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TITLE

FINAL REPORT - RESEARCH PROJECT CHEMICAL
MILLING OF COUNTERBORE RECESSES IN THE
URANIUM WALL OF ZIRCALOY-2 CLAD URANIUM
TUBES

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FINAL REPORT

RESEARCH PROJECT

CHEMICAL MILLING OF COUNTERBORE RECESSES

IN THE

URANIUM MALL OF KINCALOT-2 CLAD URANIUM TUBES

FOR

THE GENERAL ELECTRIC COMPANY

Classification Cancelled and Changed To

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By Authority of DS Lewis,

PR-24, 4-21-94

By DK Hanson, 5-9-94

Verified By J.E. Samsky 5-16-94

UNITED STATES CHEMICAL MILLING CORPORATION

1700 Rosecrans Avenue

Manhattan Beach, California

Don C. Atkins, Jr.
Technical Director

DECLASSIFIED
MASTER

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HW-1150 M-5

CHEMICAL MILLING OF COUNTERBORE RECESSES IN
THE URANIUM WALL OF ZIRCALOY-2 CLAD URANIUM TUBES

FINAL REPORT

REFERENCE

Document HW-64695 of April 11, 1960. Purchase Order No. DDR-98.
Prime Contract at (45-1)-1350.

SCOPE OF WORK

The original scope of the work, as set forth in the referenced document, called for the development of an etchant composition to chemically mill counterbore recesses in each end of the uranium walls of twenty-four inch (24") long samples of Zircaloy-2 clad uranium tubes. These recesses were to be to a depth of $0.100" \pm 0.005"$, with a depth variation across the uranium surface not to exceed $\pm 0.003"$. This work was to be preceded by, and based on, experiments conducted to chemically mill similar recesses in ten one-half inch (1/2") long samples of Zircaloy-2 clad uranium tubes. This original scope of work was subsequently modified, through verbal instructions, by C. H. Shaw, General Electric Engineer, during his visit to United States Chemical Milling Corporation on July 26 and 27, 1960. This modification reduced the scope of the project to the production of four inch (4") long samples of Zircaloy-2 clad uranium tubes. One pair was to be manufactured from tubes of 2.460" O.D., and the other pair from tubes of 1.050" O.D. The depth of the recesses was to be $0.100" \pm 0.005"$ for these tubes. A further request of Mr. Shaw was to attempt to chemically mill a recess in another tube to a depth of 0.250", using the same etchant and methods used for the 0.100" deep recesses.

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RESULTS AND CONCLUSIONS

This project definitely proved that it is possible to chemically mill counterbore recesses in the ends of uranium walls of Zircaloy-2 clad uranium tubes. The depths in these recesses were $0.100" \pm 0.005"$ with a depth variation across the uranium surface which did not exceed $\pm 0.003"$. When attempting to mill to a depth of $0.250"$, using the same etchant and methods, the results were not as successful because of the relatively unsatisfactory tolerances experienced.

The most satisfactory etchant developed to date consists of an aqueous mixture of sulfuric and nitric acids modified by the presence of copper ions. When the operating temperature was $160^{\circ} F$. this mixture milled the uranium at an etch rate of about $0.0015"$ per minute.

This project is considered completed and no further experimental work is contemplated on the current purchase order.

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generalities may be derived. It will be noted that the presence of chloride

these elements are presented in Table I. From these experiments, some

The composition of the elements investigated and results concerning

conducted as well in the field of elements.

the work was primarily experimental and depended heavily on previous research

moderating agents can greatly affect the results obtained. As a consequence,

of the part, and the influence of other parameters such as temperature and

to the killing of parts. The presence of alloying ingredients, configuration

often the information presented in the literature cannot be applied directly

to be alloys. Experience in chemical killing has demonstrated that very

the unalloyed metal, whereas the parts relating to this project are believed

by nitric acids. The literature refers, presumably, to the properties of

through the action of hydrochloric and sulfuric acids, but is not attached

literature further indicated that aluminum is amenable to dissolution

the metal may be dissolved through the use of nitric or sulfuric acids. The

of acids, particularly the halogen acids. Also, under certain conditions,

It is known, of course, that uranium is susceptible to the attack

of value.

by Mr. C. E. Shaw concerning experiments conducted at General Electric proved

chemical nature of the two metals involved. Also, the typed notes supplied

with a survey of the literature to react with UCN's concepts concerning the

without reaching the aluminum alloy surrounding the uranium was initiated

The development work on the element to chemically attack the uranium

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DISCUSSION

44-1150 N-5

ions in the etchant give rise to a groove at the juncture of the uranium with the inner zirconium tube wall. This effect was observed regardless of the source of chloride ions ... hydrochloric acid, ferric chloride, etc. Furthermore, variations in the temperature, or the concentration of chloride ions, did not change this effect. This groove became more pronounced at faster etch rates. The addition of wetting agents, often effective in correcting grooving of this type, did not result in any improvement. When ferric chloride was used in place of hydrochloric acid, the surface was somewhat smoother, but with accentuation of the groove.

One of the problems encountered was the formation, in most etchant mixtures, of a heavy black sludge. Although in the milling of many metals the deposition of a coating on the milling surface during the operation is of some advantage in the smoothing of the surface, such proved not to be the case during this work. Furthermore, should there be any need for having a clean milled surface, such as would be the case with welding any attachments to this area of the metal, the presence of sludge is a disadvantage. Still another problem that was characteristic of most acids was the quenching of the milling surface after removing the milling metal from the liquid. In many cases, simple rinsing of the metal surface with water or even with an alkaline solution did not suffice to stop the etching. Obviously, this is a disadvantage on any kind of a production basis since poor tolerances result.

On noting the effects of grooving and sludge formation, an etchant mixture was devised using an aqueous solution of nitric and sulfuric acid modified with the addition of copper sulfate (see Experiment 3-5). This

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etchant milled the uranium, without etching the Zircaloy to any noticeable extent. At a temperature of 150° F. the etch rate was 0.0015" per minute. The tolerances obtained on a depth of cut in the order of 0.1" were $\pm 0.0015"$. The surface finish was considerably rougher than that obtained by chemical milling of most metals, but was still thought to be acceptable. In fact, the surface condition obtained probably would be conducive to making a good welded joint.

A study was made of the effects of the concentrations of various ingredients composing the etchant. It was found that the primary etching characteristics were determined by the relative concentrations of sulfuric and nitric acids. As the ratio of sulfuric to nitric acid increases, the etch rate is decreased. A decrease in the concentration of sulfuric acid produces an increase in the etch rate, but this can be balanced by further decreases in the nitric acid. Excessive nitric acid concentration leads to the instability of the etchant and deterioration of the tolerances which can be obtained. Excessive amounts of water cause the surface to appear dull gray ... probably an oxidized condition. The simple mixture of acids, in any proportions studied, did not correct the problem of post milling of the metal after removal from the etchant. The addition of copper sulfate (as $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) did correct this problem and, at the same time, improved tolerances. Furthermore, the grooving noted when using most formulations was eliminated when copper sulfate was present in the etchant.

It should be noted that the above generalizations regarding the relative amounts of acids and their effects, as well as the effects of copper

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sulfate, must be considered to be applicable only within the limits of the concentration ranges studied. It would be incorrect to assume that the results obtained could be extrapolated to other concentration levels. A considerable amount of further work is necessary before such an assumption is valid.

The etchant composition regarded as most satisfactory is presented in Table II.

Experience has shown that the position of the part during chemical milling often can influence the results markedly. As a consequence, some attention was given to the proper positioning of the uranium-30 alloy tubes during the etching operation. Parts were milled vertically and, also, horizontally using the best etchant formulation (24-1) obtained to date. It was found that horizontal rolling of the parts tended to cause formation of enlarged pits and generally was not satisfactory. These tests included both slow and fast rotation rates while the part was in the horizontal (longitudinal axis of the tube parallel with respect to the surface of the etchant) position. Vertical rotation (with longitudinal axis of the tube perpendicular to the surface of the etchant) had a definite smoothing effect, but it promoted etching toward the center of the exposed uranium. The best and most practical system was vertical etching in a quiescent bath in which the etchant attacks the bottom end of a vertically held tube. Such an operation would eliminate, entirely, the necessity for masking. Simultaneous milling of both ends of a totally immersed tube may be feasible, but a full investigation of methods of milling was not conducted since it was beyond the

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current score of this project. The results obtained by milling the bottom
end of a vertically held tube are presented in Table III.

Don C. Atkins, Jr.
Technical Director

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TABLE I

Experiment Number	Ingredients					
	Water	Nitric acid	Hydrochloric acid	Ferric chloride	Potassium persulfate	Sodium dichromate
<u>1-1</u>		100%				
<p>Considerable gas evolution; U seemed to dissolve preferentially at the U-Zr interface. U continued to mill after removal from the etchant and subsequent washing; probably due to absorption of etchant into the metal.</p> <p>Operating temperature: 150-170° F.</p>						
<u>1-2</u>	50%	50%				
<p>Decrease in concentration of nitric acid; not as rapid milling as 1-1. U continued to mill after removal from the etchant and subsequent washing; probably due to absorption of etchant into the metal.</p> <p>Operating temperature: 150-170° F.</p>						
<u>1-3</u>		90%				10%
<p>Strong oxides; etchant fixture decomposed; results inconclusive.</p> <p>Operating temperature:</p>						
<u>1-4</u>	50%				10%	
<p>No reaction; system not active.</p>						

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TABLE I

Experiment Number	Ingredients				
	Water	Nitric acid	Hydrochloric acid	Ferric chloride	Potassium persulfate Sodium dichromate

1-5

Rapid reaction; rough surface; U seemed to mill preferentially at the U-Zr interface. U continued to mill after removal from the etchant and subsequent washing; probably due to absorption of etchant into the metal.

Operating temperature: 110° F.

100%

1-6

Rapid reaction; rough surface; U seemed to mill preferentially at the U-Zr interface; some improvement in surface smoothness. U continued to mill after removal from the etchant and subsequent washing; probably due to absorption of etchant into the metal.

Operating temperature: 110° F.

100%

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AKA-41SCM-5

TABLE I

Experiment Number	Ingredients					
	Nitric acid	Sulfuric acid	Phosphoric acid (75%)	Hydrochloric acid	Ammonium chloride	Copper sulfate

90%

10%

1-1 Slow etch rate; deposits left on U surface. U continued to mill after removal from the etchant and subsequent washing; probably due to absorption of etchant into the metal.

Operating temperature: 150° F.

.2%

99.8%

1-2 Use of wetting agent to improve surface configuration; again preferential milling of U at U-2g interface. U continued to mill after removal from the etchant and subsequent washing; probably due to absorption of etchant into the metal.

Operating temperature: 110° F.

10%

90%

1-3 Slow etch rate. U continued to mill after removal from the etchant and subsequent washing; probably due to absorption of etchant into the metal.

Operating temperature: 110-150° F.

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TABLE I

Experiment Number	Nitric acid	Sulfuric acid	Phosphoric acid (75%)	Monochloroacetic acid	Ferric chloride	Ammonium chloride	Cupric sulfate	Dowfax 2A1
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1-1

100%

Slow etch rate; sludge deposits. U continued to mill after removal from the etchant and subsequent washing; probably due to absorption of etchant into the metal.

Operating temperature: 130° F.

1-2

25.9% 17.6% 46.1%

10.4%

Fast etch rate; no sludge, shiny surface; no preferential attack of the U at U-Zr interface. Depth of cut in the order of 0.1" made. See Table 1A. U continued to mill after removal from the etchant and subsequent washing; probably due to absorption of etchant into the metal.

Operating temperature: 150° F.

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Am-150 M-5

TABLE I

Experiment Number	Ingredients			
	Water	Nitric acid	Sulfuric acid	Cupric sulfate
<u>6-1</u> Apparently less action than 3-5; surface bright but pitted; no grooving. Operating temperature: 150° F.	27.3%	37.1%	24.5%	11.1%
<u>6-2</u> System not chemically stable; some pitting of the milled U surface. Operating temperature: 150° F.	21.9%	29.8%	39.4%	8.9%

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HR-450M-5

TABLE I

Experiment Number	Ingredients						
	Nitric acid	Sulfuric acid	Citric acid	Ferric chloride	Cupric sulfate	Ammonium nitrate	Ammonium persulfate
7-1 Ferric chloride added in increments to final percentage indicated. No significant etch rate. Operating temperature: 150° F.	28.7%	52.1%		7.9%	11.3%		
7-2 No observable etch rate; system decomposed. Operating temperature: 150° F.	90.0%						10%
7-3 Etch rate too slow; possible surface smoothness improvement. Operating temperature: 150° F.	29.3%	53.3%			11.6%	5.8%	
7-4 Some increase in etch rate, but still too slow; possible improvement in surface smoothness. Operating temperature: 150° F.	27.7%	50.3%			11.0%	11.0%	
7-5 No etching. Operating temperature: 150° F.	43.3%	39.5%			8.6%	8.6%	

Nav-USC-M-5

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Rev-45C M-5

TABLE I

Experiment Number	Ingredients					
	Water	Nitric acid	Sulfuric acid	Citric acid	Ferric chloride	Cupric sulfate
7-6	29.2%	10.7%	54.2%			5.9%
Sulfuric acid added in increments to final percentage indicated; slow etch rate.						
Operating temperature: 150° F.						
7-7	25.00%	17.3%	45.3%	2.4%		9.9%
No improvement over 3-5; pitting still exists.						
Operating temperature: 150° F.						

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Her-45C M-5

TABLE I

Experiment Number	Ingredients					
	Water	Nitric acid	Sulfuric acid	Glacial acetic acid	Citric acid	Monochloroacetic acid
2-1 Etch rate satisfactory; no improvement in pitting problem. Operating temperature: 150° F.	25.00%	17.3%	45.3%		2.4%	
2-2 Etch rate satisfactory; possible slight improvement in surface. Operating temperature: 150° F.	24.48%	16.9%	44.2%		2.4%	2.4%
2-3 Results comparable to 3-5; suggest possibility of reducing copper sulfate content. Operating temperature: 150° F.	27.0%	18.7%	49.0%			
2-4 Some surface improvement; questionable advantage. Operating temperature: 150° F.	23.7%	16.4%	43.0%	12.2%		
2-5 Very slow etch rate; apparent precipitation of copper sulfate. Operating temperature: 150° F.	18.2%	12.5%	65.8%			

Cupric sulfate 2Al

9.9% .02%

9.6% .02%

5.3%

4.7%

3.5%

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TABLE I

Experiment Number	Ingre dients					
	Water	Nitric acid	Sulfuric acid	Glacial acetic acid	Citric Monochloroacetic acid	Cupric sulfate 2Al
2-6 No improvement over 2-5. Operating temperature: 150° F.	16.1%	22.3%	58.5%			3.1%
2-7 Etch rate increased; still pitting. Operating temperature:	22.3%	20.5%	54.2%			2.9%
2-8 Etch rate increased; surface pitting occurs. Operating temperature:	27.7%	19.2%	50.4%			2.7%

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TABLE I

Experiment Number	Ingredients				
	Water	Nitric acid	Hydrochloric acid	Sulfuric acid	Glacial acetic acid
11-1	20.7%	39.8%	25.9%		
Fast etch rate; rough surface.					
Operating temperature: 150° F.					
11-2	25.3%	18.2%	47.7%		
Reduction of temperature did not improve surface.					
Operating temperature: 110° F.					
11-3	23.4%	16.2%	42.6%	12.0%	
Reduced etch rate; no apparent advantage of organic acid.					
Operating temperature: 150° F.					
11-4	26.3%	18.2%	47.7%		
Grooving at U-Zr interface appearing.					
11-5	81.3%		18.7%		
Sput formation; smooth surface; extract tolerance variations.					
Operating temperature: 125° F.					

11-1 11-2 11-3 11-4 11-5

TABLE I

Experiment Number	Ingredients				
	Water	Nitric acid	Hydrochloric acid	Sulfuric acid	Other
11-6	28.4%	19.8%		51.8%	Cupric sulfate
No smut; poor tolerances.					
Operating temperature: 150° F.					
11-7	34.7%	10.4%		54.9%	
Smooth surface, no smut; slow etch rate (0.24 mm ²). Grooving at U-Zr interface.					
Operating temperature: 150° F.					
11-8	42.9%	45.0%		12.1%	
Smut formation; rapid etch rate; alloyed towards center of U core.					
Operating temperature:					

Mils per surface per minute.

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TABLE I

Experiment Number	Ingredients				
	Water	Nitric acid	Sulfuric acid	Phosphoric acid (75%)	Calcic sulfate Calcic sulfonate Sodium sulfonate
13-1 Slow etch rate; rough surface; effect of higher relative concentration of sulfuric to nitric acid observed. Operating temperature: 150° F.	44.3%	15.4%	40.3%		
13-2 Moderate etch rate; increased surface roughness as compared to 3-5. No benefit from phosphoric acid. Operating temperature: 150° F.	14.5%	10.0%	26.2%	45.1%	4.2%
13-3 Tolerances not as good as 3-5; grooving. Operating temperature: 150° F.	25.3%	10.2%	47.7%		5.2% 2.6%
13-4 No reaction. Operating temperature: 150° F.	42.5%	14.7%	38.6%		4.2%

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TABLE I

Experiment Number	Ingredients			
	Water	Nitric acid	Sulfuric acid	Cupric sulfate
15-1 No benefit due to citric acid. Operating temperature: 150° F.	25.7%	17.8%	46.5%	5.0%
17-1 Large batch prepared to test method of part rotation. See Table II. Operating temperature: 150° F.	27.0%	18.7%	49.0%	5.3%
20-1 Equipment failure, test discontinued. Operating temperature: 150° F.	28.7%	39.8%	25.9%	5.6%
21-1 Good tolerances; steel rate of 1.7 mm ² ; formula variation regarded as an improvement over 3-5. Operating temperature: 160° F.	40.6%	14.2%	37.1%	8.1%
24-1 Satisfactory etched surface; results comparable to 21-1. Operating temperature: 160° F.	40.6%	14.2%	37.1%	8.1%

* Miles per surface per minute.

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TABLE II

ETCHANT FOR MILLING URANIUM-ZIRCALOY-2 TUBES

FORMULA: 24-1

<u>Ingredient</u>	<u>Weight Percent</u>
Water (tap)	40.6
Nitric acid (42 %)	14.2
Sulfuric acid (66 %)	37.1
Copper II Sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$)	8.1
	100.0

PREPARATION OF FORMULA

The copper sulfate should be dissolved in the tap water. To this solution is added the sulfuric acid, followed by the nitric acid. When used at the operating temperature the copper sulfate will remain dissolved, but on cooling to ambient temperature there may be some precipitation of the copper sulfate. No difficulty was experienced with this precipitation since the material redissolved at operating temperatures. However, at high dissolved metal content ranges there is the possibility of problems developing.

OPERATING TEMPERATURE

1600 F.

ANTICIPATED ETCH RATE

0.0015 - 0.002 inches per minute.

ETCHANT LIFE

Not established.

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TABLE III

DEPTH MEASUREMENTS OF MILLED TUBES

Tube No. 47-100
(small tube)

Tube No. 47-115
(small tube)

top

top

bottom

bottom

Notes

- (1) Etchant was 24-1, operating at 160° F.
- (2) One end of the tube was milled at a time, then the tubes were inverted to mill the other end.

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TABLE III - Continued

DEPTH MEASUREMENTS OF MILLED TUBES

Tube No. 20-60
(large tube)

Tube No. 27-64
(large tube)

top

top

bottom

bottom

Notes

- (1) Etchant was 24-1, operating at 160° F.
- (2) One end of the tube was milled at a time, then the tubes were inverted to mill the other end.

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UNITED STATES CHEMICAL MILLING CORPORATION

PERSONNEL PARTICIPATING ON THIS PROJECT

[REDACTED]

Charles J. Vinton, Research Supervisor

G. D. Pearson, Manufacturing Research Engineer

D. C. Atkins, Jr., Technical Director

[REDACTED]

[REDACTED]

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ADDITIONAL

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The chemical milling of parts to achieve a particular configuration involves more than just the immersion of the part into a solution which will dissolve the metal. The development of the etchant is, in itself, a complicated program which, if properly fulfilled, will include the careful examination of the various factors influencing the milling. It may be relatively simple to devise a mixture which will attack a given metal, but the uniformity of etching, effects of temperature, concentrations of etchant ingredients, and the important effect of dissolved metal content pose problems which are complex and which cannot be determined from a relatively brief investigation. It is apparent that chemical milling is a dynamic operation in that the media removing the metal is continually changing as the metal dissolves. This raises questions concerning the life of the etchant, the effect of dissolved metal or metal complexes on the surface finish, etch rate, etc. It is evident, then, that a certain degree of control of the chemical system must be established before the simple process of etching can be extrapolated into production practice. Furthermore, the type of control for the etchant and the factors influencing that control will vary with the metal being milled. In the case of uranium, which heretofore had not been chemically milled and hence required the development of a new etchant, many problems must be identified, considered, and solved before a satisfactory manufacturing method can be determined.

Although the many aspects of devising, developing, and proving the necessary control procedures to establish an etchant system may present a complicated problem to those not familiar with chemical milling, USCM has

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conducted research to develop answers to this problem in the case of many metals. Simplification of the interrelationships of the etchant ingredient concentrations, temperature, etc., often can best be expressed in terms of a graph which depicts satisfactory operating regions, when to recharge the etchant mixture, and other pertinent information. Typical of the graphs is that presented in Figure 1. The upper and lower control limit curves for each of the hypothetical ingredients define an operating range within which satisfactory chemical milling results may be expected over the range of dissolved metal content falling beneath these curves. It will be noted that there is an area of operation where the dissolved metal content may be too low to give satisfactory results independent of the concentrations of the hypothetical ingredients "A" and "B". At the other end of the curve is an area where unsatisfactory results are obtained when the dissolved metal content is too high. Thus, it is apparent that within certain regions of the etchant composition the optimum results are obtained.

From this graph it is evident that as the dissolved metal content increases, during the normal milling operation, there will be a time at which good production practice would indicate that part of the bath should be discarded and fresh etchant added to the mixture. By knowing the behavior of the etchant it is possible to schedule dumping and recharging of the etchant at a time when it will interfere least with production of parts. There is, of course, the possibility of transferring (perhaps on a continuous or semicontinuous basis) part of the etchant to a holding tank where the dissolved metal content could be lowered by precipitation of some of the

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metal salts. This precipitation of metal salts can be enhanced by decreasing the temperature of the etchant. Although such a procedure has not yet proved economical because of the relatively little value of the metal salts originating from metals currently being chemically milled on a production basis, this may not be the case if the metal salts are high in uranium content.

One of the other facets of chemical milling that becomes more apparent when research of this nature is undertaken is the identification of causes of unsatisfactory milling results. For example, in the case of many metals it is possible to relate poor surface finish or tolerances to an excess, or perhaps a shortage, of one or more ingredients. In production if one of the phenomena starts to occur, the chemist may save valuable time by analyzing for the ingredient most likely to be causing the problem.

The etch rate is directly related to the temperature of etching, although the relationship is modified by other considerations, such as tolerances, surface finish, etc. Temperature adjustments can be made to gain the optimum benefits. Since experience has shown that the actual etching step is not the most time consuming part of the chemical milling operation, it makes little difference to the over-all production time if the etch rate is 0.5 mils per minute, 1.0 mil per minute, or 1.5 mil per minute. Other factors, particularly tolerances required, usually determine the most desirable etch rate.

The positioning of the parts in the etchant also plays an important role in the success obtained during chemical milling. Wherever possible, it is desirable to avoid gas streaming over the part where such gas evolution can cause erosion of the metal being milled. Rotation of the parts during

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milling often is necessary, but results in increased costs.

From these comments it is evident that there are many factors affecting the success of chemically milling a particular part made from a given alloy. The data and information that have been accumulated for one alloy are not necessarily wholly valid for another alloy, although the general concepts will afford a background of knowledge which will expedite the work. When a project to develop an etchant system is complete (from the standpoint of production practicality), it is to be expected that the main factors influencing the chemical milling operation will be identified and controlled. Experience has shown that the greatest success can be achieved when this information is available. The research and development project which WCCM has suggested is directed to gathering that information.

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FIGURE 1

Typical Etchant Control Graph

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