MEASUREMENT OF REACTOR TUBE CLADDING THICKNESS BY X-RAY FLUORESCENCE SPECTROMETRY

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ABSTRACT

An x-ray fluorescence spectrometer was designed and fabricated which nondestructively determines the thickness of aluminum cladding at small suspected thin spots in the inner or outer surface of actinide reactor tubes. The analysis method is based on the difference in absorption of actinide L_{α} and L_{β} fluorescent x-rays in passing through the cladding. Calibration plots of the logarithm of the L_{β}/L_{α} x-ray intensity ratio versus cladding thickness are linear to at least 40 mils for U-Al, U_3O_8-Al, and PuO_2-Al substrates. Accuracy and precision of the experimentally determined cladding thickness are evaluated for both uranium and plutonium substrates. Experimental thickness data are reported for 618 quality assurance analyses on six Mark 41 PuO_2-Al target tubes.

An x-ray fluorescence cladding thickness monitor operated with a computer-controlled fluoroscope holds considerable promise for quality assurance because 1) a permanent record of cladding thickness for each reactor tube would be provided and 2) the cladding integrity of each tube would be assured before irradiation in the reactor.

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MEASUREMENT OF REACTOR TUBE CLADDING THICKNESS BY X-RAY FLUORESCENCE SPECTROMETRY

INTRODUCTION

Tubular elements containing cores of fuel or target material dispersed in an aluminum matrix are fabricated at the Savannah River Plant (SRP) for use in the production reactors. The tubes are formed by coextrusion of fuel or target cores encased in aluminum. The fuel or target material is dispersed in the aluminum core matrix as discrete particles of actinide oxides in powder metallurgy cores or as intermetallic actinide metal-aluminum compounds in alloy cores. During the extrusion process, the presence of large core particles, or agglomerates, can penetrate the softer aluminum and reduce the cladding thickness over these agglomerates below nominal specifications. Inclusion of foreign material from process components can also result in thin cladding. The most common example of the latter cause is reaction of the core melt with the graphite crucibles to form actinide-aluminum carbides. A transverse section of an extruded tube is shown in Figure 1, and photomicrographs showing cladding thinning caused by core agglomerates are shown in Figure 2. Cladding thinning can also occur at the interfaces between the tube core and the end plugs.

Thinning of the cladding below the specified thickness can create severe operating problems. If the cladding is not sufficiently thick, it can be penetrated by mechanical abrasion, erosion, or corrosion during subsequent fabrication steps or irradiation. Fission products can then be released to the moderator and the environment during irradiation of the tube.

In the past, nondestructive inspection of fuel and target tubes for cladding thickness has been performed using a fluoroscope for visual comparison of relative x-ray densities of suspect core regions. Standards of acceptability were established by destructive examination of cladding thickness over similar high-density core regions in other tubes. This technique has been successfully applied at Savannah River Plant, but it cannot be applied with assurance when the x-ray density of a core defect is too high to permit grading of relative severity. The technique is also not capable of detecting thinning of the cladding at core - end plug interfaces where high core densities do not necessarily cause the thinning. Defect areas have been detected in recent production target tubes which are too dense for proper fluoroscope inspection.

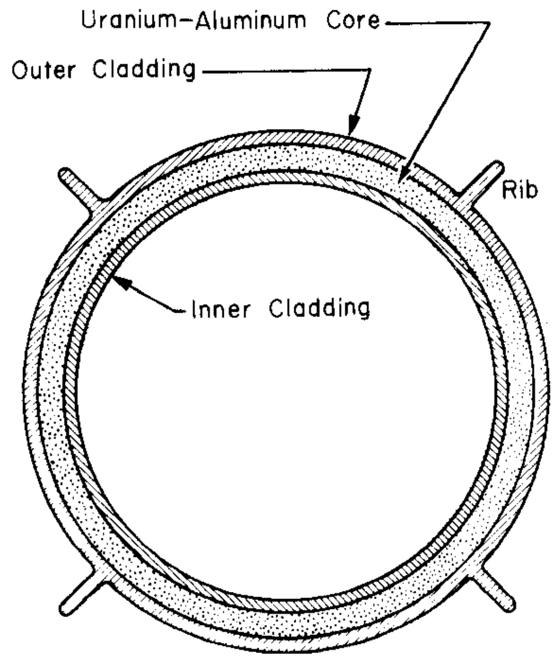

FIGURE 1. Transverse Section of Ribbed Reactor Tube

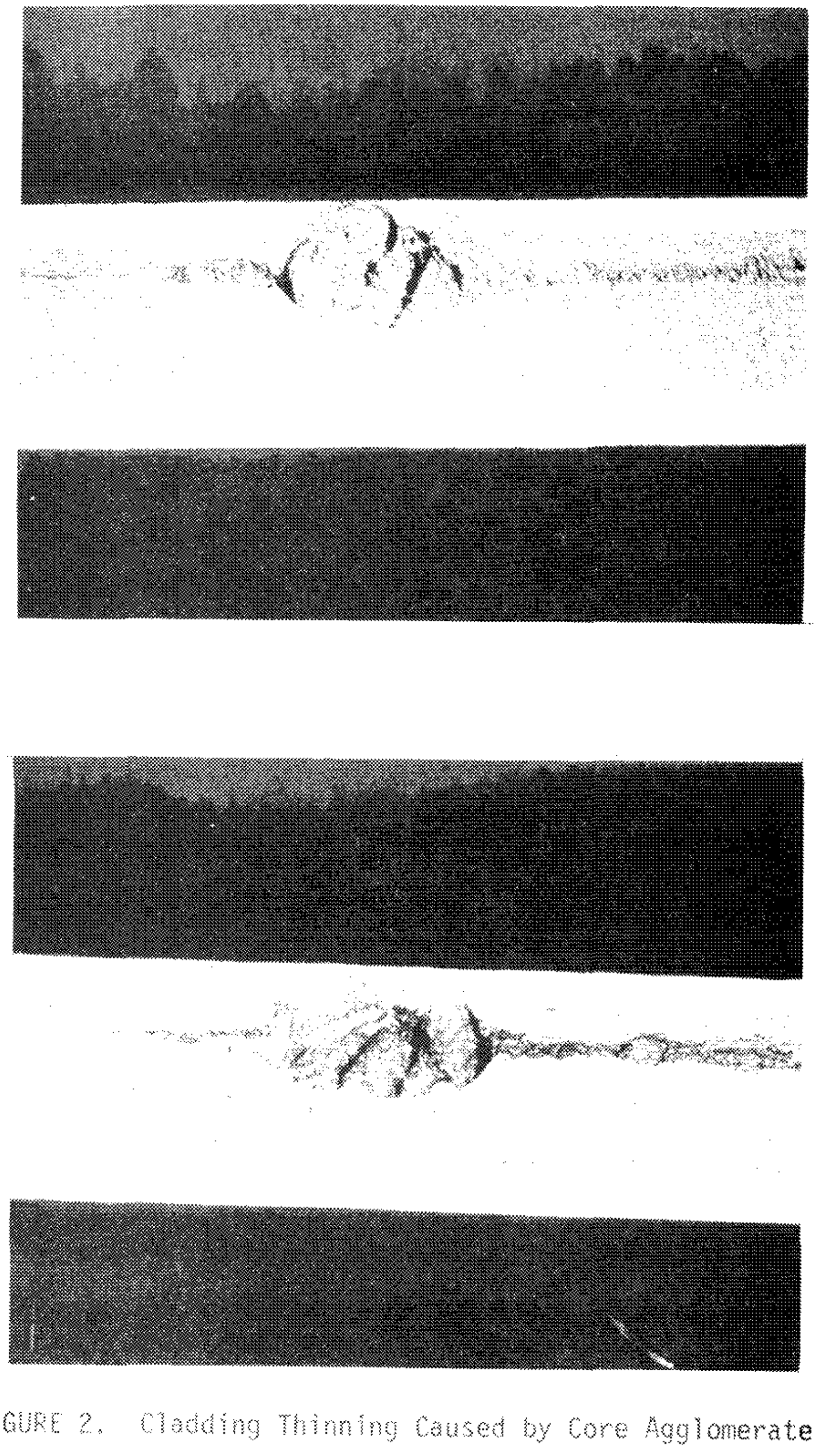

FIGURE 2. Cladding Thinning Caused by Core Agglomerates

The limitations of fluoroscopic measurement of cladding thickness led to the development of a nondestructive method. With this method, the absolute thickness of the cladding could be determined 1) over areas designated suspect by fluoroscopic examination and 2) at core - end plug interfaces. Of the several existing methods considered for adaptation to measuring tubular cladding thicknesses, only the x-ray fluorescence (XRF) method appeared feasible for measurements on SRP production tubes.

Eddy current, pulse echo, and β-autoradiographic techniques have been used at other sites to measure cladding thickness on flat plates;[1] however, our feasibility investigation indicated that none of these techniques was adequate for the tubular SRP elements. The XRF method has also been previously used to measure cladding thicknesses on flat fuel plates.[1,2] The capability of this method to measure cladding thickness on SRP tubular elements was proven by measurements of sections cut from tubular elements. These measurements were made by Savannah River Laboratory (SRL) personnel at the laboratories of Aerojet Nuclear Corp., Idaho Falls, Idaho, using an XRF instrument designed by them.

Based on the foregoing study, a manually operated cladding thickness monitor was fabricated for laboratory evaluation and use. This instrument was used to demonstrate the feasibility and advantages of x-ray fluorescence cladding thickness measurements for quality assurance of reactor tubes. After calibration it was used to determine the thickness at suspected thin spots in the cladding of six PuO_2-aluminum core target tubes. This work should provide an information and experience basis for fabrication of a more sophisticated production instrument for routine quality evaluation.

EXPERIMENTAL SECTION

Instrumentation

The cladding thickness monitor is an x-ray fluorescence spectrometer of special design*; it consists of an x-ray detector and an isotope excitation source compactly mounted at the end of a long probe (Figure 3). The cladding thickness monitor includes a mechanical system which rotates and translates the reactor tube to predetermined coordinates to position the analysis spot precisely under the detector. The probe is stationary during

* Cryostat, excitation source, and Si(Li) detector were designed and fabricated for SRL by Kevex Corporation, Burlingame, CA.

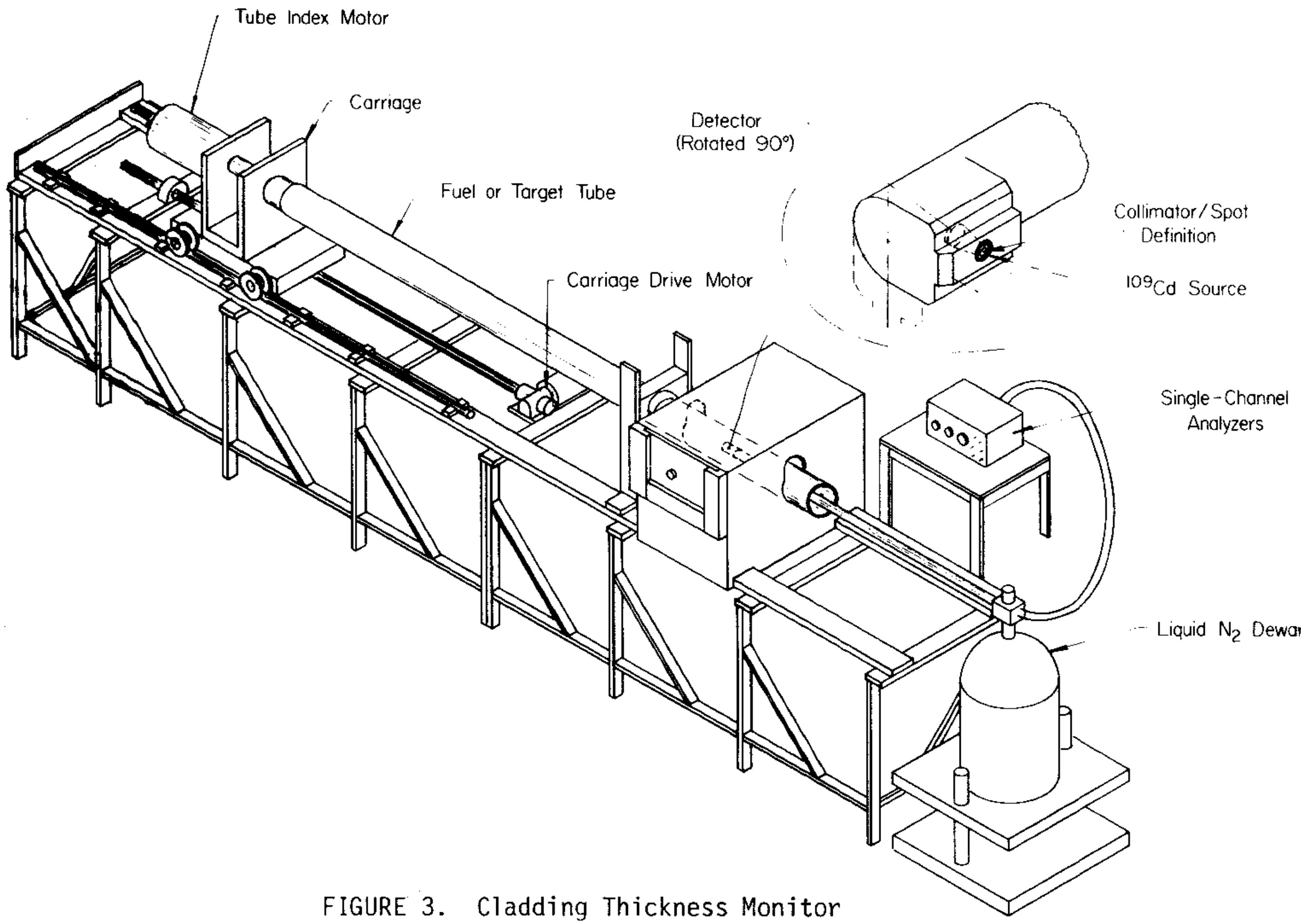

FIGURE 3. Cladding Thickness Monitor

analysis but can be moved vertically to permit analysis of internal or external cladding. The probe is positioned inside the reactor tube to measure internal cladding thickness, or above the tube to measure external cladding thickness.

An 80-mm^2 lithium-drifted silicon detector, a conical x-ray beam collimator, and a 100-mCi ^{109}Cd excitation source are concentrically positioned around a vertical axis through the end of a 7.5-ft-long horizontal probe. A remotely controlled tantalum shutter permits use of the detector with or without the ^{109}Cd excitation. The analysis area is 0.06 in. in diameter when the probe is in contact with the cladding, and somewhat larger when the probe is positioned above the cladding as during analysis of a ribbed tube. With a ^{109}Cd source, the actinide fluorescence is excited by Ag K_α x-rays (22.1 keV) which are emitted following radioactive decay of ^{109}Cd to ^{109}Ag by orbital electron capture. The electronic instrumentation used to process the detector signals for U_3O_8-Al and U-Al substrates was different from that used for PuO_2-Al substrates. The instrumentation for uranium substrates consisted of four single channel analyzers; that for plutonium substrates consisted of a multichannel analyzer which had a cathode ray tube display of the spectrum as its only output.

Calibration Standards

Calibration standards for cladding thickness analysis of U_3O_8-Al substrate reactor tubes consisted of U_3O_8-Al disks of various compositions, each approximately 3 in. in diameter and 1/8 in. thick. The disks were prepared by thoroughly blending and compacting known weights of aluminum and U_3O_8 powders. Calibration standards for uranium-aluminum alloy tubes were 3.5-in. diameter cylinders cast from U-Al melts of several known compositions.

A single standard was used in the calibration for cladding thickness analysis of PuO_2-Al substrate tubes. This standard was fabricated by mounting the rectangular surface of a PuO_2 quarter cylinder against the accurately machined 13.4-mil-thick aluminum window in the end of an aluminum can. The approximate dimensions of the rectangular surface were 0.18 in. and 0.27 in. The aluminum can was welded shut to ensure containment of the plutonium. The isotopic composition of the plutonium was determined by gamma spectrometry to be:

^{238}Pu	0.04 at. %
^{239}Pu	>98.
^{240}Pu	0.5
^{241}Pu	0.8
^{242}Pu	below detection limit

Calibration Procedure

The cladding thickness monitor was calibrated to determine the thickness of aluminum cladding on U_3O_8-Al and U-Al substrate tubes using U_3O_8-Al disks and U-Al castings respectively as standards to simulate the reactor tube substrate. Aluminum shim-stock was used to simulate the aluminum cladding. Each disk or casting standard was successively positioned below the detector with a known thickness of shim stock resting on the standard. The standard and shim were then raised to contact the detector or to give a predetermined separation between the shim and detector. The detector shutter was opened to expose the standard and shim to ^{109}Cd radiation. X-ray intensities of the uranium L_α peak, L_α background, $L_{\beta 1}$ and $L_{\beta 2}$ peaks, and L_β background were determined with the four single-channel analyzers for 300-second counts of each standard-shim combination. The peak backgrounds were determined immediately adjacent to the peaks on the high energy side. The net x-ray intensity ratio L_β/L_α was calculated from the accumulated counts of the four analyzers.

The cladding thickness monitor was calibrated to determine the thickness of aluminum cladding on PuO_2-Al substrate tubes with the plutonium standard positioned directly below the detector. Aluminum shims of known thickness were successively placed on the plutonium standard window between the standard and detector. Analytical data were collected during ^{109}Cd irradiation with the detector contacting the shim. Each accumulated spectrum was displayed on the cathode ray tube. A measure of the net intensities of the plutonium L_α and $L_{\beta 1}$ peaks was obtained for each analysis by photographing the spectral display, defining the peak backgrounds on the photograph by drawing a smoothly curving extension of the inelastic scatter peak under the L_α and $L_{\beta 1}$ peaks, and measuring the net heights of the peaks with a small finely-calibrated ruler. The intensity ratio $L_{\beta 1}/L_\alpha$ was calculated directly from the net peak heights.

RESULTS AND DISCUSSION

Technical Basis

The technical basis for the cladding thickness monitor arises from the quantitative nature of x-ray absorbance. Monoenergetic x-rays are absorbed by any homogeneous material according to the Lambert Law expressed in Equation 1.

$$I = I_o e^{-\mu T} \tag{1}$$

I_o and I are respectively the intensities of the incident and emergent x-ray beams, T is absorber thickness, and μ is the linear absorption coefficient of the absorbing material. Since the value of μ depends upon the x-ray energy, the intensity ratio for uranium (or plutonium) L_β/L_α x-rays emerging from the reactor tube cladding provides a measure of the cladding thickness.

The equation relating the L_β/L_α intensity ratio to absorber thickness, calculated directly from the Lambert Law is given in Equation 2.

$$\ln (L_\beta/L_\alpha) = \ln(L_{\beta o}/L_{\alpha o}) + T (\mu_\alpha - \mu_\beta) \tag{2}$$

$L_{\beta o}/L_{\alpha o}$ is the intensity ratio of x-rays incident upon the absorber, and μ_α and μ_β are the linear absorption coefficients for the L_α and L_β x-rays, respectively. Equation 2 provides the basis for the cladding thickness monitor. Only those uranium or plutonium L_α and L_β x-rays originating at the cladding-core interface pass out through the cladding. Nearly all x-rays originating from deeper in the core are absorbed by the core before they can escape because the linear absorption coefficients for these x-rays are more than 50 times greater for absorption by uranium or plutonium than by aluminum.

The linear relation between ln (L_β/L_α) and absorber thickness T predicted by Equation 2 was confirmed by calibration data for U_3O_8-Al and U-Al substrates. These data showed that the slopes $(\mu_\alpha - \mu_\beta)$, for each substrate was constant and independent of uranium content. The intercept, ln $(L_{\beta o}/L_{\alpha o})$, of ln (L_β/L_α) vs T plots were shown by the U_3O_8-Al and U-Al calibration data to vary slightly with composition. This variation is satisfactorily described in the investigated composition range by Equation 3 where B and C are constants determined empirically for each substrate and [% U] is the substrate bulk uranium content.

$$\ln (L_{\beta o}/L_{\alpha o}) = B [\% U] + C \tag{3}$$

Combining Equations 2 and 3, solving for absorber thickness T, and defining $A = (\mu_\alpha - \mu_\beta)$ gives Equation 4.

$$T = \frac{\ln (L_\beta/L_\alpha) - B\,[\%\,U] - C}{A} \tag{4}$$

Equation 4 permits direct calculation of cladding thickness from the experimentally determined (L_β/L_α) x-ray intensity ratio, the substrate uranium content known from tube fabrication specifications, and the empirical constants A, B, and C.

U_3O_8-Al Substrate

An x-ray fluorescence spectrum of uranium excited through and attenuated by 42 mils of aluminum is shown in Figure 4. This spectrum is typical of those obtained with the cladding thickness monitor for relatively thick aluminum cladding. For thinner cladding the L_α peak is significantly more intense and the L_β peaks are moderately more intense. In addition to the uranium L_α and L_β peaks, two large scatter peaks are present in this spectrum. These peaks result from elastic (22.15 keV) and inelastic (20.64 keV) scatter of the silver K_α x-rays used to excite uranium fluorescence during thickness analysis.

The experimental calibration data for analysis of aluminum cladding thickness of U_3O_8-Al substrate tubes are presented in Tables 1 through 8. Calibration plots of $\ln (L_\beta/L_\alpha)$ vs aluminum thickness (T) are linear for thicknesses up to 40 mils for each U_3O_8-Al composition investigated. The plots for 17.9 and 44.9 wt % uranium in U_3O_8-Al compact disks are shown in Figure 5. Calibration plots for U_3O_8-Al disks having intermediate uranium contents lay between these extremes.

The slopes of the U_3O_8-Al calibration plots are identical and constant within the investigated range as shown in Figure 6. The intercepts of the calibration plots, however, are clearly dependent upon uranium content as shown in Figure 7. Within the experimental uncertainty, the intercepts decrease linearly with uranium content in the composition range investigated.

The effect of detector-to-cladding distance on the experimentally determined x-ray intensity ratio (L_β/L_α) was determined for 19.7 mil aluminum on U_3O_8-Al substrate containing 34.0 wt % uranium. These data, presented in Table 9 and Figure 8, indicate that the ratio is independent of detector-to-cladding distance. The cladding thickness monitor can therefore be used to determine cladding thickness of ribbed reactor tubes which may not permit direct contact between cladding and the detector.

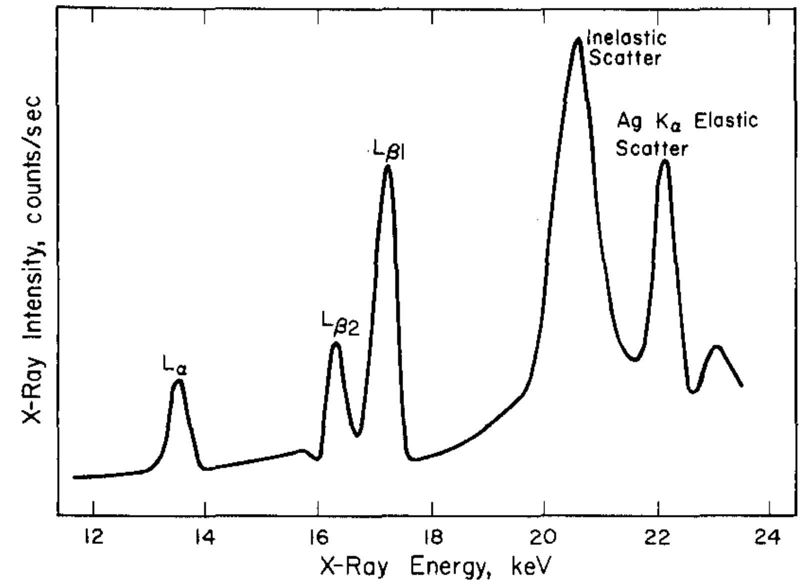

FIGURE 4. ^{109}Cd Excited Uranium Spectrum, Attenuated by 42 Mils of Aluminum

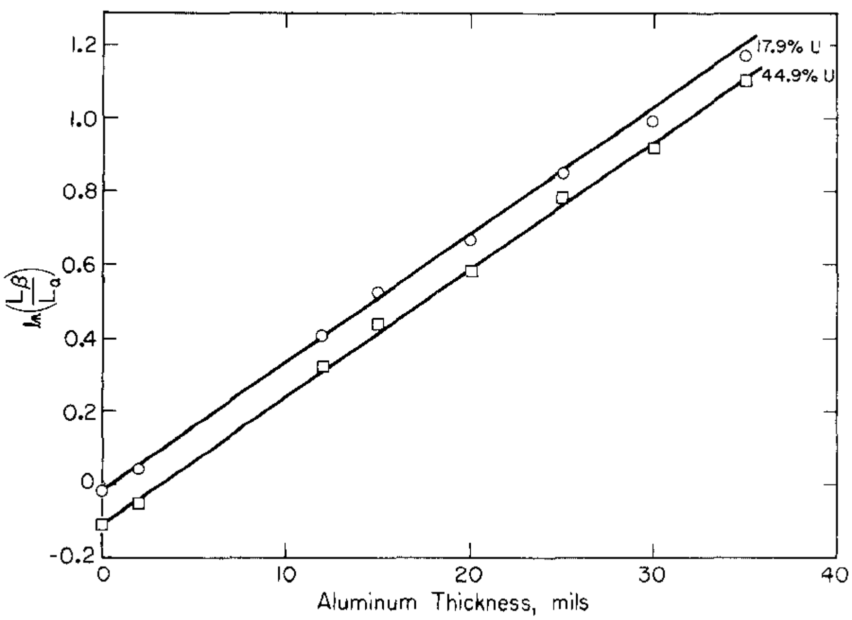

FIGURE 5. Calibration Plots of ln (L_β/L_α) vs. Aluminum Thickness for 17.9% and 44.9% U in U_3O_8-Al Substrates

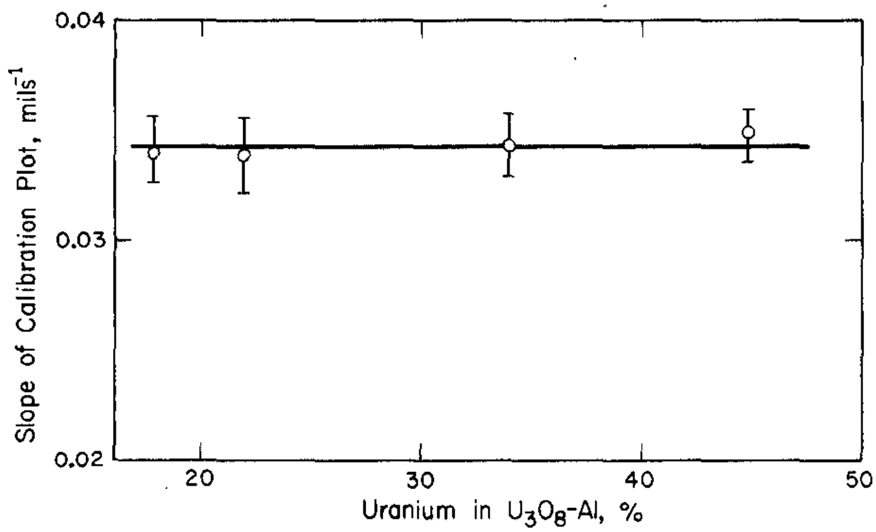

FIGURE 6. Effect of Uranium Content on Slopes of ln (L_β/L_α) vs. Aluminum Thickness Calibration Plots for U_3O_8-Al Substrates

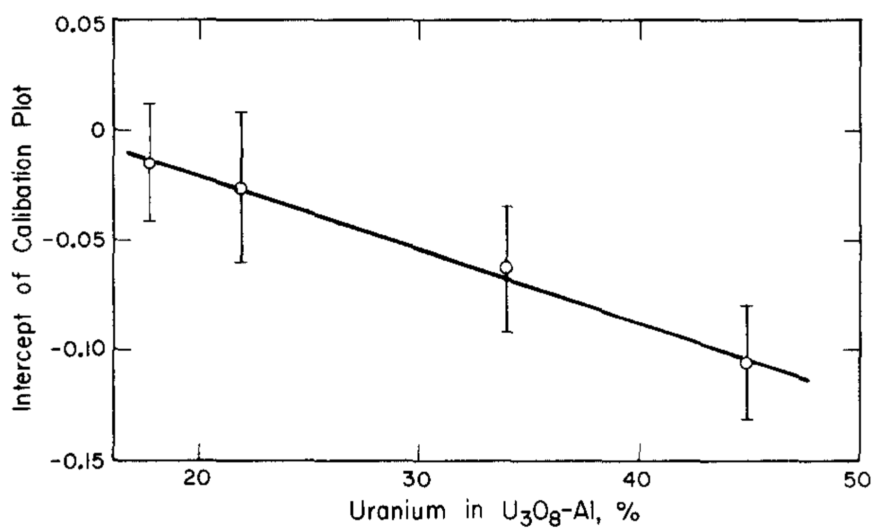

FIGURE 7. Effect of Uranium Content on Intercepts of ln (L_β/L_α) vs. Aluminum Thickness Calibration Plots for U_3O_8-Al Substrates

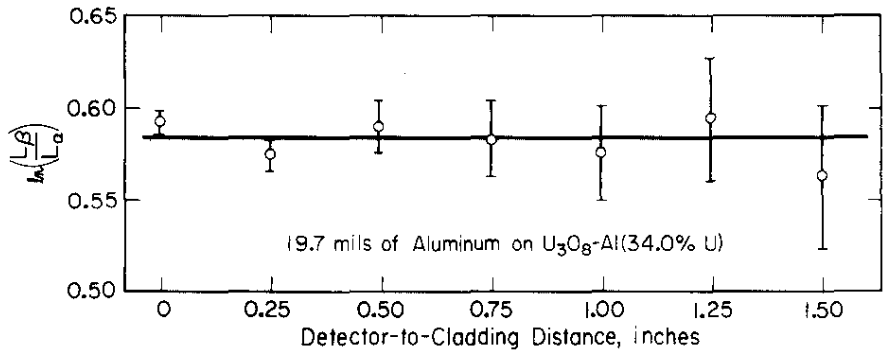

FIGURE 8. Effect of Detector-to-Cladding Distance on X-Ray Intensity Ratio L_β/L_α

TABLE 1

Calibration Data for U_3O_8-Al Substrate
Uranium Content = 17.9 wt %
Detector-to-Aluminum Distance = 0.50 in.

Total Counts, 300 Seconds					
L_α	Bkg_α	L_β	Bkg_β	T,[a] *mils*	$\ln \frac{L_\beta - Bkg_\beta}{L_\alpha - Bkg_\alpha}$
550061	21687	546675	28520	.0	-0.01952
475696	20180	504821	29844	2.0	0.04183
199689	14292	312697	35083	12.0	0.40373
148063	13484	265512	38551	15.0	0.52262
102475	12772	216346	41376	20.0	0.66811
65654	11369	168667	41720	25.0	0.84952
46034	10743	137323	42833	30.0	0.98486
29107	10242	105458	44965	35.0	1.16521
21899	10457	88421	49540	40.0	1.22321

a. T = Thickness of aluminum shim.

TABLE 2

Calibration Data for U_3O_8-Al Substrate
Uranium Content = 22.0 wt %
Detector-to-Aluminum Distance = 0.50 in.

Total Counts, 300 Seconds					
L_α	Bkg_α	L_β	Bkg_β	*T, mils*	$ln \frac{L_\beta - Bkg_\beta}{L_\alpha - Bkg_\alpha}$
560120	21980	551026	27906	.0	-0.02830
484486	20726	508689	32068	2.0	0.02735
205791	14491	318945	34525	12.0	0.39660
153496	13962	271347	38996	15.0	0.50994
-	-	-	-	20.0	-
68195	11397	172672	41727	25.0	0.83527
48328	10608	140885	42696	30.0	0.95670
30420	10383	108512	45027	35.0	1.15322
23231	11483	93276	53752	40.0	1.21322

TABLE 3

Calibration Data for U_3O_8-Al Substrate
Uranium Content = 34:0 wt %
Detector-to-Aluminum Distance = 0.50 in.

Total Counts, 300 Seconds					
L_α	Bkg_α	L_β	Bkg_β	*T, mils*	$ln \frac{L_\beta - Bkg_\beta}{L_\alpha - Bkg_\alpha}$
631038	23492	598502	28417	.0	-0.06364
552974	21727	556988	29284	2.0	-0.00669
233338	15586	349994	34271	12.0	0.37150
177290	14584	299440	38017	15.0	0.47419
124852	13396	245391	40506	20.0	0.60881
78591	11825	189490	41384	25.0	0.79673
54607	11130	153501	42098	30.0	0.94092
33500	10280	117152	44208	35.0	1.14467
25097	11462	100341	52436	40.0	1.25657

TABLE 4

Calibration Data for U_3O_8-Al Substrate
Uranium Content = 44.9 wt %
Detector-to-Aluminum Distance = 0.50 in.

Total Counts, 300 Seconds					
L_α	Bkg_α	L_β	Bkg_β	*T, mils*	$ln\ \frac{L_\beta - Bkg_\beta}{L_\alpha - Bkg_\alpha}$
714839	25122	649298	28375	.0	-0.10507
629447	23508	605973	29314	2.0	-0.04952
272435	16392	386413	33467	12.0	0.32096
203102	15312	330001	38809	15.0	0.43865
141275	14104	268743	40438	20.0	0.58514
88695	12100	207027	40149	25.0	0.77873
61478	11151	168404	42133	30.0	0.91988
38093	10521	126632	44003	35.0	1.09755
27862	11322	107047	51474	40.0	1.21191

TABLE 5

Calibration Data for U_3O_8-Al Substrate
Uranium Content = 17.9 wt %
Detector-to-Aluminum Distance = 0.0 in.

Total Counts, 300 Seconds					
L_α	Bkg_α	L_β	Bkg_β	*T, mils*	$ln\ \frac{L_\beta - Bkg_\beta}{L_\alpha - Bkg_\alpha}$
573539	54245	870146	121927	12.4	0.36522
509889	55128	866480	136438	15.6	0.47333
401218	55352	803058	155967	19.7	0.62643
286747	53512	692260	168883	25.0	0.80825
189220	51322	550971	188276	31.6	0.96704
139084	50602	462398	194248	36.0	1.10874
97918	49018	371681	206159	41.0	1.21932

TABLE 6

Calibration Data for U_3O_8-Al Substrate
Uranium Content = 22.0 wt %
Detector-to-Aluminum Distance = 0.0 in.

Total Counts, 300 Seconds					
L_α	Bkg_α	L_β	Bkg_β	T, mils	$ln \frac{L_\beta - Bkg_\beta}{L_\alpha - Bkg_\alpha}$
454041	35821	443166	62957	.0	-0.09528
585770	54670	878862	122235	12.4	0.35392
520907	55671	872155	136660	15.6	0.45799
415149	54871	814155	151557	19.7	0.60929
289977	53479	694290	168406	25.0	0.79914
188702	51563	551720	188885	31.6	0.97295
136201	50262	457285	194327	36.0	1.11835
66255	47499	280406	212759	41.0	1.28278

TABLE 7

Calibration Data for U_3O_8-Al Substrate
Uranium Content = 34.0 wt %
Detector-to-Aluminum Distance = 0.0 in.

Total Counts, 300 Seconds					
L_α	Bkg_α	L_β	Bkg_β	T, mils	$ln \frac{L_\beta - Bkg_\beta}{L_\alpha - Bkg_\alpha}$
598974	53857	870054	117206	12.4	0.32286
545880	55272	879917	129070	15.6	0.42555
438658	55304	835934	147495	19.7	0.58546
314432	53952	728250	164239	25.0	0.77254
205584	51587	579085	187290	31.6	0.93380
147245	50792	485340	191141	36.0	1.11520
72004	48072	302319	207543	41.0	1.37629

TABLE 8

Calibration Data for U_3O_8-Al Substrate
Uranium Content = 44.9 wt %
Detector-to-Aluminum Distance = 0.0 in.

Total Counts, 300 Seconds					
L_α	Bkg_α	L_β	Bkg_β	*T, mils*	$ln \frac{L_\beta - Bkg_\beta}{L_\alpha - Bkg_\alpha}$
597382	51481	831384	106512	12.4	0.28355
566121	54462	880400	124251	15.6	0.39058
467660	54876	859228	143587	19.7	0.55025
343808	54538	768516	160312	25.0	0.74314
228206	52585	620165	182663	31.6	0.91275
162813	50858	522461	189175	36.0	1.09090
118617	49199	426986	205093	41.0	1.16204

TABLE 9

Effect of Detector-to-Cladding Distance (D) on Experimentally Determined X-Ray Intensity Ratio (L_β/L_α)
Uranium Content of U_3O_8-Al Substrate = 34.0 wt %
Thickness of Al Shim = 19.7 mils

Total Counts, 300 Seconds					
L_α	Bkg_α	L_β	Bkg_β	*D, in.*	$ln \frac{L_\beta - Bkg_\beta}{L_\alpha - Bkg_\alpha}$
446917	56783	850735	146066	0.00	0.59123
252218	27990	481541	83453	0.25	0.57400
99528	10873	192258	32464	0.50	0.58913
52263	5839	100797	17776	0.75	0.58127
31571	3806	60975	11674	1.00	0.57416
19939	2633	39534	8220	1.25	0.59301
14880	2119	29063	6691	1.50	0.56141

The empirical constants A, B, and C were determined by a least squares fit of the U_3O_8-Al calibration data to Equation 4. Their respective values based on natural logarithms are +0.0342/mil, -0.002937/% U, and +0.03424. These constants are valid in Equation 4 for determination of aluminum cladding thickness up to 40 mils on U_3O_8-Al tubes having uranium contents of 17.9 to 44.9 wt %.

U-Al Substrate

The spectrum of Figure 4 is typical of those produced by ^{109}Cd excitation through aluminum cladding of U-Al and U_3O_8-Al substrates. The spectra of U-Al and U_3O_8-Al are visibly indistinguishable, with uranium L_α and L_β peaks superimposed on the low energy tail of the silver K_α inelastic scatter peak. The scatter peak is formed primarily by the aluminum cladding or shim; the aluminum of the substrate contributes very little to the scatter peak.

Experimental data are given in Tables 10 through 15 for calibration of the cladding thickness monitor to determine aluminum cladding thickness on U-Al alloy reactor tubes. Calibration plots of ln (L_β/L_α) vs aluminum shim thickness T are given in Figure 9 for U-Al alloy substrates containing 20.0 and 45.0 wt % uranium. These plots and those for intermediate uranium contents are linear as predicted by Equation 2, and all have identical slopes as shown in Figure 10.

The intercepts of the U-Al calibration plots were determined from linear least squares fits for each data set of Tables 10 through 15. The intercepts were redetermined by direct cladding thickness monitor analysis with no aluminum shim present and with the detector raised to 0.50 in. above the U-Al cylinder to reduce the count rate to an acceptable value. The intercept data are presented in Table 16 and are shown graphically in Figure 11. As with the U_3O_8-Al intercepts, these data show a decreasing intercept with increasing uranium content of the substrate. Although the experimental variation of replicate intercept determinations is greater than predicted from the statistical precision of each individual determination, the uncertainty of the intercepts is still relatively small. The intercepts of the calibration plots can be satisfactorily represented as a linear function of uranium content by Equation 3.

TABLE 10

Calibration Data for U-Al Substrate
Uranium Content = 20.0 wt %
Detector-to-Aluminum Distance = 0.0 in.

Total Counts, 300 Seconds					
L_α	Bkg_α	L_β	Bkg_β	*T, mils*	$\ln \frac{L_\beta - Bkg_\beta}{L_\alpha - Bkg_\alpha}$
550126	60454	848379	114981	12.4	0.40395
501877	62802	861571	129037	15.6	0.51183
405626	62270	817685	147192	19.7	0.66924
290679	61365	715709	163290	25.0	0.87921
212957	59626	610901	176971	30.2	1.04028
134443	57060	464810	191436	36.0	1.26207
100582	56344	389833	196447	40.6	1.47510
65105	54280	285110	206894	48.4	1.97761

TABLE 11

Calibration Data for U-Al Substrate
Uranium Content = 25.0 wt %
Detector-to-Aluminum Distance = 0.0 in.

Total Counts, 300 Seconds					
L_α	Bkg_α	L_β	Bkg_β	*T, mils*	$\ln \frac{L_\beta - Bkg_\beta}{L_\alpha - Bkg_\alpha}$
571385	60526	858225	115309	12.4	0.37448
527239	63036	876298	129556	15.6	0.47539
430178	63789	840042	147308	19.7	0.63695
308939	62616	734853	165781	25.0	0.83736
225027	60847	626940	179561	30.2	1.00244
142065	58411	483523	196148	36.0	1.23409
109231	57827	410101	202448	40.6	1.39615
72011	56034	305842	209362	48.4	1.79818

TABLE 12

Calibration Data for U-Al Substrate
Uranium Content = 30.0 wt %
Detector-to-Aluminum Distance = 0.0 in.

Total Counts, 300 Seconds					
L_α	Bkg_α	L_β	Bkg_β	*T, mils*	$\ln \frac{L_\beta - Bkg_\beta}{L_\alpha - Bkg_\alpha}$
568288	59993	838736	111160	12.4	0.35865
528888	62349	865966	123673	15.6	0.46440
434047	63453	840343	142202	19.7	0.63331
316119	61819	745506	159784	25.0	0.83433
227882	60338	634313	173874	30.2	1.01093
148099	57725	499896	187026	36.0	1.24183
108145	56362	410097	198449	40.6	1.40786
70767	54382	305961	203065	48.4	1.83735

TABLE 13

Calibration Data for U-Al Substrate
Uranium Content = 35.0 wt %
Detector-to-Aluminum Distance = 0.0 in.

Total Counts, 300 Seconds					
L_α	Bkg_α	L_β	Bkg_β	*T, mils*	$\ln \frac{L_\beta - Bkg_\beta}{L_\alpha - Bkg_\alpha}$
548388	60051	844434	114988	12.4	0.40127
504172	62118	867201	129774	15.6	0.51173
410766	63008	830178	149882	19.7	0.67102
293403	61734	721680	166488	25.0	0.87400
215810	60100	623172	179089	30.2	1.04801
139877	58365	488755	192692	36.0	1.28982
101680	57121	393138	201673	40.6	1.45789
69490	55501	304432	207815	48.4	1.93248

TABLE 14

Calibration Data for U-Al Substrate
Uranium Content = 40.0 wt %
Detector-to-Aluminum Distance = 0.0 in.

Total Counts, 300 Seconds					
L_α	Bkg_α	L_β	Bkg_β	*T, mils*	$ln \frac{L_\beta - Bkg_\beta}{L_\alpha - Bkg_\alpha}$
576262	60434	851173	112574	12.4	0.35898
542633	62472	881226	124408	15.6	0.45500
448774	63622	854351	143695	19.7	0.61255
326918	62641	761210	162297	25.0	0.81811
242684	60814	655905	175075	30.2	0.97222
157165	59032	515073	190773	36.0	1.19534
112413	56859	416260	203538	40.6	1.34263
73555	55450	314531	207804	48.4	1.77408

TABLE 15

Calibration Data for U-Al Substrate
Uranium Content = 45.0 wt %
Detector-to-Aluminum Distance = 0.0 in.

Total Counts, 300 Seconds					
L_α	Bkg_α	L_β	Bkg_β	*T, mils*	$ln \frac{L_\beta - Bkg_\beta}{L_\alpha - Bkg_\alpha}$
575506	67856	825597	119581	12.4	0.32984
542451	67745	869760	131025	15.6	0.44224
455500	64169	856722	143688	19.7	0.59997
329684	63771	754647	165000	25.0	0.79635
243743	62345	653875	178427	30.2	0.96356
157541	59587	516057	192375	36.0	1.19526
114144	57437	422528	200739	40.6	1.36382
73808	55539	313024	209690	48.4	1.73276

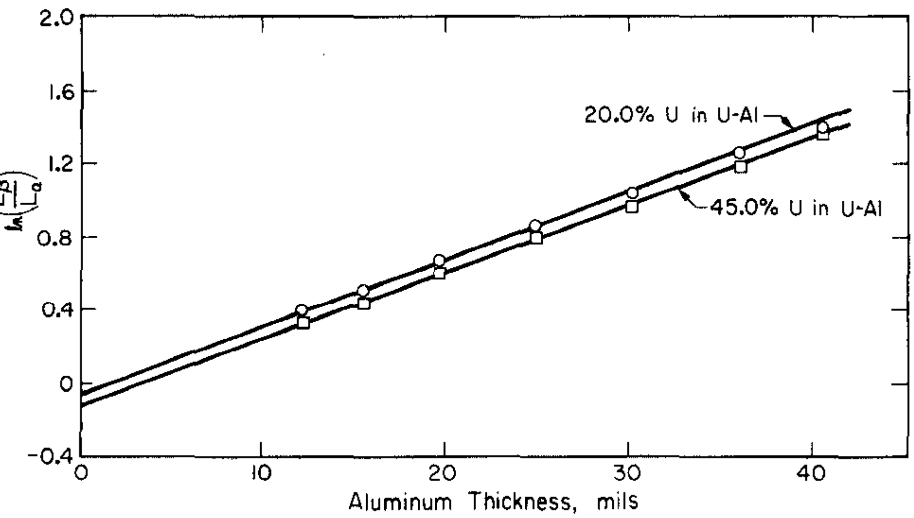

FIGURE 9. Calibration Plots of ln (L_β/L_α) vs. Aluminum Thickness for 20.0% and 45.0% U in U-Al Substrates

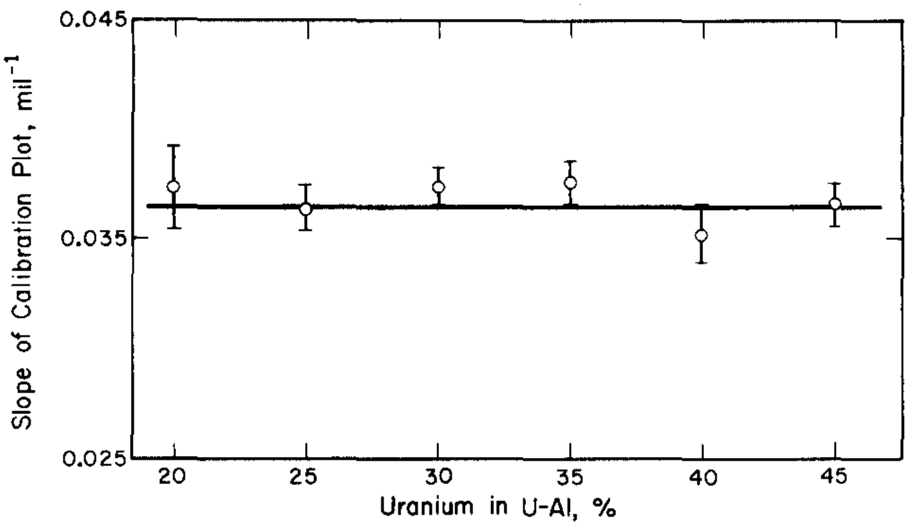

FIGURE 10. Effect of Uranium Content on Slopes of ln (L_β/L_α) vs. Thickness Calibration Plots for U-Al Substrates

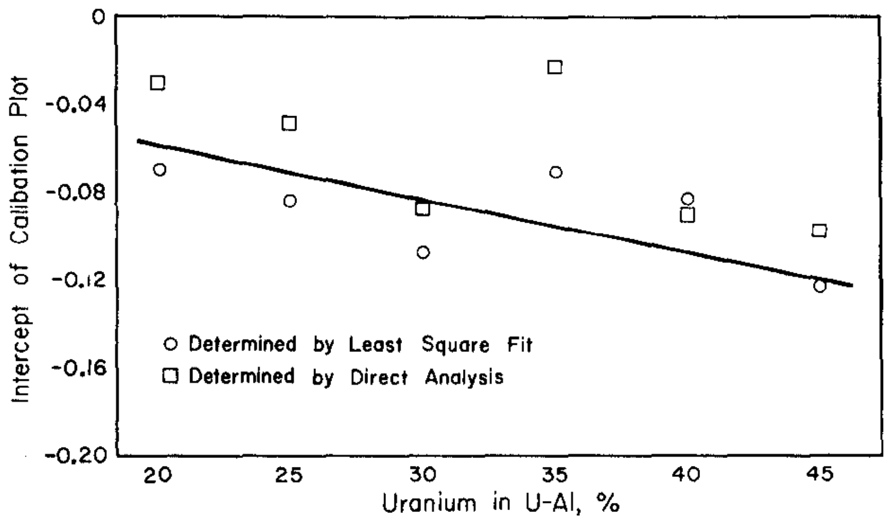

FIGURE 11. Effect of Uranium Content on Intercepts of ln (L_β/L_α) vs. Aluminum Thickness Calibration Plots for U-Al Substrates

TABLE 16

Effect of Uranium Content on Intercepts of Calibration Plots for U-Al Substrates

	Intercept, $ln\left(\frac{L_{\beta o}}{L_{\alpha o}}\right)$	
U %	*Least Sq. Fit*	*Direct Determination*
20.0	-0.0682	-0.0300
25.0	-0.0830	-0.0484
30.0	-0.1085	-0.0867
35.0	-0.0701	-0.0225
40.0	-0.0821	-0.0912
45.0	-0.1251	-0.0978

The empirical constants of Equation 4 were determined by a least squares fit of the U-Al data of Tables 10 through 15. The respective values of A, B, and C based on natural logarithms are +0.03677/mil, -0.002474/% U, and -0.008770. These values permit direct calculation of aluminum cladding thickness up to 40 mils on U-Al substrate reactor tubes having uranium contents from 20.0 to 45 wt %.

Precision and Accuracy of Measurements — U_3O_8-Al and U-Al Substrates

Experimental data demonstrate that cladding thickness can be determined by the cladding thickness monitor to ±1 mil accuracy for U_3O_8-Al substrates containing 18 to 45 wt % uranium and ±2 mils accuracy for U-Al substrates containing 20 to 25 wt % uranium using a five-minute count for substrates of known composition.

The precision and accuracy were determined for thickness measurements of aluminum shims on U_3O_8-Al and U-Al substrates using the cladding thickness monitor. The experimental data are reported in Tables 17 and 18. The L_β/L_α x-ray intensity ratios were determined experimentally for substrates having known uranium contents. The aluminum thickness, determined with a micrometer (T_M) and by the cladding thickness monitor (T_C) using Equation 4, are compared.

For U_3O_8-Al substrates containing 17.9 to 44.9 wt % uranium, T_M and T_C are in excellent agreement. The standard deviation of their differences is about 0.4 mil. These differences are in the general range predicted by Poisson statistics due to random variations in the L_α and L_β measured intensities.

For U-Al substrates containing 20.0 to 45.0 wt % uranium, the thickness measured with the cladding thickness monitor is accurate but less precise than that for the U_3O_8-Al substrate. The standard deviation of T_M-T_C is 0.72 mil, slightly greater than predicted from random statistical variation in the experimentally determined L_α and L_β x-ray intensities. The small decrease in precision of the measured aluminum thickness for U-Al substrates arises primarily from the uncertainty with which the B and C terms of Equation 4 define the calibration plot intercepts.

Uncertainty in the local uranium content of the substrate constitutes a potential source of error in the experimentally determined thickness. Although the bulk composition of the substrate may be accurately known, the composition at a suspect spot in the cladding may be only approximately known. The

TABLE 17

Precision and Accuracy of Thickness Determination for U_3O_8-Al Substrates

X-Ray Intensity Ratio		*Measured Thickness, mils*		
(L_β/L_α)	% U	*Micrometer* (T_M)	*CTM* (T_C)	$T_M - T_C$
1.0427	17.9	2.0	1.8	0.2
1.5014	17.9	12.0	12.4	-0.4
1.9505	17.9	20.0	20.1	-0.1
3.2066	17.9	35.0	34.6	0.4
1.0277	22.0	2.0	1.7	0.3
1.4868	22.0	12.0	12.5	-0.5
2.3054	22.0	25.0	25.3	-0.3
3.1684	22.0	35.0	34.6	0.4
0.9933	34.0	2.0	1.7	0.3
1.4499	34.0	12.0	12.8	-0.8
1.8382	34.0	20.0	19.7	0.3
3.1414	34.0	35.0	35.4	-0.4
0.9517	44.9	2.0	1.4	0.6
1.3785	44.9	12.0	12.2	-0.2
1.7952	44.9	20.0	20.0	0.0
2.9968	44.9	35.0	34.9	0.1
			MEAN =	-0.01
			STD DEV =	0.39

TABLE 18

Precision and Accuracy of Thickness Determination for U-Al Substrates

X-Ray Intensity Ratio		*Measured Thickness, mils*		
Ratio (L_β/L_α)	*% U*	*Micrometer* (T_M)	*CTM* (T_C)	$T_M - T_C$
1.4977	20.0	12.4	12.6	-0.2
1.9528	20.0	19.7	19.8	-0.1
2.8300	20.0	30.2	29.9	0.3
4.3715	20.0	40.6	41.7	-1.1
1.4542	25.0	12.4	12.1	0.3
1.8907	25.0	19.7	19.2	0.5
2.7249	25.0	30.2	29.2	1.0
4.0396	25.0	40.6	39.9	0.7
1.4314	30.0	12.4	12.0	0.4
1.8838	30.0	19.7	19.5	0.2
2.7482	30.0	30.2	29.8	0.4
4.0872	30.0	40.6	40.5	0.1
1.4937	35.0	12.4	13.5	-1.1
1.9562	35.0	19.7	20.8	-1.1
2.8520	35.0	30.2	31.1	-0.9
4.2969	35.0	40.6	42.2	-1.6
1.4319	40.0	12.4	12.7	-0.3
1.8451	40.0	19.7	19.6	0.1
2.6438	40.0	30.2	29.4	0.8
3.8291	40.0	40.6	39.4	1.2
1.3907	45.0	12.4	12.2	0.2
1.8314	45.0	19.7	19.7	0.0
2.6210	45.0	30.2	29.5	0.7
3.9111	45.0	40.6	40.4	0.2
			MEAN =	0.03
			STD DEV =	0.72

magnitude of this error is given by $\partial T/\partial$ [% U] in Equation 4 as -B/A or approximately 0.07 mil/% U. An error of 14% absolute in [% U] is thus required to produce a one mil error in the experimentally determined thickness. Although an error of a few percent in the uranium content will not significantly affect the measured thickness, the effect of a large error must be considered.

Thinning of reactor tube cladding is normally caused by hard particles such as carbon, U_3O_8, or UAl_4 in the substrate. With the exception of carbon which can be fluoroscopically distinguished from the more dense particles, these particles have a higher uranium content than that of the bulk substrate. Use of the bulk uranium content in Equation 4 therefore gives a conservative or minimum value for the cladding thickness.

PuO_2-Al Substrates

A typical x-ray fluorescence spectrum for aluminum cladding thickness analysis by ^{109}Cd excitation of a PuO_2-Al substrate reactor tube is shown in Figure 12. This spectrum has three components: plutonium fluorescent x-rays excited by silver x-rays from ^{109}Cd radioactive decay, silver x-rays scattered by the aluminum cladding, and x-rays from radioactive decay of tube substrate. Plutonium in the substrate decays by alpha emission to form uranium which subsequently fluoresces and produces uranium x-rays. The uranium $L_{\beta 1}$ x-rays, which are dominant in the uranium spectrum produced by this mechanism, have the same energy as the plutonium $L_{\beta 2}$ x-rays and interfere with the accurate determination of the plutonium $L_{\beta 2}$ intensity. The plutonium $L_{\beta 1}$ x-rays, however, are unaffected by this interference. The intensity of the plutonium $L_{\beta 1}$ and L_{α} x-rays were therefore selected to monitor aluminum cladding thickness on PuO_2-Al substrate tubes.

The experimental calibration data for aluminum cladding thickness analysis of PuO_2-Al substrate reactor tubes are given in Table 19 and a calibration plot of ln $(L_{\beta 1}/L_{\alpha})$ vs aluminum thickness (T) is shown in Figure 13. The calibration plot is linear as predicted by Equation 2. The intercept and slope of this calibration plot based on natural logarithms were determined by linear least squares fit of the data to be -1.828 and +0.03417/mil, respectively. Substitution of these values into Equation 2 gives Equation 5 which expresses the aluminum cladding thickness (T) as a function of the experimentally determined plutonium $L_{\beta 1}/L_{\alpha}$ x-ray intensity ratio.

$$T = \frac{\ln (L_{\beta 1}/L_{\alpha}) + 1.828}{0.03417} \quad (5)$$

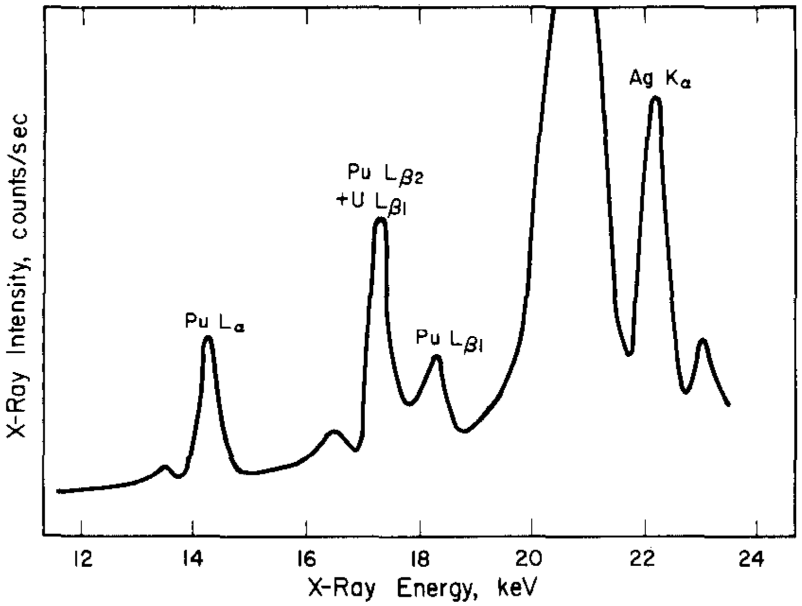

FIGURE 12. ^{109}Cd Excited X-Ray Fluorescence Spectrum of PuO_2-Al Substrate Reactor Tube

TABLE 19

Calibration Data for PuO_2-Al Substrate
Plutonium Content = 88.3 wt %
Detector-to-Aluminum Distance = 0.0 in.

Count Time, min	*CRT Vertical Scale*	*Net Peak Height, in.* L_{β_1}	L_α	*T, mils*	$\ln \frac{L_{\beta_1}}{L_\alpha}$
3.0	1024	0.44 ±.02	1.72 ±.03	13.4	-1.3633
6.0	1024	0.53 ±.03	1.41 ±.03	23.9	-0.9785
10.0	1024	0.68 ±.02	1.76 ±.03	26.9	-0.9510
15.0	1024	0.63 ±.03	1.22 ±.03	34.3	-0.6609
32.0	2048	0.35 ±.03	0.46 ±.03	45.1	-0.2733
2.0	512	0.53 ±.02	2.04 ±.04	13.4	-1.3478
2.0	256	0.65 ±.03	1.70 ±.05	24.3	-0.9614
2.0	256	0.48 ±.03	1.20 ±.03	27.0	-0.9163
2.0	256	0.38 ±.03	0.72 ±.03	34.5	-0.6391
2.0	256	0.21 ±.03	0.24 ±.04	45.5	-0.1335

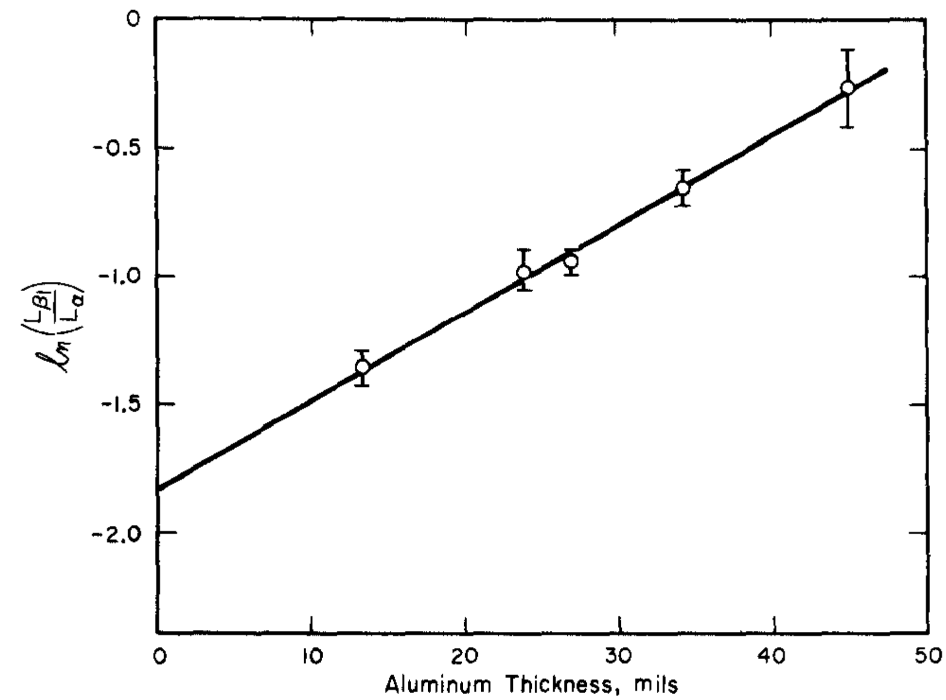

FIGURE 13. Calibration Plot of ln (L_β/L_α) vs. Aluminum Thickness for 88.3% Pu in PuO_2-Al Substrate

Equation 5 is strictly valid only for 100% PuO_2 (88.3% Pu) substrate. Expansion of the equation to include substrates having other plutonium contents requires a knowledge of the variation of the calibration intercept and slope with substrate plutonium content. This could not be determined with the single standard available. However, since this analysis is very similar to those for U_3O_8-Al and U-Al substrates, the effect of substrate composition on the calibration slope and intercept can be estimated by analogy. The calibration slope should then be independent of substrate plutonium content, and the intercept should change only slightly with plutonium content. The limits of precision and accuracy predicted on the basis of these assumptions are discussed in the next section of this report.

The cladding thickness monitor and Equation 5 were used to monitor the internal and external cladding thickness at 309 randomly sampled suspect spots on six Mark 41 PuO_2-Al reactor target tubes. These spots were detected prior to cladding thickness analysis by their low x-ray transmittance using a fluoroscope. The nominal cladding thickness for these tubes was 40 mils, and the substrate bulk plutonium content was 23.6 wt %.

Experimental data are presented in Table 20 for cladding thickness analysis of Mark 41 PuO_2-Al target tube H01034. These data demonstrate the consistency of the cladding thickness monitor analytical method within the stated uncertainty. Only three of the 80 measurements are less than 30 mils, and two of these (29.1 and 28.7 mils) are only slightly less. Significant thinning of the interior cladding (15.0 mils) was detected at one spot, coordinates 67.62 inches, 167 degrees. This value was confirmed by relocating the spot on the tube and by redetermining the thickness three times. Consistent replicate analyses of 15.9, 16.1, and 15.0 mils at this spot demonstrate the capability of the cladding thickness monitor for precise analyses at thin spots of the cladding.

Cladding thickness data for all six Mark 41 tubes are compiled in Table 21. These data further demonstrate the consistency of the analysis method and indicate that cladding on these tubes is quite uniform and relatively free of thin spots.

Precision and Accuracy of Measurements — PuO_2-Al Substrates

The accuracy of Equation 5 was estimated by assuming that

$$\frac{\partial T}{\partial[\%\ U]} = \frac{\partial T}{\partial[\%\ Pu]} = \frac{0.07\ \text{mil}}{\%\ Pu}$$

Since the bulk plutonium content of the Mark 41 PuO_2-Al substrate tubes is 23.6 wt % and that for the 100% PuO_2 calibration standard is 88.3 wt %, the maximum possible error in the cladding thickness due to composition effects is 4.5 mils for these tubes. However, since suspect thin spots are detected fluoroscopically by the high plutonium content of the substrate, the experimental error due to substrate composition effects must be less than the predicted 4.5 mils maximum.

The overall precision of the cladding thickness analyses for these PuO_2-Al substrate tubes depends upon the precision of the accumulated multichannel spectrum, the precision of the ruler measurement technique to determine the net intensity ratio, and upon the uniformity of the substrate composition. The relative standard deviation of about 3 mils for thickness analysis of tube H01034 is a good estimate for the minimum overall precision. This is because the estimate includes random errors in experimental measurements, and errors due to local variation of the plutonium content of the substrate. Since the precision of the thickness analysis of any specified spot is independent of the substrate plutonium content, the relative standard deviation for replicate analyses of a single spot with the cladding thickness monitor should not exceed 3 mils.

TABLE 20

Cladding Thickness Data for Mark 41 PuO_2-Al Target Tube H01034

Tube Coordinates		*Exterior Cladding*			*Interior Cladding*		
Inches	*Degrees*	$L_{\beta 1}$,[a] *Inches*	L_α,[a] *Inches*	*T*,[b] *Mils*	$L_{\beta 1}$,[a] *Inches*	L_α,[a] *Inches*	*T*,[b] *Mils*
Unnumbered End of Tube							
51.60	1.5	0.48	0.99	32.3	0.45	0.91	32.8
51.70	23.5	0.56	1.12	33.2	0.52	0.95	35.8
53.85	18.0	0.51	0.98	34.3	0.49	1.00	32.6
54.85	16.5	0.54	0.95	36.9	0.51	1.02	33.2
56.90	88.0	0.50	0.93	35.3	0.45	0.93	32.2
58.12	5.5	0.48	0.95	33.5	0.48	0.94	33.8
61.25	35.5	0.48	0.92	34.4	0.46	0.92	33.2
62.30	32.0	0.43	0.91	31.5	0.53	0.91	37.6
63.37	12.0	0.46	0.90	33.8	0.53	0.97	35.8
63.54	203.0	0.63	1.45	29.1	0.52	0.98	34.9
63.77	35.5	0.43	0.90	31.8	0.49	1.00	32.6
69.02	114.0	0.45	0.80	36.6	0.52	0.98	34.9
69.17	304.5	0.46	0.95	32.2	0.45	0.83	35.5
70.75	177.0	0.50	0.94	35.0	0.50	0.90	36.2
72.37	227.5	0.50	0.94	35.0	0.44	0.93	31.5
Numbered End of Tube							
16.79	184.0	0.54	0.96	36.6	0.50	0.98	33.8
18.65	159.5	0.50	0.97	34.1	0.46	0.88	34.5
21.72	195.0	0.53	0.84	40.0	0.50	0.89	36.6
23.47	358.0	0.56	0.94	38.3	0.43	0.78	36.0
27.47	345.0	0.50	0.86	37.6	0.48	0.89	35.4
29.60	195.0	0.44	0.75	37.8	0.50	0.95	34.7
30.27	171.5	0.50	0.76	41.2	0.50	0.84	38.3
32.05	192.5	0.40	0.73	35.8	0.60	1.13	34.9
34.95	189.5	0.42	0.76	36.1	0.58	1.07	35.5
43.00	203.0	0.40	0.80	33.2	0.58	1.04	36.4
49.72	188.5	0.48	0.82	37.8	0.55	1.07	34.0
52.57	23.0	0.59	1.14	34.2	0.50	0.89	36.6
57.00	223.0	0.52	1.07	32.3	0.48	0.94	33.8
57.92	195.0	0.53	0.97	35.8	0.47	0.90	34.4
63.30	213.5	0.45	0.81	36.2	0.45	0.84	35.2
64.37	170.5	0.48	0.91	34.7	0.45	0.88	33.8
65.42	222.0	0.58	1.35	28.7	0.46	0.77	38.4
66.80	178.0	0.50	0.97	34.1	0.43	0.85	33.5
66.80	180.0	0.49	0.85	37.3	0.48	0.88	35.7
67.62	167.0	0.46	0.84	35.8	0.58	2.16	15.0
68.05	342.0	0.48	0.78	39.2	0.49	1.05	31.1
68.79	30.0	0.42	0.92	30.5	0.41	0.88	31.1
69.92	185.0	0.46	0.80	37.3	0.48	0.91	34.7
71.70	230.5	0.45	0.81	36.2	0.40	0.82	32.4
71.72	66.5	0.49	0.85	37.3	0.50	0.93	35.3
			MEAN =	35.1			34.1
			STD DEV =	2.8			3.6

a. Height of peak on cathode ray tube photograph.

b. Cladding thickness.

TABLE 21

Cladding Thickness Data for Six Mark 41 PuO_2-Al Target Tubes

Tube Number	*Number of Analyses in the Indicated Thickness Range*					
	<15 mils	*15-20 mils*	*20-25 mils*	*25-30 mils*	*30-35 mils*	*>35 mils*
H01007	0	0	1	4	6	109
H01008	0	0	0	3	5	56
H01012	0	0	0	1	8	111
H01032	0	0	0	4	4	106
H01034	0	1	0	2	39	38
H01035	0	0	0	1	11	108

CONCLUSIONS

The thickness of aluminum cladding on uranium and plutonium reactor tubes can be accurately, precisely, and nondestructively determined from the relative attenuation of the L_α and L_β fluorescent x-rays. The method is also applicable to ribbed tubes because the experimentally determined thickness of uniform cladding is independent of detector-to-cladding distance.

The cladding thickness (T) is linearly related to the logarithm of the x-ray intensity ratio [$\ln (L_\beta/L_\alpha)$] and to the substrate uranium content [% U] by the empirical calibration constants A, B, and C:

$$T = \frac{\ln (L\beta/L\alpha) - B\ [\%\ U] - C}{A}$$

This equation is valid for cladding thicknesses up to 40 mils for U_3O_8-Al and U-Al substrates containing 20 to 45 wt % uranium. An error of a few percent in the uranium content will not significantly affect the calculated thickness because the constant B is relatively small.

The linear relation between cladding thickness and $\ln (L_{\beta 1}/L_\alpha)$ was also confirmed for a PuO_2-Al substrate containing 88.3 wt % plutonium. The constant B could not be evaluated for PuO_2-Al substrates with the single available standard.

Radioactive decay of plutonium produces intense uranium $L_{\beta 1}$ x-rays which interfere with the intensity measurement of the plutonium $L_{\beta 2}$ x-rays but do not interfere with measurement of plutonium $L_{\beta 1}$ x-rays. The plutonium $L_{\beta 1}/L_\alpha$ x-ray intensity ratio provides a satisfactory measure of the cladding thickness for plutonium tubes.

X-ray fluorescence analysis may also be applicable to cladding thickness analysis of reactor tubes having substrates othen than uranium and plutonium, especially other actinide elements.*

An x-ray fluorescence cladding thickness monitor could be employed to accurately determine the cladding thickness at each suspect spot during fluoroscopic evaluation of reactor tubes. The cladding thickness monitor-fluoroscope could be computer controlled to fluoroscopically survey the tube, determine cladding thickness wherever x-ray transmission is less than a specified value, and print out a permanent record of tube coordinates and cladding thickness.

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* Since this report was written, the cladding thickness monitor has been successfully used to measure cladding thicknesses in neptunium oxide target tubes.