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Ruthenium Oxide Deposition and Scouring in a Rotary Kiln Voloxidizer

J. A. Klein

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RUTHENIUM OXIDE DEPOSITION AND SCOURING IN A ROTARY KILN VOLOXIDIZER

J. A. Klein

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J. A. Klein

ABSTRACT

An experimental procedure was developed to produce reproducible, dense coatings of RuO_2 on a variety of substrates. Tracer experiments were performed to monitor the scouring of RuO_2 coatings under conditions simulating those expected to be encountered in the voloxidation step during LMFBR fuel reprocessing. It was concluded that RuO_2 may build up under repeated deposition and scouring cycles and might possibly present a problem in an actual LMFBR fuel voloxidizer.

1. INTRODUCTION

Various nuclear fuel reprocessing schemes for Liquid Metal Fast Breeder Reactors (LMFBRs) are presently being developed at the Oak Ridge National Laboratory. In order to remove tritium from the fuel prior to aqueous processing, the oxide fuel is heated in flowing oxygen or air -- a process termed voloxidation. In this process, sheared fuel rod segments are passed through a rotary kiln voloxidizer, where they constantly scrape, tumble, and impact on the inside surface. There is a possibility that, during this treatment, certain volatile fission products (principally ruthenium) will deposit on the voloxidizer walls.

The purpose of this study was to determine whether the abrasive action of the solids (stainless steel hulls) within the voloxidizer will scour the surface clean of any ruthenium oxide coating or whether this coating, which is highly radioactive, will continually accumulate.

Several different experimental procedures and equipment configurations were tested[1,2] before the present setup was selected. These procedures produced very light to moderate RuO_2 coatings; none of them were dense or adherent. All of the previous methods were deficient in certain respects; for example, the test specimens were located at an appreciable distance from the RuO_4 vapors and the specimen temperature could not be

controlled. The present procedure successfully produces reproducible, dense coatings on a variety of substrates.

2. EXPERIMENTAL METHOD

2.1 Materials Used

Standard reagent-grade chemicals were used in the study reported here. The ruthenium, which was available in the form of $RuCl_3$, needed to be converted to $Ru(NO_3)_3$ in order to prevent a large-scale chlorine corrosion problem when the ruthenium solution was heated. A slight excess of $AgNO_3$ was added to precipitate the chlorides present, and the resulting solution was filtered to remove AgCl and other insolubles. Although the chloride concentration was not determined after precipitation, no further problems were noted. The final $Ru(NO_3)_3$ solution was approximately 0.2 M in ruthenium.

The $NaBiO_3$ oxidizing solution was prepared as a 1 M mixture. This solution was saturated and contained undissolved $NaBiO_3$; thus it needed to be well mixed before each use.

The ^{106}Ru-^{106}Rh tracer was supplied as a chloride solution containing approximately 1 mCi/ml. This solution was diluted with distilled water to 20 μCi/ml before satisfactory results were obtained. The small amount of chloride present in the final solution presented no problems.

2.2 Apparatus

Figure 1 shows the experimental equipment used in the tests. The apparatus consisted of a standard 50-mm glass tee with a Teflon base attached to the bottom section to provide a vessel for the reacting liquids. A heating tape was wrapped around this end to provide heat for the reaction producing volatile RuO_4 from $Ru(NO_3)_3$ and $NaBiO_3$. Rubber stoppers were fitted in the remaining ends of the tee. The specimen heater and support consisted of a 3.2-mm-diam alumina tube that allowed for the insertion of an internal thermocouple and for wrapping with 200 mm of 6.4-mm-wide, 0.13-mm-thick stainless steel ribbon. Resistive heating in the stainless ribbon provided the necessary specimen temperature.

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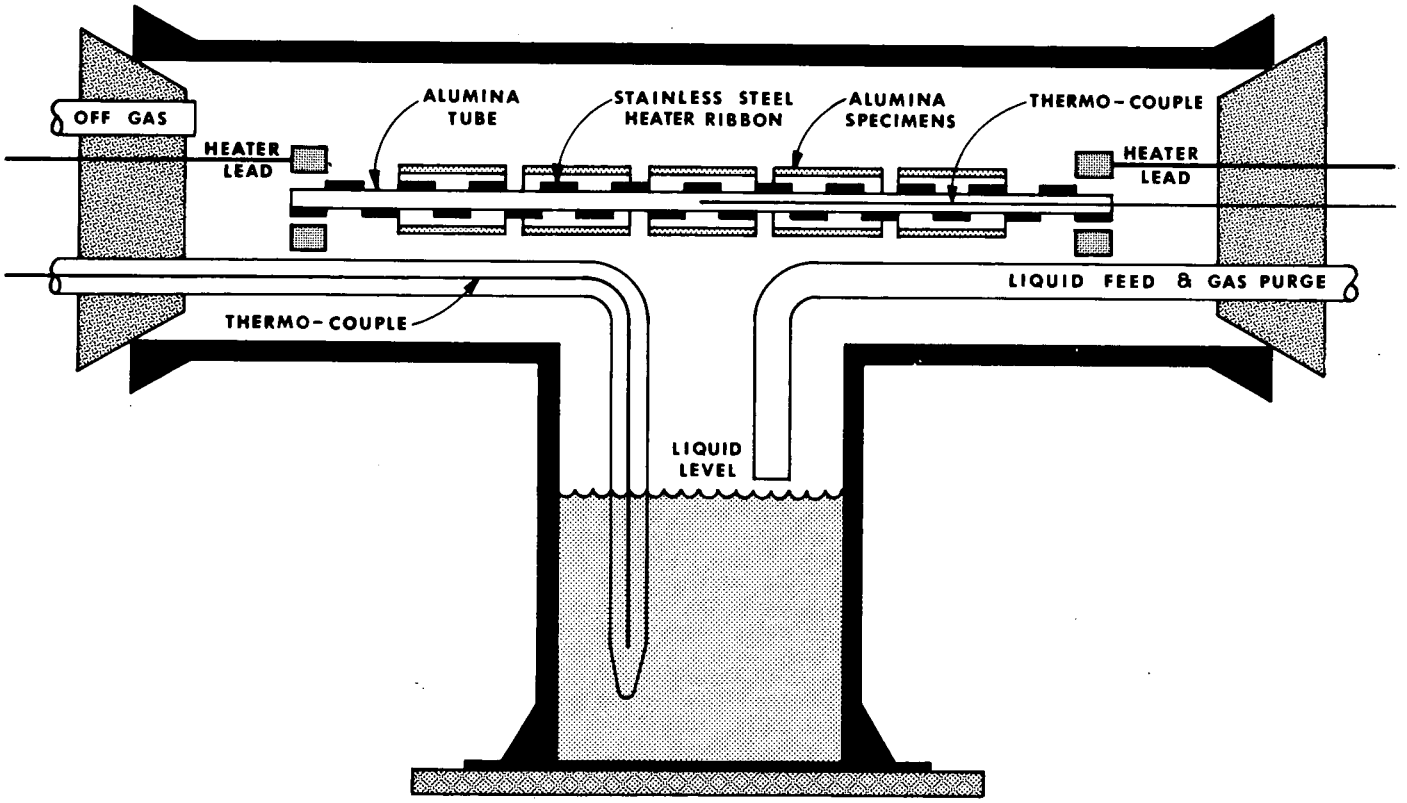

Fig. 1. Experimental reaction vessel.

For those runs in which alumina samples were desired, five 25-mm-long sections of 7.1-mm-diam alumina tubing were placed over the heater and became the specimens to be coated. In the most recent experiments, metal specimens were prepared using five 25-mm-long sections of 9.5-mm-diam stainless steel tubing fitted over the alumina tubing. Various openings in the rubber stoppers allowed for electrical contacts for the resistive heaters, for insertion of thermocouples to sense specimen and reaction vessel liquid temperatures, and for feed and purge gas flows. These openings were sealed with Duct Seal prior to the experimental runs.

Figure 2 shows the reaction vessel as well as the associated tubing and electrical hookups. The $NaBiO_3$ solution was added through the same inlet that allowed for a small continuous air purge of the system.

The coated alumina and stainless steel specimens were tumbled in a 165-mm-diam by 75-mm-deep bench-scale minivoloxidizer. This unit contained several internal baffles to provide adequate tumbling action and was housed in a small oven capable of reaching a temperature of $490^{\circ}K$. A single-speed, 8-rpm drive was used for each scouring test.

3. PROCEDURE

The experimental procedure used for generating RuO_4 was a modification of that suggested by Larsen.[3] In a typical experimental run, 80 ml of the 0.2 $\underline{M}$ $Ru(NO_3)_3$ stock solution and 15 ml of concentrated H_2SO_4 were added to the reaction vessel; then both heaters were turned on. When tracer runs were performed, 2 μCi of ^{106}Ru was added to the $Ru(NO_3)_3$ solution before it was transferred to the reaction vessel.

After the temperature of the reaction vessel had reached approximately $363^{\circ}K$ and the stainless ribbon $523^{\circ}K$ ($573^{\circ}K$ in the case of alumina and stainless specimens) as determined by the internal heater thermocouple, 25 ml of the 1 $\underline{M}$ $NaBiO_3$ solution was added to the reaction vessel in 2-ml increments over a period of approximately 45 min. When the final amount of oxidant had been added, the apparatus was allowed to remain at constant temperature for 2 hr before cooling and disassembly.

The stainless steel ribbon was then removed and microscopically inspected and photographed when appropriate. In the tracer runs, the alumina and stainless steel specimens were removed and counted using

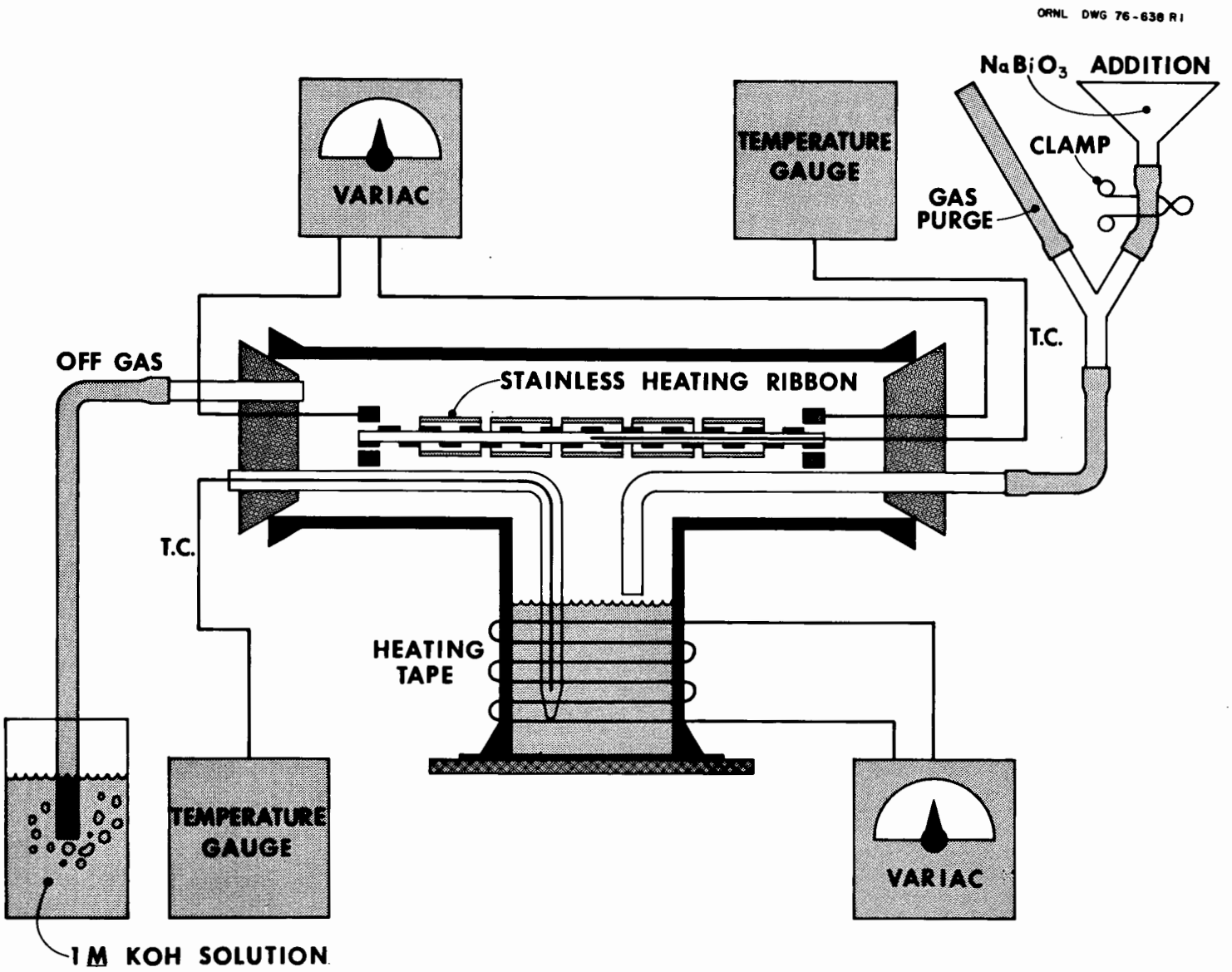

Fig. 2. Experimental equipment for ruthenium oxide deposition.

a standard well-type NaI scintillation crystal at the 0.513-keV peak of ^{106}Ru-^{106}Rh. Four of the five specimens in each run were subsequently added to the minivoloxidizer. After 80 ml of 25-mm-long sheared prototype fuel hulls and 20 ml of beach sand had also been added, the minivoloxidizer was sealed, heated to approximately 490°K, and tumbled for varying periods of time at 8 rpm. The fifth specimen, which served as a control, was not tumbled but was heated to the same temperature as the other specimens. At predetermined intervals, the samples were successively removed, tapped lightly to remove any dust particles, counted, and replaced in the minivoloxidizer. A new set of specimens was used for each run.

4. RESULTS AND DISCUSSION

4.1 Runs Made at Optimum Deposition Temperature

Literature searches indicated that RuO_2 has two temperature ranges (∿ 424°K and ∿ 775°K) at which decomposition is particularly favored.[4] In addition to these two temperatures, RuO_2 decomposition is self-catalyzed at any temperature.[4] An initial series of eight experimental runs (see Table 1) was performed to determine the optimum temperature for RuO_2 deposition. Of the various temperatures used, 525°K appeared to give the densest deposits. The higher temperatures (725 to 825°K) produced large amounts of RuO_2, but the deposits were so crystalline [see Fig. 3(a)] that slight air movements dislodged much of them. Several of the dense deposits formed at 525°K [Fig. 3(b)] were weighed and found to contain 11 to 13% of the initial ruthenium present in the reactants. A sample of the deposit from run 51 was analyzed semi quantitatively and found to be at least 90% RuO_2. Two additional experimental runs were made in which the stainless strip was etched and/or abraded prior to RuO_2 deposition. No difference in amount or type of coating could be observed. No tumbling of the stainless steel ribbons in the minivoloxidizer was attempted due to the brittleness of the specimens, even those prepared at 525°K. However, other stainless steel specimens were prepared, coated, and tumbled (see Sect. 4.4).

Table 1. Summary of experimental runs performed

Run No.	Specimen temp. (^{o}K)	Remarks
		Nonradioactive Runs
50	825	Sword-shaped crystalline deposits; very fragile and easily dislodged.
51	525	Hard, solid coating but very bittle; large pieces easily flaked off.
52	610	Moderate amounts of "popcorn" type deposits; semicrystalline.
53	475	Thin scales; very little coating.
54	525	Dense, brittle coating; 11% of ruthenium deposited.
55	725	Long crystalline deposits similar to those obtained in run 50.
57	525	Deposits very similar to those obtained in runs 51 and 54.
58	525–775	RuO_2 deposited at 525°K; specimen maintained at 775°K for 2 hr; coating similar to 525°K type coating; 13% of ruthenium deposited.
59	525	Stainless strip etched; no difference from other 525°K runs noted.
60	525	Stainless strip abraded and etched; no difference from other 525°K runs noted.
61	575	Strong, dense, adherent coating on alumina specimen.
61	575	Alumina specimen recoated; strong, dense coating; 16% of ruthenium deposited.
		Tracer Runs
62	575	Alumina specimen used; approximately 1 mCi of ^{106}Ru added; count rate overloaded NaI crystal.
63	575	Alumina specimen used; approximately 100 μCi of ^{106}Ru added; count rate overloaded NaI crystal.
64	575	Alumina specimen used; approximately 2 μCi of ^{106}Ru added; count rate acceptable, but coating was such that scouring was not attempted.
65	575	Alumina specimen used; 2 μCi of ^{106}Ru added (see Table 2).
66	575	Similar to run 66 (see Table 2).
67	575	Stainless steel specimen.
68	575	Stainless steel specimen.
69	575	Alternate coating and scouring run.

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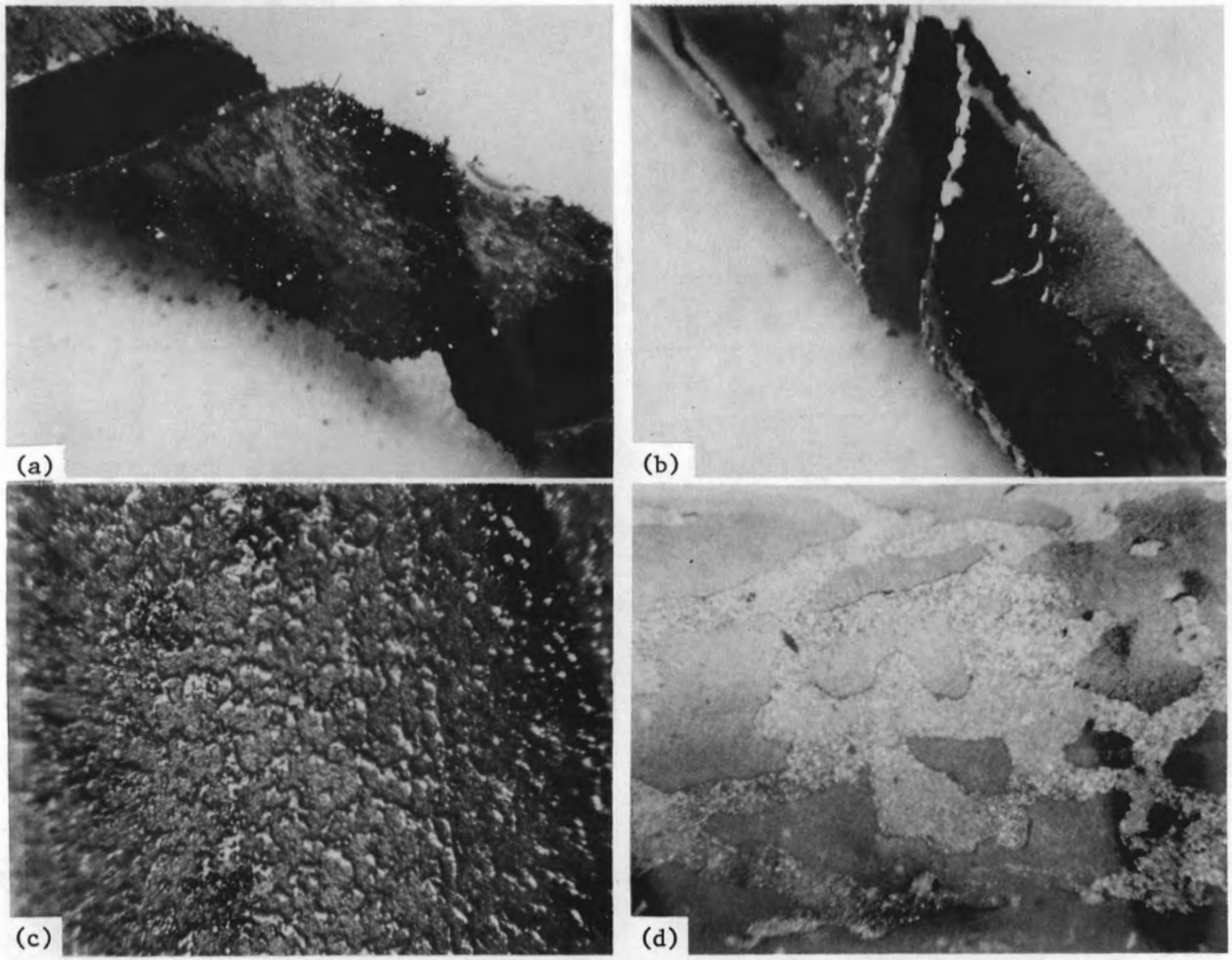

Fig. 3. Experimental rughenium oxide coatings. (a) Stainless steel ribbon, 825°K. (b) Stainless steel ribbon, 525°K. (c) Alumina specimen, 575°K. (d) Scoured stainless steel specimen, 575°K.

4.2 Deposition on Alumina

In an actual fuel reprocessing situation, it is possible that some existing mechanism will allow an adherent coating to form. Therefore, a porous substrate, alumina, was coated in order to obtain test specimens for scouring studies.

An initial run (No. 61) was made with a single 127-mm-long alumina specimen at 575°K. The temperature of the internal thermocouple was increased to allow for the temperature difference between the alumina sample and the stainless heater ribbon. This experiment produced a strong, adherent, dense coating of RuO_2 [see Fig. 3(c)] containing approximately 10% of the total ruthenium present. During our attempts to remove some of this coating, the alumina specimen cracked before any significant amount of RuO_2 could be flaked off.

4.3 Ruthenium-106 Tracer Deposits on Alumina

On determining that a dense, adherent coating of RuO_2 could be produced on alumina specimens, three experimental runs (Nos. 62-64) were performed in order to determine an acceptable level of ^{106}Ru tracer to add to the $Ru(NO_3)_3$ solution. Approximately 2 μCi proved to be satisfactory, giving between 1000 and 10,000 dis/min without saturating the counter.

Alumina specimens in runs 65 and 66 were coated with RuO_2 containing ^{106}Ru and scoured for varying lengths of time. The results are shown in Table 2 and Fig. 4. Both runs indicate that only about 20 to 30% of the RuO_2 can be scoured off, even with extremely long tumbling times. It is possible that RuO_2 scoured off during previous tumbling cycles might redeposit on the surfaces of some of the samples; however, the stainless steel experiments (runs 67 and 68 discussed below) were performed under similar conditions, and no leveling off of count rate occurred until only a small fraction of the original activity remained. A significant fraction of the RuO_2 appears to be embedded in the alumina pore structure and is thus relatively immune to scouring action.

Table 2. Data obtained in runs 65-69: ^{106}Ru tracer experiments

Type of samples	Run number	Sample number	0 hr dis/min	2 hr dis/min	2 hr % of 0 hr	18 hr dis/min	18 hr % of 0 hr	34 hr dis/min	34 hr % of 0 hr
Alumina	65	1	8978	8748	97	7892	88	7915	88
		2	7676	7411	97	6603	86	6544	85
		3	3105	2206	71	2263	73	2558	82
		4	1430	1493	104	1281	90	1134	79
		Ave. (1-4)	5297	4965	94	4509	85	4538	86
		5	714	685	102	658	98	677	101

Type of samples	Run number	Sample number	0 hr dis/min	2 hr dis/min	2 hr % of 0 hr	4 hr dis/min	4 hr % of 0 hr	20 hr dis/min	20 hr % of 0 hr	38 hr dis/min	38 hr % of 0 hr	56 hr dis/min	56 hr % of 0 hr
Alumina	66	1	2878	2556	89	2414	84	1703	59	1310	46	1061	37
		2	1469	1193	81	1226	83	962	65	941	64	846	57
		3	11383	10744	94	10699	94	10144	89	9455	83	9063	80
		4	6196	5735	93	5503	89	4995	81	4552	73	4147	67
		Ave. (1-4)	5482	5056	92	4961	90	4451	81	4065	74	3778	69
		5	1701	1658	97	1625	96	1722	101	1698	100	1669	98

Type of samples	Run number	Sample number	0 hr dis/min	1 hr dis/min	1 hr % of 0 hr	3 hr dis/min	3 hr % of 0 hr	7 hr dis/min	7 hr % of 0 hr	11 hr dis/min	11 hr % of 0 hr	15 hr dis/min	15 hr %of 0 hr
Stainless Steel	67	1	5552	4943	89	3442	62	1819	33	574	10	215	4
		2	3098	2630	85	2089	67	964	31	440	14	249	8
		3	8342	4170	50	3450	41	1093	13	454	5	200	2
		4	5357	4997	93	4196	78	1341	25	386	7	242	5
		Ave. (1-4)	5586	4185	75	3294	59	1304	23	464	8	227	4
		5	4965	4879	98	4953	100	5034	101	4928	99	4853	98

Type of samples	Run number	Sample number	0 hr dis/min	1 hr dis/min	1 hr % of 0 hr	3 hr dis/min	3 hr % of 0 hr	7 hr dis/min	7 hr % of 0 hr	11 hr dis/min	11 hr % of 0 hr
Stainless Steel	68	1	4896	1652	34	796	16	318	6	184	3
		2	7727	1837	24	963	12	159	2	123	2
		3	10477	7552	72	4902	47	3391	32	1657	16
		4	3199	644	20	395	12	262	8	215	7
		Ave. (1-4)	6575	2921	44	1764	27	1042	16	539	8
		5	6457	6335	96	6299	98	6372	99	6179	96

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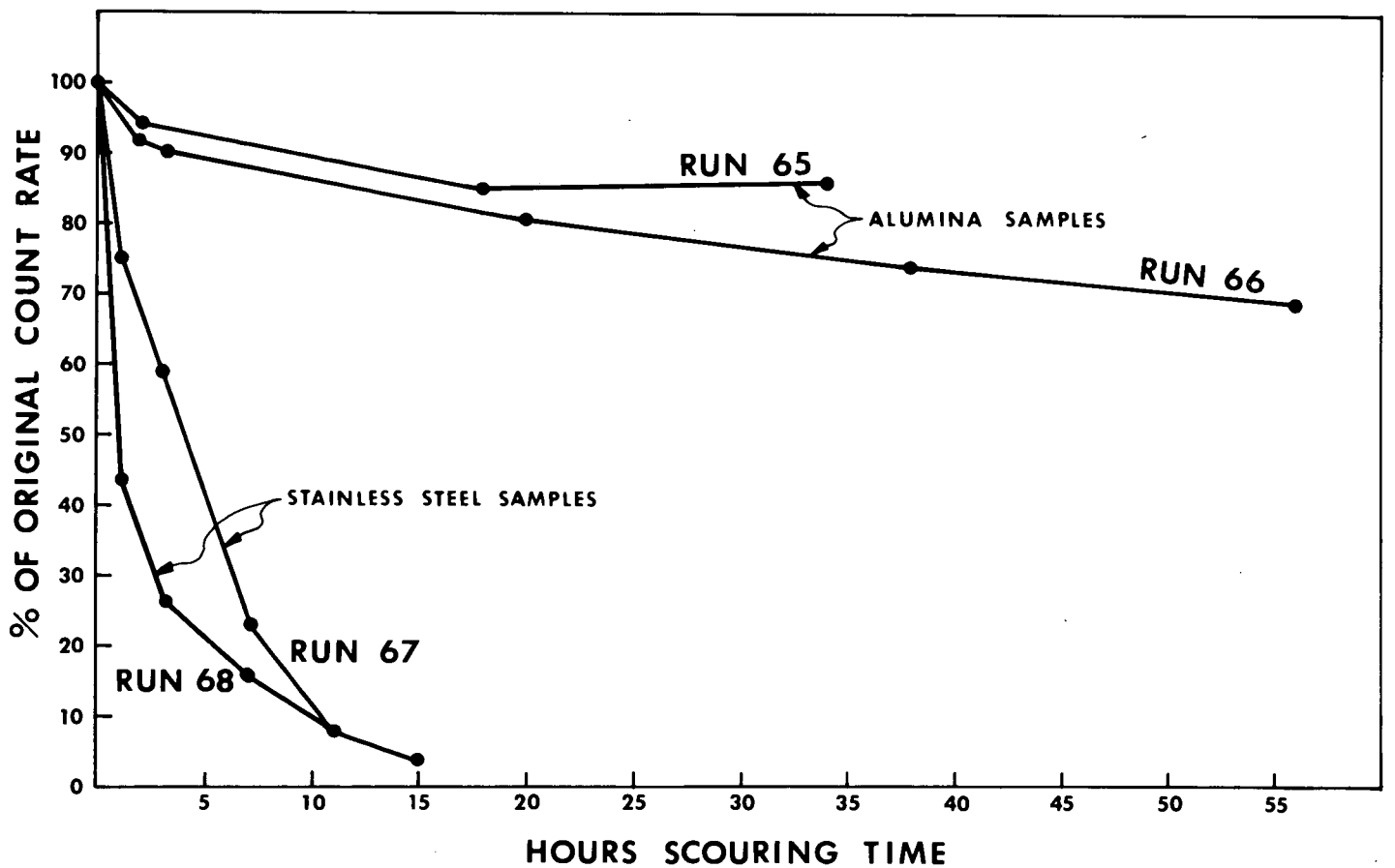

Fig. 4. Ruthenium-106 tracer experiments with alumina and stainless steel specimens.

4.4 Ruthenium-106 Tracer Deposits on Stainless Steel

Stainless steel specimens were substituted for the alumina samples in an effort to obtain RuO_2 coatings on a nonporous substrate. Results from these runs are also shown in Table 2 and Fig. 4. As expected, the RuO_2 was scoured from the stainless steel samples at a significantly faster rate than it was removed from the alumina samples. Figure 3(d) shows one of those specimens after 3 hr of tumbling time. Essentially all the coating could be removed with scouring times on the order of 15 to 20 hr. A significant amount (20 to 60%), however, did remain after 3 to 4 hr of tumbling. This was somewhat unexpected since the original RuO_2 coatings were quite nonadherent to stainless steel. Hence it appears that, while a significant portion of the RuO_2 coating is nonadherent, more tenaciously held areas are also present and these latter areas resist extensive scouring.

4.5 Experiment with Multiple Coating and Scouring

One experiment, run 69, consisted of a set of stainless steel samples that were coated with RuO_2 in the normal manner and successively scoured for 4 hr, recoated, scoured, etc. This sequence was repeated for a total of six cycles. As an afterthought, on completion of the experiment, all five samples were abraded by filing on the inside surfaces for removal of internal ^{106}Ru and counted for five cleanings. Data and results are shown in Tables 3 and 4 and Fig. 5.

Although the results obtained in this experiment are somewhat difficult to interpret, it appears, that contrary to runs 67 and 68, a large percentage (average, 75%) of the ruthenium deposited on the inside of the specimens. This may have been due to slightly different temperature conditions, purge gas flows, etc., or the stainless steel specimens may have been slightly oversized, thereby allowing RuO_4 vapor to enter the insides of the specimens.

The scouring sand and prototype fuel hulls were not replaced between scouring cycles; thus it is possible that the increasing quantity of RuO_2 in the minivoloxidizer could be a factor in the buildup of ^{106}Ru on the sample. However, in experimental runs 67 and 68, the stainless steel

Table 3. Multicycle ruthenium-coated and scoured samples of run 69

^{106}Ru activity (dis/min) after:	Sample number 1	2	3	4	Average of Samples 1-4	Control[a]
Coating 1	421	453	209	372	364	259
Scour 1	374	432	190	331	332	253
Coating 2	1027	875	407	653	741	312
Scour 2	837	800	365	579	645	312
Coating 3	1264	1097	508	818	922	382
Scour 3	1152	1071	493	761	869	377
Coating 4	1437	1205	618	1082	1085	417
Scour 4	1328	1131	552	991	1001	390
Coating 5	1525	1189	661	1210	1146	418
Scour 5	1479	1148	631	1171	1107	421
Coating 6	1773	1539	773	1520	1401	520
Scour 6	1686	1473	713	1471	1336	507
Cleaning 1	1358	921	454	1049	946	338
Cleaning 2	1008	726	339	784	714	257
Cleaning 3	803	566	328	566	566	223
Cleaning 4	483	415	183	322	351	179
Cleaning 5	468	421	137	255	320	167

[a]Control was not scoured.

Table 4. Analysis of data from run 69

	Sample number 1	2	3	4	Average of samples 1-4	Control[a]
Change in ^{106}Ru activity (dis/min) due to:						
Coating 1	421	453	209	372	364	259
Coating 2	653	443	217	322	409	59
Coating 3	427	297	143	239	277	70
Coating 4	285	134	125	321	216	40
Coating 5	197	58	109	219	146	28
Coating 6	294	391	142	349	294	99
Average	379	296	157	304	284	93
Scour 1	-47	-21	-19	-41	-32	-6
Scour 2	-190	-75	-42	-74	-95	0
Scour 3	-112	-26	-15	-57	-53	-5
Scour 4	-109	-74	-66	-91	-85	-27
Scour 5	-46	-41	-30	-39	-39	3
Scour 6	-87	-66	-60	-49	-65	-13
Average	-99	-51	-39	-59	-62	-8
Cleaning	-1218	-1052	-576	-1216	-1016	-340
Percent of coating on outer surface after Scour 6	28	29	19	17	24	33
Increase in ^{106}Ru activity on outer surface per cycle, Average	78	70	23	43	53	28
^{106}Ru activity deposited on outer surface per cycle, dis/min	105	85	30	53	68	30
Percent removal per cycle	26	18	23	19	22	7

[a]Control was not scoured.

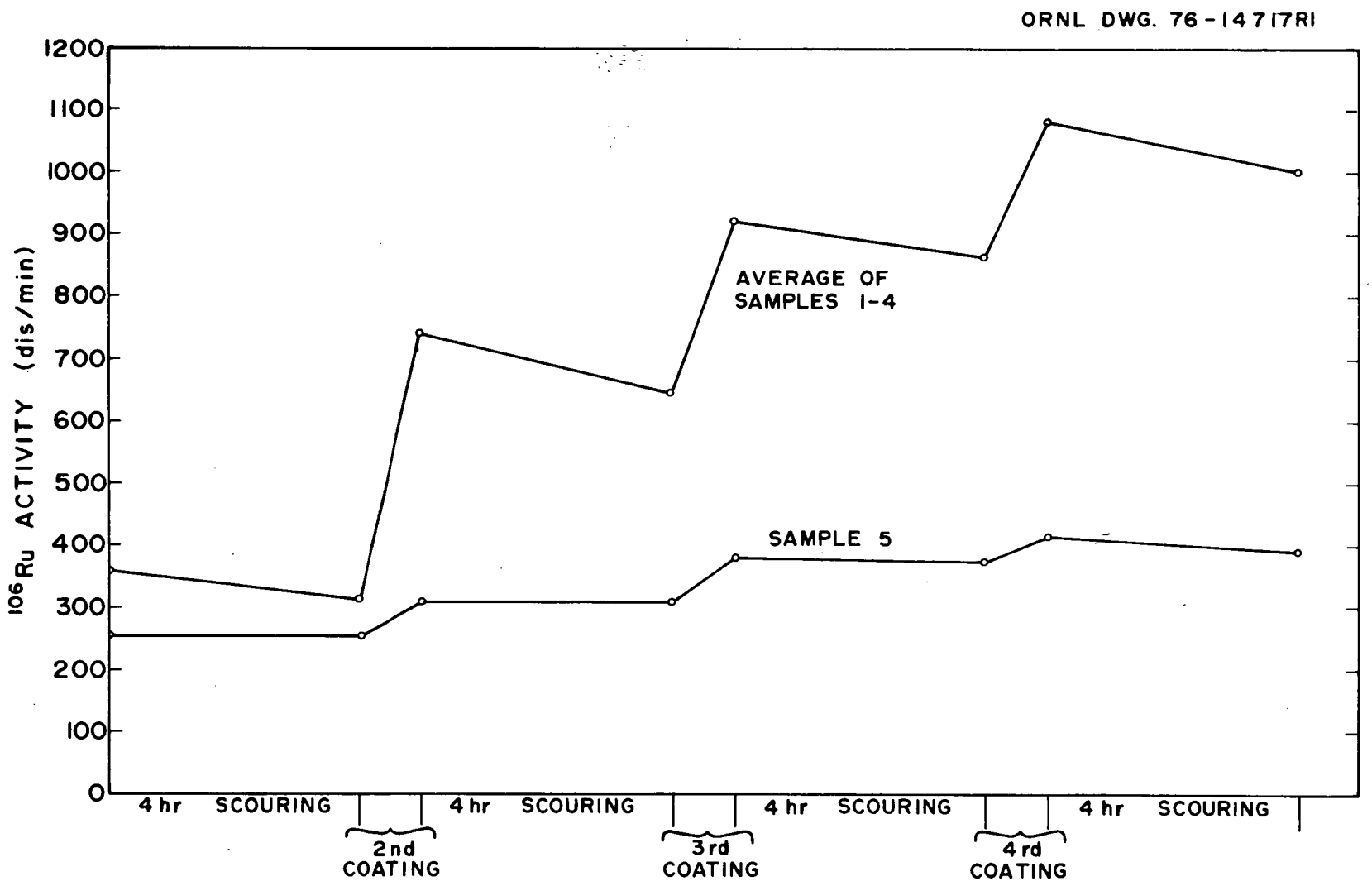

Fig. 5. Results of multiple coating-scouring experiment, run 69.

samples lost greater than 90% of their ^{106}Ru activity during 15 hr of scouring even though the scouring sand and hulls were constantly increasing in ^{106}Ru activity. In any case, the amount of RuO_2 remaining on the outside of the specimens after cleaning indicates that the RuO_2 coating does not reach a steady state in spite of the alternate scouring cycles.

5. CONCLUSION

Reproducible, dense layers of RuO_2 can be coated on a variety of substrates. These coatings will stick tenaciously to porous materials such as alumina but much less so to stainless steel substrates. Even a stainless steel coating, however, will accumulate RuO_2 under repeated scouring-coating cycles. Accumulation of such highly radioactive coatings may cause considerable difficulties in an actual LMFBR fuel reprocessing voloxidizer. These problems would arise in any direct maintenance operations. In addition, the coating would be come a substantial heat source, making temperature monitoring and controlling difficult.

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