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PROCESSING OF LEU TARGETS FOR ^{99}Mo PRODUCTION -- DISSOLUTION OF U_3Si_2 TARGETS BY ALKALINE HYDROGEN PEROXIDE

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ABSTRACT

Low-enriched uranium silicide targets designed to recover fission product ^{99}Mo were dissolved in alkaline hydrogen peroxide (H_2O_2 plus NaOH) at about 90°C. Sintering of matrix aluminum powder during irradiation and heat treatment retarded aluminum dissolution and prevented silicide particle dispersion. Gas evolved during dissolution is suspected to adhere to particles and block hydroxide ion contact with aluminum. Reduction of base concentrations from 5M to 0.1M NaOH yielded similar silicide dissolution and peroxide destruction rates, simplifying later processing. Future work in particle dispersion enhancement, ^{99}Mo separation, and waste disposal is also discussed.

INTRODUCTION

Concerns over nuclear proliferation are driving research to replace high-enriched uranium (HEU) with low-enriched uranium (LEU). Currently, ^{99}Mo, the precursor of ^{99m}Tc used in several medical applications, is almost exclusively produced from HEU. Commercial production of ^{99}Mo exploits the 6% yield achieved by the thermal neutron fission of ^{235}U:

$$^{235}\mathrm{U}\ (n,f)\ ^{99}\mathrm{Mo} \qquad (1)$$

The HEU targets are typically 93% enriched uranium oxide, uranium-aluminum alloy, or uranium aluminide. The ^{99}Mo is recovered by dissolving the irradiated target and separating the ^{99}Mo from the uranium and other fission products in the dissolver solution.

We are investigating the consequences of reducing enrichment to less than 20% by replacing HEU aluminide (UAl_x) targets with LEU silicide (U_3Si_2) targets. The substitution of LEU requires the target mass to be increased by a factor of six to achieve the same yield of ^{99}Mo under the same irradiation conditions. The increase in ^{238}U in the LEU targets produces more than 50 times the ^{239}Pu over existing HEU designs. Differences encountered in dissolving HEU and LEU targets are discussed.

URANIUM SILICIDE TARGETS

Over the last several years, uranium silicide fuels have been developed as LEU targets for ^{99}Mo production. The LEU silicide is aimed to replace the UAl_x in the HEU dissolution process used by the Institut National des Radioelements (IRE), Fleurus, Belgium [1]; Comision Nacional de Energia Atomica, Buenos Aires, Argentina [2]; and the Atomic Energy Corporation of

South Africa. Replacement of UAl_x with U_3Si_2 targets requires development of a more aggressive dissolution process. Unlike UAl_x, U_3Si_2 targets do not readily dissolve in base. In acid dissolution, silica is precipitated, and the ^{99}Mo cannot be recovered from the solution [3]. In 1987, workers at Argonne National Laboratory (ANL) were able to dissolve uranium silicide in alkaline hydrogen peroxide at 70°C, dissolving 0.3 g U_3Si_2 in 100 mL of liquid [4]. The target was initially placed in 3.0$\underline{M}$ NaOH to remove the cladding. The cladding solution was then removed, and a 1:1 ratio of 3$\underline{M}$ NaOH and 30 wt % H_2O_2 was used to dissolve the uranium silicide [4].

An optimized procedure for dissolving uranium silicide targets was proposed two years later [5]. After lying dormant for several years, the project resumed in 1993, and an improved dissolution process with separate steps for aluminum cladding removal and uranium silicide dissolution was developed and tested using unirradiated targets (discussed below) [6]. A process reporting combined dissolution of aluminum cladding and uranium silicide could not be reproduced in our laboratory [7]. Initially, the target was placed in 3$\underline{M}$ NaOH-3$\underline{M}$ $NaNO_3$ in a glass vessel. This solution dissolved the cladding and the aluminum in the fuel matrix. After the aluminum was dissolved, the newly formed flocculent and the solution were removed from the dissolver vessel, leaving the dense uranium silicide behind. The vessel was heated to 90-95°C, and 5 mL each of 30 wt % hydrogen peroxide (9.56$\underline{M}$) and 10$\underline{M}$ NaOH was added to 3 g of silicide in the dissolver vessel. This combination produced close to the optimum dissolving solution of 5$\underline{M}$ H_2O_2-NaOH. The solution foamed vigorously, subsided within about 3 min, and produced a dark red uranium solution. To avoid dilution, the solution was removed from the dissolver vessel, leaving behind the undissolved U_3Si_2. Then, 10 mL each of 30 wt % hydrogen peroxide (9.56$\underline{M}$) and 10$\underline{M}$ NaOH was added to the vessel. The reagent volume was increased because the foaming decreased with subsequent reagent additions. As before, the solution foamed, subsided within about 3 min, and yielded a dark red uranium solution. This solution was removed from the vessel, and the process was repeated until the entire target was dissolved. It took about 150 mL of reagents to dissolve 3 g of U_3Si_2 in 1 h [6]. This procedure achieved concentrations up to 0.16$\underline{M}$ U, sufficient for LEU targets to produce ^{99}Mo concentrations equivalent to current HEU processes. Unfortunately, this procedure failed to dissolve a 9-year-old (~40% burnup) LEU silicide target; nearly 10 h and 800 mL of 5$\underline{M}$ H_2O_2-NaOH were required to complete dissolution [6].

The procedure to remove the aluminum cladding was developed in the fifties at Oak Ridge National Laboratory (ORNL) [8], and slight variations of it are practiced all over the world. It has worked well for removing cladding from nonirradiated-unannealled ("cold"), thermally annealed, and irradiated targets. However, the aluminum powder in the meat of the thermally annealed and irradiated targets did not dissolve well after the cladding was removed. Also, the silicide particles did not disperse but remained affixed with matrix aluminum powder in a thin wafer. The annealed targets required approximately double the time of the cold targets to dissolve the matrix aluminum and disperse the particles. After the silicide dispersed, it dissolved readily, as in the cold targets. Experiments to optimize U_3Si_2 dissolution after complete aluminum dissolution (cladding and matrix) are in progress at our laboratory.

OPTIMIZED PROCEDURE FOR ALUMINUM DISSOLUTION

We have found that aluminum cladding alloys used in the targets for production of ^{99}Mo dissolved easily in a solution of 3M NaOH-3M $NaNO_3$ at 88°C. Most of the alloying elements precipitate, but the sodium aluminate remains in solution under the optimized dissolution conditions for molar ratios of Al:NaOH:$NaNO_3$ at 1.00:1.66:1.47.

During development of the optimized procedure, samples of dispersed U_3Si_2 targets were thermally annealed to simulate the effects produced during irradiation. During this annealing, the aluminum sintered, and limited diffusion of U_3Si_2/Al across grain boundaries occurred. Sample targets thermally annealed at 450°C for 0, 12, 24, and 72 h failed to dissolve as easily as old unannealed depleted uranium (DU) targets. The aluminum cladding dissolved in 5-10 min for all samples. The silicide particles in an old unannealed plate broke apart into a loose powder upon dissolution of the cladding. The particles did not disperse when the cladding of the thermally annealed plates dissolved. The particles retained the shape of a plate, and the matrix or "meat" aluminum slowly dissolved in an hour. The major difference in the targets was the type of aluminum used in the meat. The old unannealed plate used pure aluminum powder while the annealled samples used the alloy Al 6061. All targets had Al 6061 cladding.

Samples of U_3Si_2 plates with pure aluminum powder in the meat were then obtained and annealed at 450°C for 0, 3, 6, and 12 h. These samples contained 35% uranium by mass sandwiched in Al 6061 cladding. A control and each annealed sample were dissolved using the procedure specified by Hutter when dissolving the high-burnup target in September 1994 [6]. Thermal annealing retarded particle dispersion.

The cladding dissolution procedure used 20 mL of 3M NaOH-3M $NaNO_3$ per gram of aluminum, and the resulting solution was agitated continuously at 88°C until dissolution. The mass of aluminum in the samples ranged from 0.41 to 0.48 g and 10 mL of cladding dissolving solution was used in each case. The cladding dissolved in 10-12 min in each case, yielding the meat as a wafer containing U_3Si_2 particles and pure aluminum powder. The solutions became opaque dark-gray due to suspended flocculent material from the Al 6061 alloy and lost approximately 40% of their volumes due to boiling. The aluminum dissolution reaction nearly stopped after the cladding was dissolved.

The solutions were removed from the dissolver vessels, and a fresh 10 mL of 3M NaOH-3M $NaNO_3$ was added. As the cladding dissolving solution warmed from room temperature to 88°C, bubbles formed on the surface of the wafer and entered the solution. The control sample dispersed into powder 25-30 min after the beginning of the aluminum dissolution. The annealed samples did not disperse as quickly. The bubbles formed on the meat wafer were less frequent, and the wafer had not broken up after 40-45 min. Again the solution was removed (60% recovery) and replaced with 10 mL of 3M NaOH-3M $NaNO_3$. The wafer slowly broke into smaller pieces and dispersed into particles 60-70 min after the beginning of the aluminum dissolution. The different annealing times (3, 6, or 12 h) did not appear to affect the aluminum dissolution time other than doubling that required by the control sample.

The aluminum cladding was removed, and another set of annealed samples was prepared for metallographic analysis. High temperature was determined to have caused the aluminum powder to sinter together and block the channels needed for the dissolving solution to reach the aluminum. It is also believed that bubbles formed during the dissolution of aluminum, adhered to the meat matrix, and blocked the path of the dissolving solution. A sonicating bath proved ineffective in dislodging these bubbles. We plan to investigate two additional methods for dislodging gas bubbles: employing a powerful sonicating wand and adding surfactants to the solution to reduce surface tension.

We found that the hydroxide flocculent of Al 6061 alloying elements must be removed since it catalyzes the autodestruction of hydrogen peroxide in the subsequent silicide dissolution. The first contact with 3M NaOH, 3M $NaNO_3$ that dissolves the cladding has the most flocculent and the least recoil fission products. The solutions used to dissolve the matrix aluminum contain less flocculent material and much higher levels of fission products. Depending on the size of the silicide particle, between 8 and 30% of the ^{99}Mo produced will be lost to the aluminum dissolution solutions due to fission recoil. Economic concerns dictate ^{99}Mo recovery from the aluminum dissolving solution, and we are investigating the yield of ^{99}Mo in the fractions. Because the aluminum in the meat dissolves more slowly than the cladding, it may be possible to discard the cladding removal solution without ^{99}Mo recovery.

CONSUMPTION OF HYDROGEN PEROXIDE DURING URANIUM SILICIDE DISSOLUTION IN ALKALINE PEROXIDE SOLUTIONS

Two chemical reactions occur during the silicide dissolution process, the autodestruction of hydrogen peroxide and the dissolution of uranium silicide. A literature search revealed very little information about the autodestruction of hydrogen peroxide in sodium hydroxide solutions. One source simply identified that the autodestruction reaction is catalyzed in base, but no quantitative data were given [9]. A limited kinetic study of dilute hydrogen peroxide (0.01M) in 0.5-6.0M NaOH at room temperature indicated that hydrogen peroxide was stable in highly basic solutions [10]. The rate of autodestruction of hydrogen peroxide:

$$2\,H_2O_2 \rightarrow 2\,H_2O + O_2 \qquad (2)$$

depends upon the concentrations of base and peroxide and the temperature. The peroxide consumption rate for solution in contact with particles is given by:

$$\frac{d[H_2O_2]}{dt} = -k_m[H_2O_2]^n \qquad (3)$$

where,

$[H_2O_2]$ = concentration of hydrogen peroxide, mol/L
t = time, min
n = order of reaction of hydrogen peroxide
k_m = rate constant, $min^{-1}\cdot(mol/L)^{1-n}$

For Eq. 3, the values of n and k_m vary with base concentration in the alkaline peroxide dissolution of uranium silicide. Similar variation was observed in the alkaline peroxide dissolution of LEU foil targets [11]. Experiments without silicide particles indicated first-order dependence of peroxide concentration on the peroxide destruction rate [6].

The destruction of peroxide is catalyzed by surfaces and increases significantly with increases in temperature. The addition of silicide particles provides active surfaces that consume peroxide during silicide dissolution and destroy peroxide due to local heating. If autodestruction did not occur, 25 moles of H_2O_2 would be consumed per mole of U_3Si_2 dissolved. In a typical dissolution, approximately an order of magnitude more H_2O_2 is consumed. Figure 1 depicts the variation in peroxide consumption rate with starting base concentration at 40, 50, and 60°C. At all three temperatures, the peroxide consumption rate reached a maximum when the starting base concentration was 1.0-1.5M NaOH.

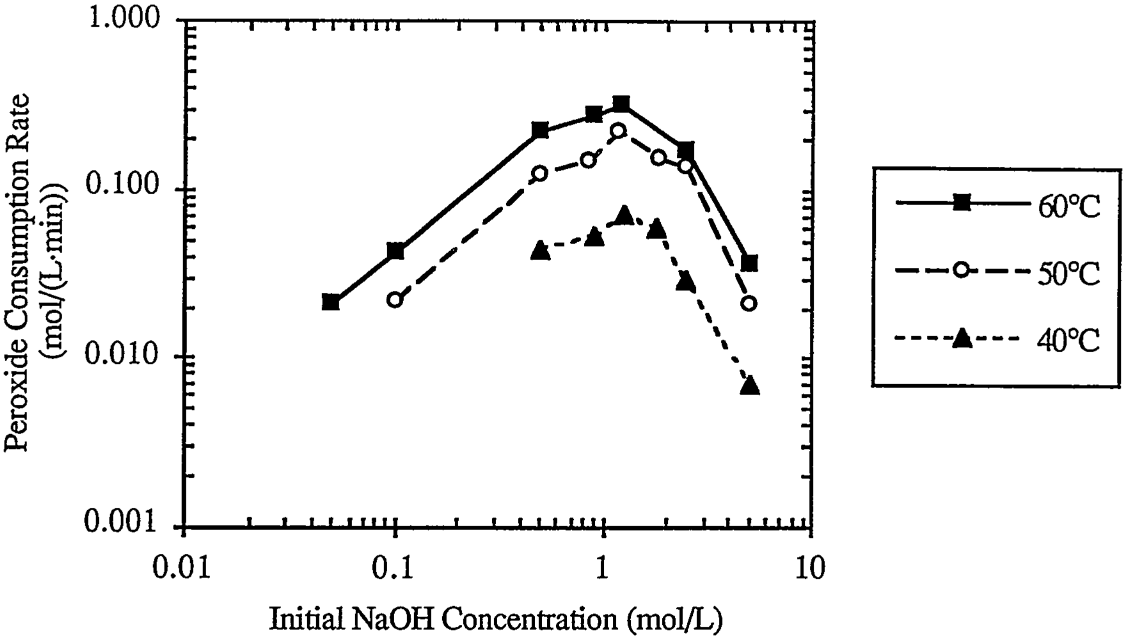

Figure 1. Effect of Initial Base Concentration and Temperature on the Initial Peroxide Consumption Rates in the Presence of U_3Si_2 Particles. Initial H_2O_2 concentration was 5.5M.

At equilibrium, the concentrations of H_2O_2 and OH^- are significantly lower than the starting concentrations. The equilibrium equation

$$H_2O_2 + OH^- \xleftrightarrow{K=160} HO_2^- + H_2O \tag{4}$$

governs the actual concentrations of H_2O_2 and OH^- during dissolution. Using equilibrium concentrations for hydrogen peroxide and base reduces the variation of peroxide reaction order and rate constant. The maximum peroxide destruction rate occurs at the equilibrium base concentration of ~0.1M OH^-, which corresponds to a starting concentration of ~1.2M NaOH. Peroxide concentrations were determined by placing 0.1 mL of dissolving solution into 50 mL of 1M H_2SO_4 and 0.15M KI for titration with 0.1M sodium thiosulfate [12].

URANIUM SILICIDE DISSOLUTION

The kinetics of uranium silicide dissolution were determined by the initial rate method [13]. The dissolution was done in a 250 mL jacketed glass beaker. Temperature was controlled by circulating a 50/50 mixture of propylene glycol and water in an external beaker jacket using a Brinkman RC 6 refrigerator/heater. By this method, the temperature of the beaker contents was easily controlled within ±2°C during the experiments. The temperature of the beaker contents was monitored by a thermometer. The liquid phase was continuously stirred with a magnetic stirrer. The stirring was not adequate to suspend the dense silicide particles, but the liquid phase was well mixed. During a typical experiment, the hydrogen peroxide and sodium hydroxide mixture was initially thermally equilibrated in the beaker. When the silicide was introduced, the first sample was taken, and the clock was started. Grab samples were taken at predetermined intervals during a 15-25 min experiment. The grab samples were analyzed for hydrogen peroxide by titration and for dissolved uranium by inductively coupled plasma-mass spectroscopy (ICP-MS). During the experiment, the heat generated by the autodestruction of hydrogen peroxide was continuously removed by the circulating heat-transfer fluid. At temperatures higher than 60°C, the heat released by the autodestruction reaction was greater than the capacity of the jacketed beaker to remove it. The liquid temperature increased rapidly, and the dissolution could not be controlled, preventing completion of accurate initial rate experiments.

The dissolution experiments used 30-100 mg samples of 45-53 µm (325-270 mesh) depleted U_3Si_2 particles in 100 mL of dissolving solution. The small particle mass was required to control the dissolver temperature. Sample masses of 500-1500 mg used in earlier work [6] produced run-away reactions in which the temperature increased 20-30°C and the contents of the dissolver beaker foamed over the top. Smaller samples prevented run-away reactions and allowed more accurate measurement of reaction rates. Dissolution rates remained constant as long as the peroxide concentration was not depleted (generally the first 10 min of the reaction). Starting solution concentrations were varied from 0.05 to 5.0M NaOH with 5.5M H_2O_2 at 40, 50, and 60°C. Uranium dissolution rates are plotted against initial base concentrations in Figure 2. Maximum U_3Si_2 dissolution rates occurred for initial base concentrations of 1.2-1.8M NaOH, corresponding to the peroxide consumption curves depicted in Figure 1.

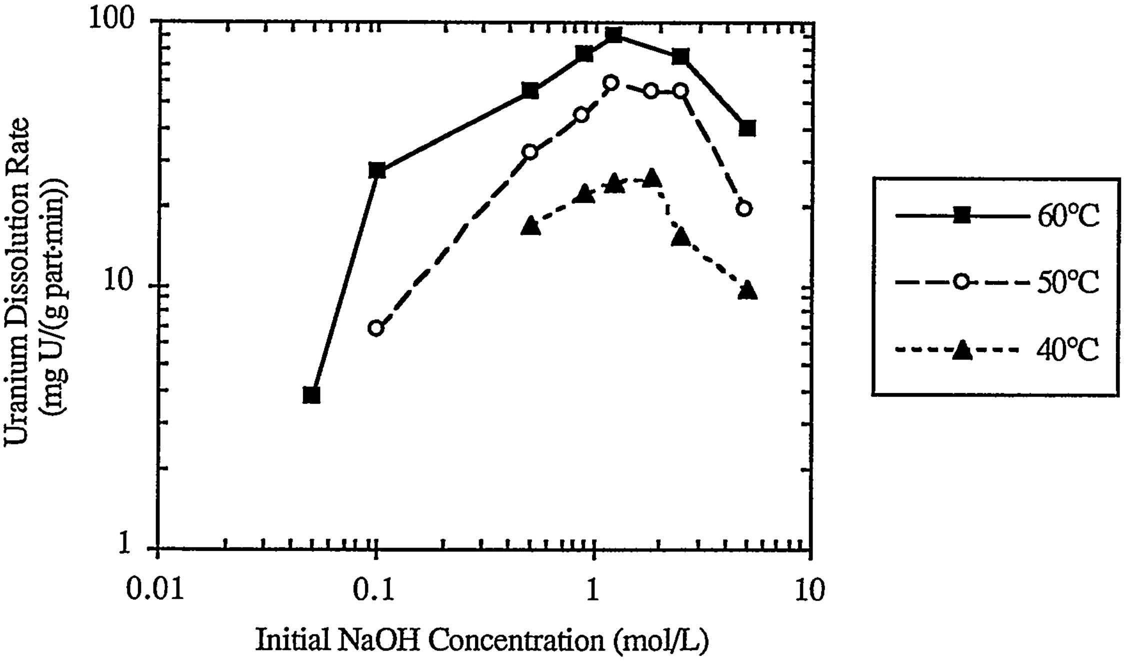

Figure 2. Effect of Initial Base Concentration and Temperature on the Initial Uranium Dissolution Rates in the Presence of U_3Si_2 Particles. Initial H_2O_2 concentration was 5.5M.

The uranium dissolution reaction rate varied with base concentration similarly to the foil dissolution [11]. The reaction rate can be written

$$\frac{dU}{dt} = k_U [H_2O_2]^n \qquad (5)$$

where,

U = uranium mass dissolved per g U_3Si_2 particles, mg/g
t = time, min
n = reaction order
k_U = dissolution rate constant, mg U/g U_3Si_2/min/ $[H_2O_2]^n$
$[H_2O_2]$ = concentration of hydrogen peroxide, mol/L

The dissolution rate constant also has a hydroxide-ion-dependent term embedded in it, since OH^- is consumed with H_2O_2 in the dissolution of U_3Si_2. Earlier work that indicated a second-order correlation of peroxide concentration to uranium dissolution rate for initial base concentrations of 2.57M NaOH [6] agrees with our more recent data.

OPTIMIZED URANIUM SILICIDE DISSOLUTION PROCEDURE

Last year we proposed an optimum dissolution procedure that employed 5M NaOH-5M H_2O_2 at 90-95°C [6]. Further experiments demonstrated that similar results could be obtained at lower base concentrations. The uranium dissolution and peroxide destruction rates can be preserved by reducing the base concentration from 5 to 0.1-0.2M NaOH. This change in base reduces later acid addition during processing by a factor of 25-50. Slightly higher base concentrations (e.g., 0.5M NaOH) increase uranium dissolution modestly but suffer much higher peroxide destruction. Although optimum conditions for an irradiated target are not yet determined, initial conditions will likely be 0.2-0.5M NaOH and 5-7M H_2O_2 at 80-90°C. The dissolution kinetics at these conditions are ideal for a plug-flow reactor configuration. Spent solution loaded with dissolved target can be drained and replaced. If the solution is boiled away, the base should remain, and a H_2O_2 drip may replace solution volume and produce a more concentrated solution.

CONTINUED DEVELOPMENT OF URANIUM SILICIDE PROCESS

The technical and economic feasibility of an LEU silicide process for ^{99}Mo production depends on progress on chemical processing of hot targets. We plan to focus on the following processing issues.

Dispersion of annealed and irradiated LEU targets. Increase rate of matrix aluminum dissolution through use of surfactants and/or focused sonic energy.

Molybdenum-99 loss during aluminum dissolution. Conduct low burnup tracer tests to determine loss of ^{99}Mo to cladding and matrix aluminum. Verify quantity of U, ^{99}Mo, and other fission products dissolved with cladding. Attempt to show that initial cladding removal solution can be disposed as low-level waste, and only the aluminum dissolved in meat requires processing for ^{99}Mo recovery.

Acidification of the cladding solution. Acidify cladding solution for recovery of ^{99}Mo by ion chromatography. The HEU processes used at IRE [1] acidify the dissolved solution (cladding and fuel are dissolved in one step) to 1M by using concentrated nitric acid. Work at Argonne showed that ^{99}Mo is better recovered with 0.5M nitric acid [14]. Experiments to verify this procedure are planned.

Acidification of the dissolver solution. Acidify dissolved target solution and keep the silicon concentration less than 0.1M in the acidified dissolver solution [4] to prevent precipitation of gelatinous silica. Concentrated nitric acid will be used to acidify this solution.

Recovery of the uranium. Precipitate uranium as sodium diuranate by destroying the peroxide complex after the silicide target is dissolved. Experiments are planned to determine the parameters that control this process, including the effect of carbonates on interfering with the uranium precipitation. Since coprecipitation loss of ^{99}Mo was experienced with bulky uranyl hydroxide [15], coprecipitation with sodium diuranate will be assessed. Processes for recycling the recovered uranium and their cost effectiveness will be investigated.

Ion chromatography recovery of ^{99}Mo. This procedure has been used at IRE on HEU targets for more than 10 years. A detailed description of the process is given elsewhere [1]. Unlike the IRE process, the LEU process will produce a slightly different composition solution. The procedure described by Sameh and Bertram-Berg using an anion exchange resin followed by a chelating resin column may also be used [16]. Experiments are planned in which an irradiated miniplate will be used to determine the material balance for molybdenum, uranium, activation products, and fission products. These experiments will employ the optimized dissolution procedure and the published ion chromatographic procedure to recover the ^{99}Mo.

Waste disposal. The liquid and solid wastes generated from the process must be characterized and disposed. A material balance on the optimized process will be done, and the waste streams identified.

CONCLUSIONS

Target dissolution continues to be the primary development need for the replacement of conventional HEU aluminide with LEU silicide targets for ^{99}Mo production. Aluminum alloy cladding dissolved easily in base, but sintering of matrix aluminum powder and adhesion of gas bubbles to the silicide matrix during dissolution retarded aluminum dissolution and prevented silicide particle dispersion. Once dispersed, silicide particles dissolved in alkaline hydrogen peroxide solutions. Reduction of base concentrations from 5$\underline{M}$ to 0.1$\underline{M}$ NaOH yielded similar silicide dissolution and peroxide destruction rates, simplifying later processing. The kinetics of the dissolution process favor a plug-flow reactor configuration that frequently replaces a small volume of the dissolving solution or continuously replenishes hydrogen peroxide. Downstream process development remains to be done, including ion chromatographic recovery of ^{99}Mo and waste disposal.

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