

MTL TR 90-43

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AD-A228 237

ASSESSMENT OF CORROSION RESISTANT COATINGS FOR A DEPLETED U-0.75 Ti ALLOY

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September 1990

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM	
1. REPORT NUMBER MTL TR 90-43		2. GOVT ACCESSION NO.	
3. RECIPIENT'S CATALOG NUMBER			
4. TITLE (and Subtitle) ASSESSMENT OF CORROSION RESISTANT COATINGS FOR A DEPLETED U-0.75 Ti ALLOY		5. TYPE OF REPORT & PERIOD COVERED	
6. PERFORMING ORG. REPORT NUMBER			
7. AUTHOR(s) F. Chang, M. Levy, B. Jackman*, and W. B. Nowak†		8. CONTRACT OR GRANT NUMBER(s)	
9. PERFORMING ORGANIZATION NAME AND ADDRESS U.S. Army Materials Technology Laboratory Watertown, Massachusetts 02172-0001 SLCMT-EMM		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS	
11. CONTROLLING OFFICE NAME AND ADDRESS U.S. Army Laboratory Command 2800 Powder Mill Road Adelphi, Maryland 20783-1145		12. REPORT DATE September 1990	
13. NUMBER OF PAGES 13			
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified	
15a. DECLASSIFICATION/DOWNGRADING SCHEDULE			
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited.			
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)			
18. SUPPLEMENTARY NOTES *Geo-Centers, Inc., Newton, MA †Northeastern University, Boston, MA			
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Uranium alloys Corrosion protection U-Ti alloys Coatings Physical vapor deposition (PVD) Scanning electron microscopy (SEM)			
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) (SEE REVERSE SIDE)			

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Block No. 20

ABSTRACT

Aluminum, zinc, magnesium, Al-Zn, Al-Mg, nickel, titanium, TiN and Al/TiN coatings were applied by the arc plasma physical vapor deposition (PVD) technique to a depleted uranium (DU) alloy for corrosion protection assessment. The as-deposited specimens were examined by scanning electron microscopy (SEM) for surface morphology and tested for adhesion. Electrochemical polarization tests and immersion tests were conducted in aerated 3.5 wt.% NaCl solution. Results of electrochemical polarization scans and observations after long-term exposure tests indicated that both Al-Zn and Al-Mg alloys appear to be the best sacrificial coating materials for improving the corrosion resistance of DU-0.75 Ti.

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(Received March 22, 1989)

Summary

Aluminum, zinc, magnesium, Al-Zn, Al-Mg, nickel, titanium, TiN and Al/TiN coatings were applied by the arc plasma physical vapor deposition (PVD) technique to a depleted uranium (DU) alloy for corrosion protection assessment. The as-deposited specimens were examined by scanning electron microscopy (SEM) for surface morphology and tested for adhesion. Electrochemical polarization tests and immersion tests were conducted in aerated 3.5 wt.% NaCl solution. Results of electrochemical polarization scans and observations after long-term exposure tests indicated that both Al-Zn and Al-Mg alloys appear to be the best sacrificial coating materials for improving the corrosion resistance of DU-0.75 Ti.

1. Introduction

High density depleted uranium (DU) alloys possess superior armor piercing capabilities but they are susceptible to corrosion when exposed to high humidity and salt laden environments. In order to mitigate corrosion, various metallic coatings such as aluminum and TiN have been applied by a cathodic arc plasma physical vapor deposition (PVD) process. The ions in the plasma have a high vapor energy level (50 - 150 eV) and the plasma has a high ratio of ions to neutral atoms (70 - 95%) [1]. This PVD system can operate over a wide range of pressures (high vacuum to 1×10^{-1} Torr) and has the ability to deposit many materials. The high vapor energy level and the high ion ratio are the most attractive features among others which may be controlled to optimize the microstructure and composition of the deposited layer to produce coatings with better corrosion resistance.

In an earlier paper [2] on the corrosion behavior of an IVD aluminum coated* DU-0.75 Ti alloy, it was found that the coating provided general corrosion protection after 12 days of exposure to a 5% salt fog environment except for pits that initiated and grew at pin-hole defects in the coating. Defects such as cracks or porosity, either pre-existing or induced by the environment may be a controlling factor affecting corrosion behavior of coated specimens. For deposited metals which are anodic (sacrificial) with respect to DU-0.75 Ti, the presence of a small number of defects in the coated layer may be acceptable. For cathodic type (noble) coatings, the existence of a single pore or crack in the coated layer will be detrimental.

In this study, the following nine different metallic coatings were deposited on DU-0.75 Ti alloy specimens: zinc, magnesium, Al-Zn, Al-Mg (anodic coatings); aluminum, nickel, titanium, TiN (cathodic); and a dual layered coating of aluminum and TiN (Al/TiN). Reported here are SEM examinations of the deposit surface morphology, results of adhesion tests, and assessment of the corrosion resistance of the as-coated specimens by short-term electrochemical polarization tests and long-term immersion tests up to 120 days in 3.5 wt.% NaCl aqueous solution. Data obtained for the uncoated DU-0.75 Ti alloy are also included for comparison.

2. Experimental procedure

Specimens were fabricated from a long rod of DU-0.75 Ti alloy of diameter 25.40 mm, heat treated at 850 °C, water quenched at 0.46 m min⁻¹, and aged at 350 °C for 16 h to obtain the desired microstructure, which consisted of a major α phase and a minor U₂Ti phase. This alloy had a martensitic microstructure and a grain size of about 400 μ m. Two types of disk specimens were cut from the treated rods; larger ones with a diameter of 25.40 mm and thickness of 6.350 mm for immersion and adhesion tests, smaller ones with a diameter of 15.885 mm and thickness of 3.175 mm for electrochemical polarization tests. The disks were ground and polished through 600 grit silicon carbide paper to a surface roughness of about 0.25 μ m and ultrasonically cleaned.

The coatings were applied by Nuclear Metals, Concord, MA, employing the cathodic arc plasma (PVD) process in a Multi-Arc Vacuum System (Multi-Arc Vacuum Systems, St. Paul, MN). Before placement into the deposition chamber, the specimens were degreased in ethyl alcohol, ultrasonically cleaned in detergent, rinsed and air dried. Electro-polishing and acid cleaning were also used as needed.

The specimens were loaded into the chamber and pumped down to a vacuum of 1.0×10^{-5} Torr prior to starting the arc. This was followed by

*Applied by the McDonnell Douglas Ivdizer System; McDonnell Douglas Corp., P.O. Box 576, St. Louis, MO 63166, U.S.A.

nitrogen ion bombardment to the substrate for about 1 h to sputter off remaining contaminants on the surface and to heat the specimen. During deposition, the temperature of the specimen was controlled to remain below 300 °C and the vacuum was maintained at 20×10^{-3} Torr. The thickness of the coatings ranged from 3 to 13 μm , deposition usually taking 1–2 h to complete, depending on the coating metals and the type of coatings: one or two layers, single metal or two-component alloy selected. The as-coated specimens were ultrasonically cleaned, then examined by SEM for surface morphology. It should be noted that coating parameters employed for deposition of these coatings were based on limited trials and do not represent optimum conditions.

Electrochemical corrosion testing of the coatings was conducted in aerated 3.5 wt.% NaCl aqueous solutions, pH 5.5, at room temperature (25 °C). A Princeton Applied Research potentiostat–galvanostat model 273 was used for potentiodynamic polarization of the specimens. An anodic scan rate of 20 mV min⁻¹ was used with a saturated calomel reference electrode (SCE), two high density, non-permeable graphite rod counter electrodes, and a PAR standard flat specimen holder model K105, which employed a sealing knife edge washer of Teflon to expose 1 cm² of specimen area to the test solution. Tests began after 1 h of immersion to achieve steady state. All electrochemical potentials are reported against SCE.

Adhesion tests were qualitative and consisted of placing transparent Scotch Magic Tape onto the coating, finger rubbing the tape many times until the air between the tape and coating seemed to be removed and then rapidly peeling the tape off the coating.

An immersion test was carried out to assess the relative corrosion resistance of the coatings that were tested electrochemically on other samples. The coated specimens used were the larger disks, containing an uncoated 1.600 mm diameter hole, drilled 9.525 mm deep in a direction normal to the through thickness. This hole represented a coating defect that exposed the substrate DU–0.75 Ti alloy to the NaCl solution and concomitant galvanic corrosion. Each disk was placed flat surfaces horizontal, separately in its own 250 ml Pyrex beaker filled to the 200 ml mark with a 3.5 wt.% NaCl aqueous solution. Each beaker was sealed by a parafilm sheet punctured with a pinhole, and left standing at ambient temperature (about 25 °C). Physical appearances were noted as a function of time and recorded.

3. Results and discussion

3.1. SEM examination of as-deposited coatings

The surface morphology of representative coatings are shown in Fig. 1 (a–c). Generally the deposits contain microdroplets and defects such as pits or pores and cracks. Microdroplets are often contained in coatings deposited by the cathodic arc process [3]. The concentration of microdroplets can be controlled by optimization of process parameters such as cathode–substrate

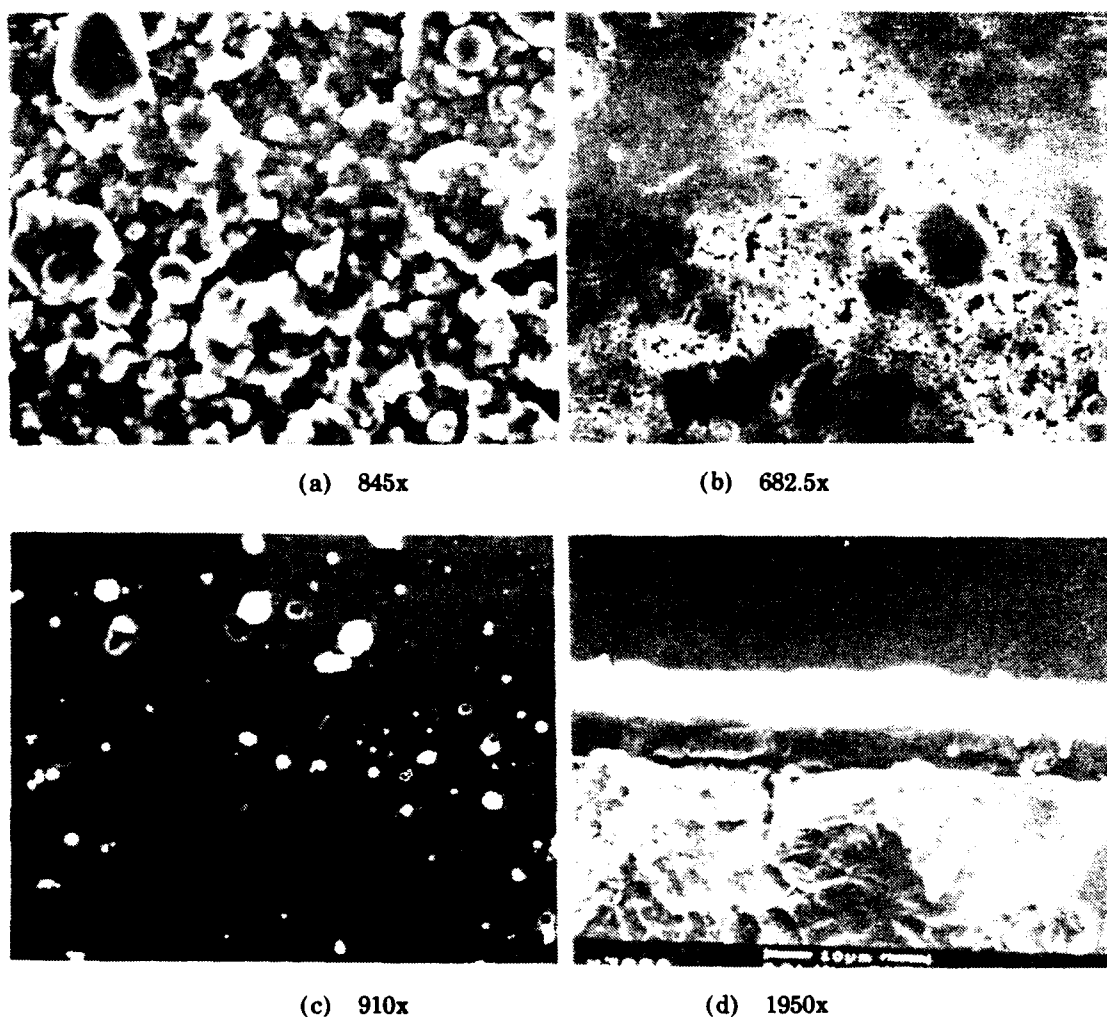


Fig. 1. SEM photomicrographs of cathodic arc plasma PVD coated DU-0.75 Ti alloy specimens: (a) aluminum, (b) Al-Zn, (c) TiN, (d) cross-section of representative coating-substrate (TiN/DU-0.75 Ti).

geometry, arc current, cathode temperature, etc. The cracks are likely caused by the difference in thermal expansion coefficients of coating and substrate. The surface morphology of each coating is described in detail in Table 1. A cross-section of a representative coating/DU-0.75 Ti substrate is shown in Fig. 1(d). The coating is adherent, uniform in thickness and fine grained. A columnar structure was not observed.

3.2. Adhesion test

The adhesion of all coatings was excellent as judged by the Scotch Tape test. No removal of coating from substrates was evident. Excellent adherence of the coating to its substrate is reported as one of the main advantages of the cathodic arc process due to the higher degree of ionization and energy level of the coating material leaving the arc source [1].

TABLE 1
Morphology of coatings (from SEM evaluation)

Coating	Morphology
Aluminum (Fig. 1(a))	Rough surface. Appears to be synthesis of a spray of droplets and an atomic vapor stream condensing on substrate. Particle size: 0.7 - 5.0 μm . Larger "droplets" are clusters.
Zinc	Fewer droplets than aluminum. Irregular pits.
Magnesium	Agglomeration of droplets and layering, with irregular islands of agglomerates with smooth surface. Deeper layers of agglomerates seen between islands.
Al-Zn (Fig. 1(b))	Similar to zinc. No particles. Spongy structure with holes of diameter 1 μm and smaller.
Al-Mg	Similar to aluminum. Characteristic agglomerated particles. Porous, low density structure visible at surface.
Al/TiN	Identical to aluminum. Large agglomerated masses. Small particles of diameter 1.5 - 4 μm . Aluminum coating appears to have interconnected porosity.
Nickel	Some deep pits, large and small agglomerates, some with cracks surrounding them. Underlying smooth dense film. Particle size: 0.5 - 2.5 μm .
Titanium	Underlying dense, smooth film with numerous pits. Overlayer of particles 1.5 - 7 μm . Voids surrounding some agglomerates.
TiN (Fig. 1(c))	Gold color. Scratches, droplets, some pits (may be sites where droplet broke away during handling and cleaning).

3.3. Electrochemical polarization tests

The anodic polarization curves for the coatings-alloy systems in 3.5 wt.% NaCl solution are shown in Figs. 2, 3 and 4. The important electrochemical parameters are corrosion rest potential (E_C), pitting potential (E_P) and protection range (E_{PR}); these have been extrapolated from these curves and are listed in Table 2. The protection range has been defined by Nowak *et al.* [4] as the difference in potential between the corrosion potential and the onset of pitting. The corrosion potential is the equilibrium or steady state potential of the alloy or coating.

If the corrosion potential of the coating is more active or electronegative than the substrate DU-0.75 Ti alloy, which is the case for zinc, magnesium, Al-Zn, and Al-Mg, then the coating is considered as sacrificial, that is, it provides galvanic protection to the DU-0.75 Ti. The direction of galvanic current through the NaCl solution is from coating to DU-0.75 Ti alloy and as a consequence the alloy is cathodically protected. McIntyre *et al.* [5] and

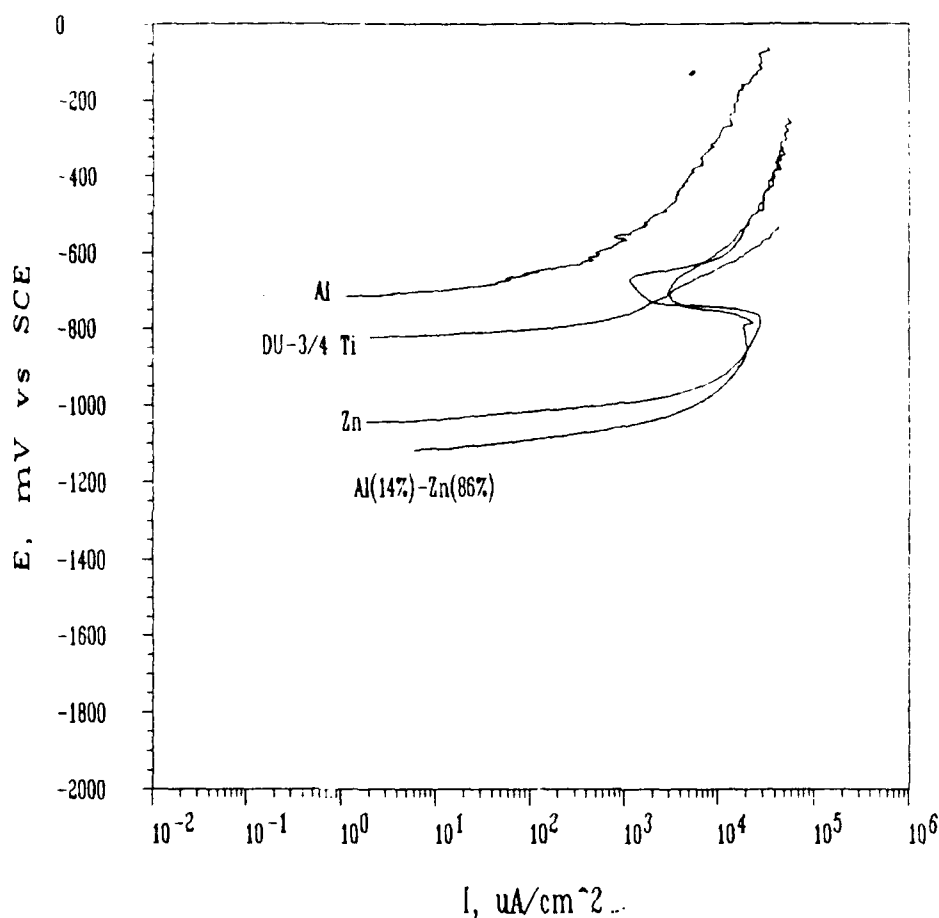


Fig. 2. Electrochemical anodic polarization curves of uncoated and coated (aluminum, zinc, Al-Zn) DU-0.75 Ti alloy in aerated 3.5 wt.% NaCl solution, 20 mV min⁻¹.

TABLE 2

Results from potentiodynamic polarization tests

Specimen	E_C^a (V _{SCE})	E_P^b (V _{SCE})	E_{PR}^c (V)
Titanium ^d	-0.27 C ^e	0.20	0.47
Nickel ^d	-0.39 C ^e	-0.08	0.31
Al/TiN ^e	-0.71 C ^e	-0.60	0.11
Aluminum ^d	-0.72 C ^e	-0.64	0.08
TiN ^f	-0.78 C ^e	-0.78	0.00
DU-0.75 Ti	-0.82	-0.71	0.11
Zinc ^g	-1.05 A ^h	-0.67	0.38
Al-Zn ^g	-1.12 A ^h	-0.65	0.47
Al-Mg ^g	-1.22 A ^h	-0.65	0.57
Magnesium ^g	-1.45 A ^h	-0.75	0.70

^aCorrosion (rest) potential at 55 min after specimen introduction to corrosion cell.

^bPitting potential. Designated as potential observed at onset of pitting.

^cProtection range. Designated as the potential difference $E_P - E_C$.

^dFilm delaminated above E_P .

^eFilm broke and flaked off above E_P .

^fSpontaneous pitting occurred upon specimen immersion in test solution; substrate visible through coating.

^g E_C is Anodic with respect to DU-0.75 Ti; coating is sacrificial.

^h E_C is Cathodic with respect to DU-0.75 Ti; coating is noble.

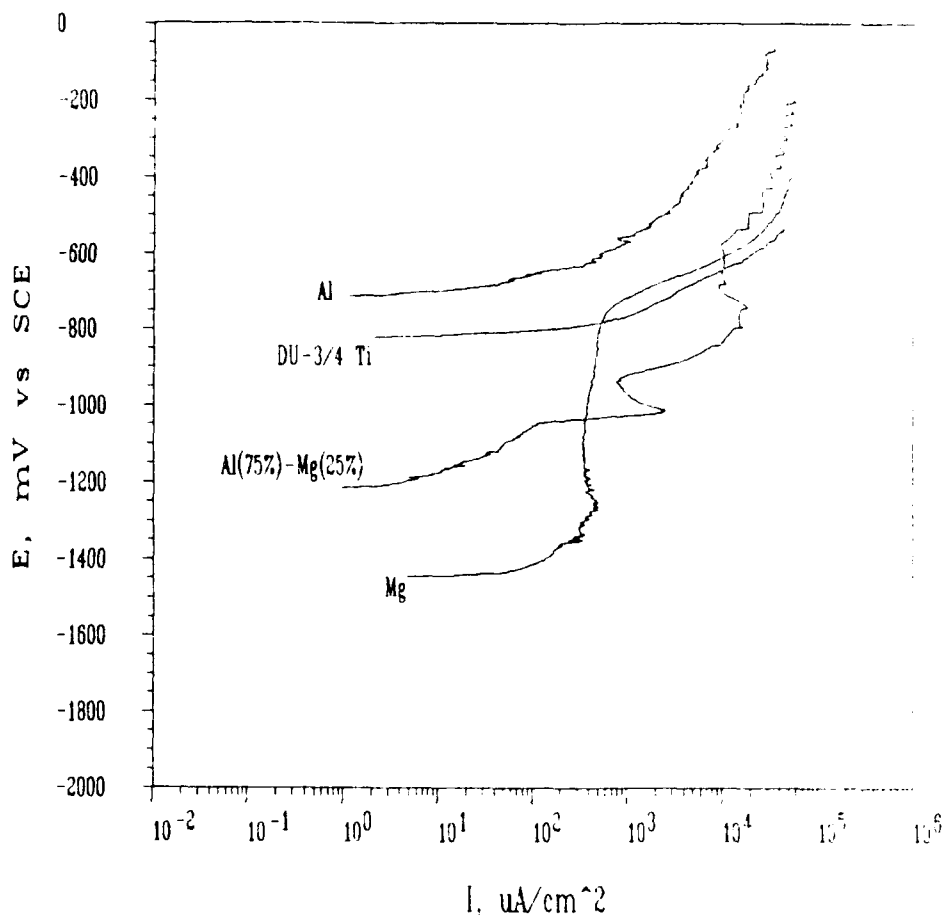


Fig. 3. Electrochemical anodic polarization curves of uncoated and coated (aluminum, magnesium, Al-Mg) DU-0.75 Ti alloy in aerated 3.5 wt.% NaCl solution, 20 mV min^{-1} .

Briggs [6] have also reported that zinc protective layers were active to DU-0.75 Ti and preferentially corroded at an accelerated rate. The degree of porosity of sacrificial coatings therefore is not of great significance but long term cathodic protection will depend on the thickness of the coating [7]. In comparison, the remaining coatings, i.e. aluminum, nickel, titanium and TiN, exhibit a more noble or electropositive potential with respect to the DU-0.75 Ti alloy. If any pores or cracks are present in the noble coating then the direction of galvanic current accelerates attack of the DU-0.75 Ti at these defects and eventually will undermine the coating. Weirick [8] also reported that aluminum and nickel coatings on DU-0.75 Ti alloy were noble and any flaws therein could result in an increased local corrosion rate.

The protection range of all the coatings listed in Table 2 indicates that passive behavior is observed here due to the formation of oxide films and in the case of the Al-Zn and Al-Mg coatings spinels may also form [4, 9]. Above this range pitting was observed. The sacrificial coatings Zn, Mg, Al-Zn, and Al-Mg exhibited the largest protection ranges with 14 at.% Al-86 at.% Zn offering the protection exceeding that observed by the individual films of aluminum and zinc. Nowak *et al.* [4] speculated that the excellent

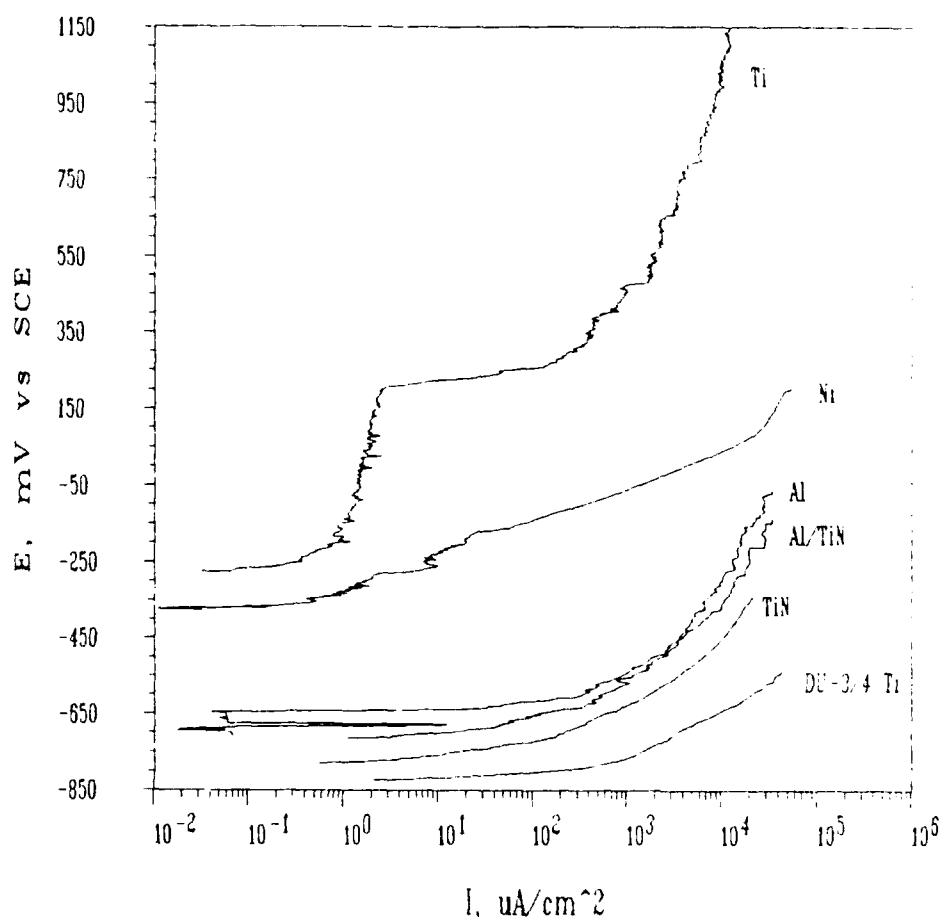


Fig. 4. Electrochemical anodic polarization curves of uncoated and coated (nickel, titanium, aluminum, TiN, Al/TiN) DU-0.75 Ti alloy in aerated 3.5 wt.% NaCl solution, 20 mV min^{-1} .

corrosion resistance of ion plated Al-Zn films was due to the Al-Zn alloys initially corroding preferentially at the zinc-rich sites followed by an aluminum corrosion character (formation of a hydrated Al_2O_3 passive layer) that delayed and was mediated by the presence of zinc. Also a very thin Al_2ZnO_4 spinel barrier layer may exist at the metal-oxide interface, providing enhanced passivity similar to the Fe_3O_4 spinel layer between iron and Fe_2O_3 .

The 75 at.% Al-25 at.% Mg coating also exhibited a relatively large protection range. The surface protective layer could consist of a mixture of Al_2O_3 and MgO with different degrees of hydration or could be a spinel structure of Al_2MgO_4 . The work of Nowak and Seyyedi [9] on ion plated Al-Mg alloy films with up to 52 at.% Mg showed that these alloys could develop the spinel structure which at higher magnesium content was an approximately stoichiometric spinel.

3.4. Immersion tests

Photographs of specimens taken through the beaker walls and from above after 32 days of exposure are shown in Fig. 5(a - j). Visual observations conducted after 120 days of exposure are reported for each coating system in

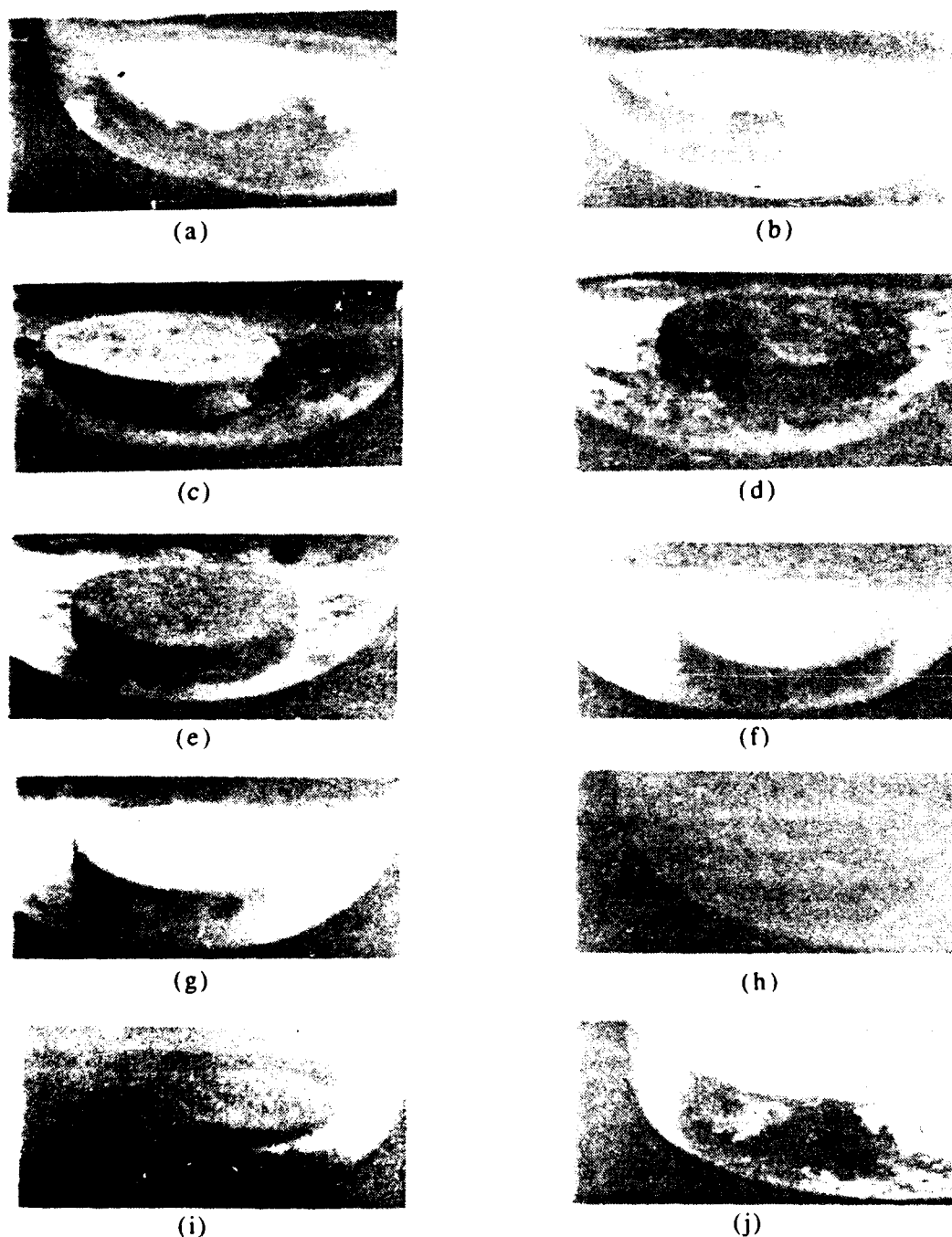


Fig. 5. Photographs of immersion test specimens 32 days after placement in aerated 3.5 wt.% NaCl solution at 25°C: (a) DU-0.75 Ti, (b) aluminum coating, (c) zinc coating, (d) magnesium coating, (e) Al-Zn coating, (f) Al-Mg coating, (g) Al/TiN coating, (h) titanium coating, (i) nickel coating, (j) TiN coating.

Table 3. The degree of corrosion was rated on a scale of 0 (severe) to 10 (negligible). The DU-0.75 Ti alloy was considered baseline with a rating of 0.

A review of the observations in Table 3 showed that the sacrificial coatings such as Al-Zn and Al-Mg provided better corrosion protection to DU-0.75 Ti. The Al-Zn coating appeared to be the best of the sacrificial

TABLE 3

Observations after 120 days of immersion tests

<i>Material</i>	<i>Rating</i>	<i>Observations</i>
DU-0.75 Ti	0	Black corrosion product over entire specimen (UO_2 or UH_3).
Aluminum	6	White corrosion product (Al_2O_3 hydrate). DU corrosion products: yellow (UO_3 hydrate), black (UO_2). No cathodic protection around defect.
Zinc	4	Black corrosion product over 75% of surface (UO_2), yellow deposits (UO_3 hydrate), some cathodic protection around defect. No evidence of white ZnO or Zn_2OCl .
Magnesium	4	Entire surface covered with black corrosion product (UO_2), some cathodic protection around defect. No evidence of white MgO hydrate.
Al-Zn	10	Some white corrosion product (mixed oxides or spinel), no evidence of DU corrosion. Cathodic protection around defect.
Al-Mg	8	Some white corrosion product (spinel), small area of black corrosion product (UO_2). Cathodic protection around defect.
Al/TiN	6	Same as aluminum.
Titanium	2	Black corrosion product (UO_2). Severe galvanic attack around defect.
Nickel	2	Black corrosion product (UO_2). Severe galvanic attack around defect.
TiN	3	Black corrosion product (UO_2), delamination of coating, galvanic attack around defect.

coatings, forming little or no corrosion products with a clear solution. This coating afforded cathodic protection to the defect introduced into the specimen. There was no evidence of corrosion around the defect area. The Al-Mg coating provided the DU-0.75 Ti with similar cathodic protection. For the aluminum coating, the defect was corroded suggesting no cathodic protection was provided, thus its corrosion resistance should depend on the integrity of the deposited coating. This also will be true for the Al/TiN coating, but the Al/TiN coating system was more protective than the TiN coating, because of the presence of the aluminum top layer.

It is interesting to note that the zinc and magnesium coatings did not provide good corrosion protection to DU-0.75 Ti even though they are sacrificial to the substrate and exhibit a large protection range. The aluminum may extend the protective life of zinc and magnesium by moderating the rate at which each of these elements preferentially corrodes. Also, titanium performed poorly despite the relatively large protection range listed in Table 2.

4. Conclusions

It is known that most commercially prepared metal coatings contain porosity in some degree. The present work involving nine different coatings applied by the cathodic arc plasma PVD process also showed by SEM examination that fully dense coatings free of defects were not produced. From the corrosion standpoint, the galvanic action at the base of a defect such as a pit or crack which penetrates through the coating thickness will be an important factor in determining coating efficiency. Since the corrosion potential of the DU-0.75 Ti is relatively active, only materials such as zinc, magnesium, Al-Zn and Al-Mg may be used as sacrificial coatings. The other coatings, *i.e.* nickel, titanium, aluminum and TiN, are shown to be more noble than DU-0.75 Ti. Therefore they should only be used if a defect free film can be obtained. Accordingly, the useful life of sacrificial coatings is governed by the thickness whereas that of noble coatings is controlled by the amount of defects.

Of the nine metallic coatings studied, it is apparent that the Al-Zn and Al-Mg alloy systems have the best potential for protecting DU-0.75 Ti. However, additional work is needed to optimize these coatings with respect to composition and producing a more desirable dense morphological structure which would exhibit an equiaxed grain structure.

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ASSESSMENT OF CORROSION RESISTANT COATINGS
FOR A DEPLETED U-0.75 Ti ALLOY -
F. Chang, M. Levy, B. Jackman, and
W. B. Nowak

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