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PRELIMINARY INVESTIGATION OF CERAMIC-COATED ANODES FOR
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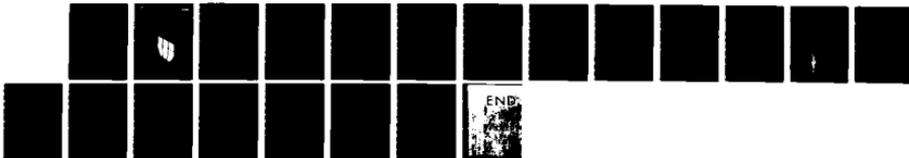
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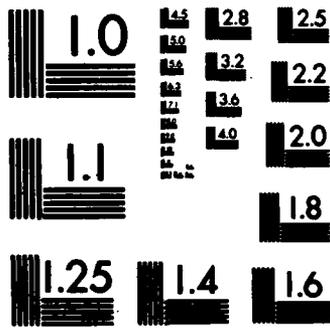
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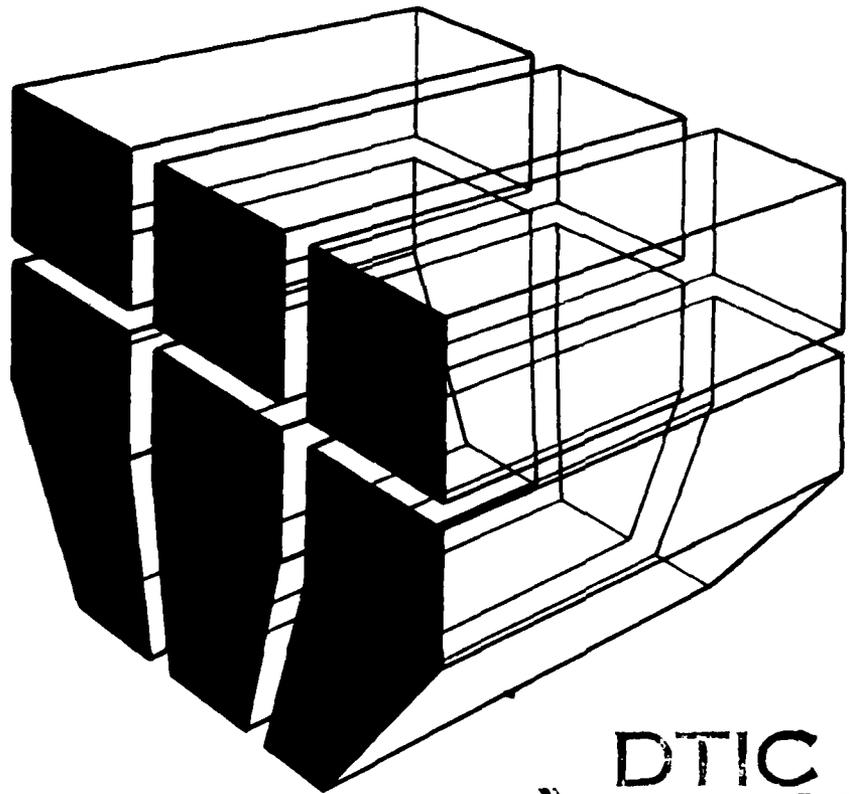
Technical Report M-333
August 1983

Corrosion Mitigation in Civil Works Projects

- AD-A133440

**PRELIMINARY INVESTIGATION
OF CERAMIC-COATED ANODES
FOR CATHODIC PROTECTION**

by
**E. G. Segan
A. Kumar**



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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A cathodic protection anode was manufactured by plasma-spraying an electrically conducting lithium ferrite coating on button-shaped 99 percent pure titanium and niobium substrates. Lithium ferrite was selected because of its unique adherence to titanium and niobium substrates, as well as its good electrical conductivity, low dissolution rate, and long-range stability. The pitting behavior of the titanium substrate in chloride solutions was characterized; the worst case of pitting potential was 9.66 V. The dissolution rate of the lithium-		

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↘ ferrite-coated anode was measured over a 2-month period and was determined to be 1 to 2 g per ampere year in an aerated 3.5 percent sodium chloride solution. No damaging effects from the tests were observed on the coating.

The button-shaped anode was designed so it could be installed easily on underground pipes or on structures in water, such as waterway lock gates and elevated water storage tanks, without dewatering the structure. The ceramic coating's low dissolution rate provides the advantage that the anode can be relatively small and still provide considerable protection from corrosion. A small anode can be installed and replaced relatively easily and is less vulnerable to damage.



FOREWORD

This study was conducted for the Directorate of Civil Works, Office of the Chief of Engineers (OCE), 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 (CERL). The OCE Technical Monitor was Mr. J. Robertson (DAEN-CWE-E). Dr. R. Quattrone is Chief of EM.

COL Louis J. Circeo is Commander and Director of CERL, and Dr. L. R. Shaffer is Technical Director.

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PRELIMINARY INVESTIGATION OF CERAMIC-COATED ANODES FOR CATHODIC PROTECTION

1 INTRODUCTION

Background

The corrosion of metallic structures immersed in water or buried in soil can be stopped by cathodic protection, i.e., by applying a small electric current from an outside source to the corroding structure. Cathodic protection has been used since 1824 when Sir Humphrey Davy introduced it as a means of protecting copper sheathing in ships.

The paint or coating applied to a metal is the primary means of defense against corrosion. However, paint films are never perfect; defects and holidays are always present. Furthermore, the corrosion of metal at isolated pinholes in paint films is usually more severe than that on uncoated surfaces; therefore, supplementary protection of structures is often needed. The corrosion of metal structures submersed or buried in ionically conducting mediums, such as water or soil, can be stopped using cathodic protection. The corrosion of 1 m² of bare carbon steel in fresh water can be stopped by applying 0.2 mA of direct current. Cathodic protection can be used as a sole means of corrosion mitigation, although it is usually used with paint films for maximum efficiency.

The U.S. Army Corps of Engineers is responsible for maintaining many types of structures including lock gates and water storage tanks, which are subject to corrosion. Effectively protecting these structures from corrosion would afford considerable savings, both in time and in maintenance dollars. Many cathodic protection systems are available which can be used for these structures. In low-resistivity mediums, sacrificial systems that do not require an external power source are used. These systems are usually simple and easy to maintain. However, most Corps structures cannot be adequately protected by sacrificial systems and must make use of impressed current systems.

Traditionally, impressed current systems have used anodes that are either inexpensive and very large, or small and expensive. High-silicon, chromium-bearing cast iron (HSCBCI) and graphite anodes often weigh more than 60 lb (24 kg). These anodes have posed

many installation and maintenance problems, and are often damaged by ice and debris on hydraulic structures. An alternative system to the heavy anodes is the use of platinized anodes. These consist of a thin platinum layer on a passive substrate like niobium or titanium. They are much lighter because the anodic dissolution rate of platinum is very low; for example, the dissolution rate of platinum is less than one ten-thousandth that of HSCBCI. However, platinized anodes are vulnerable to abrasion and erosion corrosion damage, and the platinum makes them very expensive. Because of these problems, there is an acute need for improved cathodic protection systems with affordable anodes that can provide reliable protection with a minimum of installation and maintenance problems.

The possibility of using electronically conducting ceramics for anodes has been investigated.¹ Many conducting oxides have been successfully used as electrode materials in high-temperature electrochemical cells and anodes for electrochemical processing. However, the use of ceramics for cathodic protection anodes has only recently been examined. Some oxide ceramics, such as ferrites, titanates, and cobaltates, as well as some carbides and nitrides, exhibit semiconducting behavior at ambient temperatures. Often, the conductivities of these ceramics approach that of impure metals. The semiconducting ceramics are largely made of inexpensive and abundant raw materials. Anodes coated with these ceramics contain a large proportion of oxygen, carbon, or nitrogen and a relatively low proportion of expensive or strategic metals. The expense of making ceramics at the high temperatures usually required has been greatly reduced by the development of processing techniques which minimize energy expenditures. These factors make ceramic anodes a promising option for impressed current cathodic protection systems.

Objective

The objectives of this investigation were: (1) to evaluate the feasibility of using electronically conducting ceramics for anodes in impressed current cathodic protection systems and identify applications where ceramic anodes can offer improved perform-

¹S. Wakabayashi and T. Aoki, "Characteristics of Ferrite Electrodes," *Journal De Physique*, Vol 4, p C1 (1977); R. Itai and H. Kunai, U.S. Patent No. 3,850,701, "Anode Coated with Magnetite and the Manufacture Thereof" (November 1974); T. Fujii, T. Kodama, H. Baba, and S. Kitahara, "Anodes Behavior of Ferrite Coated Titanium Electrodes," *Boskoko Gijutsu* (Corrosion Engineering) Vol 29, No. 4 (1980), pp 180-184.

ance, and (2) to manufacture and test anodes coated with low-resistivity ceramics that have low anodic dissolution rates and provide characteristics such as freedom of anode configuration, easy installation and replacement, small size, and toughness.

Approach

The state of the art of impressed current cathodic protection systems and the application of conducting ceramics for such systems were reviewed. The performance of lithium ferrite and other conducting oxides prepared by various techniques under anodic polarization in various environments was then evaluated to determine the ceramic systems and stoichiometry best suited for impressed current anodes. Designs for field anodes were developed and field tests of prototype anodes were conducted at several locations.

Mode of Technology Transfer

It is recommended that the information in this report be used to develop procurement specifications for incorporation into Corps of Engineers Guide Specification 2310.

2 FEASIBILITY OF USING CERAMIC ANODES IN CATHODIC PROTECTION SYSTEMS

Cathodic Protection State of the Art

The potential benefit of using cathodic protection for corrosion mitigation is well known,² and in-depth discussions of the various systems are available.³ The Corps of Engineers has been using cathodic protection since the 1950s when experimental systems were introduced. Properly designed and maintained systems can extend a structure's life and minimize maintenance and operating costs.

Many factors are involved in selecting an appropriate cathodic protection system, including:

1. Corrosivity of the electrolyte (i.e., water or soil)
2. Anode selection

²H. H. Uhlig, *Corrosion and Corrosion Control* (John Wiley and Sons, Inc., 1963).

³A. Kumar, R. Lampo, and F. Kearney, *Cathodic Protection of Civil Works Structures*, Technical Report M-276/ADA080057 (U.S. Army Construction Engineering Research Laboratory [CERL], 1979; *Electrical Design, Corrosion Control*, Technical Manual 5-811-4 (Department of the Army, 1 August 1962).

3. Coating selection
4. Condition of the coating
5. Current distribution
6. Resistance of the cathodic protection circuit
7. Structural configuration.

Many of these factors are interrelated.

Cathodic protection may be achieved by sacrificial or impressed current systems. Tables 1 and 2 summarize the characteristics of sacrificial and impressed current anodes. Sacrificial systems require both electrical connection of the anode (i.e., zinc or magnesium) to the structure and the presence of an electrolyte. Over time, the anode dissolves, providing the current for cathodically protecting the structure. This system does not require an external power source and is easy to install and maintain. However, it is usually used in seawater, brackish water, and low-resistivity soils. This is because in high-resistivity media, the "throw distance" of the anodes (the distance from the anode where cathodic protection current flows to protect the structure) is decreased to less than 1 to 2 ft. (.3 to .6 m).

In impressed current systems, electrical current is passed from an anode through the electrolyte (water or soil) into the corroding structure. An outside source of direct current (usually a rectifier) is used to apply 5 to 25 V to the anode. The positive lead of the rectifier is connected to the anode, which is eventually consumed, and the negative lead is connected to the structure. Many types of impressed current anodes are available. Graphite and high-silicon, chromium bearing cast iron (HSCBCI) anodes have traditionally been used to protect metallic structures in salt-water, freshwater, and soil. However, these anodes are somewhat brittle, cannot be machined easily, and have dissolution rates of about 1000 g per ampere year. The high dissolution rates require use of large anodes that are vulnerable to debris and ice damage and prone to field installation problems.

Platinized anodes using a 0.001-cm layer of platinum on metal substrates such as titanium, niobium, and tantalum that passivate during anodic polarization have recently been developed.⁴ When immersed in water, these metals exhibit active-passive behavior and form electrically insulating oxide films that do

⁴R. Baboin, "Performance of Platinum Anodes in Impressed Current Cathodic Protection," *Proceedings of the Fourth Inter-*

Table 1
Characteristics of Sacrificial Anodes

Metal	A-hour/lb (actual)*	Efficiency (%)	Corrosion Potential (V vs. Cu/CuSO ₄)	Price Factor**
Aluminum	1250	95	-1.2	1.50
Zinc	355	95	-1.1	1.00
Magnesium	500	50	-1.6	2.00

*Metric conversion: 1 A-hour/lb = 2.2 A-hour/kg; 1 lb/A-year = 2.2 kg/A-year; 1 in. = 25.4 mm; 1 lb = 0.453 kg; 1 sq ft = 0.09 m².

**Current price of zinc = 1.00.

not break down at the normal operative voltages encountered in cathodic protection. A thin layer of platinum deposited on the clean metal substrates prevents the formation of the passive insulating film and allows the cathodic protection current to flow. The consumption rate of platinum is only about 5 mg per ampere year. However, its cost is high, and the thin layer of platinum (0.0001 cm) makes the platinized anodes susceptible to abrasion and erosion corrosion damage. If the outer platinum layer gets scratched, the freshly exposed substrate passivates (i.e., forms the insulating oxide film) and stops passing current from the scratched area. Investigations of the passivation of titanium substrates have shown that the applied voltage should not exceed 9.17 V (vs. saturated calomel electrode [SCE]) for cathodic protection of hydraulic structures.⁵

Using Ceramics in Impressed Current Cathodic Protection Systems

Electrical and Electrochemical Properties of Conducting Ceramics

Many ceramic materials exhibit high electrical conductivities because of their semiconducting properties. Semiconductors are generally classified as materials in which the electrical conductivity is electronic in nature and has values in the range of 10⁻³

national Congress on Marine Corrosion and Fouling, Antibes, France (June 1976); M. A. Warne and P. C. S. Haywood, "Platinized Titanium Anodes for Use in Cathodic Protection," Materials Protection and Performance, Vol 15, No. 3 (March 1976), pp 39-42; M. A. Warne, "Precious Metal Anodes—The Options for Cathodic Protection," Paper No. 142, 1978 Corrosion Conference, Houston, TX (National Association of Corrosion Engineers, March 1978); E. W. Dreyman, "Precious Metal Anodes: State-of-the-Art," Materials Protection and Performance, Vol II, No. 9 (September 1972), pp 17-20; R. Baboian, "Platinum Consumption in Cathodic Protection Anodes," Materials Protection and Performance, Vol 16, No. 3 (March 1977), pp 20-22.

⁵E. G. Segal, J. Bukowski, and A. Kumar, *Titanium Anodes in Cathodic Protection*, Technical Report M-303/ADA111366 (CERL, 1982).

Table 2
Characteristics of Impressed Current Anodes

Material	Maximum Working Capacity Amperes/Sq Ft		Approximate Wastage (g/Ampere-year)
	Soil	Water	
Scrap steel	0-5	0-5	15-20
Scrap cast iron	0-5	0-5	10-15
Silicon iron	3	3-4	1-2
Graphite	1	2	2
Lead	—	10-20	—
Lead/platinum	—	1000	—
Platinum	—	<1000	—
Platinized titanium	—	<1000	—
Platinized niobium	—	<1000	—
Aluminum	—	2	9

to 10⁻⁹ ohm-cm. This is between that of metallic conductors (about 10⁻⁶ ohm-cm) and insulators (>10¹² ohm-cm).

The total electrical conductivity of any material is the sum of the conductivities of its individual charge carriers. In most ceramics, electrical conductivity is largely due to ionic conductivity, in which anions, such as O⁻², pass through the structure (crystalline or amorphous) by diffusion in an applied electric field. The mobility of cations and anions in ceramics is very low at ambient temperatures and increases significantly only at high temperatures (usually above one half of the melting point in °K). Most ceramic materials are covalently bonded, which causes localization of electrons in the structure. This results in very low electronic conductivity because electrons are "pinned" to their sites in the ceramic. At ambient temperatures, the ionic and electronic conductivities of most ceramics are very low. Thus, the traditional use of ceramics has been for insulators. For example, ceramic insulators are frequently used for high-tension power transmission lines.

Some ceramic materials exhibit semiconducting behavior. This occurs because in some crystal struc-

tures, electrons may become mobile by transferring from one cation site to the next. This is a complicated process that gives them other unique magnetic and optical properties. Semiconduction is common in oxides that crystallize in spinel, hematite, and perovskite structures as well as in some carbides and nitrides. At ambient temperatures, ionic conduction in these materials is usually low in comparison to electronic conductivity.

The ratio of electronic to ionic conduction in ceramics is usually quantified by the electronic transference number, t_e , defined as:

$$t_e = \frac{T_e}{T} \quad [\text{Eq 1}]$$

where: T_e = fraction of conductivity contributed by electronic conduction

T = total conductivity.

For an electrochemical cell to function properly, the electrode must conduct electrons, not ions. Significant ionic conductivity can cause ionic short-circuit of the cell. Since the electronic and ionic conduction of charge in an electrode offer parallel conduction paths, the ratio of electronic to ionic conductivities (i.e., the transference number) must be maximized. Although the transference number of conducting oxides is very close to 1.0 at ambient temperatures, even a very high conductivity material will not perform properly as an electrode if its transference number falls below 0.9.

Conducting ceramics have been used for some time as electrodes in high-temperature electrochemical cells and in chemical processing reactors. However, the use of ceramics for impressed current cathodic protection system anodes was only begun recently. Two types of systems have been tested. The first is a sintered ceramic anode, either with or without a metal core; the second is a plasma-sprayed ceramic on substrates such as titanium, niobium, or tantalum. Table 3 gives the dissolution rates of different anode materials.

Magnetite anodes having a dissolution rate of 40 g per ampere year have been used for impressed current cathodic protection systems.⁶ These anodes exhibit chemical inhomogeneity, high porosity, and low mechanical strength. The density and mechanical strength can be improved somewhat by sintering NiO

with Fe_2O_3 , forming a spinel nickel ferrite. Best results have been achieved by using 40 mol percent NiO or 60 mol percent Fe_2O_3 , thereby reducing the dissolution rate to 0.4 g per ampere year. However, these anodes do not have the required toughness and do not yield freedom of configuration for environments like locks and miter gates.

Table 4 shows typical properties of ferrite and cast magnetite sintered anodes investigated by Wakabayashi and Aoki. Nominal ferrite compositions were $0.1 \text{ MO} - 0.9 \text{ Fe}_2\text{O}_3$, where M represents a divalent ion such as magnesium, zinc, manganese, cobalt, nickel, and iron ions. The table shows that nickel ferrite offered the lowest dissolution rate (1.56 g per ampere year). Wakabayashi and Aoki also found that the dissolution rate was reduced to 0.4 g per ampere year when nickel ferrous ferrite containing 60 mol percent Fe_2O_3 and 40 mol percent NiO was used. The high resistivity (0.3 ohm-cm) of nickel-ferrite containing 40 percent NiO is acceptable in cathodic protection, because the anode resistance can be reduced by using special geometrical modifications such as tubular anodes.

Sintered ceramic anodes have good corrosion resistance and low resistivities. However, the toughness of the sintered ferrite anodes is low and is not acceptable in many applications where the anode can be mechanically damaged. The anodic dissolution rates of these anodes increases with decreasing current density and with decreasing chloride content in the water. The dissolution rate is also relatively constant in the pH range of 1 to 10, increasing markedly at $\text{pH} < 1$.

The marginal properties of sintered ceramic anodes have led to the development of ceramic-coated anodes. Various attempts have been made to manufacture anodes coated with magnetite. Itai and Kanai used electrodeposition to coat titanium and other substrates with magnetite. The coating techniques employed electrodeposition of iron, dipping the iron-deposited titanium substrate into a solution of ammonium ferric oxalate under a reduced pressure, and then heating the treated substrate in an atmosphere of hydrogen and steam. The electrodes are suitable for producing chlorine and chlorates and for electrowinning metals. However, the maximum coating thickness obtained was only 20 microns; this does not meet the long-life requirement (10 to 20 years) of cathodic protection systems.

In another approach, Fujii, et al., plasma-sprayed several 50-micron (0.002-in.) coatings of several

⁶Wakabayashi and Aoki, 1977.

Table 3
Dissolution Rates of Different Anode Materials

Electrode Material	Anodic Dissolution Rate* (g/Ampere-year)
Platinum-coated titanium	0.01
Nickel ferrite (0.4 NiO - 0.6 Fe ₂ O ₃)	0.40
Nickel ferrite (0.1 NiO - 0.9 Fe ₂ O ₃)	1.56
Lead silver alloy (1.5 Ag)	30
Cast magnetite	40
Graphite	200
High-silicon, chromium-bearing cast iron	450

*Current density: 500 A/m² in 3 percent sodium chloride solution.

Table 4
Typical Properties of Ferrites and Cast Magnetite

Material	Electric Resistivity (ohm-cm)	Anodic Dissolution Rate* (g/Ampere-year)
Mg-ferrite**	0.3	3.47
Zn-ferrite	0.02	3.28
Mn-ferrite	0.02	2.67
Co-ferrite	0.015	2.19
Ni-ferrite	0.02	1.56
Cast magnetite	0.11	50

*3 percent sodium chloride; anodic current: 500 A/m².

**Nominal ferrite composition: 0.1 MO-0.9 Fe₂O₃.

spinel ferrites (magnetite, nickel, and cobalt ferrites) on titanium substrates and determined their anodic behaviors in sodium chloride solutions. The dissolution rates of the ferrite-coated anodes ranged from 0.1 to 8.7 g per ampere year, which is comparable to that of sintered ferrites. However, adhesion of the ferrite films to the substrate metals was not satisfactory. The process used reducing atmospheres during plasma-spraying, and the coatings produced were rather thin (50 microns [0.002 in.]). Plasma-spraying with 10 percent titanium dioxide mixtures or using a tantalum undercoating did not improve adhesion of the coating, and a workable anode could not be produced.

Optimization of Ceramic Anode Cathodic Protection Systems

Ceramic anodes show promise for use in impressed current cathodic protection systems. The sintered anodes appear to be usable in systems where the possibility of mechanical damage is small (i.e., buried pipelines and structures), while ceramic-coated anodes with high toughness can be used in environ-

ments where mechanical damage can occur (i.e., lock gates and the interior of water tanks).

Titanium and niobium—the two types of substrates most often used for coated anodes—are best suited for ceramic-coated anodes. Both exhibit excellent physical, electrical, and electrochemical properties. Niobium anodes can be used at applied voltages as high as 50 V in salt water, and titanium anodes can be used at up to 9 V (vs. SCE). However, if there is a risk of chloride pitting, and the operating voltage is expected to exceed 9 V (vs. SCE), niobium should be used.

Good ceramic coatings can be prepared by a variety of techniques. Recent developments in plasma-spraying, chemical vapor deposition, hot pressing, and other techniques can produce thick, adhesive coatings.

Ceramic-coated anodes must exhibit many properties, including:

1. Low electrical resistance to minimize power consumption (i.e., substrate and coating resistivities must be minimized and the geometrical design should yield low resistance).

2. Capability of providing an effective barrier to oxygen ions (i.e., the coating must be nonporous and have a high electronic transference number).

3. An active surface area for oxidation that does not passivate or become poisoned by the environment during operation.

4. Toughness and good adherence to the substrate.

3 TESTING PROCEDURES

Laboratory Tests

Ceramic anodes were manufactured for CERL by plasma-spraying lithium ferrite on titanium and niobium substrates. Lithium ferrite raw materials were produced by grinding hot-pressed bars; the materials were deposited by plasma-spraying at a power level at 26.25 kW with an argon forming gas at a flow rate of 1.7 m³ per hour. Spray distance was maintained at 8.89 cm. The powder gas used was

oxygen at a flow rate of 0.226 m³ per hour. Strong, fine-grained adherent coatings of various thicknesses were deposited on titanium and niobium substrates. The substrates were machined out of solid rods.

Figure 1 shows the anode configuration used for testing the anodes in the field. The ceramic-coated anode consists of a plasma-sprayed lithium ferrite coating having an active surface area of about 0.001 m², 500 to 2500 microns thick, deposited on niobium or titanium substrates that are machined to a button shape, 2.54 cm thick, and threaded to fit a plastic gland. A weep hole is drilled in the plastic gland which is fastened by a stainless steel nut to the structure to be protected. The ceramic coating faces the water side of the structure. Sealant is squeezed through the weep hole to waterproof the inside of the anode assembly. The cathodic protection cable is connected to the metallic threaded plug, which is threaded into the substrate. A nylon ferrule (tapered washer) and plug are screwed into the plastic gland until the excess sealant is forced through the weep hole. The ceramic-coated anode is installed by drilling a hole in the steel structure to be protected and attaching the anode assembly. The nut and the washer are removed, and the full length of wire is pulled through the hole in the steel structure. The washer is put on and the nut is threaded and tightened to 30 foot-pounds of torque. Installation of the anode assembly is then complete and the anode lead is connected to the junction box or rectifier.

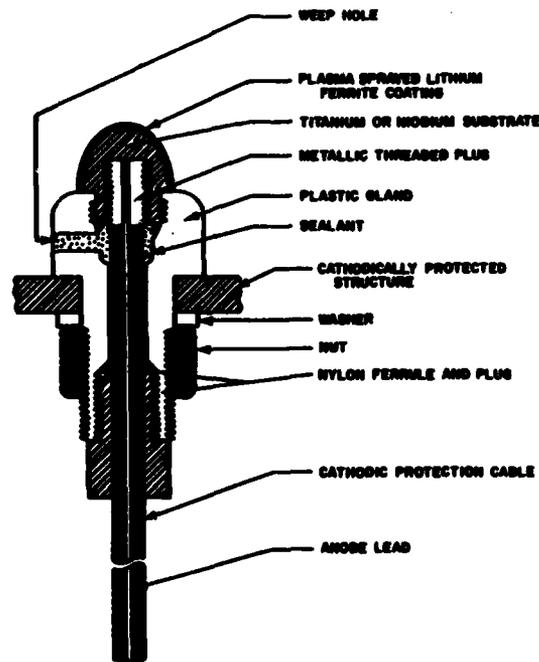


Figure 1. Ceramic-coated anode assembly.

The coatings were chemically analyzed by Chicago Spectro Service Laboratory, Inc. The composition of the plasma-sprayed materials was determined by x-ray fluorescence of a fused sample. The dissolution rates of the anodes were determined in the laboratory by immersing the anodes for 1 to 2 weeks at various anodic current loadings both in tap water and in distilled water containing 3.5 percent reagent grade sodium chloride. Table 5 shows typical chemical analysis of Champaign, Illinois, tap water, which was used for those tests.

The amount of dissolved lithium and iron in the solutions was determined by atomic absorption analysis. Long-range tests of lithium ferrite were conducted by measuring the weight loss of an anode immersed in 3.5 percent sodium chloride in distilled water for 2 months at a current loading of 2000 A/m². The solution was replenished weekly.

Field Tests

Test anodes (25.4 mm in diameter) fabricated as shown in Figure 1 were installed at the Racine Lock gate structure on the Ohio River in West Virginia and at the Miller's Ferry Lock gate on the Alabama River in Alabama. Test anodes were also installed inside water storage tanks at Fort Eustis, Virginia, and Fort Hunter Liggett, California. Anode assemblies were embedded in coke breeze contained in steel casings and installed to protect underground pipes at Fort Carson, Colorado, and at Fort Polk, Louisiana. The test anodes were installed, both in parallel and side by side, in existing cathodic protection systems designed with HSCBCI anodes. The current passing through the ceramic-coated anode and the applied voltage were measured.

Table 5
Typical Chemical Analysis of Champaign, Illinois, Tap Water

pH	8.8 to 9.0 units
Total Alkalinity	100 to 120 mg/L as CaCO ₃
Calcium	30 to 35 mg/L as CaCO ₃
Magnesium	45 to 50 mg/L as CaCO ₃
Hardness	75 to 85 mg/L as CaCO ₃
Fluoride	0.90 to 1.20 mg/L
Iron	Less than 0.02 mg/L
Sodium	Less than 40 mg/L
Sulfate	Less than 40 mg/L
Nitrate	Less than 2.0 mg/L
Chloride	Less than 5.0 mg/L
Total Solids	Less than 180 mg/L
Ammonia	Less than 2 mg/L
Chlorine	Combined residual 2.5 ± 0.1 mg/L
Turbidity	Less than 0.1 JTU
Color	0 units
Odor	0 units

4 TEST RESULTS

Laboratory Tests

For coating thicknesses up to 30 mils, the plasma-sprayed lithium ferrite adhered to the niobium and titanium substrates for the duration of the tests. Thicker coatings were found to have extensive micro-cracking that apparently resulted from thermal expansion mismatches, which caused thermal cracking during cooling. Thus, it appears that the present coating process will have to be improved to produce thicker, nonporous coatings. Although anodes may have either niobium or titanium substrates, for this investigation anodes made of 20 mils of lithium ferrite plasma sprayed on titanium substrates were used unless otherwise noted.

Chemical analysis of the plasma-sprayed lithium ferrite for elements other than lithium was conducted by x-ray fluorescence on a fused sample. Lithium content was determined by atomic absorption, since its low molecular weight precluded analysis by x-ray fluorescence.

Table 6 shows the composition of the plasma-sprayed lithium ferrite by both weight and mole percent. The oxygen content was estimated by assuming that the nonmetallic portion of the ceramic is oxygen. Analysis showed that the lithium ferrite contains significant amounts (molar proportions) of manganese, zinc, and calcium. These elements can all participate in the spinel structure on cation sites.

The chemical formula of stoichiometric lithium ferrite is Li_{0.5}Fe_{2.5}O₄, yielding an iron-to-lithium ratio of 5. The plasma-sprayed lithium ferrite has an iron-to-lithium ratio of 5.89, making it rich in iron. This is probably the result of preferential volatilization of lithium during the plasma-spraying process, since lithium is much less stable than iron at elevated temperatures. The chemical formula of the plasma-sprayed lithium ferrite can be determined by normalizing the proportions of the elements to yield 4 moles of oxygen. The following are formulas for some simple stoichiometric ferrites, and for the plasma-sprayed lithium ferrite:

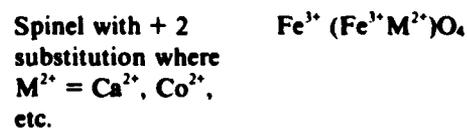


Table 6
Chemical Analysis of Plasma-Sprayed Lithium Ferrite on Titanium Substrate*
 (From Chicago Spectro Service Laboratory, Inc., Chicago, Illinois)

	Weight Percent	Mole Percent
Iron	69.22	40.49
Manganese	3.05	1.81
Lithium**	1.46	6.87
Zinc	1.43	0.72
Magnesium	0.008	1.08×10^{-2}
Calcium	0.27	0.22
Nickel	0.082	0.46
Oxygen (estimated)	24.375	49.77
Others Approximate:		
Titanium	0.02	1.36×10^{-2}
Aluminum	0.01	1.21×10^{-2}
Boron	0.01	3.02×10^{-2}
Silicon	0.005	5.82×10^{-3}
Chromium	0.003	1.88×10^{-3}
Copper	0.003	1.54×10^{-3}
Molybdenum	0.003	1.02×10^{-3}
Cobalt	0.001	5.54×10^{-4}
Silver	0.0005	1.51×10^{-4}
Sodium - Questionable, If Present	0.05	≈0

*Analysis performed by x-ray fluorescence on a fused sample of plasma sprayed lithium ferrite.

**Lithium analysis performed by atomic absorption.

Plasma-sprayed lithium ferrite $\text{Li}_{0.55}\text{Fe}_{3.25}\text{O}_4$

Plasma-sprayed lithium ferrite (major impurities included) $\text{Li}_{0.55}\text{Zn}_{0.6}\text{Mn}_{0.14}\text{Ca}_{0.02}\text{Fe}_{3.25}\text{O}_4$

The plasma-sprayed lithium ferrite appeared to be oxygen-deficient, as shown by the high cation to anion ratio $(\text{Li} + \text{Fe})/\text{O}_4 = 0.95$, compared to the stoichiometric ratio of 0.75. This effect is probably due to the reducing environment of the argon forming gas used during plasma-spraying. Furthermore, the plasma-sprayed powders are deficient in lithium (note the low lithium-to-iron ratio—0.17 as compared to the stoichiometric ratio of 0.20). This is most likely the result of preferential volatilization of lithium during plasma spraying. However, the impurities and nonstoichiometry of the lithium ferrite do not pose serious problems in using it as an anode for cathodic protection.

The dissolution rates of ceramic anodes prepared from crushed lithium ferrite bars and from spray-dried powders were determined by calculating the overall anode dissolution rate from the proportions of lithium and iron in the test solutions. It was found

that plasma-sprayed, spray-dried powders gave lower dissolution rates than the coatings prepared from crushed bars. (Only the results for the spray-dried powders are presented here.)

Chemical analysis (see Table 6) was used to calculate mole fractions of lithium and iron in the anodes. Table 7 shows the dissolution rates of the ceramic anodes at various current loadings of 3.5 percent sodium chloride in distilled water and in tap water. Figure 2 illustrates the effect of varying current density on the anode dissolution rates, based on both lithium and iron dissolution rates for tap water and saltwater. Error bars are shown for points where more than one test was performed; these error bars reflect the accuracy of all the data presented. It appears that in short-term tests, the anode dissolution rates are maximum at currents of 20 mA ($\approx 20 \text{ A/m}^2$) in both saltwater and tap water. The anode dissolution rates approach unacceptable limits at 20 mA in saltwater (111 g per ampere year), and the dissolution rate of the ceramic anodes decreases with increasing current density at total currents above 20 mA. This is very desirable, because it suggests that the ceramic anodes may be well-suited to operation at very high-current densities, thus reducing size requirements.

Table 7
Dissolution Rates of Ceramic Anodes in Tap Water and 3.5 Percent Sodium Chloride in Distilled Water
 (From Chicago Spectro Service Laboratory, Inc., Chicago, Illinois)

No. of Tests	Solution	Current Density (A/m ²)	Total Current (A)	Fe/Li Ratio in Solution	Anode Dissolution Rate Based on Fe (g/A-yr)	Anode Dissolution Rate Based on Li (g/A-yr)	Average Dissolution Rate Based on Averaged Li and Fe Dissolution Rates (g/A-yr)
1	Tap Water	15	15	14.7	3.9	11.6	7.8
3	Tap Water	20	20	43.7 ± 4.6	17.6 ± 5.0	20.8 ± 4.0	19.2 ± 9.0
1	Tap Water	44	44	47.1	8.01	8.1	8.05
3	Tap Water	100	100	84.3 ± 8.4	6.9 ± 1.2	16.7 ± 4.81	11.8 ± 6.0
1	Saltwater	15	15	41	13.26	15.1	14.2
3	Tap Water	20	20	9.7 ± 0.3	110.7 ± 22.5	545.7 ± 126.8	328 ± 150
3	Tap Water	100	100	151.2 ± 24.7	21.72 ± 0.9	29.3 ± 7.7	22.5 ± 8.6
1	Tap Water	200	200	20.0	0.46	1.09	1.55

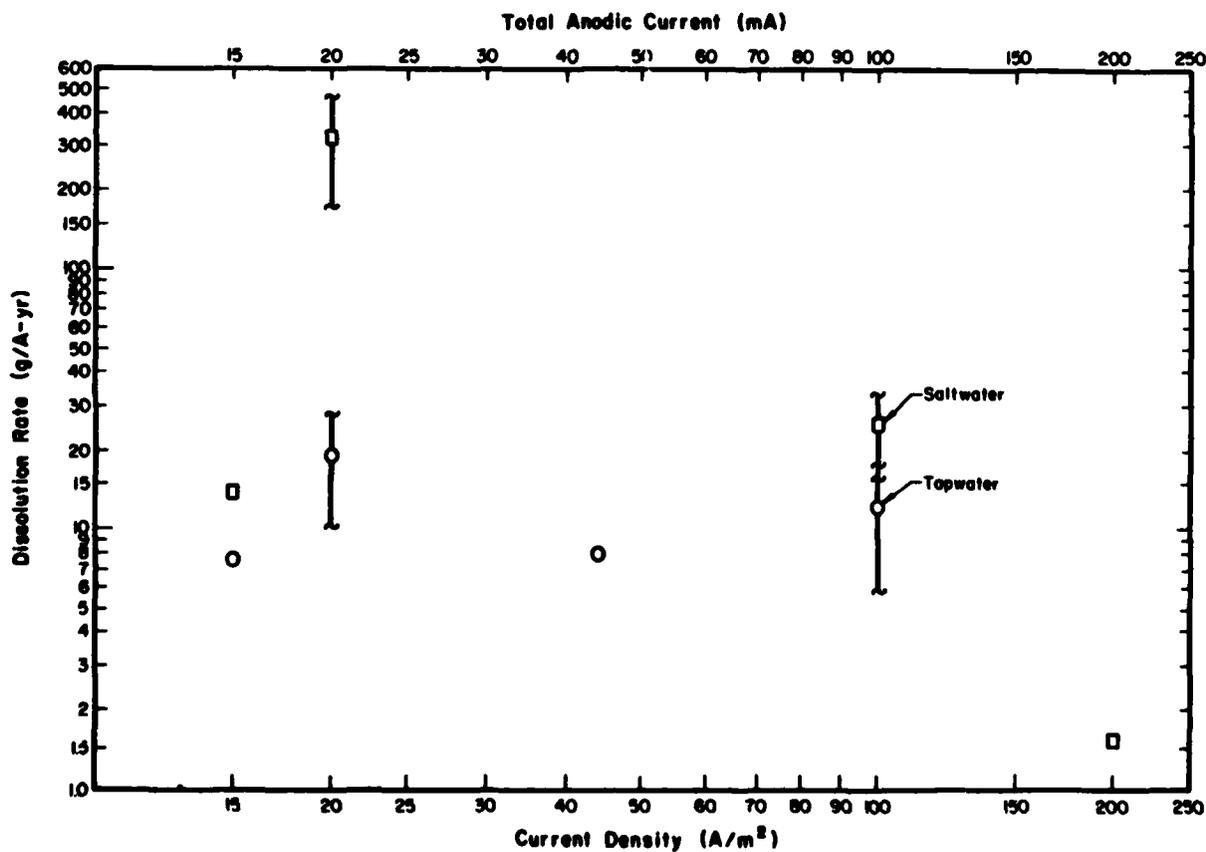


Figure 2. The effect of varying current density on the dissolution rate of lithium ferrite coated titanium anodes in tap water and in 3.5 percent sodium chloride in distilled water.

The dissolution rate of lithium ferrite, found by weight loss measurements, was only 1.7 g/ampere year at a current loading of 2000 A/m² during the 2-month, long-term test. This also shows that the dissolution rate of the oxide decreases with increasing current density.

Field Tests

The average current flowing through the test ceramic anodes in the water storage tanks and in the fresh water canal gates was found to be 20 mA at 5 V applied potential. The resistivity of the water was between 3000 and 3500 ohm-cm.

The average current passing through the ceramic anode installed near an underground coated pipe in soil of 5000 ohm-cm resistivity was 20 mA at 5 V. Higher currents can be obtained by increasing the rectifier voltage or by using media of lower resistivity. This shows that one ceramic anode can protect 9 m² of coated steel (assumes 10 percent base area).

Advantages of Ceramic Anodes

On the basis of the information provided by these tests, ceramic anodes appear to have the following advantages over the presently used anodes:

1. The substrate metals used for ceramic anodes, such as titanium or niobium, can be easily fabricated in any shape or form. The substrates also offer a good strength-to-weight ratio.

2. The consumption rate of ceramics is about 1/100 that of graphite or HSCBI. Therefore, smaller anodes can be used which are less vulnerable to damage. Preliminary evidence suggests that the dissolution rate decreases with increasing current density for total anode currents of 20 to 100 mA. This suggests that very small anodes with high current loadings may perform best.

3. Small anodes can be manufactured in a factory, so less fabrication will be required in the field. This increases the reliability of the cathodic protection system.

4. The smaller size of the anodes makes their replacement much easier.

5. Lightweight metal-ceramic anodes can be installed using plastic supports. This reduces installation problems which arise from the difficulty of providing electrical isolation of metallic supports used for the heavier button-type anodes which can weigh more than 60 lb.

6. Ceramic coatings are naturally resistant to abrasion; the inherent problem of ceramics brittleness can be overcome with appropriate design modifications.

5 CONCLUSIONS AND RECOMMENDATIONS

This research has produced the following conclusions:

1. Anodes made with lithium ferrite coatings on a pure titanium or niobium substrate exhibit good electrical conductivities and long-range stability and therefore have proven to have excellent applicability to cathodic protection systems.

2. The dissolution rate of the ferrite field anodes during short-term tests at a current loading of 20 A/m² is about 10 g per ampere-year in fresh water (1/100 of the currently used HSCBI and graphite anodes).

3. The dissolution rate of plasma-sprayed lithium ferrite in 3.5 percent NaCl in distilled water was 1.7 g per ampere-year at 2000 A/m² over a 2-month period.

4. Lithium ferrite anodes exhibit decreasing dissolution rates with increasing current densities when the current densities are above 20 A/m².

5. A unique button-shaped anode was developed which can be easily installed on underground pipes and submerged structures. Structures in water, such as waterway lock gates and elevated water storage tanks, can be protected without dewatering the structure. These anodes are tough and are small enough to be recessed in locations where damage from debris and ice is less likely.

The following recommendations are made concerning the further development of these anodes:

1. Investigate and develop improved materials and designs for ceramic anodes for use in specialized environments encountered in hydraulic structures.

2. Continue field and laboratory testing of lithium ferrite and other semiconducting ceramics to provide a full understanding of the short- and long-range behavior of ceramic-coated anodes.

3. Investigate the mechanisms of dissolution and the effect of structure and composition on the electrochemical stability of the ceramic semiconductors now being used as anodes in impressed current cathodic protection systems. Use the results of the mechanistic studies to develop new ceramics for anodes that offer improved properties. The development of materials that exhibit substantially improved dissolution characteristics hinges on an understanding of the factors that affect dissolution. Unlike the extensive base of knowledge of the electrochemistry of metals, the electrochemistry of ceramic semiconductor anodes for cathodic protection is not well understood.

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