An Interim Evaluation of the Thermal Stability of Cu-Y-⁸⁵Kr Sputter Deposits and Plans for Eventual Disposal

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April 1988

**Prepared for the U.S. Department of Energy
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SUMMARY

Pacific Northwest Laboratory has developed and demonstrated a pilot process for trapping and storing ^{85}Kr recovered and purified from the dissolver off-gas stream of a nuclear fuel reprocessing plant. Ions produced in a low-pressure krypton discharge are implanted in a growing sputtered film deposited on the inner wall of a krypton trapping storage device (KTSD). The main objectives of this work are to do the following:

- measure the release of ^{85}Kr implanted in a copper-yttrium alloy
- outline the characterization work to be performed on the Cu-Y- ^{85}Kr waste form
- identify the options available for disposing of the remaining ^{85}Kr .

Release rates were obtained from periodic measurements of the krypton released from both radioactive and nonradioactive krypton-loaded KTSDs. Sampling is expected to continue as long as the extrapolated release rates remain acceptable^(a) for a period not to exceed one half-life (~10 yr). Seven tests were performed on a nonradioactive KTSD over the temperature range 180-450°C. Three KTSDs loaded with 80-150 curies of ^{85}Kr stored at 150, 250, and 350°C were sampled four times each.

Proposed post-elevated-temperature storage characterization of the Cu-Y- ^{85}Kr waste form includes: identification of the surface composition and structure, metallographic examination of its cross-section, electron microscopy of thinned sections, and the susceptibility to corrosion in air and ground water.

(a) Current U.S. regulations limit the release of ^{85}Kr to 50,000 Ci/10⁹ watt yr of electrical energy, or about 15% of the total ^{85}Kr produced in light water (LWR) fuels.

Both the radioactive and nonradioactive test specimens stored at temperatures not exceeding 350°C have shown that the release rates measured to date are less than 0.1% yr⁻¹. The release rates for the radioactive specimens were obtained for storage periods of 1.6, 1.1, and 0.4 yr for 150, 250 and 350°C storage temperatures, respectively. The nonradioactive specimen has been held at temperatures ranging between 180 and 450°C, but mainly at 350°C, for a total storage time of 2.1 yr. Tests should continue for a period not exceeding 10 yr as long as the measured release rates remain acceptable in order to evaluate the effects of long-term storage on release. Following the ~10 yr monitoring period, the remaining Cu-Y-⁸⁵Kr waste form should be evaluated to better predict the overall stability for the full storage period (50-100 yr). Three options for the disposal of the remaining ⁸⁵Kr were identified which met present state and federal regulations. The preferred choice is best made at the time of disposal as these regulations are subject to change.

CONTENTS

SUMMARY	iii
INTRODUCTION	1
CONCLUSIONS AND RECOMMENDATIONS	3
METHODS	5
ELEVATED-TEMPERATURE STORAGE	5
PROCEDURE FOR KRYPTON-RELEASE MEASUREMENTS	7
RESULTS AND DISCUSSION	11
NONRADIOACTIVE KRYPTON-RELEASE MEASUREMENTS	11
RADIOACTIVE KRYPTON-RELEASE MEASUREMENTS	13
POST-ELEVATED-TEMPERATURE STORAGE CHARACTERIZATION	17
DISPOSAL OF RADIOACTIVE KRYPTON	19
REFERENCES	21
ACKNOWLEDGEMENTS	23

FIGURES

1	Krypton Trapping Storage Device Storage Cask and Heater	.	.	6
2	Schematic Diagram of Piping for Measuring ^{85}Kr Release	.	.	8

TABLES

1	Gas-Collection Results with Nonradioactive Surrogate	.	.	.	12
2	Long-Term ^{85}Kr Release Data Obtained with $\text{Cu}_{.67}\text{Y}_{.25}\text{Kr}_{.08}$ Stored 150°-350°C	.	.	.	14

INTRODUCTION

Testing the long-term stability of sputter-deposited krypton-metal matrix is needed to evaluate the ion implantation/sputter-deposition process for immobilizing radioactive ^{85}Kr gas. Current regulations (EPA 1977) will limit the release of ^{85}Kr to about 15% of the ^{85}Kr in light water reactor fuels or 8% of that generated in high-temperature gas-cooled reactor fuels. After 50 yr of storage only 4% of the original ^{85}Kr will remain. Laboratory experiments using nonradioactive krypton trapped in a nickel-lanthanum sputter-deposited matrix have shown that krypton gas is released from the metal matrix at low rates by a diffusion process (Knoll, et al. 1986). Gas release rates and the stability of the metal matrix may, however, be affected by the radiation field and transmutation products in an actual storage container of ^{85}Kr . Pacific Northwest Laboratory (PNL) is currently evaluating the thermal and radiation stability of ion-implanted ^{85}Kr in a sputter-deposited matrix (McClanahan, et al. 1987) for the Department of Energy (DOE). Experiments are in progress to measure the release of ^{85}Kr gas from three multicurie copper-yttrium-krypton deposits and to confirm the stability of the Cu-Y-Kr alloy at 150, 250, and 350°C. This temperature range should bracket the anticipated long-term storage conditions. If the release rate is adversely related to the beta dose or the rubidium produced by decay, it should be detectable in the period of one half-life. Thus a period of 10 yr was selected for monitoring the krypton release.

As part of the current evaluation program a plan has been made for characterizing, if the measured release rates remain acceptable, and ultimately disposing of the remaining Cu-Y- ^{85}Kr waste form. It is not very practical or cost-effective to monitor ^{85}Kr release under the actual radiation and chemical environment anticipated for the KTSDs for the full length of the expected storage period. Therefore, to follow the 10-yr release measurements task with a program characterizing the copper-yttrium-krypton deposit will enable researchers to better estimate the overall radiation and thermal stability of the waste form for the full storage period. The characterization program includes the following:

- corrosion tests in air and ground water

- scanning electron microscopy and x-ray diffraction examination of the surface
- transmission electron microscopy of thinned sections
- metallographic examination of polished cross-sections of the Cu-Y-Kr deposit.

Three alternative schemes for disposal of the remaining ^{85}Kr are presented.

This report summarizes the storage conditions and the release measurements and visual observations made on four krypton trapping storage devices since the beginning of their storage period. We have also included a plan for destructive evaluation and eventual disposal of the radioactive material.

CONCLUSIONS AND RECOMMENDATIONS

Based on the data from both radioactive and nonradioactive test capsules, release rates measured to date are less than $0.1\% \text{ yr}^{-1}$ for storage temperatures not exceeding 350°C . The higher initial release rates observed for the multicurie KTSD without a closeout layer and the nonradioactive KTSD are probably due to the loosely bound krypton held on the surface. As long as the measured release rates remain low, monitoring should be continued for a period of about one half-life (10.7 yr).

Since it is impractical to carry out the release tests for the full storage life (50-100 yr) it is recommended that the remaining Cu-Y- ^{85}Kr deposits be characterized after completion of the 10-yr test program in order to better estimate their stability for a period of 50-100 yr.

Three options for the disposal of the residual ^{85}Kr were identified which meet current federal and state regulations. The preferred choice would be best made at the time of disposal as these regulations are subject to change.

METHODS

Three multicurie and one nonradioactive copper-yttrium-krypton deposits that are contained in krypton trapping storage devices (KTSDs) are being tested to determine the stability of the metal matrix during storage at elevated temperatures over a period of up to 10 yr.

ELEVATED-TEMPERATURE STORAGE

Temperatures of 150, 250, and 350°C were selected to bracket anticipated storage conditions. The four units are currently being stored at their respective temperatures. The krypton released from each sputtered deposit was measured at ~3-month intervals during the initial heat-up periods and at ~6-month intervals thereafter.

Figure 1 is a diagram of the storage cask configuration used for the multicurie capsules. The cylindrical cask was fabricated from stainless steel sheet in a double-walled configuration. Lead shot was used to fill the 1.50-in.-thick wall cavity and, combined with a solid lead lid (1.5 in. thick), provides the necessary radiation shielding. Radiation levels were reduced from about 50 R/hr at contact with the KTSD to less than 25 mR/hr at the cask surface. The nonradioactive KTSD capsule is contained in a similar configuration without the lead shielding.

The KTSD is positioned within a clam-shell heating element that is centered near the bottom of the cylindrical cask. Inlet and exhaust lines with valves outside the cask are provided for gas sampling. Prior to storage, the KTSDs were flushed with pure argon until no appreciable activity was detected by an inline Triton^R monitor in series with the effluent stream. The unit containing argon gas at 1 pressure is sealed off by closing the valves and the temperature raised to the test value. The internal pressure of the unit is measured with a compound pressure gauge attached to the exhaust line. This gauge is monitored weekly and provides early warning of

^RTriton is a registered tradename of Johnston Laboratories, Inc., Cockeysville, Maryland.

FIGURE 1. Krypton Trapping Storage Device, Storage Cask and Heater

unexpectedly large krypton release from the metal matrix or of a leak in the cask. Thus a significant increase or decrease in pressure would indicate a problem with the unit, and the experiment could be terminated. For the nonradioactive KTSD, elevated-temperature storage is conducted under vacuum with the KTSD evacuated to $<10^{-4}$ Pascals before adding heat. Pressure increases during storage are measured with a sensitive capacitance manometer.

Temperature of the storage units is measured with chromel-alumel thermocouples and controlled with variable transformers. The temperature-power characteristics of each storage cask were experimentally determined by spot-welding thermocouples directly to the side of a nonradioactive KTSD and measuring the wall and furnace temperatures as a function of power level. The data from these experiments were used to establish the proper furnace temperature and power level to obtain the desired KTSD wall temperature for the long-term storage tests.

PROCEDURE FOR KRYPTON-RELEASE MEASUREMENTS

For the nonradioactive deposit, the krypton release was estimated from the pressure increase measured after the unit had been cooled to ambient temperature. The KTSD was connected to a residual gas analyzer, and the mass spectrum was obtained to qualitatively estimate the krypton content. The unit was re-evacuated before it was returned to its test temperature.

Figure 2 is a schematic diagram of the gas-measuring system for the multicurie KTSDs. The radioactive deposit is contained within the KTSD, which has inlet and exhaust lines with valves for connecting to the gas-measuring system. The system consists of a stainless steel expansion chamber of known volume, a capacitance manometer to measure the system pressure, two small ($\sim 20 \text{ cm}^3$) sample containers for gas analysis, a dry nitrogen or argon gas source for purging the system, and a Triton gas monitor calibrated for ^{85}Kr .

The ^{85}Kr release measurements were based on the technique commonly used to measure the release of fission gas from the oxide fuels in nuclear fuel rods. Gas from the KTSD was expanded into the evacuated system, and the resulting pressure was measured with the manometer. Volume of the expansion

FIGURE 2. Schematic Diagram of Piping for Measuring ^{85}Kr Release

system was designed to provide subatmospheric pressure in the system even for the unlikely release of large quantities of krypton during storage (up to 20% for the 150-Ci deposits). The KTSDs are typically stored under 1 atm argon gas (at ambient temperature), and for the expected low release of krypton gas the system pressure after expansion would be less than 0.5 atm. This negative pressure minimizes the potential for accidental release of ^{85}Kr from the glass-sampling containers during transport and analysis.

The ^{85}Kr concentration of the gas was measured by multidimensional gamma-ray spectrometry of two gas samples. Sample containers were fabricated from 16-mm-dia Pyrex^R tubing, ~10 cm long, sealed at one end and fitted with a vacuum stopcock and a ground glass joint at the other end. The volume of each sample container was determined by the difference in weight between the container when it was evacuated and when it was filled with water. The measured volumes of five containers ranged from 18.8 to 19.5 cm³.

Gas samples, removed from the system after expansion, were counted by multidimensional gamma-ray spectrometry. The spectrometer system was calibrated using a gas mixture of known ^{85}Kr content. Identical geometries for the sample and standard gas containers were used to minimize the effects of geometry on counting efficiency. Agreement between duplicate samples has been within 10%, which is consistent with the estimated uncertainty of the counting statistics, $\pm 5\%$.

The total ^{85}Kr gas released from the metal matrix was calculated from the sample gas analysis and the combined volumes of the gas expansion system and KTSD by the relation

$$\text{Total } ^{85}\text{Kr Release} = C(V_1 + V_2)$$

where C = ^{85}Kr concentration, $\mu\text{Ci}/\text{cm}^3$

V_1 = Expansion system volume, cm³

V_2 = KTSD volume, cm³.

Volumes of the expansion system and KTSD were determined from the well-established pressure/volume relationships of ideal gases. Gas from a

^RPyrex is a registered tradename of Corning Glass, Corning, New York.

calibrated volume at atmospheric pressure was expanded into the evacuated expansion system, and the resulting pressure was measured with the manometer to determine the volume of the expansion system. This expansion was made after the system had been connected to the KTSD and included the volume of the piping up to the KTSD valve. Internal volume of the KTSD was determined, after the gas-release measurements were completed, by expanding nitrogen gas at atmospheric pressure from the KTSD into the evacuated system. Another method, using a Triton gas monitor calibrated for ^{85}Kr to measure gas release, was initially set up as a back-up method. Early experiments, however, showed that the Triton monitor was too sensitive for even the lowest ^{85}Kr releases, and this method was abandoned. Thereafter, the Triton monitor was used to ensure that >>99% of the released ^{85}Kr was purged from each KTSD before returning the vessel to elevated-temperature storage.

RESULTS AND DISCUSSION

Seven gas-release tests were performed on the nonradioactive copper-yttrium-krypton specimen at storage temperatures ranging from 180 to 450°C.

NONRADIOACTIVE KRYPTON-RELEASE MEASUREMENTS

The initial tests were conducted to establish an upper temperature limit for safe storage of the radioactive deposits. Testing at 350°C, the maximum temperature at which one of the three radioactive specimens is being held, is continuing. This test could provide an early warning of a significant increase in gas release as a function of time at 350°C. This warning would prompt additional monitoring of the radioactive specimens to ensure a safe termination to the experiment.

Table 1 summarizes the gas collection results of these experiments. The quantity of gas collected in each test was measured continuously with a sensitive mechanical manometer accurate to ~1 Pa. The value for the quantity collected was computed from the known volume of the apparatus (746.7 cc) and the final pressure measured in the KTSD after each cool-down phase. The total release fraction was computed by dividing the gas collected by 2700 cc-atm (the quantity of krypton trapped in the copper-yttrium deposit). The total release and the maximum release rate given in Table 1 were based on the assumption that all the gas collected is krypton. Results from the residual gas analyzer instrument showed the primary gases to be nitrogen and krypton, with lesser amounts of hydrogen and water. Since the residual gas analyzer employed was meant to be used as a qualitative indication of the gases present, the [Kr] in Table 1 is reported to the nearest 50%. The presence of nitrogen probably represents a small inleakage to the apparatus. Note that very little nitrogen was present in test 6, the briefest test. A gross leak developed in test 7 during the cool-down phase. The quantity of gas reported was estimated by reducing the final pressure at the storage temperature using Charles' Law and assuming a uniform KTSD temperature.

TABLE 1. Gas-Collection Results with Nonradioactive Surrogate

Test No.	Storage Temperature, °C	Storage Period, Days	Quantity of Gas Collected, cc-atm	Total ^(a) Release Fraction, $\times 10^5$	Maximum ^(a) Release Rate, % yr ⁻¹	Qualitative Estimate [Kr], %	Release Rate Corrected for [Kr], % yr ⁻¹
1	180	34	0.10	3.7	0.040	~ 50	0.02
2	243	26	0.15	5.5	0.078	~ 50	0.04
3	300	25	0.06	2.2	0.032	> 50	0.02
4	350	29	0.06	2.2	0.028	~ 50	0.01
5	400	39	0.24	8.9	0.083	~ 50	0.04
6	450	6	3.2	118	7.2	~ 100	7.2
7	350	504	(2.4) ^(b)	89	0.064	Gross leak during cool-down phase	----
8	350	100+	Test still in progress				

(a) Assumes all gas collected to be krypton.

(b) Final pressure after KTSD cool-down calculated from Charles' Law assuming a uniform KTSD temperature.

Even when the "worst case" results are considered, the release rates given in Table 1 are well within acceptable limits for temperatures up to 350°C and may be satisfactory even up to 400°C. At 450°C, however, the release rate corresponds to 7% per year, which is considerably above an acceptable value for long-term storage.

Visual examination after each elevated-temperature storage period showed the deposit to be intact, with only a few flakes in the bottom of the KTSD. A slight tarnish indicates that some oxidation of the surface of the copper-yttrium-krypton deposit has occurred. The nonradioactive deposit is presently at 350°C (test 8). After 100 days the pressure of the KTSD reduced to room temperature is ~40 Pa. For comparison, the KTSD pressure during test 7, also held at 350°C for 100 days, was ~130 Pa. The continued reduction in release rate provides additional evidence that the bulk of the gas released is from near the surface.

RADIOACTIVE KRYPTON-RELEASE MEASUREMENTS

Gas-release measurements from the three multicurie Cu-Y-⁸⁵Kr deposits have shown minimal ⁸⁵Kr release after storage at 150°, 250° and 350°C for periods up to 2 yr. All three KTSDs have been sampled on four occasions, with intervals of 3 to 8.5 months between samplings. Table 2 summarizes the data obtained and the calculated release rates for each storage interval. The total release fraction was obtained by dividing the quantity measured by the total amount implanted and corrected for radioactive decay.

These data show the marked effect of a closeout layer of Cu-Y-⁸⁴Kr. The deposit in KTSD #3 does not contain the nonradioactive surface layer because of experimental difficulties during deposition. The higher release rate is thought to be related to the release of weakly bound ⁸⁵Kr from near the surface of the deposit. It is encouraging to note the similar release results for the KTSD #3 and the nonradioactive release results for corresponding temperature ranges. The steady decrease in release rates for KTSD #3 and the nonradioactive KTSD with time also suggests that the bulk of the early gas released is due to surface release and not to long-range diffusion.

TABLE 2. Long-Term ^{85}Kr Release Data Obtained with Cu_{0.67}Y_{0.25}Kr_{0.08} Stored 150⁰-350⁰C

KTSD No.	Storage Temperature °C	Implanted 9-22-85, Ci	Total ^{85}Kr Storage Period, Yr	Date Analyzed	^{85}Kr Released, Ci X 10 ⁴	Release Rate, (%)yr ⁻¹	Closeout Layer of Nonradioactive Krypton
1	150	146.5	0.27	1-22-86	2.38	0.00061	Yes
1	150	146.5	0.26	4-30-86	1.14	0.00031	Yes
1	150	146.5	0.70	1-14-87	2.19	0.00022	Yes
14	150	146.5	0.39	6-10-87	1.74	0.00033	Yes
2	150	146.5	0.27	1-22-86	2.47	0.00067	Yes
2	250	146.5	0.26	4-30-86	3.52	0.00094	Yes
2	350	146.5	0.70	1-14-87	11.21	0.0011	Yes
2	350	146.5	0.39	6-10-87	3.28	0.00062	Yes
3	150	80.7	0.27	1-22-86	15.96	0.0072	No
3	150	80.7	0.26	4-30-86	58.3	0.028	No
3	250	80.7	0.70	1-14-87	98.7	0.018	No
3	250	80.7	0.39	6-10-87	13.03	0.0044	No

Only the KTSD #1 has been at a constant temperature long enough to look for long-term effects. Within the accuracy of the release data, no significant change in release rate was observed during the past 1.4 yr at 150°C.

POST-ELEVATED-TEMPERATURE STORAGE CHARACTERIZATION

It is impractical and not cost-effective to carry out thermal release measurements under the actual radiation environment expected for the KTSDs for the full length of the anticipated storage period. However, following up the 10-yr release measurements task with a program aimed at characterizing the krypton-copper-yttrium deposit would make it possible to better estimate the radiation, environmental and thermal stability of the waste form for the full storage period.

To accomplish this goal it is important to know the following:

1. Was there significant oxidation of the copper-yttrium-krypton deposit during the storage tests which could have affected the gas release results?
2. Is the material still amorphous or has it partially recrystallized?
3. Is the krypton held in atomic sites or has it migrated and formed bubbles?
4. What effect, if any, has the ingrown rubidium on the corrosion resistance?

The answers to these questions can be obtained only by post-storage characterization. All of the characterization work can be carried out in the 325 hot cell facility or the 327 radiometallurgy facility, both in the 300 area and managed by PNL. Extensive surface oxidation resulting from the elevated-temperature storage is not expected, but small leaks or impurities in the fill gas could make this possible. The amount of oxidation will be determined by visual observation and with the scanning electron microscope. Metallographic examination of polished cross-sections of samples removed from the KTSDs would be used to estimate how much oxidation and recrystallization has occurred in elevated-temperature storage. X-ray diffraction will be used to determine the crystallinity of the copper-yttrium-krypton deposit. Electron microscopy of thin cross-sections of the copper-yttrium-krypton deposit will be used to determine whether krypton has migrated and formed large bubbles in the alloys. To perform destructive examination on the nonradioactive sample well ahead of the work on the radioactive samples may be considered advisable as it would provide advance information on what is

occurring. Moreover, it will be more cost-effective to develop the technique for making thin cross-sections for transmission electron microscopy on the nonradioactive material before attempting to examine radioactive samples.

DISPOSAL OF RADIOACTIVE KRYPTON

State and federal regulations applicable to the disposal of radioactive and chemical substances are changing rapidly. Thus the rules that will apply at the actual time of disposal are an unknown. For this reason several plans are being considered for the eventual disposal of the ^{85}Kr remaining after completing the long-term testing and characterization tasks.

The preferred method of disposal would combine accelerated thermal and/or corrosion tests with krypton being released to the atmosphere at acceptable rates. These tests are described in the previous section.

Probably the simplest and cheapest method would be to place the ^{85}Kr -containing KTSDs and all sections remaining from the characterization effort in a polyvinyl chloride pipe encased in concrete inside an approved galvanized steel drum for land burial. Presently it takes 1 month for a burial permit. The present burial cost for a standard drum is about \$75. This cost may double every 2 yr.

If there is a critical need and the extra expense is justified, the ^{85}Kr could be released by chemically dissolving or heating the Cu-Y- ^{85}Kr deposit to 600-800°C. After preliminary purification, the ^{85}Kr would be returned to Oak Ridge National Laboratory for additional purification.

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