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FINAL REPORT, RESEARCH PROJECT, CONTRACT
CA-264, CHEMICAL MILLING OF COUNTERBORE
RECESSES IN THE URANIUM WALL OF ZIRCALOY-2
CLAD URANIUM TUBES.

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FINAL REPORT

RESEARCH PROJECT

CONTRACT CA-66

CHEMICAL MILLING OF COMBUSTIBLE MATERIALS

IN THE

WASTING MILL OF KINCALOT-2 CLASS WASTING TUBES

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By Authority of DS Lewis
PR-24, 4-19-94

By DK Hansen, 5-10-94

Verified By J E Savvy 5-16-94

FOR

THE CHEMICAL MILLING COMPANY

UNITED STATES CHEMICAL MILLING CORPORATION

1700 RIVERSIDE AVENUE

MANHATTAN BEACH, CALIFORNIA

[REDACTED]

[REDACTED]

Don C. Atkins, Jr.
Technical Director

March 28, 1961

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ACKNOWLEDGMENT

The participation of Mr. W. L. Hanson and Mr. Wayne Hendrichsen, General Electric Engineers, in monitoring and conducting General Electric's part of this research program is most gratefully appreciated.

The actual laboratory work, data collected, and tests performed at United States Chemical Milling Corporation were under the direct supervision of Mr. Charles J. Vinton, Research Supervisor. In this work, he was assisted by Mr. John Fourness, Laboratory Technician.

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TABLE OF CONTENTS

	<u>Page</u>
Scope of Work	1
Results and Conclusions	2
Discussion	3
Experimental Procedures	11
Recommended Production Procedure	13
Corrective Measures	16
Operational Control Graph - Figure 3	19
Analytical Procedures	20
Adjustment of Constituents	25
Effect on Ion Concentration by Addition of Various Ingredients - Figure 4	27
Equipment	28
Through-put	30
Table I - Recipe for Milling Uranium Zirconium-2 Tubs	32
Table II - Data on Effects of Chemical Variations in Recipe 77-2	33
Table III - Summarized Data	40

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REFERENCE

Document EN-4540 of August 16, 1958. Consultant Agreement CA-254.
Prime Contract AT(49-1)1350.

SCOPE OF WORK

The scope of the work as set forth in the referenced documents was essentially predicated on the basic formulas developed under a previous contract (Document EN-4465; Purchase Order No. ENH-98). This work called for, on a best effort basis, the definition of the chemical operating parameters of an etchant system and the establishment of a practical production process to permit operation by General Electric production personnel. This investigation was to include a comprehensive study of as many of the chemical factors which could be isolated and their effect on the operating system. Control of these factors also was to involve the development of suitable analytical procedures to permit adequate chemical control of the process. Furthermore, the substantiation of the work was to be demonstrated by the production of at least ten (10) large OD and ten (10) small OD tubes with tube end recesses of $0.250" \pm 0.005"$. The original scope of this work was changed, through verbal instructions, by Walt Ruppen and Wayne Rudrickson of General Electric during their several visits to United States Chemical Milling Corporation and during a visit by ENH personnel to General Electric's facilities. The nature of these changes was to increase the depth of cut to $0.300"$ and to demonstrate the effectivity of the process by producing several tubes of each diameter having recesses to this depth of cut. Several other minor changes were made which did not materially affect the project but which are discussed in this report.

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RESULTS AND CONCLUSIONS

This project demonstrated the feasibility to chemically mill counterbore recesses in the ends of uranium walls of Kirealay-2 clad uranium tubes on a production basis. It was proved that under controlled conditions recess depths of $0.300" \pm 0.015"$ can be obtained although it is necessary to physically move the parts during the milling action to achieve optimum results. With lesser depths of milling, closer tolerances can be achieved. The tests conducted showed that the part movement most conducive to good tolerances is a rotation of the parts around a central axis at 4 rpm. The etchant that proved most satisfactory was a modification of the etchant developed under P.O. DED-98. This is an aqueous mixture of sulfuric and nitric acids whose action is enhanced through the presence of copper ions. It is identified as Etchant J7-2. The operating temperature of this system is $165^{\circ} F.$ under which condition the average etch rate will be $0.0013"$ per minute. The capacity of this etchant to chemically mill uranium and provide satisfactory surfaces is 600 lbs. per thousand gallons.

Although the funding for this contract had been completely utilized by January 31, 1961, by mutual agreement with General Electric engineers, USCM alerted to complete, at its own expense, the studies presented in this report. There is no obligation to General Electric for this additional work.

Attention is invited to the fact that chemical milling of a relatively deep recess confined by vertical walls represents a significant advancement in this method of manufacture of parts. Prior to this project, it generally had been considered impractical to undertake the production of such recesses in even the more common metals. The results of the research accomplished through General Electric's sponsorship of this project undoubtedly represents a forward step in chemical milling. United States Chemical Milling Corporation is most pleased to have participated with The General Electric Company in this work.

DISCUSSION

The production of relatively deep recesses in the uranium wall of a Circaloy-2 clad uranium tube implies more than the simple removal of metal. Certain requirements with respect to surface finish, tolerances, etch rate, chemical balance of etchants, and practicality of operation had to be considered. The formula previously developed under P.O. DMR-98 did not, in itself, permit chemical milling to this depth of cut. Accordingly, experiments were undertaken to improve this formula to the extent that the proper depth of cut could be obtained while at the same time achieving the desired tolerances, surface finish, and etch rate. This revised formula is presented in Table I along with general comments regarding its properties.

Previous experience at ORNL has shown that production of parts can be facilitated if the behavior characteristics of the etchant are known when the etchant deviates from the desired operating range. Such characteristics are reflected, in the case of milling uranium, in inferior tolerances, pitting, and slower etch rate. In recognition of these possible effects, research was undertaken to establish the type of phenomena that would occur should the etchant system be allowed to stray from the prescribed compositions.

In these experiments, the composition of the etchant was varied by changing one component at a time while holding all others constant. If this resulted in a tolerance or smoothness improvement it was further explored. If one of these characteristics was improved while the changes caused a detrimental effect on the other, a compromise was made. Of course, the continuous objective was to establish the optimum control range in which good parts could be produced. In this work considerable pertinent and valuable data were obtained as shown in Table II, and summarized in Table III. For example, it is necessary to keep the

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sulfate ion between 4000 and 5100 lbs. per thousand gallons while, at the same time, maintaining a nitrate to sulfate (NO_3/SO_4) ratio of 0.170 to 0.185. An increase in the ratio within the prescribed sulfate limits results in a deterioration of tolerances and a probability of pitting of the milled surface. If this nitrate to sulfate ratio is reduced, there will be further tolerance improvements but at the expense of a decrease in etch rate. The sulfate ion concentration range of 4000 to 5100 lbs. per thousand gallons represents a region where freedom from pitting may be obtained. Should the sulfate ion concentration be allowed to increase beyond this range while maintaining the proper nitrate to sulfate ratio (0.170 to 0.185), the etch rate will be somewhat retarded and the tolerances will become inferior. If the sulfate ion concentration is decreased below this range while maintaining the proper nitrate to sulfate ratio, pitting of the milled surface will occur. An additional effect of increasing the sulfate ion concentration, while maintaining the fixed range for the nitrate to sulfate ratio, is to overcome pitting problems. This may be desirable, or even necessary, should there be any porosity of the uranium beyond that which was evident in the uranium used in these experiments. Also, since the presence of iron contamination in the uranium is a likely cause of pitting, varying the sulfate concentration affords an additional means of converting other production problems.

The nitrate to sulfate ratio is the mechanism of maintaining tolerances through chemical control. The ratio obviously specifies the desired nitrate ion concentration for a given sulfate ion concentration. If the sulfate ion is increased while maintaining the fixed ranges for the nitrate to sulfate ratio there will be a deeper penetration toward the center of the tube. This deeper penetration toward the center of the tube has arbitrarily been defined as a positive slope. When the milled surface shows a deeper penetration toward the

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The effect of dissolved uranium metal on the physical appearance of the electrolyte surface was examined over a range of 0 to 100 lbs. of uranium per thousand gallons of electrolyte. It was found that the uranium metal (probably as a catalyst) serves to improve surface appearance and tolerance. When a concentration of approximately 100 lbs. of uranium per thousand gallons of electrolyte was reached, further dissolution of uranium results in a gradual reduction of each peak, such as to be exposed through some action of the dissolved ions. When the uranium concentration is 100, the surface is entirely flat and may be described as having a smooth appearance. When the dissolved uranium concentration is 100 lbs. per thousand gallons there may be a precipitation of uranium salts, especially if the electrolyte is allowed to cool below the operating temperature. The range of dissolved uranium is 100 to 100 lbs. per thousand gallons for optimum plating results. The copper ion concentration may be adjusted to vary between 175 to 200 lbs. per thousand gallons. There is a slight increase of each peak when the

FIGURE 1

results of the test, the slope is defined as being negative. This becomes more readily apparent by referring to Figure 1.

concentration exceeds 175 lbs. per thousand gallons, probably because of the action of the copper as a depolarizer. There are no other apparent benefits. If the copper ion concentration is permitted to go below 175 lbs. per thousand gallons there is a significant reduction in etch rate and a possibility of non-uniform etching. Precipitation of copper sulfide is not a problem when the copper ion is within this prescribed range, even when the bath is allowed to cool to ambient temperature.

Reagent No. 57-2 was found to perform best when operating at a temperature of approximately 167° F. A temperature range from 140° F. to 180° F. was examined. As would be expected, lower temperatures result in decreased etch rates. Also, the slope of the milled surface varies unsatisfactorily. Increasing the temperature, of course, increases the etch rate but definitely complicates the normal firing problem characteristic of volatile acid etchants. There is a direct relationship between etch rate and tolerances of the chemically milled surface. It was found that when the etch rate exceeds 0.0013" per minute the tolerances were unacceptable. As would be expected when the uranium ion concentration increases, its mass action retards the etch rate. By increasing the temperature slightly, compensation can be made for this mass action effect. Since, apparently, uranium forms a complex with nitrate ions in this etchant system, the increase of dissolved uranium will reduce the loss of this volatile acid even with an increase in temperature. By adjustment of the temperatures it is possible to maintain essentially a uniform etch rate over a dissolved uranium content range varying from 100 to 700 lbs. of uranium per thousand gallons of etchant.

It is evident, of course, that in the examination of the various chemical factors affecting the chemical milling reactions it was necessary to establish suitable analytical chemical procedures which permit rapid and accurate examination

to be made of the chemical etchant's balance. In deriving these procedures attempts were made to reach a satisfactory compromise between the most accurate and precise analytical procedure available and the important necessity of performing rapid analyses so that data would be available on which to base changes in the etchant composition. Production experience over many years of successful operation has proved that no matter how good the analyses may be, if they are not available in time to take corrective action on an etchant system, the results are of little value. In WEC's research work it was decided not to devote any appreciable time to development of analytical procedures for determining the uranium content of the etchant since General Electric already had adequate techniques for determining the concentration of this material. In the experiments conducted at WEC the uranium content was determined by close and accurate records of the amount of metal removed from the test pieces as indicated by their weight and the depth of cut. Analyses were developed for the other ingredients. The details of the analytical procedures which were derived are presented in the section entitled Analytical Procedures.

Since the physical handling of parts during the milling action often will be as important as the chemical balance of the system, considerable attention was given to positioning of the parts during chemical milling. Experience at WEC has shown that gas entrapment may play an important role in tolerance problems. For that reason, initial work on this project and on the previous research project considered milling of the tubes in an upright position such that the milling surface was essentially parallel to the surface of the etchant. However, when the depth of etch approaches 0.200" some difficulties with tolerances are experienced, and, furthermore, there is a restriction to milling one end of the tube at a time. The parallel testing at General Electric's facilities resulted in the development of a

rotating device whereby the parts were rotated around the center axis at speeds which could be varied through changes in the rotating equipment. The rotating device at General Electric was constructed of teflon plastic; however, it was decided that IGCN's equipment would be made from stainless steel because of the more immediate availability of this material. Using this equipment, the rate of rotation was studied from 4 rpm to 24 rpm. During these studies the position of the tube from the axis was maintained constant at 3-1/2" as measured radially from the axis of the equipment. A fast rate of rotation gave rise to a positive slope effect and produced severe tolerance limits. Thus, it is apparent that slope problems can be remedied by adjustments of the rate of rotation as well as changes in the chemical balance of the etchant. The minimum rate of rotation that could be achieved because of mechanical restrictions of the equipment was 4 rpm. However, a projection of the information gained suggests that a more ideal rotation rate would be about 3 rpm with the tube placed 3-1/2", radially, from the axis of rotation. Obviously, a longer radius would require a slower rate of rotation to maintain the same relative movement of part with the etchant.

As one would anticipate, the reaction of uranium with the acidic etchant is exothermic with the result that the temperature at the surface of the etching uranium may be far above the actual etchant temperature as measured by a thermometer in the etchant solution. It is possible that the effect of rotation on the slope of the milled surface may be due to the cooling effect of the etchant media on the Zircaloy-2 cladding holding the uranium. If etching is permitted to occur such that the uranium dissolves faster than 0.0013" per minute, the rate at which the heat is generated in the milling reaction apparently is faster than the heat transfer through the uranium to the Zircaloy-2 and then to the surrounding etchant

media. The result is that the actual temperature of the medium rises locally at the center of the cut due to heat concentration effects caused by the geometry of the tube. This, in turn, causes an accelerated etching at the center of the milling medium or a tolerance fault known as dishing. Similarly, an increase in the rate of rotation removes heat more rapidly from the Siverley-2 causing a slope variation and increased dishing. Thus, it is apparent that the contour of the milled surface not only is affected by the chemical constituents in the etchant but, also, by physical considerations during the milling of the part.

Consideration was given to varying the actual milling time since previous experience at UGM has proved that characteristics of the milled surface may be changed by the duration of actual etching. The time cycle test indicated that the tolerance was not noticeably dependent upon the length of time of the etch period. This substantiated the concept that this is indeed a balanced etchant and is amenable to mass production of parts. There was a slight etch rate acceleration as the etchant penetrated deeper into the part. This, most probably, is a heat effect similar to that described above. This is interpreted to mean that the total cut could be made when the part is in the Ferris wheel type of fixture, in one step, without any tolerance disadvantages. However, it is possible that mass production of parts could be facilitated by a two stage process. In the first stage, parts would be etched for a fixed period of approximately two hours on the Ferris wheel in order to obtain an indentation of somewhat greater than 0.100". The parts then could be removed and completed in another etchant tank by being placed vertically on a suitable plastic surface in a basket. The etching then would occur from the top surface only, as was demonstrated at General Electric's pilot facilities and later substantiated at UGM. By maintaining the basket near the surface of the etchant a ready inspection of individual parts would be possible as

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they near completion. The time that the parts are in the Ferris wheel could be increased to the extent where the finishing process would amount to approximately thirty minutes. As has been previously noted, the rotation of the parts in the Ferris wheel fixture contributes to the final smoothness of the milled surface, whereas the parts etched in a vertical position in a placement tank tend to have a somewhat rougher surface. This problem could be overcome by increasing the sulfate ion concentration in the second, or finishing, tank to about 5500 lbs. per thousand gallons. Of course, the advantages of close control in the last step of the milling operation must be weighed against the inconvenience of removing the tubes from the rotating equipment. It is probable that the answer to this question can best be determined through actual production practice.

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EXPERIMENTAL PROCEDURE

The research work at ORNL was conducted in a fiberglass-polyester tank approximately 18" in diameter and 24" deep¹. The etchant solutions were heated with a Raymont Type, Kartate Heat Exchanger², utilizing steam at approximately 20 to 100 psi. Other equipment directly pertinent to the tests conducted consisted of a stainless steel device developed for rotating the parts and for and such that the milling on any given end would occur primarily while the axis of the part is in the vertical position. The other fixture was a rotating device patterned after that developed at General Electric, as previously described. Each of these pieces of equipment would permit rotating the parts either in the solution or withdrawing them and rotating them out of the solution. Other standard laboratory equipment, such as beakers, brushes, ventilation hoods, were utilized in this work.

In conducting these tests, the part was affixed in its proper position in the device and placed in the etchant solution which previously had been brought to the desired operating temperature. Depending upon the particular experiment, the part was withdrawn at intervals and examined for the properties of the milled surface. Such observations include measurement of depth of cut, examination for variations in slope, determination of etch rate, and presence or absence of pitting. It is to be noted that in all of the tests the weight of each individual part was recorded before and after milling so as to provide a clear record of uranium content and accountability of this material.

As is evident from Table II, the actual etchant composition with respect to nitrate, copper, and sulfate ion, as well as dissolved uranium, was purposely varied in order to characterize the system. However, considerable care was given

1 - Supplied by Riverfield Plastics, Inc., 2129 East Florence Avenue, Huntington Park, California.

2 - National Carter Company, 30 East 42nd Street, New York 17, New York.

to vary only one factor at a time and assess its influence on the end results. When this procedure was applied a sufficient number of times to present a picture of the effect on the properties of the chemically milled surface caused by variations in one ingredient, then other ingredients would be altered to establish their effects. Thus, when the effects of the ingredients became more evident and the trend of changes in the concentrations was apparent, the concentration of the entire group of ingredients would be adjusted to obtain more optimum results.

After conducting several different types of tests, it became apparent that the physical positioning of the parts during the milling action often could overshadow some of the chemical effects that were obtained. Thus, it was decided to standardize on using General Electric's rotating device as the technique for final evaluation of the etchant system and it is upon the experiments using this equipment that the conclusions and recommendations are based. More detailed information concerning the individual tests is presented in Table II. A diagram of the General Electric rotating device is shown in Figure 2.

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RECOMMENDED PRODUCTION PROCEDURE

It is obvious, of course, that the ultimate production procedure which will prove to be most amenable to General Electric's conditions of operation will be derived subsequent to the actual manufacture of the recessed tubes. However, as a basis for developing final manufacturing techniques, the following production procedure is offered:

- Step 1. Incoming parts to be chemically milled should be cleaned and free of surface contaminants, particularly on the ends to be milled. Oleophilic ingredients may be removed by alkaline cleaning or by vapor degreasing³.
- Step 2. Parts should be positioned in the rotating device and secured.
- Step 3. Inquiries should be made of Chemical Process Control to ascertain that the etchant bath is within the recommended concentration ranges and at the proper operating temperature.
- Step 4. Prior to making a production run, it may be desirable to check the etch rate to substantiate that the bath is operating correctly. As more experience is gained, it may be possible to omit this step.
- Step 5. The rotating device should be immersed in the hot etchant and continuously rotated at the prescribed rpm until the parts are within the last 0.005" of the desired depth of cut. As experience is gained, this can be

3 - Teco Products, Inc., Los Angeles, California.
(Vitonium)

measured by a final depth gauge, checking on the part as the rotating device is momentarily brought out of the etching solution. The device should be re-immersed into the etchant and allowed to continue to mill to the final depth of cut. Although the etch rate for the final 0.000", or less, of cut may not vary too significantly, it is important that several additional checks be made prior to reaching the final depth in order that the desired depth will not be exceeded. It also is very important that the etching parts not be left unattended.

Step 6. When the final depth of cut has been achieved, the rotating device should be removed from the etchant and thoroughly rinsed with water. Water rinsing may be accomplished by immersion of the entire device in a suitable tank containing water.

It is fully expected that this production procedure will permit General Electric to manufacture their parts satisfactorily. However, the success of this operation will depend to a great extent upon accurate chemical analyses of all ingredients in the etchant tank, and adjustment of the etchant to the optimum area of the prescribed control chart within a minimum amount of time. The frequency to which these analyses should be made will depend upon the number of parts being processed. It is suggested that analyses and adjustment of the tank be made at least once in every eight hour shift, preferably at the beginning of the shift. If these adjustments are not made and if the tank is allowed to vary from the recommended range, it is to be expected that inferior tolerances and, possibly, pitting will

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result. To facilitate performing these analyses and making the adjustments, it is highly recommended that suitable laboratory facilities and apparatus be located in the immediate area of the production tanks.

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CORRECTIVE MEASURES

As a characteristic of every production operation involving chemicals, there will be occasions when apparent anomalies may occur, as indicated by the characteristics of the chemically milled surfaces. In recognition of this fact, attention is invited to the following suggested corrective measures as related to observed characteristics of the etchant and milled surfaces. It is imperative that only one corrective measure be taken at a time, otherwise it may be difficult to bring the operation back into balance.

Observation

1. Slow etch rate.

Corrective Measure

- (a) Increase the temperature.
- (b) Bring the nitrate to sulfate ratio to its maximum of 0.185.
- (c) Check for a possible overconcentration of uranium ions (above 700 lbs. per thousand gallons).
- (d) Reduce the sulfate ion to the bottom of its range of 4800-5100 lbs. per thousand gallons, while maintaining the nitrate to sulfate ratio at the required 0.170 to 0.185 range.
- (e) Check for a possible loss of copper ions and adjust the dissolved copper content to 175 lbs. per thousand gallons.

2. Fitting.

- (a) Reduce the nitrate to sulfate ratio.
- (b) Increase the sulfate ion concentration.
- (c) Ascertain that the uranium ion concentration is between 100 to 700 lbs. per thousand gallons.
- (d) Verify that the copper ion concentration is not less than 175 lbs. per thousand gallons in order that copper ion concentration may be reduced through drag-out losses.

3. Four tolerances.

- (a) Reduce the nitrate to sulfate ratio to the bottom of its 0.170 to 0.185 range.
- (b) If the etch rate is above 0.0013" per minute, reduce the temperature.
- (c) Check for a possible deficiency of copper ions.

4. Negative slope effect.
(Depression toward the outer edge of the part.)

- (a) Increase the sulfate ion to the maximum of 5100 lbs. per thousand gallons while maintaining the nitrate to sulfate range of 0.170 to 0.185.

5. Positive slope effect.

(Depression toward the center of the part.)

6. Fast etch rate. (Above 0.003" per minute.)

- (b) Increase the rate of rotation of the Ferris wheel rotating device.
- (c) Reduce the etchant temperature.
- (a) Decrease the sulfate ion concentration to the minimum value of 1000 lbs. per thousand gallons while maintaining the nitrate to sulfate ratio range of 0.170 to 0.185.
- (b) Decrease the rate of rotation of the Ferris wheel rotating device.
- (c) Increase the etchant temperature.
- (a) Reduce the nitrate to sulfate ratio to its minimum of 0.170. In extreme cases of fast etch rate, this ratio may be reduced to 0.160.
- (b) Increasing the sulfate ion concentration to the maximum of 5100 lbs. per thousand gallons.
- (c) If operation has been above 165° F. reduce the etchant temperature to 165° F.

The information discussed above also is presented in an Operational Control Graph shown in Figure 3.

OPERATIONAL CONTROL GRAPH

FIGURE 3

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ANALYTICAL PROCEDURES

The analytical procedures which have been found satisfactory for maintaining proper control of Reagent 77-1 are presented below.

REAGENT 77-2

(A-1) PREPARATION OF SAMPLE

Object: To prepare a diluted sample of uranium reagent for control analysis.

Apparatus: 1 - 25 ml pipet equipped with cushion bulb
1 - 250 ml volumetric flask

Procedure: 1. Pipet exactly 25 ml of uranium reagent into the 250 ml volumetric flask and dilute to volume with distilled water.

2. Use samples of this diluted solution as required in the following procedures.

(A-2) NITRATE TEST (Method for standardization)

Object: To determine the amount of nitrate ion in Reagent 77-2.

Apparatus: 2 - 250 ml Erlenmeyer flasks
1 - 50 ml graduated cylinder
1 - 5 ml pipet
1 - 25 ml pipet
1 - 50 ml burette

Reagents: Ferrous indicator, 0.005 N.

Ferrous ammonium sulfate solution:

Prepare a stock solution by dissolving 393 grams of ferrous ammonium sulfate, $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$, in 1300 ml of distilled water plus 500 ml of concentrated sulfuric acid, H_2SO_4 . Cool and dilute to two liters.

Dilute one liter of stock solution to 2-1/2 liters with distilled water for standard solution.

0.0005 N potassium dichromate solution:

Dissolve exactly 19.615 grams of primary standard grade $\text{K}_2\text{Cr}_2\text{O}_7$ in sufficient distilled water to make two liters in a volumetric flask at 25° C.

Sulfuric acid, concentrated.

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- Procedure:**
1. Pipet exactly 5 ml of the diluted sample (A-1) into a 250 ml Erlenmeyer flask containing a number of glass beads.
 2. Add, via a pipet, exactly 25 ml of ferrous ammonium sulfate solution.
 3. Slowly add, while swirling, 25 ml of concentrated sulfuric acid.
 4. Add 30 ml of distilled water.
 5. Boil the solution at least ten minutes.
 6. Cool, add 30 ml of H_2O , 3 drops of furcain indicator and titrate with standard potassium dichromate solution.
 7. At the same time a sample is run, run a blank in the same manner as above except substitute 5 ml of distilled water for the sample.
 8. Record the burette readings for both blank and sample.

Calculations: (blank titration - sample titration) $\times$ 69.0 = lbs. NO_3^-/M gallons

(A-3) RAPID VOLHARDT'S NITRATE TEST

Object: To determine the amount of nitrate ion in the extract as lbs./M gallons NO_3^- .

Apparatus:

- 1 - 2 ml pipet
- 2 - 250 ml Erlenmeyer flasks
- 1 - 25 ml automatic burette
- 1 - 4 liter Griffin beaker

Reagents: Ferrous sulfate solution:
Add 250.7 g $FeSO_4 \cdot 7H_2O$ to 500 ml H_2O containing 50 ml H_2SO_4 . Dilute to 1000 ml.

Standardize against a mixture of 100 ml HNO_3 diluted to one liter. The exact value is determined by titration of the acid against caustic. Handbooks indicate the lbs./gal. for any normality of HNO_3 . Multiply this by the factor $\frac{NO_3 \times 1000}{HNO_3}$ to get lbs./M gal. of NO_3^- . Divide by cc of titer

to get lbs./M gal. per cc of titer.

Sulfuric acid, concentrated.

- Procedure: 1. Add 2 ml of etchant to a 250 ml Erlenmeyer flask, and then add 25 ml reagent grade concentrated H_2SO_4 .
2. Titrate with the standard $Fe SO_4$ to a brown color while keeping the flask partially immersed in water in the 4 liter beaker during the titrating and swirling process.

Calculations: ml titer $\times$ lbs./M gal. SO_4^{2-} per cc of titer = lbs./M gal. SO_4^{2-}

Reactions: $4FeSO_4 + 2HNO_3 + 2H_2SO_4 = 2Fe_2(SO_4)_3 + H_2O_2 + 2H_2O$

(A-4) SULFATE TEST

Object: To determine the amount of sulfate ion in Etchant 37-2.

Apparatus: 1 - 2 ml pipet
1 - 75 ml graduated cylinder
1 - 10 ml graduated cylinder
1 - 250 ml beaker
1 - 250 ml Erlenmeyer flask
1 - 3 inch 60° funnel

Reagents: Barium chromate:

Add 107 ml concentrated hydrochloric acid, HCl, to approximately one liter of distilled water. Add 20 grams of barium chromate $BaCrO_4$, and mix to dissolve. Dilute to 2000 ml with distilled water.

Sodium thiosulfate - 0.2N, standardized.

Sodium hydroxide - 20% solution.

Ammonium hydroxide - reagent grade.

Hydrochloric acid - reagent grade.

Potassium iodide - reagent grade.

Starch indicator:

Dissolve 150 grams of sodium chloride in approximately 800 ml of water. Add 100 ml of acetic acid and dilute to one liter. Bring solution to a boil. Make a paste of 5 grams of starch in 25-30 ml of water and pour into boiling solution. Allow to boil 2-3 minutes before cooking.

Whatman filtering paper #30.

Filtering tablets.

- Procedure: 1. Pipet exactly 2 ml of the diluted etchant (A-1) into a 250 ml beaker.

2. Dissolve all copper from the cathode in 20% HNO₃ under fume, and dry, preferably under a heat lamp.
1. Place 25 ml cathode into a 250 ml beaker and dilute with water to approximately 1/2 of volume.

Reagents: 20/20 HNO₃

- 1 - Analytical balance
- 2 - 250 ml distilled beakers
- 1 - 25 ml pipette

Object: To determine the concentration of copper ion in solution 77-2.

(4-2) Copper Ion

Calculations: (Molarity of Fe^{2+}) $\times$ (ml used) $\times$ 1336.1 = 100. 20% / ml.

2. Add 5 ml of 0.1% KI solution and dilute the solution. The end point is a sudden change from purple to light green.
1. Add the titrant to form a precipitate and then add about 2 g of KI. The solution becomes, deepened and dilute with water (dilution to a clear color).
6. Add 10 ml of concentrated HCl to the solution. Dilute the precipitate.
5. Wash and the beaker with small portions of boiling distilled water onto the precipitate until the solution coming from the funnel is clear.
4. Add a filtering paper, pour to boiling and filter through Whatman #50 paper into a 250 ml volumetric flask.
3. Remove the flask and then with distilled water with HNO₃ (a small portion precipitate will form and not so much later solution when boiled. Then add 4 ml of HNO₃ in excess).
2. Add 20 ml of HNO₃ and heat to boiling.

ADJUSTMENT OF CONCENTRATIONS IN ETCHANT JT-2

As production proceeds there will be a necessity of adjusting the etchant to the optimum operating ranges. The following equations present a means whereby computations may be readily made.

I. When concentrations are too low:

Nitrate Ion

$$\left(\begin{array}{l} \text{Gallons of } 42^{\circ} \text{ Be'} \text{ HNO}_3 \\ \text{to be added} \end{array} \right) = \frac{\left[900 - \left(\begin{array}{l} \text{Lbs. NO}_3^-/\text{M gal.} \\ \text{determined by} \\ \text{analysis} \end{array} \right) \right] \times \left(\begin{array}{l} \text{Gals. etchant in tank} \\ 1000 \end{array} \right)}{8}$$

Where: 900 = Lbs. NO₃⁻/M gal. selected as an optimum value
8 = Lbs. NO₃⁻/gal. of 42° Be' HNO₃

Sulfate Ion

$$\left(\begin{array}{l} \text{Gallons of } 66^{\circ} \text{ Be'} \text{ H}_2\text{SO}_4 \\ \text{to be added} \end{array} \right) = \frac{\left[9000 - \left(\begin{array}{l} \text{Lbs. SO}_4^{--}/\text{M gal.} \\ \text{determined by} \\ \text{analysis} \end{array} \right) \right] \times \left(\begin{array}{l} \text{Gals. etchant in tank} \\ 1000 \end{array} \right)}{10.5}$$

Where: 9000 = Lbs. SO₄⁻⁻/M gal. selected as an optimum value
10.5 = Lbs. SO₄⁻⁻/gal. of 66° Be' H₂SO₄

Oxide Ion

$$\left(\begin{array}{l} \text{Lbs. CuSO}_4 \cdot 5\text{H}_2\text{O} \\ \text{to be added} \end{array} \right) = \left[175 - \left(\begin{array}{l} \text{Lbs. Cu}^{++}/\text{M gal.} \\ \text{determined by} \\ \text{analysis} \end{array} \right) \right] 3.92 \times 10^{-3} \times \left(\begin{array}{l} \text{Gals. etchant} \\ \text{in tank} \end{array} \right)$$

$$\text{Lbs. CuSO}_4 \cdot 5\text{H}_2\text{O} = \frac{66}{249.5} \times \frac{2.895}{\text{Gals. etchant}} = \text{Lbs./M gal. SO}_4^{--} \text{ contributed}$$

$$\text{Where: } 3.92 \times 10^{-3} = \frac{249.6 (\text{A.W. CuSO}_4 \cdot 5\text{H}_2\text{O})}{1000 \times 63.5 (\text{A.W. Cu})}$$

II. When concentrations are too high:

Nitrate Ion

$$\left(\text{Adjusted volume of} \right. \\ \left. \text{standard in gals.} \right) = \frac{\left(\text{lbs. NO}_3^-/\text{M gals.} \right. \\ \left. \text{determined by} \right. \\ \left. \text{analysis} \right)}{200} \times \left(\text{Present volume} \right. \\ \left. \text{standard in gals.} \right)$$

Sulfate Ion

$$\left(\text{Adjusted volume of} \right. \\ \left. \text{standard in gals.} \right) = \frac{\left(\text{lbs. SO}_4^{2-}/\text{M gals.} \right. \\ \left. \text{determined by} \right. \\ \left. \text{analysis} \right)}{200} \times \left(\text{Present volume} \right. \\ \left. \text{standard in gals.} \right)$$

Note: (1) Water may be used to dilute standard to adjusted volume.

(2) If standard tank is reasonably symmetrical, the depth of solution may be substituted for "present volume" and "adjusted volume".

(3) It must be recognized that unless a correction is made for the effect of one of the above equations on the other, a slight error will result.

The effect on ion concentration of the addition of various ingredients to the standard is shown on Figure 4.

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EFFECT OF ADDITIONS TO 1000 GALLONS OF URANIUM ETCHANT

FIGURE 4

REQUIREMENT

The equipment recommended for protection chemical milling of the uranium metal, of necessity, be resistant to the acid environment comprising the etchant and its fumes. Materials of construction which could suffice for these purposes are well known in the field of chemical engineering. However, experience at UCCN suggests that a fiberglass-polyester tank offers considerable advantages for holding the acid etchant. Depending upon the space available and the equipment to be used in the tank, either a self-supporting fiberglass-polyester tank or a fiberglass lining in a mild steel tank can be used. If, as has been indicated in discussions with General Electric personnel, the equipment for rotating the uranium tubes is to rest on the bottom of the tank, most certainly the bottom should be reinforced with steel. In such case, the lined tank would be more advantageous. In addition to the lining itself, it is recommended that suitable ducting be provided for removal of the acidic vapors emanating from the etchant bath. Here again, fiberglass and polyester can be used to advantage, although duct systems manufactured from rigid PVC are equally as good.

Heating of the etchant can be accomplished through Kirtate Heat Exchangers. If this type of heat exchanger is selected, it is recommended that the circulating acid metal be selected. Kirtate Heat Exchangers, Type VVE-1X, have been found most satisfactory for this purpose. Steam for heating will prove most efficient, although hot water could be used.

In chemical processing systems, especially one involving such a rigorous environment as a chemical milling etchant, it is to be expected that the materials of construction will have a finite operating life. The system just recommended may be anticipated to have an operating life of at least one year and probably two to

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three years with proper maintenance. To achieve this objective it will be necessary, of course, not only to have selected high quality materials for the tank construction but, also, to insure that fabrication is of high quality. Of course, an all stainless steel tank would contain the etchant currently recommended for this chemical milling operation. However, should other etchants be developed which attack stainless steel, or should it later be desired to use this equipment for other purposes requiring an acid resistant lining in a tank, a stainless steel tank may not offer the degree of flexibility as will one constructed of fiberglass and polyester. Obviously, if someone drops a basket or a fixture full of parts into a fiberglass tank there is the possibility of fracture of the material should the impact be sufficiently great. However, with even reasonable care and safety devices, this possibility can be kept at a minimum.

It is suggested that consideration be given to a tank that is approximately eleven feet (11') long by three feet (3') wide by five feet (5') deep. Such a tank, when filled to a depth of slightly over four feet will contain approximately one thousand gallons of etchant. A rinse tank also will be needed to flush the etchant free from the milled parts. Depending upon the actual size and configuration of the holding fixture for the tubes, the rinse tank may be either the same size and shape or, perhaps, it may be scaled down somewhat should space be a problem. The rinse tank may be of mild steel coated with a corrosion resistant paint or, perhaps, a thin layer of fiberglass-polyester.

THROUGH-PUT

Although actual computations for determining the through-put of this proposed production line will depend upon factors which may not be fully recognized at this time, it is possible to offer some information concerning the anticipated through-put. Accordingly, attention is invited to the following calculations.

If the following assumptions are made:

- (1) The average etch rate is 0.0013" per minute;
- (2) The depth of cut to be 0.388";
- (3) The capacity of the etchant to dissolve uranium is 600 lbs. per thousand gallons;
- (4) It is possible to devise a rack which will hold one hundred (100) tubes;
- (5) Three racks may be placed in the etchant at a given time, and
- (6) That the tubes being milled are two feet (2') long,

then the following appears logical:

$$\text{Time to remove metal} = \frac{0.388"}{0.0013"/\text{min.}} \times \frac{1 \text{ hr.}}{60 \text{ min.}} = 4 \text{ hours.}$$

This means that three hundred (300) tubes would be processed in four hours. As calculated on a depth of cut of 0.388", the approximate amount of uranium removed per tube (both ends) is:

Inner Tube
0.3779 lbs.

Outer Tube
0.7086 lbs.

Thus, in milling three hundred (300) inner tubes the amount of uranium dissolved will be about 112.6 lbs. This amount of uranium then would be removed every four hours. Since the capacity of the etchant is 600 lbs. of dissolved uranium per one thousand gallons and the tank holds one thousand gallons of etchant, then the number of cycles of four hours which may be experienced before it is necessary to

changed as follows:

$$\frac{\text{---}}{24 \text{ hours}} = 5.3 \text{ cycles}$$

This means that about every twenty hours the board must recharge the batteries two changes a week.

Using the outer tube show that number of hours and, accordingly, that each may be expected to recharge tubes it is probable to be an eight hour shift.

Putting these calculations together with the number of other type of an of

is indicated that it is desired and can in some prescribed steps recharged from the number of power

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[illegible]

 	

TABLE II

Test Number	Part Nos.	Objective of Experiments	Reagent Concentrations lbs./1000 Gallons				OBSERVATIONS	
			Temp. °F.	H ₂ O ₂	Ca	HNO ₃		
1	40 40A	To etch both parts for three 20 minute periods without rotation or agitation.	150	0	3% *	1000	4450	Initial milling of uranium. Etch rate too slow, 0.55 mm. Fitting of milled surface.
2	39 39A	Increase in temperature without altering previous mixture.	160	0	3% *	1000	4450	Etch rate increased to 1.7-1.9 mm. Surface smoothness improved. This experiment demonstrated the unsuitability of etching from the bottom since the bottom milled surfaces were too irregular for evaluation. Etch rates were retarded on bottom milled surfaces by about 50%. Groove problems were evident.
3	38 38A	Use of new holding fixture for further study of milling from top and bottom simultaneously.	160	5% *	3.2% *	972	4512	Surface smoothness was improved by the chemical change due to coagulation and concentration. There was a tendency toward separation of the uranium and Kieselguhr at metal interfaces. Technical difficulties became apparent at the bottom of the tube when a penetration of 0.025" was attained.
4	37 37A	Parts to be etched vertically from both ends and rotated 180° every five minutes.	160	something greater than 5%.	3.5% *	1010	4474	No improvement over Test No. 3. Trenches generated while etching from the bottom were distributed over both surfaces.

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* Experimental analyses used.

TABLE II (Continued)

Test Number	Part Nos.	Objective of Experiments	Ingredient Concentrations lbs./1000 Gallons					OBSERVATIONS			
			Temp. °F.	H ₂ O ₂	Cu	NO ₃	SO ₄				
5	36 36A	Repetition of previous tests at slightly different control ranges.	160	constant	325 *	962	4166	Extreme pitting and slow etch rate occurred. Parts were not completed.			
6	35 35A	Parts etched vertically from both ends and rotated 180° every five minutes. Temperature varied to increase etch rate. Additions of ingredients made to improve surfaces.	160-180	constant	325 *	1007	4483	Slow etch rate despite increase in sulfate concentration.			
					328 *	1116	4576	Persistent slow etch rate even after increase in nitrate concentration. Temperature increased to 180° F. did not produce appreciable etch rate increase.			
7	36 36A	Rotation of Part No. 36 180° under solution while being milled vertically. Rotation made every ten minutes, readings taken every sixty minutes. Later sealed after each ten minute period and then rotated 180°.	160	219.7**	376	1048	4893	Dishing and variation of tolerances were only slightly improved by sealing the part after each etch period. The severe damage due to etching from the bottom makes a direct comparison difficult.			
						Part No. 36 after 239 min. etch			Variation		
						Outer area	263	261	259	260	4
						Center area	260	260	261	260	13
						Inner area	257	258	256	260	10
						Part No. 36A after 200 min. etch					
						Outer area	252	236	247	248	16
						Center area	272	244	265	258	28
						Inner area	269	236	252	251	23

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* Experimental analyses used.

** Electrolytic determination of copper on this and all subsequent tests.

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TABLE II (continued)

Test No.	Objective of Experiment	Temperature, °C	Uranium Concentration, %	Time, hrs.	Observations
8	Reduction of copper concentration as a possible means of etch rate increase. Examination of effect of copper on surface.	165	0	1500 1800 1900	Etch rate increased to over 2 mm.
9	Examination of effect of increasing uranium content on surface conditions.	165	150 160	1800 1900	Surfaces were about the same as in Test No. 8. Pits formed preferentially on outer part. Etch rate 2.0-2.5 mm.
10	Examination of further reduction of copper and reduction of temperature on milled surface.	160	190 144 sec	1870 1900	Reduction of copper concentration and temperature lowered the etch rate to 0.7-0.8 mm. A groove formed at the center of the outer part. This groove believed to be due to too low a copper concentration.
11	Examination of effect of increasing copper, uranium content, and temperature.	165	206 161	1850 1900	Etch rate increased to 1.8-1.9 mm. The inner part continued to be smooth while the outer part pitted. No groove formation observed.
12	To study effect of increasing uranium content in Test No. 11 type system.	150	347 162	1806 1900	An indurated lower temperature reduced etch rate to 1.5-1.7 mm. Surface effects about the same as Test No. 11.

ee Uranium content calculated by weight loss determination on this and all subsequent tests shown.

see Electrolytic determination of copper on this and all subsequent tests.

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TABLE II (Continued)

Test Number	Objective of Experiment	Temperature, °C.	Time, min.	Pressure, mm. Hg.	Volume, ml.	Observations
13	To study effects of increasing amount of washing solvent to 600 lbs. per thousand gallons.	165	600	156	1400	With rate of 1.5 mm on outer tube and 1.7 mm on inner tube. The solvent was placed and surface conditions were similar to those obtained in Test No. 11.
14	To examine surface effects on increasing washing solvent.	165	900	156	1304	There is addition recorded in liquid within three minutes. Large also flow found on outer part. The inner part was not affected.
15	To study the effect of high velocity flow on surface with washing solvent at the top of undisturbed range.	165	940	156	1504	Penetration of liquid 2 observed on outer part. Large jet formed at penetration and there was general jetting of the outer tube. The inner tube was relatively smooth.
16	To locate a minimum acceptable limit of dissolved material.	165	1250	155	1210	Smooth surface obtained on inner and outer tubes. No jets. Washing was apparent. The tube was slightly rounded to 1.5 mm for outer tube and 1.6 mm for inner tube.
17	To increase washing solvent to 1500 lbs. per thousand gallons.	165	not determined	not determined	not determined	Attempting to increase washing to 1500 lbs. per thousand gallons resulted in severe tube wear. Then and great formation on part. Apparent reduction of solution with respect to dissolved material present. At further filling of the vessel. Further tests suggested that on the average in water less would result a washing concentration of 1500 lbs. per 1000 gal. to be reached. However, this was not reached in the first 1000 gal. time tested since the material occurred.

TABLE II (Continued)

Test Number	Part No.	Objective of Experiments	Ingredient Concentrations lbs./1000 Gallons				OBSERVATIONS	
			Temp. °F.	U	Ca	NO ₃		
18	23 23A	Variations of formula modification suggested by G.H.	165	0	202	1058	4379	Pits on outer tube, no pits on inner tube. Slight improvement in tolerances.
19	22 22A	Increase in uranic content in Test No. 18 type formula.	165	997	209	987	4060	Pits on outer tube, no pits on inner tube. Some improvement in tolerances. Etch rate retarded; 0.9 mm.
20	21 21A	Attempt to overcome pitting problems through increase of sulfates.	165	536	187	981	9908	No pits on either tube. Smooth surfaces obtained. Pitting and tolerance problems evident. Etch rate approximately 1 mm.
21	20 20A	Observation of copper ion increase in Test No. 20 type formula.	165	981	254	997	9661	Etch rate increased to 1.45-1.5 mm. Slight pitting on outer tube.
22	19 19A	Reduction of nitrate ion concentration to improve tolerances.	165	907	202	700	4677	Parts not completed because of extremely low etch rate of 0.3-0.4 mm.
23	18 18A	To obtain smoothing effect of high sulfate ion concentration coupled with a practical etch rate through an increase of nitrate ion concentration.	165	454	176	888	9090	Both inner and outer tubes were smooth and free from pits. Etch rates were about 1.4 mm. Tolerances approaching project objective.
24	17 17A	Increase of nitrate to sulfate ratio to study tolerance effects.	165	902	176	1174	5142	Both surfaces smooth although there was some slight pitting on outer part.

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TABLE II (CONTINUED)

Test Number	Part No.	Objective of Experiments	Ingredient Concentrations lbs./1000 Gallons				OBSERVATIONS	
			Temp. °F.	U	Ca	Mg		
25	16 16A	To study tolerance behavior of system by increasing sulfate and maintaining nitrate ion concentration constant.	165	547	175	1114	5099	No significant change in tolerances. Etch rate was very fast, over 1.6 mm. Slight pitting on outer tube.
26	15 15A	Attempt to obtain smoothness and tolerance through increase of sulfate ion and decrease of nitrate ion.	165	594	174	1093	5443	Chemically milled surfaces were very smooth and free from pits. Etch rate was over 1.6 mm. Tolerances became worse.
27	14 14A	To improve tolerance by reduction of sulfate.	165	565	176	999	4803	Inner tube smooth, outer tube pitted. Some improvement in tolerance, probably due to sulfate ion reduction.
28	13 13A	To determine effect on tolerances through reduction of nitrate to sulfate ratio in Test No. 27 type formula.	165	504	177	803	4808	Smooth surfaces on both tubes. Tolerances within requirements. Etch rate 1.2 mm.
29	12 12A	To examine effect of slight increase in sulfate concentration for possible further smoothness benefits.	165	551	176	811	4925	Both parts had unusually smooth surfaces. Tolerance requirements were within limit, but etch rate was somewhat too slow; 1.0 mm.
30	11 11A	To increase etch rate through adjustment of sulfate and nitrate concentrations.	165	598	176	905	4880	A slight pitting tendency. Some orange peel effect. Apparent tendency of uremic Hircaloy-2 to separate at center of outer tube. Etch rate increased to 1.23-1.35 mm.

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TABLE II (CONTINUED)

Test Number	Test No.	Objective of Experiments	Inherent Corrosion Rate (m./1000 cells)					OBSERVATIONS
			Temp. 70°	80°	175°	200°	225°	
31	10 10A	To avoid pitting while maintaining satisfactory tolerance through reduction of nitrate to sulfate ratio.	165	644	175	822	1405	Smooth surface, no pits. Parts within one unit of tolerance.
32	9 9A	Study of effects of rotating parts for a fixed period followed by a vertical etching to complete parts.	165	690	175	922	1405	Good tolerances achieved. Still etching caused a rough surface. Bottom of tube completely protected by outgassed gas.
33	8 8A	To examine effect of short etch periods on tolerance. Etch periods to be of ten minute duration.	165	725	175	900	1370	Smooth surface, no pits. No tolerance benefits observed. Reverse slope effect caused by starting and stopping of etching process.
34	7 7A	To determine effect of rapid rotation.	165	770	174	900	1386	Test discontinued due to extreme surface tolerance difficulties. Rate of rotation was 36 rpm.

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TABLE III

Summarized Data on Effects of Chemical Variations in Stages 27-2

Part Temp. No.	Ingr. No.	Conc. %	Vol. ml.	Wt. gm.	Mol. Weight	Chemical Variations										Surface
						Inner Tube	Outer Tube	Inner Tube	Outer Tube	Inner Tube	Outer Tube	Inner Tube	Outer Tube	Inner Tube	Outer Tube	
23	165	0	200	1000	4579	.23	1.06	1.16	1.5	10	12	10	+1	+4	-5	-6 smooth pitted
10	165	604	175	898	4925	.18	1.23	1.06	1.8	10	10	11	-7	-8	-1	+1 smooth smooth
13	165	304	177	803	4808	.17	1.8	1.2	1.3	16	14	13	-7	-6	-1	-2 smooth smooth
22	165	909	209	909	4060	.23	0.97	0.98	1.6	17	14	14	-8	-9	-8	-7 slight pit pitted
11	165	398	176	905	4080	.19	1.38	1.23	1.8	19	12	12	-6	-7	+1	-1 slight pit smooth
12	165	351	176	811	4925	.16	1.06	1.08	1.5	15	17	18	-5	-4	-7	-8 smooth smooth
18	165	424	176	808	3090	.17	1.40	1.46	1.9	19	18	15	-8	-10	-4	-7 smooth smooth
14	165	365	176	999	4003	.20	1.63	1.51	2.0	18	15	17	-5	-3	-3	-8 smooth pitted
27	165	904	156	1304	5103	.26	1.72	1.53	2.1	23	16	17	-1	-4	0	-3 smooth large pits
21	165	356	187	901	5908	.17	1.10	1.01	2.3	23	18	18	+8	+5	+2	+4 smooth smooth
20	165	901	254	977	5661	.17	1.55	1.46	2.8	21	20	20	-7	-9	-8	-7 smooth slight pit
28	165	600	156	1208	4068	.26	1.76	1.55	2.1	22	13	20	0	0	-2	-6 smooth pitted
25	165	1858	155	1878	5142	.26	1.66	1.51	2.4	25	18	16	-3	-4	-6	-6 smooth smooth
17	165	901	176	1174	5142	.22	1.96	1.86	2.3	22	20	19	-4	-5	+5	+6 smooth slight pit

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TABLE III (Continued)

Summarized Data on Effects of Chemical Variations in Element 27-2

Part No.	Temp. Cr.	Impediment	Conc. g	Conc. mg/100g	Mach Rate mm/rev	Max. Toluenes Deviation				Element				Surface				
						Inner Tube	Outer Tube	Inner Tube	Outer Tube	Inner Tube	Outer Tube	Inner Tube	Outer Tube					
16	165	947	175	1114	5009	.21	1.76	1.68	24	24	19	16	-8	-7	-6	-5	smooth	slight pit
21	140	198	144	1879	4192	.30	0.88	0.78	24	25	18	18	+28	+16	+21	+20	smooth	(slight groove)
15	165	974	174	1803	5443	.20	1.78	1.63	23	23	28	20	-6	-7	-5	-6	smooth	smooth
30	165	206	161	1890	4537	.30	1.96	1.81	26	24	19	15	+1	+1	0	-1	pitted	smooth
33	165	0	152	1884	4609	.20	2.36	2.13	28	25	18	16	+3	+4	-6	-4	smooth	pitted
29	150	947	162	1886	4205	.30	1.50	1.36	25	24	22	18	+20	+8	-8	0	smooth	pitted
32	165	152	160	1884	4284	.28	2.26	2.06	28	29	20	20	+5	+9	-8	-3	smooth	pitted
26	165	948	156	1904	5103	.31	1.88	1.63	29	28	23	22	+7	+5	+1	+1	smooth	pitted

NOTES

1. Parts made by continuous rotation at 4 rpm.

2. Parts listed in order of toluene deviation; best parts at top of list. Deviation listing prepared from the sum of the maximum toluene deviation of the inner and outer tubes.

3. Maximum toluene refers to the number of mills between the deepest chemically milled areas and the least chemically milled areas in a given section of a tube. The two sets of numbers refer to each end of the tubes.

4. The slope is defined as the direction of the surface of the milled surface. When there is a deeper penetration toward the center of the tube the slope is positive. When the penetration is deeper toward the outside of the tube the slope is negative. Numerical values obtained by determining the difference between the depth of cut at the center of the section and subtracting from it the depth of cut at the edge of the section.

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