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Materials and Structures

Electroplating of Tungsten from Nonaqueous Lewis Acid Solutions

SEMIANNUAL TECHNICAL REPORT

(Period Covering 1 July-31 December 1961)

28 FEBRUARY 1962

*Prepared by F. D. HESS and L. SCHIELER
Chemical Propulsion Department
Aerodynamics and Propulsion Research Laboratory*

Prepared for DEPUTY COMMANDER AEROSPACE SYSTEMS

AIR FORCE SYSTEMS COMMAND

UNITED STATES AIR FORCE

Inglewood, California

17305



LABORATORIES DIVISION • AEROSPACE CORPORATION

CONTRACT NO. AF 04(647)-930

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ABSTRACT

An experimental investigation of the electroplating of tungsten from organic electrolytic solutions is described. The method depends on solution of a tungsten halide in a Lewis acid solvent which will leave the tungsten in the cationic state. Preliminary tests with tungsten hexachloride dissolved in a boron trifluoride etherate did not yield a metallic tungsten plate.

The work described in this document was done during the period covering 1 July - 31 December 1961.

CONTENTS

ABSTRACT	iii
I. INTRODUCTION	1
II. TECHNICAL PROGRESS	9
III. FUTURE WORK	11

Table 1	13
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ELECTROPLATING OF TUNGSTEN FROM NONAQUEOUS LEWIS ACID SOLUTIONS

1. INTRODUCTION

The high melting-point of tungsten makes it a logical choice for rocket nozzles, stimulating much recent interest in methods of fabricating this metal. A few years ago the techniques of tungsten metallurgy were extremely limited - sintering of powder compacts, extrusion, rolling, drawing, and machining being adequate to meet the requirements of tungsten-filament manufacture. Since then a variety of novel techniques has been used more or less successfully for the production of larger and more intricate shapes. Vapor-phase deposition (by reduction from a compound, rather than vacuum evaporation of the metal), arc-melting and vacuum casting, spinning, brazing, forging, plasma-jet cladding, explosive forming and electron-beam welding have all taken their places in the metallurgists' repertory.

It is singular that a relatively old method of metal forming, that of electro-deposition, has never been successfully adapted to tungsten. Its failure to plate from aqueous solutions diminished the interest of early investigators. More recently the possibilities of plating tungsten from nonaqueous solutions have been reviewed, but the results have not been promising as far as a practical process is concerned. Jonassen at Tulane tried various complexes in organic and nonaqueous solvents, while the comprehensive work under Dr. Brenner* at the National Bureau of Standards was discontinued owing to the

*A. Brenner, Private Communication, Nov. 17, 1961.

success of the vapor-phase process. The NBS review of previous attempts at tungsten plating discloses the occurrence of the following undesirable features:

- a. Plating stops after an initial flash deposit
- b. Badly pitted deposits occur
- c. Deposits contain occluded oxides
- d. Deposits have unacceptably poor mechanical properties
- e. Codeposition techniques with other metals from aqueous solutions have been utilized successfully, but the practical upper limit is 35 percent tungsten
- f. Cathode efficiency is unusually low.

To understand why plating from aqueous solutions has been unsuccessful, it should be noted that these solutions, in addition to tungsten, also contained one or more different cations. Water solutions contain hydrogen ions (H^+) and when sodium tungstate was used to supply tungsten, sodium cations were also introduced. During electrolysis competition occurs between the various cations for deposition at the cathode. In aqueous tungsten solutions, the experience has been that the hydrogen is more easily plated out than tungsten. The sodium ion is usually not considered troublesome since it is far more electronegative than tungsten or hydrogen.

Standard equilibrium half-cell potentials for the elements indicate tungsten should plate out as easily as hydrogen from aqueous solutions. Voltages can be calculated by the equilibrium half-cell equations:

$$E_m^s = E_m^o + \frac{RT}{nF} \ln A, \quad (1)$$

where

E_m^o = standard voltage,

E_m^s = voltage corresponding to a particular metal ion activity A (its active concentration),

n = the valence of the metal,

F = the Faraday electrochemical equivalent, and

R = the gas constant, 8.32 joules.

In a plating process (nonequilibrium) the above equation must be modified by inclusion of a polarization voltage (P) to account for the nonequilibrium condition. Values of P are not available in the literature and generally must be determined experimentally for each particular cell:

$$E^d = E^s - P \quad (2)$$

If half-cell equations are used to predict actual voltages, one must know specifically the ion mechanism and the activity (concentration) of the particular ion involved in the cathode film. In some cells, tungsten in particular, it is known that suboxides deposited at the cathode interfere with the plating, producing a cathode film. The presence of a cathode film can interfere with the ion migration and materially change the ion concentration and the ion mechanism. For this reason, the half-cell potentials are not particularly useful for the analysis of tungsten plating. In plating tungsten, a cathode film builds up and polarization sets in, retarding the deposition of the tungsten.

Unsuccessful efforts have been made to control the relative concentration of the tungsten and hydrogen ions in aqueous solutions to overcome the polarization tendency. A metal (M) on dissolving in water forms the simplified species MOH which may ionize to some extent into M^+ and MO^- ions as follows:



where (MOH) is the undissociated acid or base. The solution is acidic if (MOH) dissociates (ionizes) as in (3) to yield hydrogen ions, and basic if it ionizes as in (4) to yield hydroxyl ions (OH^-) . In a simplified model of and equations for the ionization of acids and bases, the following expression results:

$$(M^+) = \frac{(H^+) (MOH)}{K_a} = \frac{(H^+) K_h (MOH)}{K_w} \quad (5)$$

where (MOH) is the concentration (activity) of the undissociated metal compound, K_a and K_b are the acid and base ionization constants, and K_w is the ionization constant for water. K_a has, at a given temperature, a fixed value and (MOH) will be limited by the saturation concentration. This leaves the (H^+) ion as the one factor that can be used to adjust the metal ion concentration (M^+) . Because of the polarization problem, however, it is also desirable to achieve a large value for the (M^+) to (H^+) ion ratio. The desired parameter, containing these two effects, is obtained by dividing equation (5) by (H^+) which yields

$$\frac{M^+}{H^+} = \frac{MOH}{K_a} = \frac{K_b(MOH)}{K_w} \quad (6)$$

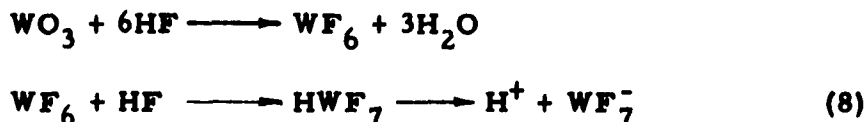
This expression is sometimes referred to as being proportioned to the cathode efficiency. It has been used successfully in relating actual plating efficiencies to electrolyte composition. For the particular model chosen here it is seen that the (M^+/H^+) ratio or cathode efficiency is not independently a function of the hydrogen ion concentration but rather is limited by the saturation concentration of the undissociated acid (MOH). While this picture may be somewhat oversimplified, it does correspond with experience that in tungsten plating, polarization and hydrogen deposition are encountered, and altering the (H^+) concentration does not provide a solution.

In any cathodic plating process for a metal it is desirable to promote cation formation and suppress the formation of an anion (MO^-) . Again, using the simplified model above the following expression results

$$\frac{(M^+)}{(MO^-)} = \left(\frac{H^2}{K_a} \right) \quad (7)$$

Consequently, the ratio for cation formation (M^+/MO^-) will be more favorable under acid conditions. Unfortunately, tungsten oxide (WO_3) or tungstic acid (H_2WO_4) is virtually insoluble in water or in solutions of noncomplexing acids.

Complexing acids are not helpful because they promote the anion form of (WF_7), as is the case with hydrofluoric acid.



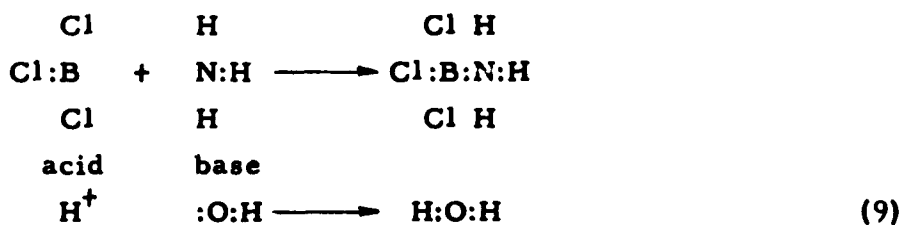
The use of hydrochloric acid is reported to cause almost complete precipitation of (H_2WO_4).

The aqueous electroplating of tungsten appears to be unfavorable because of the following effects:

- a. Suboxides of tungsten precipitate at the cathode.
- b. Polarization and hydrogen plating usually results in low cathode efficiencies regardless of the hydrogen ion concentration.
- c. Tungstic acid is insoluble in acid solution.

Because of the obstacles listed above some attention has been directed to nonaqueous solutions. Some experimenters have tried organic amines (e. g. , ethylene diamine) and a few have employed fused alkali salts (e. g. , $\text{Na}_2\text{WO}_4 + \text{Li}_2\text{WO}_4 + \text{WO}_3$) at about 1020°C without appreciable success.

To understand the ionic behavior of metal oxides in a nonaqueous solvent, consider the Lewis acid-base concept. According to Lewis, an acid is capable of accepting a share in a lone electron pair from a base to form a coordinate covalent bond, and a base donates a share in a lone electron pair to the acid. Examples are:

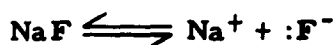


Here the boron trichloride and hydrogen act as acids to combine with their respective bases, ammonia and hydroxyl. This definition does not require the presence of a solvent. It is, however, quite usable for water solutions and has the added facility of characterizing nonaqueous and nonsolvent acid base reactions.

One form of tungsten suitable for a nonaqueous solvent is the hexafluoride. In solution it may ionize as follows:

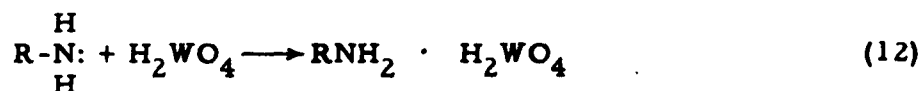


If a fused alkali salt such as sodium fluoride were used:

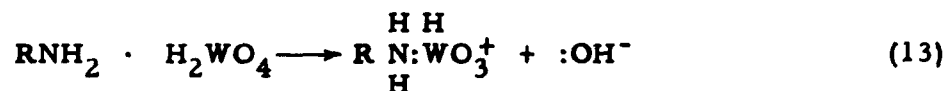


The WF_6 would be observed to act as an acid and accept the electron pair of the basic fluoride ion. If (11) characterizes the reaction correctly for an alkali salt melt then the wrong ion has been promoted. This, in fact, appears to be the case; if the salt is Na_2WO_4 or WF_6 or WCl_6 the tungsten ion will be predominately negative.

Another recommended method is the use of an organic amine (R-NH_2) to dissolve tungstic acid:



which would ionize as:



Characteristically, an amine is basic and while tungsten oxide may be thought of as being somewhat amphoteric, it is actually a mildly strong acid (like the other members of its chemical group; periodic members are chromium, molybdenum and tungsten). The reaction (12) is as expected. In (13), H_2WO_4 is a base. This is incompatible with (12) and the Lewis concept that the base R-NH_2 will donate an electron pair to an acid. Therefore, the presentation of H_2WO_4 as a base in the reaction (13) must be ruled out.

Since it is desired to promote tungsten as a base so that it may provide cations for electroplating, it follows from the above, that it should be in an acid environment. Typical of a Lewis base it will then share one of its electron pairs with a Lewis acid, which, upon ionization, will provide the required tungsten cations. In addition, in order to avoid the deleterious side-effects described previously, the acid and all other constituents to be selected should not provide ionic H^+ , (the hydrogen plating problem) nor available oxygen that could shift to the tungsten (the tungsten suboxide cathode film problem).

An approach that avoids the difficulties enumerated above is to dissolve a tungsten compound in a solvent that is an acid, that can also dissolve suitable Lewis acids, and will not provide H^+ nor available oxygen. Examples of suitable acids are BF_3 , BCl_3 , AlCl_3 , SiF_4 , PF_5 , and GeF_4 , all of which are characterized by their affinity for bases, and in solution, complex with halides which ionize to BF_4^- , BCl_4^- , AlCl_4^- , SiF_6^{2-} , PF_6^- , and GeF_6^{2-} . It would be appropriate to select either the fluoride or chloride salt of tungsten. WF_6 and WCl_6 are examples.

In a suitable solvent, WF_6 will ionize in the following manner:



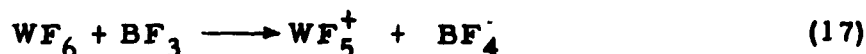
This reaction will be promoted by the Lewis acid which will coordinate with the fluoride ion



In aqueous acid solutions it is believed that tungsten complexes with the fluoride ion of hydrofluoric acid to give the anion (WF_7^-).



As is shown in (15), the proposed acids furnish the desired cation WF_5^+ of (14), and additionally suppress the formation of the undesired anion WF_7^- of (16). The reaction between the tungsten and the acid can be simply presented as follows:



If the environment allows the tungsten also to be divalent, then a second reaction may occur.



Since some of the proposed acids, listed above, are gaseous at room conditions and to allow a wide variation of solute concentrations, a solvent is probably necessary. For reasons presented earlier the solvent should not provide (H^+) nor available oxygen, and should not interfere with the reaction of Equation (17). The solvent should be capable of dissolving considerable amounts of all reactants including the reduced valence salts of tungsten (e. g., WF_4 , WF_5 , WCl_4 , WCl_5). Possible solvents are:

- a. Diethyl ether
- b. Boron trifluoride etherate
- c. Ethyl carbitol
- d. Carbon disulfide
- e. Chloroform
- f. Trichloroethylene
- g. Cyclohexane
- h. Acetone

The electrolyte may be composed of one of the suggested solvents containing dissolved tungsten hexahalide and one or more acid additives. A combination is sought that is electrically conducting and is stable in storage.

II. TECHNICAL PROGRESS

The experimental phase of the program was initiated using metal halides and solvents that were on hand or readily procurable. Tungsten was available as a hexachloride. On account of its volatile, hygroscopic, and toxic nature all transfers were made in a dry-nitrogen atmosphere inside a glove box, and solutions were kept tightly stoppered.

As a preliminary estimate of solubility and complex formation, small unweighed quantities of various chlorides were shaken with WCl_6 in 30 ml vials. After noting the appearance of the solution, electrodes of 14 gage B & S copper wire were introduced and a 5 volt direct current passed. The following observations were made:

<u>SOLVENT</u>	<u>Supporting ELECTROLYTE</u>	<u>Appearance of SOLUTION</u>	<u>Appearance of PLATE</u>
Acetone	$SbCl_3$	Clear - pale green	Heavy gray - good bond
Ether	$SbCl_3$	Milky - some WCl_6 residue	None
Acetone	$BiCl_3$	Deep blue - slt WCl_6 residue	Heavy, buffing to bright finish
Ether	$BiCl_3$	Milky - large WCl_6 residue	Light gray film
Acetone	PCl_3	Cloudy - lemon yellow	None
Ether	PCl_3	Cloudy - lemon yellow - slt WCl_6 residue	None
Acetone	$(CH_3)_3 SiCl$	Clear - pale green	None
Ether	$(CH_3)_3 SiCl$	Clear - pale yellow - slt WCl_6 residue	None

While good plates were obtained in two instances, they were soft and had a greasy, lead-like appearance, making it unlikely that they contained appreciable amounts of tungsten. Nevertheless, some confirmatory testing was done, using the same equipment but starting with one weight percent acetone solutions of SbCl_3 and BiCl_3 containing no tungsten.

The antimony chloride solution deposited a plate similar in appearance to that obtained in the previous test, when tungsten had been present. The bismuth chloride however, was largely undissolved in acetone, and no plate was obtained on electrolysis. Since 0.6 milliamp current was recorded at 5 volts, some solution had occurred. At this point a small amount of WCl_6 was introduced into the vial and the BiCl_3 residue dissolved immediately with the appearance of the deep blue color previously noted. A plate was readily obtained at 5 volts and 15 milliamps. When a few shavings of this plate were tested on the Fisher Melting Point Apparatus, the melting point was above the range of the instrument (300°C). Since pure bismuth melts at 271°C , the expectation arose that at least a codeposition with some tungsten had occurred. However, a subsequent experiment showed that this was not the case.

Two cells for electroplating tests were made from 250 ml Woulff bottles. Through one of the side necks a dial thermometer stem was inserted; through the center neck a half-inch rod tungsten anode, and through the other side neck a lead to a 2 x 5 cm copper cathode. This piece was contoured to present a concave side to the anode, and its supporting lead was sufficiently flexible to permit spacing the electrodes at any desired distance from 1/2 cm to 3 cm. A vent consisting of a glass tube with a slit rubber policeman on the upper end was provided alongside the cathode. Finally, provision was made for withdrawing samples of electrolyte through a valve attached to a ground-glass fitting in the bottom opening of the Woulff bottle. The latter connection failed the first time the bottle was exposed to a vacuum in the glove box air lock, and thereafter a plain stopper was fitted in this opening.

In the series of tests next described, since weighing and measuring operations were performed in the glove box, the accuracy of weighing was ± 0.2 percent and of liquid measurements ± 0.5 percent. Conductivity determinations were made with the Industrial Instruments Co. Conductivity Bridge, an NLS Digital Voltmeter and a Hewlett-Packard vacuum tube voltmeter providing accurate measurement of volts and amperes respectively. Plating tests were performed at ambient room temperature and no significant change in temperature occurred. (See Table I.)

To determine the nature of the deposit obtained from Tests 6 and 7, scrapings were made and fused with an acid flux. The melt was dissolved, acidified with HNO_3 and HCl , boiled and filtered, and 10 percent cinchonine solution added. No precipitate was observed, leading to the conclusion that no plating of tungsten occurred. However, after standing cold for several days a slight precipitate was observed in the sample from Test No. 6. This has not been analyzed.

Bismuth was determined in the sample from Test No. 7 by neutralizing with NH_4OH , adding $(\text{NH}_4)_2\text{S}$ and weighing the dried precipitate as Bi_2S_3 . Of the original sample, 104 percent was recovered as bismuth, the error was probably due to the presence of copper in the electrode scrapings. Assuming that the plate was pure bismuth, a cathode plating efficiency of 52 percent was obtained.

III. FUTURE WORK

Present work will be continued and amplified in some respects to thoroughly explore the feasibility of electroplating tungsten from solutions of Lewis acids such as boron halide-etherates. Other solvents discussed in the introduction will be tried, as well as other coordinating compounds such as SiF_4 . In addition to the chloride, tungsten hexafluoride will be investigated.

If deposition of tungsten is obtained, its purity, thickness, adhesion, and hardness will be determined, and the bath temperature, current density, and solute concentration optimized, to yield a good plate at high plating efficiency

The magnitude of electrode polarization effects in Lewis acid electrolytes containing dissolved tungsten halides will be investigated.

Table I

Test No.	WCl ₆	Supporting ELECTROLYTE	Specific Conductance MICROMHOS	VOLTS	MILLI-AMPS	Appearance of PLATE
1	$\frac{M}{50}$	3 parts BF ₃ * ether complex 7 parts ether	890	12	83-75	Thin, brown-black non-metallic, poor bond
2	$\frac{M}{10}$	BF ₃ ether complex	745	3-3.5	21-26	Heavy, brown-black non-metallic, poor bond
3	$\frac{M}{10}$	Acetone	2160	6	45-91	None
4	$\frac{M}{10}$	MEK	1570	5	52	None
5	$\frac{M}{10}$	3 parts BF ₃ ether complex 2 parts acetone	2590	3	150	None
6	$\frac{M}{10}$	BF ₃ ether complex**	----	9	45-35	Thin, brown-black non-metallic, poor bond - 0.0124 g deposited in 2-3/4 hr
7	$\frac{M}{12}$	Acetone	----	6	58	Heavy, rough gray, metallic, good bond, 0.3556 g deposited in 4-2/3 hr

*Technical 48 percent BF₃

**Incomplete solution of WCl₆ obtained

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