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

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DISCLAIMER

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ABSTRACT

In FY 1995, we started studies on a new process for dissolution of low-enriched uranium (LEU) targets for ^{99}Mo production. In this process, an LEU metal foil target is dissolved in a mixture of sodium hydroxide and hydrogen peroxide, then ^{99}Mo is recovered from the dissolved solution. We focused on the dissolution kinetics to develop a mechanistic model for predicting the products and the rate of uranium dissolution under process conditions. We thoroughly studied the effects of hydrogen peroxide concentration, sodium hydroxide concentration, and temperature on the rate of uranium dissolution.

It was found that uranium dissolution can be classified into a low-base (<0.2$\underline{M}$) and a high-base (>0.2$\underline{M}$) process. In the low-base process, both the equilibrium hydrogen peroxide and hydroxide concentrations affect the rate of uranium dissolution; in the high base process, uranium dissolution is a 0.25th order reaction with respect to the equilibrium hydrogen peroxide. The dissolution activation energy was experimentally determined to be 48.8 kJ/mol. Generally, the rate of uranium dissolution increases to a maximum as the hydroxide concentration is increased from 0.01 to about 1.5$\underline{M}$, then it decreases as the hydroxide concentration is further increased. The alkalinity of the dissolution solution is an important factor that affects not only the dissolution rate, but also the amount of radioactive waste.

INTRODUCTION

Argonne National Laboratory (ANL) has been developing technologies for the chemical processing of LEU targets and production of ^{99}Mo for medical applications[1-6]. Two processes are currently under development [7-8]: the dissolution of LEU-foil targets with sulfuric and nitric acids and the dissolution of LEU-uranium silicide (U$_3$Si$_2$) targets with alkaline peroxide solutions. In FY 1995 we started studies on a new process, chemical processing of LEU-metal foil targets with alkaline peroxide solutions. In this process, an irradiated LEU-foil target is dissolved in a sodium hydroxide/hydrogen peroxide solution. The fission product ^{99}Mo is then separated and purified from the dissolved solution. Low-enriched-uranium metal foil targets can replace both HEU/Al and UO$_2$ targets[9]. To produce the same amount of ^{99}Mo per target, the total amount of uranium in an LEU (~19.75% of ^{235}U) target that has to be processed will be approximately five times as much as that in an HEU (~93% of ^{235}U) target.

Unlike the well-documented acid process, dissolution of uranium metal with alkaline peroxide solutions has received little study. In the early 1940s, Warf [10] reported that "X (the X stands for uranium) metal dissolved in H_2O_2 + Na_2O_2, NaOH + H_2O_2, and Na_2O_2 + H_2O slowly, and in NaOH + Na_2O_2 very slowly." Both Gindler [11] and J. C. Warf [12], in their review papers, briefly mentioned that uranium metal dissolves in sodium hydroxide solution containing hydrogen peroxide or in sodium peroxide-water mixture, and they both cited L. Warf's report [10] as a reference. Larson [13] reported that uranium metal reacts at moderate rate with a sodium hydroxide/hydrogen peroxide mixture to form a clear solution, highly colored by the uranyl peroxide complex.

In FY 1995, we focused on the uranium dissolution kinetics to develop a mechanistic model for predicting the products and the rate of dissolution under process conditions. We determined the rate of dissolution with depleted uranium (DU) foil under various chemical conditions. Dissolution kinetics are analyzed and reported here. We also developed a closed-dissolver and gas-trap system in cooperation with BATAN researchers for dissolving irradiated/LEU in a shielded cell. The closed system was necessary to trap fission gases.

EXPERIMENTAL

Uranium Dissolution and Analysis

Dissolution experiments were carried out in an open, batch-type reactor under isothermal conditions. The dissolver used is a 250 mL jacketed beaker placed in a fume hood. In an experiment, a piece of DU foil (~0.2 g mass and ~2.0 cm^2 total surface) was contacted with 100 mL solution of hydrogen peroxide and sodium hydroxide. The reacting solution was continuously stirred during dissolution. A Brinkman RMS-6 refrigerator/heater was used to maintain a constant temperature by circulating a 50/50 propylene glycol-water mixture through the jacket. A thermometer was kept in the solution to monitor temperature. Typically, temperature fluctuation of the reactor contents was controlled within ±1.0°C.

The sodium hydroxide solution was first introduced into the dissolver and heated to a dissolution temperature. Then, a predetermined volume of 30% hydrogen peroxide solution was added to make a total volume of 100 mL. When the mixture temperature rose to and stabilized at the experimental temperature, a piece of DU foil was dropped into the solution, and a stop watch was started to record the dissolution time. The first grab sample was immediately withdrawn from the dissolver. Subsequent grab samples were taken at predetermined time intervals.

Each grab sample had a volume of about 1 mL (0.3 mL was used for uranium analysis and 0.1 mL for hydrogen peroxide determination). Upon being withdrawn from the dissolver, a grab sample was immediately quenched in a dry ice/water bath to stop the decomposition of hydrogen peroxide. Typically, we collected 9 to 10 grab samples in each experiment. Dissolved uranium was analyzed by inductively coupled plasma-mass spectrometry (ICP-MS, Fisons PlasmaQuad II, Fisons Elemental, Winsford, Cheshire, UK). The ICP-MS analyses were carried out by the Analytical Chemistry Laboratory (ACL) at ANL. Each sample was run five times, and the mean was used for the initial rate determination. The overall error of determination was estimated to be ±10% or less. Detailed instrument operating conditions can be found elsewhere [14]. In this investigation, the initial rate technique was used to determine the rate of reactions.

Hydrogen peroxide was determined by titration [15] of liberated iodine (I_2) with a standard 0.10$\underline{N}$ sodium thiosulfate solution. The detailed experimental procedure can be found elsewhere [6].

RESULTS AND DISCUSSION

Dissolution Kinetics

Figure 1 shows the effect of hydroxide concentration on the rate of uranium dissolution at fixed temperature and a fixed hydrogen peroxide concentration of about 3.5$\underline{M}$. The rate of uranium dissolution increases sharply (a slope of about 2 in the log-log plot) from a base concentration of 0.01$\underline{M}$ to about 0.2$\underline{M}$, reaches a maximum at about 1.5$\underline{M}$, then decreases as the base concentration is further increased from 1.5$\underline{M}$ to 5.0$\underline{M}$.

Hydroxide concentration is a very important factor in the processing of LEU targets for production of [99]Mo. After an irradiated target is dissolved, the dissolver solution pH will be adjusted to produce an acid solution. The [99]Mo will be separated from other components in the acidified solution by its adsorption on a bed of alumina or another anionic exchange, then stripped from the column by a concentrated alkaline solution. Because of the short half-life of [99]Mo, a high rate of dissolution is preferred to reduce processing time. On the other hand, the lower the base concentration, the less radioactive waste will be generated from the process because a low alkaline content in the dissolver solution would reduce the amount of acid solution needed to achieve the desired acidification.

Previous work [6] has reported that the optimum conditions for LEU-silicide dissolution appeared to be 5$\underline{M}$ NaOH-5$\underline{M}$ H_2O_2. However, Figure 1 indicates that a low base concentration (e.g., 1.5$\underline{M}$ or lower) is likely favored for LEU-foil dissolution. For example, at a base concentration of 1 to 1.5$\underline{M}$, a high dissolution rate can be achieved, and the processing time will be shortened. Meanwhile, the total amount of radioactive waste will be reduced.

Figure 2 shows that the effect of base concentration on the rate of overall disappearance of hydrogen peroxide is similar to that of uranium dissolution. A maximum rate of peroxide disappearance takes place at a base concentration of about 1.0 to 1.5$\underline{M}$. The rate of overall disappearance of hydrogen peroxide decreases if the base concentration is either increased or decreased from this region.

The rate of uranium dissolution as a function of hydrogen peroxide concentration is shown in Figure 3, and the rate of overall disappearance of hydrogen peroxide is plotted in Figure 4. If both uranium dissolution and the overall disappearance of hydrogen peroxide are expressed as an nth order reaction (Eqs. 1 and 2), the slopes of those lines in Figures 3 and 4 can be used to estimate the order of reactions, and the intercepts at [H_2O_2]=1$\underline{M}$ can be used to estimate the rate constants. The estimated order of reaction and the rate constants are summarized in Table 1.

$$R = \frac{1}{S}\frac{dU}{dt} = kC^n \tag{1}$$

$$R' = -\frac{dC}{dt} = k'C^{n'} \tag{2}$$
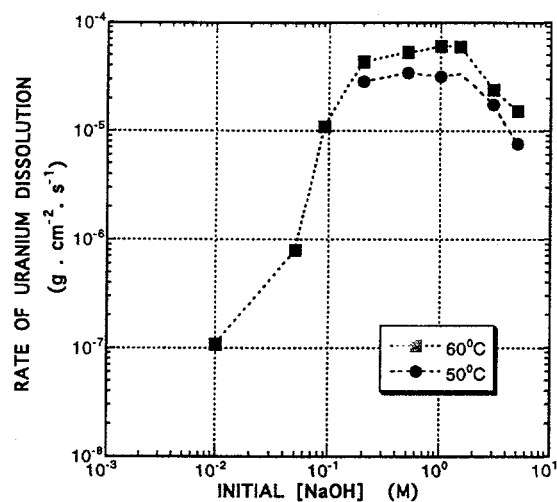
Figure 1. Effect of Initial Alkaline Concentration on Rate of Uranium Dissolution (U Surface≈2.0 cm^2, Initial [H$_2$O$_2$]≈3.5$\underline{M}$)
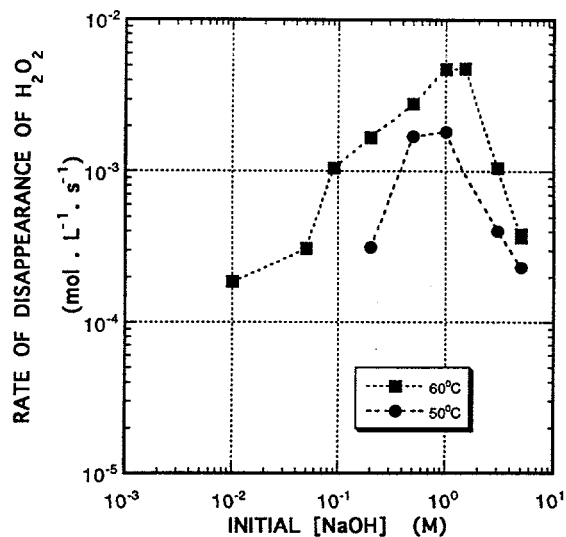
Figure 2. Effect of Alkaline Concentration on Rate of Depletion of H$_2$O$_2$ (U Surface≈2.0 cm^2, Initial[H$_2$O$_2$]≈3.5$\underline{M}$)
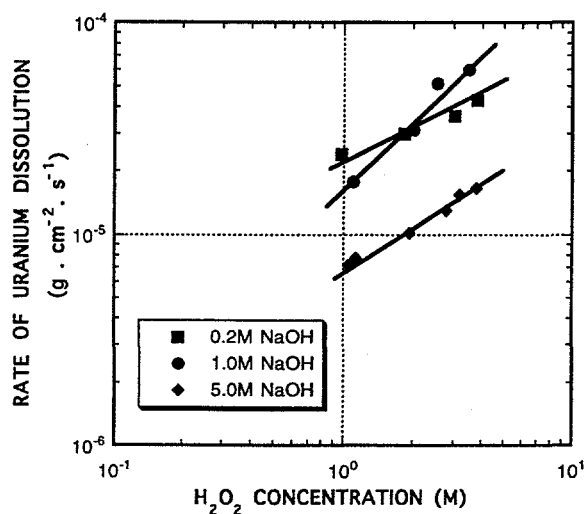
Figure 3. Effect of Hydrogen Peroxide on Rate of Uranium Dissolution (U Surface≈2 cm^2, Temp.=60°C)
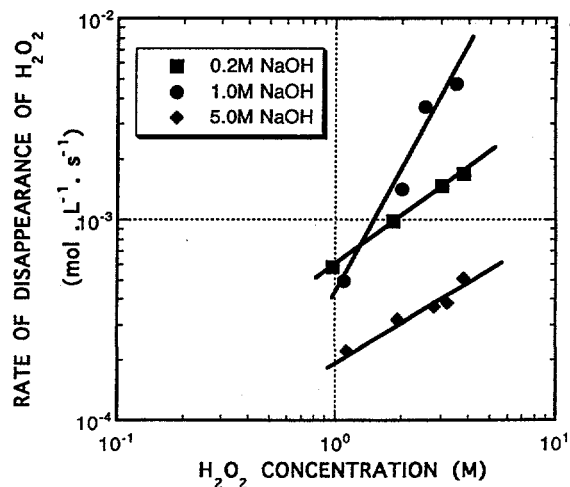
Figure 4. Effect of Hydrogen Peroxide on Rate of Its Depletion (U Surface≈2.0 cm^2, Temp.=60°C)

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In Eqs. 1 and 2, U is the amount of uranium dissolved in grams; S is the total uranium surface in cm^2; C is the hydrogen peroxide concentration in mol/L; R is the rate of uranium dissolution in [g-uranium $\cdot$ s^{-1} $\cdot$ cm^{-2}-uranium surface]; R' is the rate of overall disappearance of hydrogen peroxide in [mol-peroxide $\cdot$ L^{-1} $\cdot$ s^{-1}]; n and n' are the corresponding order of reactions; and k and k' are the corresponding rate constants, respectively. Since both n and n' are dimensionless, the k and k' are defined as [grams-uranium $\cdot$ L^n $\cdot$ $mole^{-n}$-peroxide $\cdot$ cm^{-2}-uranium surface $\cdot$ s^{-1}] and [$L^{(n'-1)}$ $\cdot$ $mol^{(1-n')}$-peroxide $\cdot$ s^{-1}], respectively.

Table 1. Apparent Kinetics Data for Uranium Dissolution ($R=kC^n$) and the Overall Disappearance of Hydrogen Peroxide ($R'=k'C^{n'}$) at Fixed Base Concentrations

NaOH	Temperature	Uranium Dissolution		Peroxide Disappearance	
M	°C	n	k	n'	k'
0.2	60	0.42≈0.5	2.385E-5	0.81≈1	5.989E-4
1.0	60	1.10≈1	1.601E-5	1.81≈2	4.611E-4
5.0	60	0.64≈0.5	7.004E-6	0.45≈0.5	2.178E-4

Table 1 indicates that the order of uranium dissolution with respect to hydrogen peroxide concentration varies at different base concentrations. Also, uranium dissolution is approximately a first order reaction in 1.0M base and an half-order reaction in 0.2M and in 5.0M base. The reason why the reaction order varies will be discussed in the next section.

Figure 5 shows the Arrhenius plots of uranium dissolution at fixed base concentrations. The lines at different base concentrations are almost parallel indicating a homogenous activation energy. The calculated activation energies of uranium dissolution are summarized in Table 2, which shows an average activation energy of 48.84 kJ/mol.

Table 2 Activation Energy of Uranium Dissolution

NaOH (M)	Activation Energy (kJ/mol)
0.2	45.48
1.0	47.79
3.0	43.56
5.0	58.52
Mean	48.84

Effect of H_2O_2 Equilibrium on Rate of Uranium Dissolution

There has been no report in the literature that the rate of uranium dissolution increases to a maximum, then decreases as the base concentration is further increased, and that the order of uranium dissolution with respect to hydrogen peroxide varies at different base concentrations. These experimental observations are explained in the following discussions.

Hydrogen peroxide is known to dissociate to perhydroxide ions in a basic solution according to the following equilibrium [16,17]:

$$H_2O_2 + OH^- \Leftrightarrow HO_2^- + H_2O \qquad (K=160) \qquad (3)$$
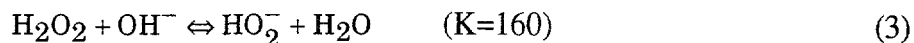
Based on Eq. 3, the presence of base (OH$^-$ ions) will strongly affect the hydrogen peroxide equilibrium. Also evident from Eq. 3 is that the equilibrium hydroxide concentration is affected by the H$_2$O$_2$ concentration. The plot of the effect of initial base concentration on the hydrogen peroxide concentration at equilibrium is shown in Figure 6. In this Figure, an initial (total) hydrogen peroxide concentration of 4.0$\underline{M}$ was assumed, and the species concentrations at equilibrium were calculated and plotted against the initial (total) base concentrations.

Figure 6 indicates that the concentration of hydrogen peroxide at equilibrium barely decreases as the initial (total) base concentration is increased up to about 0.2$\underline{M}$, gradually decreases as the initial base concentration is increased from 0.2$\underline{M}$ to 1.0$\underline{M}$, and sharply decreases as the initial base concentration is increased further. Hydrogen peroxide essentially stays in its form at a base concentration below 0.2$\underline{M}$ and undergoes an acid dissociation to perhydroxide ions (HO$_2^-$) at higher base concentrations. Figure 6 illustrates the following two facts that help understand uranium dissolution kinetics: (a) in a basic solution, the equilibrium hydrogen peroxide concentration is lower than its initial (total) concentration, and (b) the equilibrium hydrogen peroxide concentration in a high-base solution is lower than its counterpart in a low-base solution.

In the light of these results, we did equilibrium calculations for the data in Figures 1 and 3 and re-plotted the rate of uranium dissolution versus the equilibrium hydrogen peroxide concentration (instead of base concentration), as shown in Figure 7. As mentioned previously, these data cover four sets of experiments: the three sets used in Figure 3 were obtained at three fixed base concentrations (0.2, 1.0, and 5.0$\underline{M}$) with varying hydrogen peroxide concentration from 1.0 to 4.0$\underline{M}$, and the data set used in Figure 1 had a constant initial (total) peroxide concentration of about 3.5$\underline{M}$, but the base concentration varied from 0.01 to 5$\underline{M}$. In the last set, the variation in equilibrium H$_2$O$_2$ concentration was caused by different base concentrations.

Figure 7 clearly indicates that the uranium dissolutions over a broad range of base concentrations can be divided into two groups. The first group, the dashed line on the right-hand side, includes only three data points at base concentrations of 0.01, 0.05, and 0.09$\underline{M}$. The second group, the solid line, covers the remaining 19 data points at base concentrations $\geq$ 0.2$\underline{M}$. We conclude that uranium dissolution can be classified into a low-base and a high-base processes, and the transition from the low-base to high-base process takes place at a base concentration of about 0.2$\underline{M}$.

We believe that, in the low-base process (i.e., below 0.2$\underline{M}$), alkali content is the main factor that controls the rate of reaction. An increase in base concentration significantly promotes the rate of uranium dissolution. As shown in Figure 7, the equilibrium hydrogen peroxide concentration of the three data points in the first group barely varied, but the rate of dissolution varied significantly. It is obvious that this dramatic change in the rate of dissolution is caused by different base concentrations.

On the other hand, in the high-base process (i.e., above 0.2$\underline{M}$), the rate of uranium dissolution is solely controlled by the equilibrium hydrogen peroxide. Figure 7 indicates that, for base concentrations from 0.2$\underline{M}$ to 5.0$\underline{M}$, the rate of uranium dissolution increases as the equilibrium hydrogen peroxide concentration is increased, and that all 19 data points in the second group fall on the same straight line plotted against the equilibrium hydrogen peroxide concentration (in log-log plot). Therefore, the order of reaction does not vary with base concentrations. We
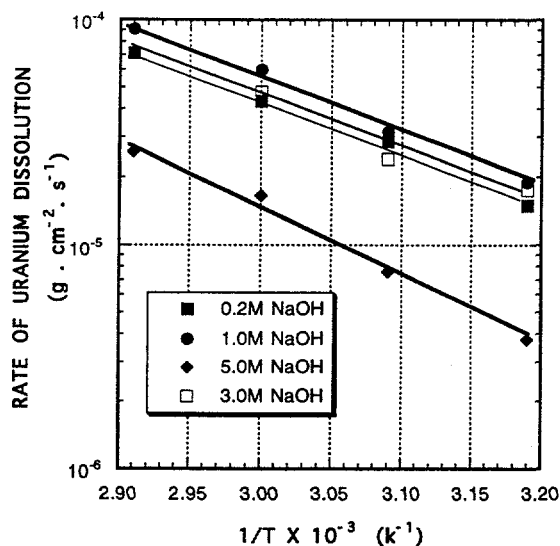
Figure 5. Effect of Temperature on Rate of
Uranium Dissolution (U
Surface≈2.0 cm^2, Initial
[H$_2$O$_2$]≈4.0$\underline{M}$)
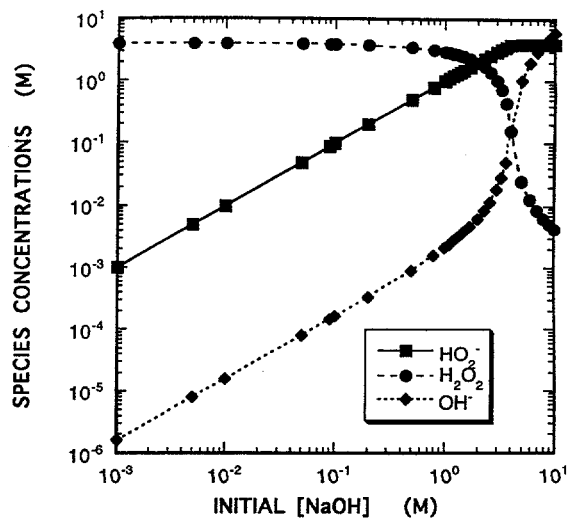
Figure 6. Effect of Hydroxide Concentration
on H$_2$O$_2$ Equilibrium (Initial
[H$_2$O$_2$] = 4.0$\underline{M}$) (H$_2$O$_2$ + OH$^-$
<=> O$_2$H$^-$ + H$_2$O (K=160))

Figure 7. Effect of Equilibrium Hydrogen Peroxide Concentration on Rate of
Uranium Dissolution (U Surface = 2 cm^2, Temp. = 60°C)

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believe that the variation in the order of reaction shown in Figure 3 is caused by the difference between the total and equilibrium concentrations of hydrogen peroxide in a basic medium.

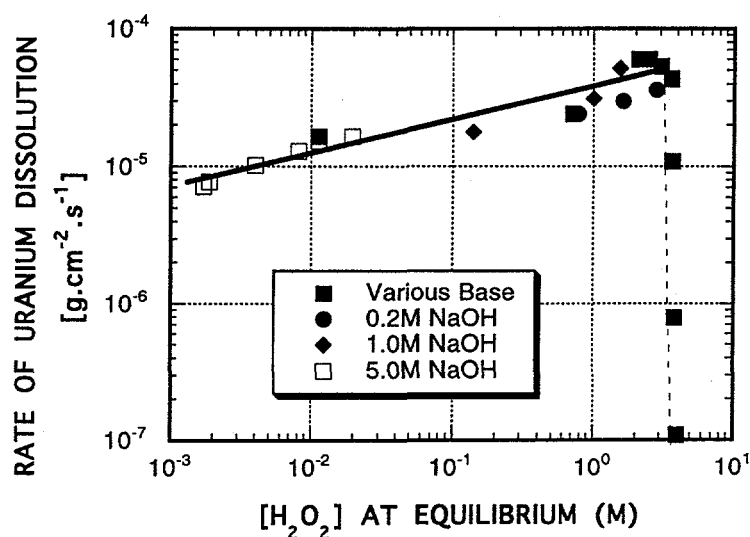Figure 7 also shows that all data points in the high-base group can be fitted to a straight line in a log-log plot (Eq. 4) that has a slope of 0.25 and a correlation coefficient of 0.89. Therefore, uranium dissolution in a high-base process is a 0.25^{th} order reaction with respect to the equilibrium hydrogen peroxide concentration. If the average activation energy listed in Table 2 is applied, the rate of uranium dissolution is defined by Eq. 5. With Eq. 5, we can predict the rate of uranium dissolution under process conditions.

$$R = \frac{1}{S}\frac{dU}{dt} = kC^n = 3.63 \times 10^{-5}[H_2O_2]^{0.25}_{equilibrium} \tag{4}$$

$$R = A(e^{\frac{-E_a}{RT}})C^n = 1.65 \times 10^3 (e^{\frac{-48.84 \times 10^3}{RT}})[H_2O_2]^{0.25}_{equilibrium} \tag{5}$$

In Eqs. 4 and 5, E_a is the activation energy (J mol^{-1}), A is a pre-exponential factor, R is the gas constant, T is the dissolution temperature in Kelvin, and $[H_2O_2]_{equilibrium}$ is the equilibrium concentration of hydrogen peroxide in a base solution.

Dissolution in Closed Dissolver System

We developed a closed-dissolver and gas-trap system for testing of small pieces of irradiated LEU foils at BATAN. The major objective of this part of work is to investigate if and how irradiation affects the rate of dissolution. To safely process irradiated targets in a closed system, radioactive gases and oxygen gas are our major concern. The radioactive gases are the fission products Iodine, Xenon, Krypton, etc., and appear in small quantity. A significant amount of oxygen gas is produced due to the decomposition of hydrogen peroxide. As a result, a pressure rise in the closed system is another problem to be solved.

The apparatus developed contains the following major functional devices: a 250 mL dissolver, a 3000-mL oxygen trap, a moisture trap, a radioactive noble gas trap, a pressure gauge, a pressure relief valve, and a 4000-mL ballast tank. These functional devices are connected together with tubings and valves so that the entire system can be easily operated in a closed environment. The total system volume is about 4500 mL.

The safety problems with oxygen gas and pressure-buildup were solved by using the oxygen trap and the ballast tank. The oxygen trap used is a cylinder filled with copper catalyst supported on aluminum-based materials, and it is able to destroy oxygen gas at ambient temperature. Experiments with cold LEU foils at ANL have shown that the oxygen trap effectively removed oxygen gas. The ballast tank was used to provide another safety means in case the oxygen trap did not function properly. It provides a large volume to reduce the system pressure.

Dissolution experiments in a closed system were carried out at both ANL and BATAN. At ANL, 100 mg of unirradiated LEU foil was tested with 50 mL of 4.0$\underline{M}$ H$_2$O$_2$/1.0$\underline{M}$ NaOH solution in each experiment. At BATAN, unirradiated LEU pieces (50 to 75 mg) were tested in 25 mL of H$_2$O$_2$/NaOH solution. The dissolution experiments at both sites were all performed at 60°C for 60 minutes. Table 3 summarizes the test results. Table 3 indicates that at ANL about 74% of uranium foil was dissolved within one hour; however, the BATAN results are significantly lower than those from ANL. We will investigate what caused this difference in the near future.

Table 3. Results of Uranium Dissolution in a Closed Dissolver System.
(U: unirradiated LEU foil; Temp = 60°C.)

Number	Uranium mg	Time minute	Solution	Volume mL	U. Dissolved %	Test Site
1	99.1	60	4.0$\underline{M}$ H_2O_2/1.0$\underline{M}$ NaOH	50	72	ANL
2	99.8	60	4.0$\underline{M}$ H_2O_2/1.0$\underline{M}$ NaOH	50	76	ANL
3	51.2	60	3.1$\underline{M}$ H_2O_2/1.0$\underline{M}$ NaOH	25	33	BATAN
4	64.2	60	3.1$\underline{M}$ H_2O_2/1.0$\underline{M}$ NaOH	25	37	BATAN
5	66.2	60	3.1$\underline{M}$ H_2O_2/1.0$\underline{M}$ NaOH	25	36	BATAN
6	73.9	60	3.1$\underline{M}$ H_2O_2/1.0$\underline{M}$ NaOH	25	52	BATAN
7	65.9	60	4.0$\underline{M}$ H_2O_2/1.0$\underline{M}$ NaOH	25	28	BATAN

CONCLUSION

Dissolution experiments have shown that alkalinity is an important factor in the uranium dissolution. It affects not only the rate of uranium dissolution, but also the total amount of radioactive waste generated from the process. The rate of uranium dissolution increases to a maximum as the alkaline concentration is increased from 0.01 to about 1.5$\underline{M}$, then it decreases as the base concentration is further increased.

Kinetics studies revealed that uranium dissolution can be classified into low and high base processes, separated at a base concentration of about 0.2$\underline{M}$. In base concentrations below 0.2$\underline{M}$, both the equilibrium hydrogen peroxide and alkali affect the rate of uranium dissolution. In base concentrations above 0.2$\underline{M}$, uranium dissolution is a 0.25^{th} order reaction with respect to the equilibrium hydrogen peroxide, and the dissolution activation energy was experimentally determined to be 48.8 kJ/mol.

Our research has shown that processing LEU metal foil with alkaline peroxide is a promising alternative for [99]Mo production. Compared to silicide (U_3Si_2) targets, for example, this process will eliminate a number of dissolution and process problems due to the presence of aluminum cladding and silicon. It also produces less radioactive waste.

For future work, we will study (1) thermodynamics and address the heat transfer issues, (2) investigate gas release and address the pressure-buildup issues, (3) optimize process parameters to reduce dissolution time and to minimize radioactive waste, (4) develop means to destroy peroxide remaining after the target dissolution, (5) develop procedures to precipitate and remove hydrated uranium and insoluble fission product, and (6) evaluate chromatographic technologies to recover [99]Mo from the dissolved solution.

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