coating was applied to a titanium substrate to yield an anode suitable for use in fresh water. Figure 5 shows the design for the mixed-metal oxide coating, titanium substrate anode. The flat substrate reduces the anode's material costs and requires less machining which allows it to be manufactured more easily and lowers its total costs.

To further reduce the damaging effects of impacts, USA-CERL and APS Materials, Inc. jointly designed and developed a protective shield for the flat anode. The ceramic anode rests in the polyurethane plastic (Figure 6a and 6b). While the shield leaves the coated anode face exposed, it protects the perimeter and the uncoated back of the anode from collisions.

Both the round and flat ceramic anodes can be used to protect many different types of metallic structures from corrosion. New configurations are being developed and tested. Ceramic anodes have been incorporated into the design of cathodic protection systems that have been field-tested on water storage tanks, underground pipes, and lock gates. They can also be incorporated into cathodic protection systems for other structures.

New fabrication techniques, such as ion plating, are also being developed to yield a perpetual anode coating, such as niobium-doped titanium oxide or other mixed metal oxides. Such techniques should yield a

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[7] *The LIDA Difference: Its Impact on Cathodic Protection, LIDA Impressed Current Systems, Seminar Manual* (General Cathodic Protection Services, Inc.).

Table 3

Locations of Impressed Current Cathodic Protection Systems Employing LIDA

Saras Jetty	Cagliari, Italy	19[illegible]
Ute Power Station	Montevideo, Uruguay	1972
Enel Nuclear Power Station	Caorso, Italy	1974
ISAB Jetty	Siracusa, Italy	1974
Moalli Sand Collection Tanks	Siracusa, Italy	[illegible]
Boke Jetty	Guinea	19[illegible]
Kenitra Power Station	Morocco	1976
Candiota II Power Station	Porto Alegre, Brazil	1976
Enel S. Gilla Power Station	Cagliari, Italy	1977
Military Arsenal	Messina, Italy	1977
Mediterranea Jetty	Milazzo, Italy	1978
Mediterranea II Jetty	Milazzo, Italy	1978
AGIP Platform	Gela, Italy	1979
Sarjah Power Station	Saudi Arabia	1979
Skikda Power Station	Libya	1979
Cassa Per Il Mazzogiorno Jetty	Manfredonia, Italy	1979
Esmeraldas Power Station	Brazil	1979
Mohammedia Power Station	Morocco	1979
AGIP Platform	Gela, Italy	1980
Progetto S. Marco Platform	Indian Ocean	1980
Punta Cugno Jetty	Augusta, Italy	1980
Enel Power Station	Montalcone, Italy	1980
AGIP Platform	Ravenna, Italy	1980
Capri Underwater Aqueduct	Capri, Italy	1980
Snam Gas Pipeline	Monselice, Italy	1981
Snam Gas Pipeline	Bologna, Italy	1981
Metanocitta' (ENI) Gas Network	Chioggia, Italy	1981
AGIP Platform	Porto Corsini, Italy	1982

Table 4

LIDA® Performance Data

Date Of Installation	Type Of Installation	Place Of Installation	No. Of Anodes	Dimensions (n.m)	Working Current Density (A/m^2)	Note
101972	Seawater Power Station Condenser	Montevideo, Uruguay	No. 105 D	15 × 300	500 A/m^2	Still working
031974	Pier I	Melilli, Sicily	No. 90 D No. 10 D	20 × 1500 20 × 600	700 A/m^2	Still working Some mechanical failures
0[illegible]1975	Sand-Water Separator	Syracuse, Sicily	No. 11 D	20 × 200	80 A/m^2	Still working
0[illegible]1976	Seawater Power Station Condenser	Porto Alegre, Brazil	No. 75 D	16 × 300	500 A/m^2	Still working
061977	Seawater Power Station Condenser	Cagliari, Sardinia	No. 8 D	12 × 400	300 A/m^2	[illegible]
021978	Pier	Milazzo, Sicily	No. 10 D	20 × 600	450 A/m^2	Still working
031979	First Platform	Gela, Sicily	No. 7 D	30 × 800	[illegible] A/m^2	Still working
011980	Second Platform	Gela, Sicily	No. 10 D	30 × 800	150 A/m^2	Still working

3 CONCLUSIONS AND RECOMMENDATIONS

New [illegible] to improve the [illegible] were investigated. The research produced the following conclusions:

1. Alternate anode configurations, including a flat anode, reduce exposure to ice and debris and reduce substrate machining costs.

2. Of the various ferrite-based compositions plasma-sprayed onto niobium substrates, the undoped ferrite (Fe_3O_4) had the lowest dissolution rates (3.3 g/ampere-year in saltwater and 9.1 g/ampere-year in fresh water).

From research, the following [illegible] made:

1. Mixed-metal flat anodes should be installed at field sites for evaluation. Since these anodes have titanium substrates, the applied voltage should be restricted to less than 10 V.

2. Techniques for depositing iridium oxide and titanium dioxide onto niobium substrates should be investigated. Such techniques should yield a perpetual anode coating for anodes which can be subjected to more than 10 V, regardless of their shape.

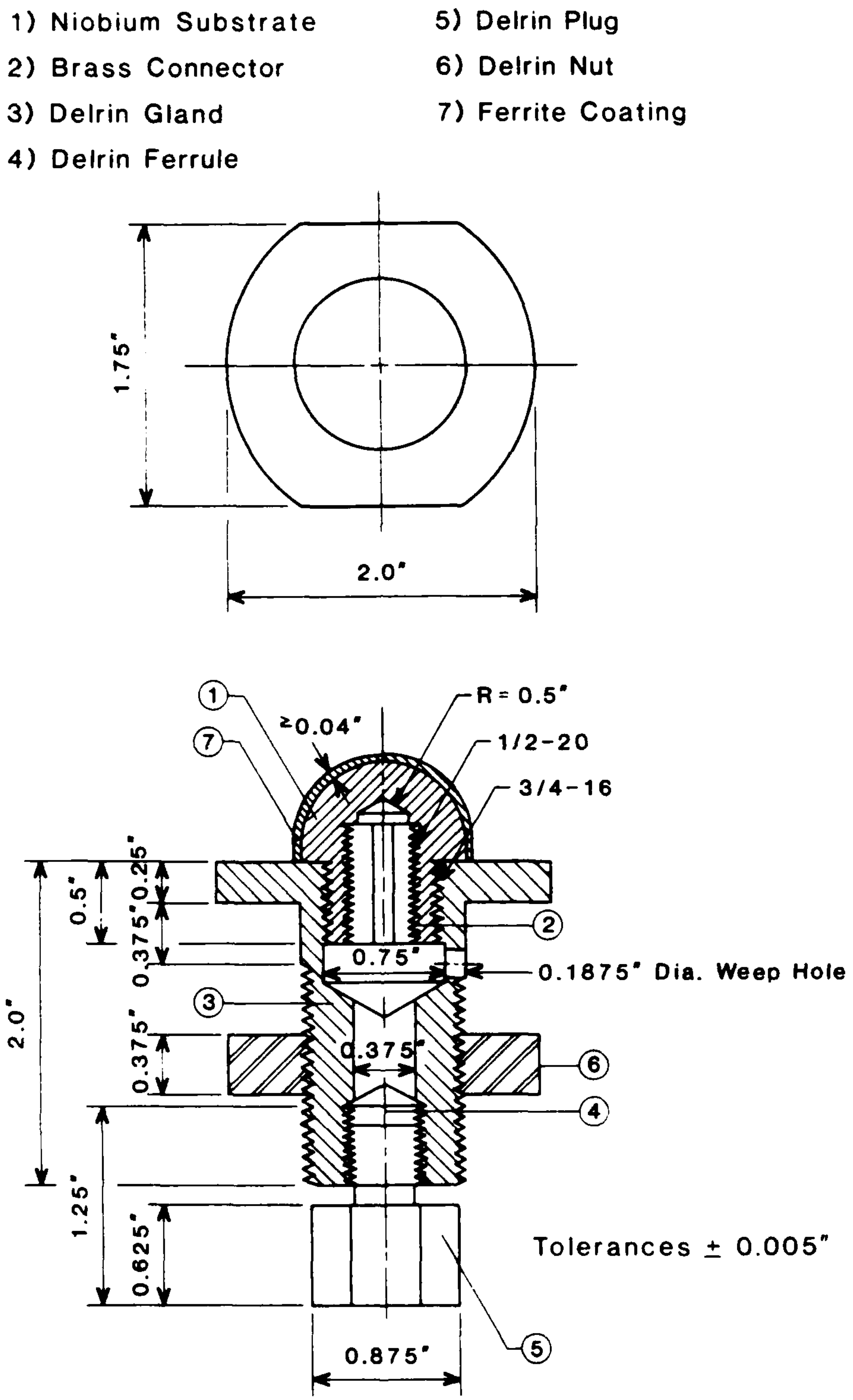

Figure 1. Design of U.S. Army Construction Engineering Research Laboratory (USA-CERL)-developed ceramic anode (CERANODE).

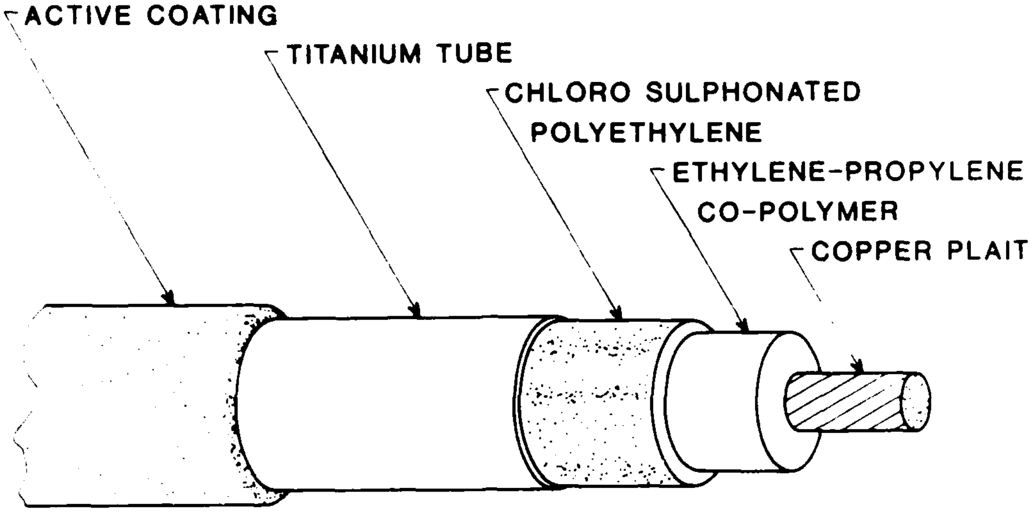

Figure 2. Schematic of LIDA® precious metal anode.

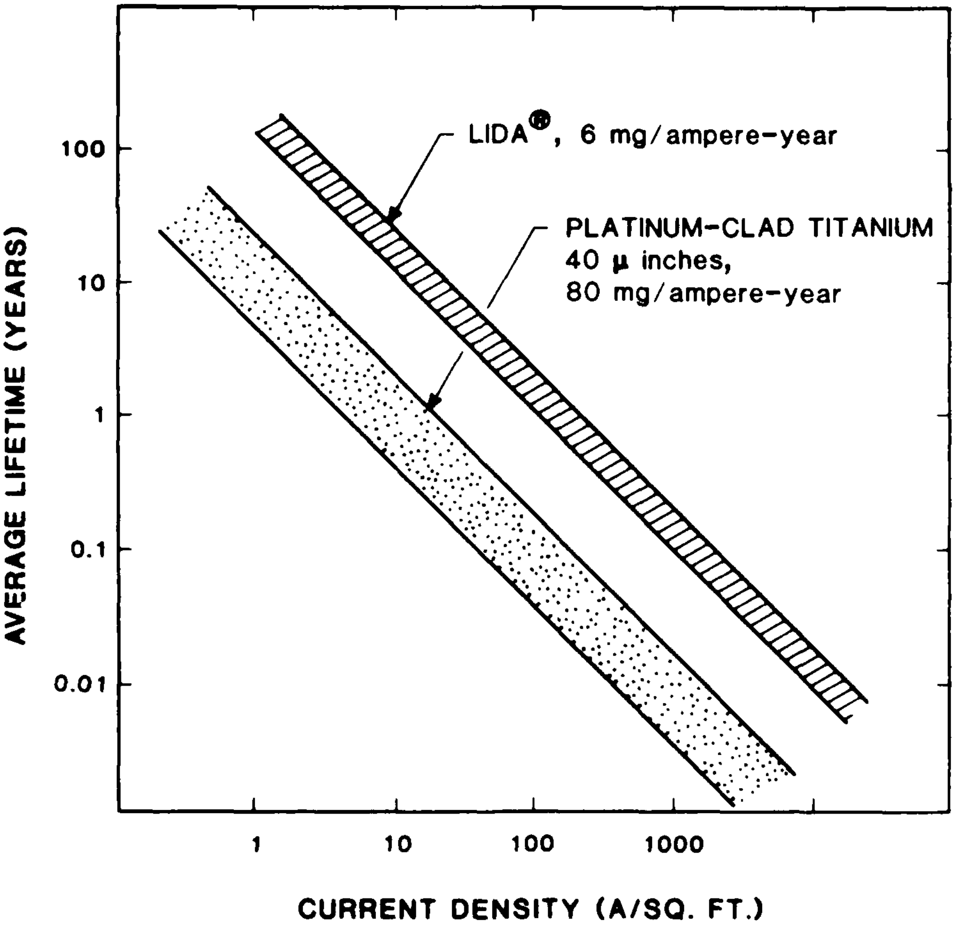

Figure 3. Service life of LIDA® precious metal anode.

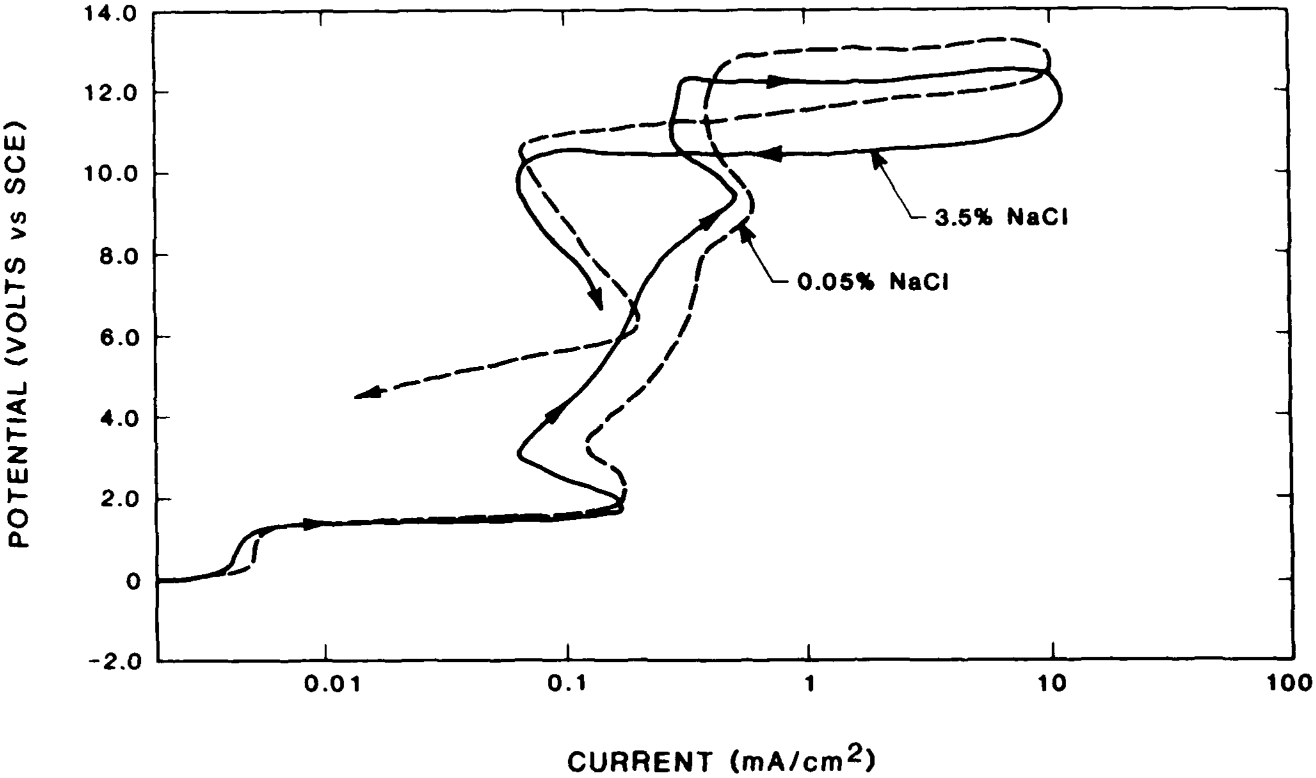

Figure 4. Potentiodynamic scans of titanium in distilled water solutions containing 3.5 and 0.05 percent NaCl.

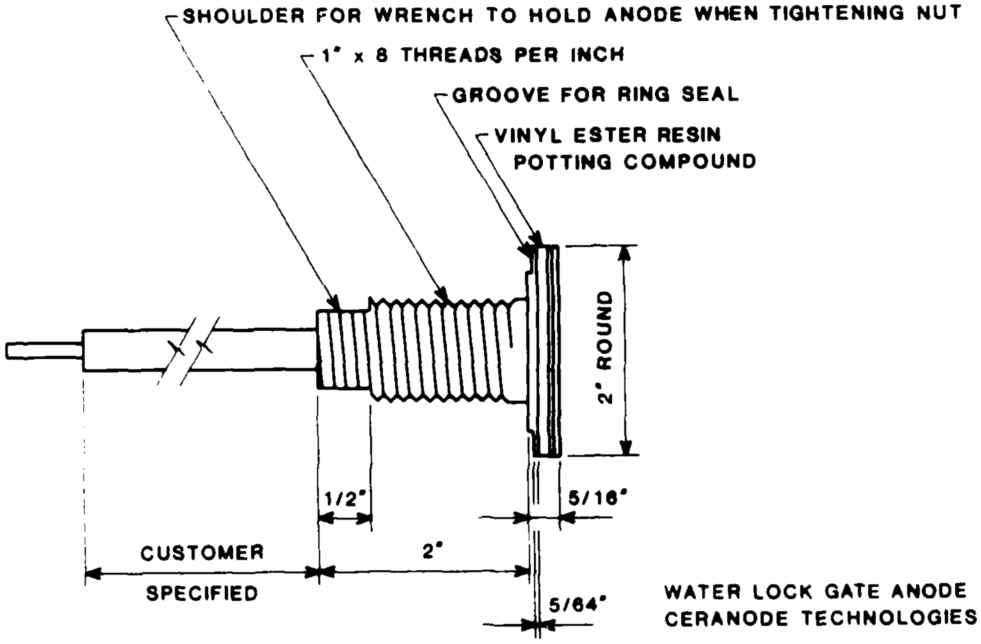

Figure 5. Schematic of new flat CERANODE design.

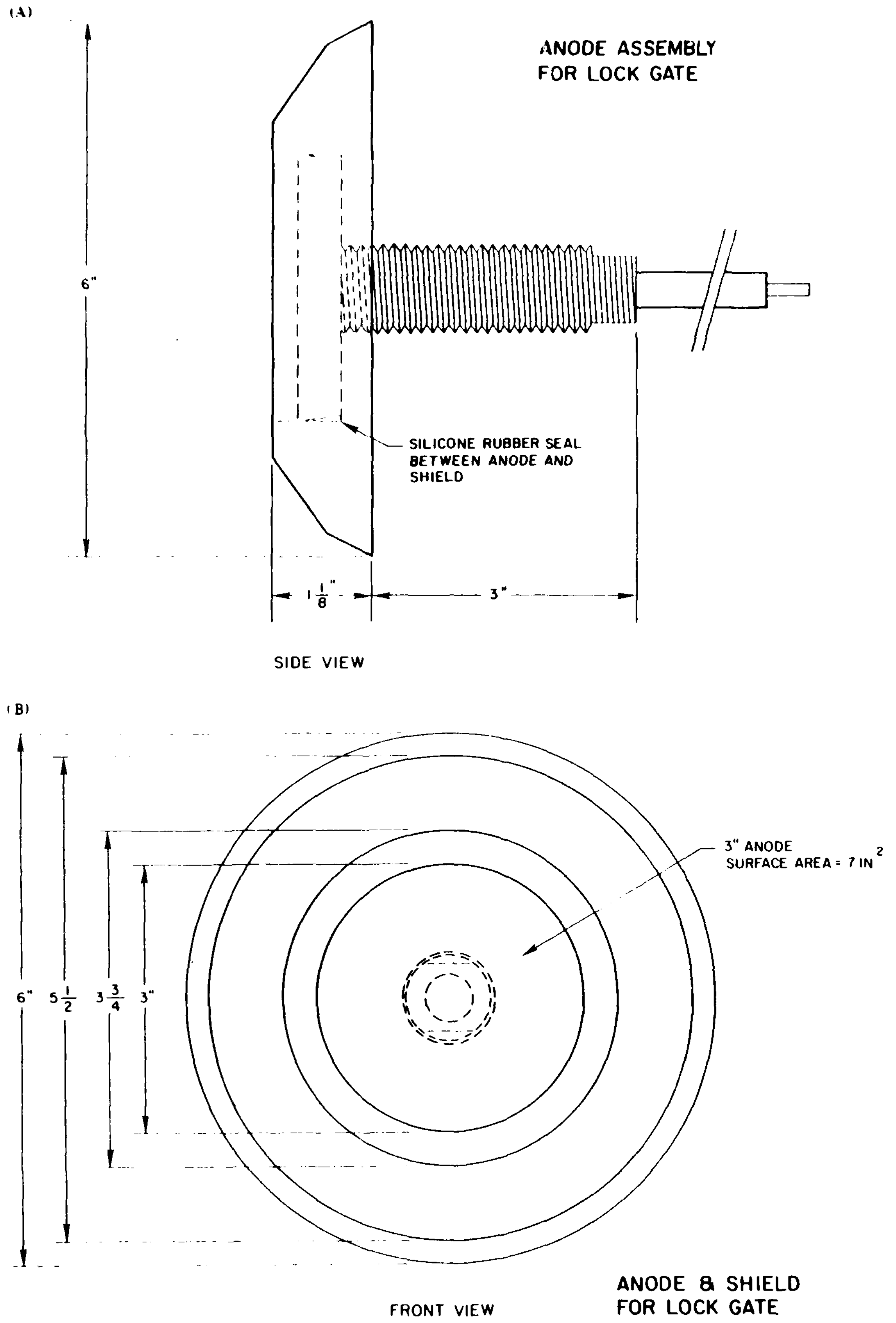

Figure 6. Flat anode shield assembly for lock gates.

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