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DECONTAMINATION OF STEEL BY MELT REFINING

A LITERATURE REVIEW

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ABSTRACT

It has been reported that a large amount of metal waste is produced annually by nuclear fuel processing and nuclear power plants. These metal wastes are contaminated with radioactive elements, such as uranium and plutonium. Current Department of Energy guidelines require retrievable storage of all metallic wastes containing transuranic elements above a certain level. Because of high cost, it is important to develop an effective decontamination and volume reduction method for low level contaminated metals. It has been shown by some investigators that a melt refining technique can be used for the processing of the contaminated metal wastes. In this process, contaminated metal is melted with a suitable flux. The radioactive elements are oxidized and transferred to a slag phase. In order to develop a commercial process it is important to have information on the thermodynamics and kinetics of the removal. Therefore, a literature search was carried out to evaluate the available information on the decontamination uranium and transuranic-contaminated plain steel, copper and stainless steel by melt a refining technique. Emphasis was given to the thermodynamics and kinetics of the removal. Data published in the literature indicate that it is possible to reduce the concentration of radioactive elements to a very low level by the melt refining method.

INTRODUCTION

Metallic wastes contaminated with transuranic elements occupy a relatively large portion of the wastes generated by nuclear power plants, fuel producing facilities and reprocessing facilities. These materials cannot be treated as combustibles. Current guidelines in many countries require retrievable storage of all wastes containing radioactive elements above a certain level. Costs associated with storage of these materials are high. Recently the focus has been on developing a better technology for the volume reduction and decontamination method. One established decontamination method is melt refining.(1-6) In this method contaminated mild steel, stainless steel, copper or other contaminated wastes are melted with a suitable flux. Since uranium and the transuranic elements have higher affinity to oxygen than iron, copper, nickel, etc. these elements are preferentially oxidized and transferred to the liquid slag phase. The slag phase is separated and treated as low level waste. The organic compounds in the waste will be decomposed at refining temperatures resulting in hydrogen, carbon and oxygen that are normal components of the metal refining operations. The amount of radioactive elements remaining in the steel depends on operating conditions and it is expected to be very low. The radioactive elements in the steel product are evenly distributed and, therefore, less hazardous than original surface contaminated waste material. The liquid steel is casted into convenient shapes. During the operation a large reduction in waste volume can be accomplished.

In order to develop a commercial melt refining technology it is necessary to have information on the kinetics and thermodynamics of the removal of uranium and transuranic elements from mild steel, stainless steel, copper and other metals under consideration. Therefore, a literature search was carried out to obtain the available information on the melt refining. Emphasis was given to the thermodynamics and kinetics of the system in order to obtain the optimum refining conditions. The results from the literature search are summarized in the report.

THERMODYNAMIC CONSIDERATIONS

In the melt refining technique the removal of uranium or transuranic elements from steel is accomplished by combining it with oxygen and transferring to a slag phase as an oxide. Depending on the chemical potentials of oxygen and uranium, compounds such as UO_2, UO_3, U_4O_9 and U_3O_8 may form. The valence of uranium changes with oxygen potential. However, under steelmaking conditions, it is expected that uranium in the slag primarily exists as U^{4+}.Therefore, the reaction for the removal of uranium in terms of stable compounds can be written as

$$U(l) + O_2 = UO_2(s) \qquad [1]$$

$$\Delta G_1^0 = -257900 + 41.87T \quad \text{cal.}^{(**)} \qquad [2]$$

Since a very small amount of contaminant is present in the steel it is convenient to change the standard state for uranium from pure liquid to 1 weight percent. The free energy of the solution of uranium in iron is estimated from the regular solution model, thus

$$U(l) = U(1\ \text{wt.\%}\ \text{in Fe}) \qquad [3]$$

$$\Delta G_3^0 = -56100 \quad \text{cal. at } 1873\ \text{K} \qquad [4]$$

The free energies of solution of gaseous O (**) in liquid iron is known and given by

$$\frac{1}{2}O_2(g) = O(1\ \text{wt.\%}\ \text{in Fe}) \qquad [5]$$

$$\Delta G_5^0 = -28000 - 0.69T \quad \text{cal.} \qquad [6]$$

Combining equations (1)-(6) gives

$$\underline{U} + 2\underline{O} = UO_2(s) \qquad [7]$$

$$\Delta G_7^0 = -65157 \quad \text{cal. at } 1873\ \text{K} \qquad [8]$$

Equations (7) and (8) can be used to calculate the final concentration of uranium in steel in equilibrium with a slag at a given oxygen activity. For the calculations it is necessary to have information on the activity coefficient of uranium oxide in slags under consideration. The information is not available and, therefore, calculations were not carried out.

The removal of uranium can also be formulated as a slag metal-reaction

$$[U] + 2(FeO) + O^{2-} = UO_3^{2-} + 2Fe \qquad [9]$$

The the reaction (9) it is assumed that the uranium in the slag exists predominantly as UO_3^{2-}. Similar reactions can also be written for other monomer uranium ions.

It is possible to relate the ability of the slag to hold uranium directly to the composition and temperature by introducing the concept of capacity. The capacity of a slag is independent of the oxygen pressure but can only be derived with the knowledge of the type of ion formed by the component in the slag. Uranium in the slag may exist as U^{4+}, UO^{2+}, UO_3^{2-}, UO_4^{4-} if the valence of uranium 4+. If one assumes uranium in the slag exists as monomers, such as U^{4+} and UO^{2+}, which show basic behavior, then the basic oxidation reactions are

$$[U] + 2[O] = U^{4+} + 2O^{2-} \qquad [10]$$

$$[U] + 2[O] = UO^{2+} + O^{2-} \qquad [11]$$

On the other hand, UO_3^{2-} and UO_4^{4-} are acid in nature and, therefore, the oxidation reactions are

$$[U] + 2[O] + O^{2-} = UO_3^{2-} \qquad [12]$$

$$[U] + 2[O] + 2O^{2-} = UO_4^{4-} \qquad [13]$$

The U^{4+} capacity of the slag can be derived from the equilibrium constant of reaction (10)

$$K_{10} = \frac{a_{U^{4+}}\ a_O^2}{a_U\ a_O^2} \qquad [14]$$

$$C_{U^{4+}} = \frac{L_U}{f_U\ a_O^2} = \frac{K_{10}^{\times}}{\gamma_{U^{4+}}\ a_{O^{2-}}^2} \qquad [15]$$

Where $L_U = L_U^{(U^{4+})} = (\% U^{4+})/[\%U]$, $K_{10}^{\cdot}$ is the equilibrium constant for reaction (10) involving a constant for conversion from mole fraction U^{4+} to weight percent, f_U is the activity coefficient of uranium in steel with respect to 1 wt.% standard state. Similar capacities can also be derived for UO^{2+}, UO_3^{2-} and UO_4^{4-}. At a given slag composition and oxygen pressure these capacities are proportional to $C_{U^{4+}}$ because only L_U on the right hand side of equation (15) changes.

If uranium in the slag exists as U^{4+} or UO^{2+} it is important to use a slag that has a high value of $C_{U^{4+}}$ or $C_{UO^{2+}}$ and low basicity in order to get a higher uranium distribution ratio. If uranium exists as UO_3^{2-} or UO_4^{4-} a highly basic slag must be used. A higher oxygen potential increases the uranium distribution ratio in both cases.

Similar reactions also can be written for plutonium. The removal of plutonium occurs according to the following reaction.

$$Pu(l) + O_2 = PuO_2 \qquad [16]$$

$$\Delta G^o_{16} = -250000 + 42.52T \quad cal \qquad [17]$$

The free energy change associated with changing standard state from pure liquid to 1 wt.% in iron is not know. The activity of plutonium oxide in steelmaking type slags has not been determined previously and, therefore, equation (16) can be used to calculate the degree of removal at a given oxygen potential.

LITERATURE REVIEW

Mild Steel

Decontamination of uranium contaminated mild steel was investigated by T. Uda, et al.(11) Metal samples (100 gr) contaminated with 500 ppm of uranium were melted with slags of different composition in an alumina crucible. After a desired equilibration time the samples were quenched to room temperature and analysed by the epithermal neutron activation method. The weight of the slag was 10% of the metal weight. The slag was not analysed for uranium. The uranium content of the slag was obtained from the mass balance. The pressure in the reaction chamber was less than 13.5 Pa, even during heating. The initial concentration of uranium was obtained by coating the metal samples with uranyl nitrate. The experimental results for the melting time versus ingot uranium level is shown in Figure 1. Uranium concentration decreases during the first 10 min. and in about 30 min. the concentration approaches a constant value. Their experimental results indicate that the system reaches equilibrium in short period of time. The effect of basicity on the removal was also investigated. In these experiments temperature, equilibration time and the amount of uranium was fixed, and only slag composition was changed. The results are presented in Figure 2. The uranium concentration decreases with increasing

CaO/(SiO_2+Al_2O_3). The minimum concentration was obtained at basicity of around 1.6. A further increase in CaO/(SiO_2+Al_2O_3) increases the decontamination level which is not well understood. The decontamination factor is defined as a ratio of the amount of uranium in slag to the amount in the metal.

The melt refining method was also used by Abe et al.(12) to investigate uranium decontamination in metallic wastes. The experimental method was identical to the one used by Uda et al.(11). Their experimental results indicate that the decontamination reaction was over less than 0.1 hours. For the majority of the experiments a slag containing 40% SiO_2, 40% CaO and 20% Al_2O_3 was used. The most effective slag basicity was around 1.5. Small additions of NiO and CaF_2 to the CaO - SiO_2 - Al_2O_3 decreased the decontamination. A decrease due to the NiO addition most likely is due to the fact that NiO increases the oxygen potential of the system.

Uda, Tsushiya and Iba(13) have investigated the removal of uranium from iron by smelting. The experimental apparatus and method was similar to the one used by Uda et al.(11) Table I shows their results. The decontamination factor increased with melting time and the temperature for the same slag composition. The decontamination factor became higher when CaF_2 was added to the mixture of CaO and SiO_2 or when a magnesia crucible was used.

The effect of slag composition on decontamination of metallic wastes was investigated by Heshmatpour and Copeland.(14) Samples of contaminated metals were melted with fluxes by resistance or induction heating using silica, alumina or zirconia crucibles. After a desired equilibration time the sample was cooled down and slag and metal were analyzed. The experimental results are given in Table II for mild steel. For some experiments, slag containing different ratio of CaO/SiO_2 with 30% Fe_2O_3 was used in alumina crucibles by resistance heating. The results from these experiments are plotted in Figure 3. The data in Figure 3 indicates that the partition ration decreases with decreasing CaO/SiO_2 ratios assuming the FeO content of the slag remain the same in all experiments. This is not in agreement with the data given in Figure 2. It was found that the degree of decontamination was not highly sensitive to the slag compositions. Using highly fluid slags with relatively high Fe_2O_3 content it could be possible to reduce contamination to 0.01 to 1 ppm.

The decontamination of iron wastes by the electroslag refining (ESR) method was studied by Uda, Ozawa and Iba(15). An uranium contaminated electrode was placed in a molten slag pool and electric power (voltage: 40 to 50 volts; current: 300 to 1000 amp) was supplied. The electrode was melted by the joule heat generated in the slag. The off gas was filtered. A flux containing 40% SiO_2, 30% CaO, 20% Al_2O_3 and 10% CaF_2 was used for the iron experiments. The uranium concentration of ingots was decreased to about 0.0125 ppm. The uranium concentration in the slag and dust after the experiments as well as the amount of slag used is not given in the paper.

A 4 tonne electric arc furnace was used to to process steel waste by melting.(16) The contamination level before the melting was 20 mC_i/ton. Atmospheric contamination was monitored during furnace feeding, melting, oxidation, reduction and casting. This operation indicates that metals with very low radioactivity can be processed. The quality of steel produced is such that it can be used for a wide range of finished products.

Seitz, Gerding and Steindler(17) determined the distribution of plutonium and americium between mild steel and $CaOSiO_3$ slags of various compositions. About 200 grams of metal and 20 grams of slag containing plutonium were equilibrated for 1 or 2 hours. All metal samples generally have concentrations below 0.010 ppm plutonium; for some samples concentrations are well below 0.001 ppm. Partition coefficients (Pu in slag/Pu in metal) of 7×10^6 were obtained with a slag containing 81% SiO_2, 13% B_2O_3, 4% Na_2O, 2% Al_2O_3 and 0.5% K_2O and 3×10^6 with calcium and magnesium silicate. Slag adhering to the metal surface and inclusions in the metal are the important contributors to Pu remaining in processed metal.

The distribution of plutonium between mild steel and slags of different compositions was investigated by Heshmatpour, Copeland and Heestand.(18) These results are presented in Table 3. Again the experimental results indicate that it is possible to remove Pu from mild steel using a melt refining technique.

Contaminated metallic wastes were treated by Kitagawa et a(19)l using the electroslag refining method. Simulated metallic wastes were melted and solidified in a 100 kg test furnace. A slag containing 47% CaO, 48% Al_2O_3 and 5% B_2O_3 was used. Hafnium oxide was used to simulate PuO_2. Waste volume was reduced 1/25 with a decontamination factor of 25. The slag and copper mold were capable of repeated use.

Stainless Steel

Removal of uranium from stainless steel was also investigated by Heshmatpour and Copeland(14) using various slag compositions. The experimental results are summarized in Table 4. They found that the decontamination factor was not highly dependent on the slag composition. However, it was found that highly fluid basic slags are more effective and result in decontamination of the metal as low as 0.01 to 0.05 ppm uranium even using 5% slag.

Removal of uranium from stainless steel was investigated by Abe et al.(12) A slag containing 40% SiO_2, 30% CaO, 20% Al_2O_3 and 10% CaF_2 was equilibrated with a stainless steel sample for 30 min. A decontamination factor of 5×10^3 was obtained which was lower than that obtained for mild steel. The decontamination was slightly higher with compare to mild steel using the same slag composition. This is might be due to the fact that the alloying elements in steel decreases activity coefficient of oxygen.

Distribution of plutonium between stainless steel and slags of different composition was studied by Heshmatpour et al(18) at 1600°C. About 500 ppm PuO_2 was throughly mixed with the slag and added to the metal in a crucible and equilibrated for 30 to 60 min. These experimental results are summarized in Table 5. Results in Table 5 shows that values less than 1 ppm Pu can be obtained by a single slag treatment. Similar type of experiments were also carried out by Seitz et al.(17) The plutonium content of the metal was decreased from 445 ppm to 0.09 ppm using 20% slag containing 81% SiO_2, 13% B_2O_3, 4% Na_2O, 2% Al_2O_3 and 0.5% K_2O. Klevin and Harris(20) used a 25 pound induction furnace to remelt contaminated stainless steel wastes. It indicated that uranium surface contamination can be removed from stainless steel, copper and nickel down to approximately 1 ppm by remelting.

The removal of U and Pu from stainless steel versus normal steel is only effected by the differences in the activity coefficient of the contaminants in various metals. Normally for metals the activity coefficients do not vary much, less than a factor of two, therefore the refining ability of a given slag will be similar in both cases.

Copper

The equilibrium distribution of uranium between copper and a slag containing 40% SiO_2, 30% CaO, 20% Al_2O_3 and 10% CaF_2 was investigated at 1500°C.(12) The final ingot uranium level was 0.083 ppm when 10% slag is used. The decontamination factor of 6×10^3 was obtained. Similar type of experiments were also carried out by Heshmatpour and Copeland(14). The experimental results are summarized in Table 6. The data in Table 6 indicates that uranium was effectively removed from copper regardless of the flux composition and the degree of decontamination was, again, not too sensitive to the slag composition, although fluid slags with high silica contents were more effective in reducing the uranium level to 0.1 to 0.8 ppm.

The removal of uranium from the other metals such as aluminum, lead, tin, zinc and lead-tin alloys was also investigated. This information can be found in the report by Heshmatpour and Copeland.(14)

CONCLUSIONS

Large quantities of metal wastes are produced annually by nuclear fuel processing and nuclear power plants. It is required that retrievable storage of all metallic wastes containing uranium and transuranic elements above a certain level be done. Because of the high cost of this operation it is important to develop an effective decontamination and volume reduction method. A literature survey was carried out to find the available information on the decontamination and volume reduction of radioactive metallic wastes. The emphasis was given to the thermodynamics and kinetics of the removal of uranium and transuranic elements from metallic wastes such as mild steel, stainless steel and copper. The data in the literature indicates that it is possible to remove uranium and transuranic elements from steel, stainless steel and copper using a melt refining technique. Depending on the waste properties it is expected that up to 30% volume reduction will occur during this operation. Processed steel can be casted and used for many applications. Based on the data available in the literature it can be concluded that the kinetics and thermodynamics of the removal has not been completely investigated. The thermodynamics of uranium in liquid steel and uranium oxide in the slags under consideration is unknown. The effect of the temperature was not investigated in detail. In order to obtain the best operating conditions for a possible large scale operation it is necessary to have more information on the kinetics and thermodynamics of the system.

FUTURE WORK

Despite the considerable amount of work cited in the report, most of it was not done under controlled conditions and useful thermodynamic data was not obtained from which a process could be optimized. Fundamental information on the activity coefficients of the contaminants in iron and various slags is required at the very least the partition ratio for given slags must be determined. Since it maybe difficult to use U or Pu in a safe manner surrogates with similar behavior could be used. in order to accomplish this the following work is suggested.

- In order to select an effective slag composition it is important to know which uranium compounds exist in the slag. If removal occurs, according to equations (10) and (11) where uranium monomers such as U^{4+} and UO^{2+} show basic behavior, it is necessary use an oxidizing acidic slag. On the other hand, if uranium exists as UO_3^{2-} and UO_4^{4-} which are acidic in nature an oxidizing basic slag should be used. Therefore, two experiments will be made using basic slags and two experiments using very acidic slags.
- Reactions (10), (11), (12) and (13) indicate that the removal of contaminants is favored at high oxygen potential. In order to determine the affect of oxygen potential a few experiments will be carried out with slags having a constant CaO/SiO_2 ratio containing different levels of FeO. The distribution ratios should increase with FeO.

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Table 1. Results on smelting experiments on iron containing 500 ppm uranium.(13)

Temp. (°C)	Time (hr)	Crucible Material	Uranium in Metal, ppm	Decon. Factor	Slag Composition (wt.%)
1580	0.5	Alumina	85-100	5	none
1580	0.5	Alumina	2.1-2.5	220	40 SiO_2-40 CaO-20 Al_2O_3
1680	0.5	Alumina	0.52	980	40 SiO_2-40 CaO-20 Al_2O_3
1580	1.2	Alumina	0.84	610	40 SiO_2-40 CaO-20 Al_2O_3
1580	5.0	Alumina	0.52	1000	40 SiO_2-40 CaO-20 Al_2O_3
1580	0.5	Alumina	4.5	110	50 SiO_2-50 CaO
1580	0.5	Alumina	0.26	1960	40 SiO_2-40 CaO-20 CaF_2
1580	0.5	Magnesia	0.40	1270	40 SiO_2-40 CaO-20 Al_2O_3

Table 2. Experimental results on removal of uranium from mild steel.(14)

Uranium, ppm in Metal	Urainum, ppm in slag	W_{Slag}/W_{Metal}	Slag Composition, wt.%
0.84	1210	10/100	70 CaO, 10 SiO_2, 20 Fe_2O_3
0.70	322	10/100	40 CaO, 40 SiO_2, 20 Fe_2O_3
1.08	1640	10/100	70 CaO, 20 SiO_2, 10 Fe_2O_3
0.14	1780	10/100	50 CaO, 40 SiO_2, 10 Fe_2O_3
2.72	1440	10/100	65 CaO, 35 SiO_2
0.21	162	10/100	25 CaO, 75 SiO_2
0.44	1290	50/500	60 CaO, 25 Al_2O_3, 10 SiO_2, 5 CaF_2
0.03	150	50/500	60 CaO,10 Al_2O_3, 25 SiO_2, 10 CaF_2
0.01	3710	50/500	50 CaO,25 Al_2O_3, 10 SiO_2, 10 CaF_2, 5 Fe_2O_3
0.91	1090	50/500	50 CaO,10 Al_2O_3, 25 SiO_2, 10 CaF_2, 5 Fe_2O_3
0.16	715	50/500	60 CaO, 35% SiO_2, 5 CaF_2
0.10	1400	50/500	80 SiO_2, 13 B_2O_3, 4 Na_2O, 1 K_2O, 2 Al_2O_3

Table 3. Experimental results on removal of plutonium from mild steel at 1600°C.(18)

Uranium, ppm in Metal	Urainum, ppm in slag	W_{Slag}/W_{Metal}	Slag Type%
0.20	1470	20/200	Borosilicate
0.11	2790	20/200	Blast Furnace
0.20	2360	20/200	High Silica
0.05	2280	10/200	Borosilicate
0.30	2670	10/200	Blast Furnace
0.20	3970	10/200	High Silica
2.00	942	20/200	Basalt
0.06	30	10/200	High Silica*

Borosilicate slag: 80% SiO_2, 13% B_2O_3, 4% Na_2O, 2% Al_2O_3, 1% K_2O.

Blast Furnace slag: 40% CaO, 30% SiO_2, 10% Al_2O_3, 15% Fe_2O_3, 5% CaF_2.

High silica slag: 60% SiO_2, 30% CaO, 10% Al_2O_3

Basalt slag: 42% SiO_2, 8% Al_2O_3, 26% Fe_3O_4, 12% CaO, 6% MgO, 4% Na_2O, 2% K_2O.

* Double refined with fresh slag.

Table 4. Experimental results on removal of uranium from stainless steel.(14)

Uranium, ppm in Metal	Urainum, ppm in slag	W_{Slag}/W_{Metal}	Slag Composition, wt.%
2.52	346	50/500	60 CaO, 40 Al_2O_3
2.39	231	50/500	60 CaO, 10 SiO_2, 25 Al_2O_3, 5 CaF_2
0.28	73	50/500	65 CaO, 35 SiO_2
0.01	2260	50/500	20 CaO, 75 SiO_2
0.05	5070	25/500	50 CaO, 30 SiO_2,10 Al_2O_3, 5 Fe_2O_3, 5 CaF_2

Table 5. Experimental results on removal of plutonium from stainless steel at 1600°C.(18)

Uranium, ppm in Metal	Urainum, ppm in slag	W_{Slag}/W_{Metal}	Slag Type%
0.60	1120	20/200	Borosilicate
0.30	1700	20/200	Blast Furnace
0.42	2240	20/200	High Silica
0.75	3680	10/200	Borosilicate
0.21	2640	10/200	High Silica
0.5	4880	10/200	Blast Furnace
0.04	140	10/200	Borosilicate*

Borosilicate slag: 80% SiO_2, 13% B_2O_3, 4% Na_2O, 2% Al_2O_3, 1% K_2O.

Blast Furnace slag: 40% CaO, 30% SiO_2, 10% Al_2O_3, 15% Fe_2O_3, 5% CaF_2.

High silica slag: 60% SiO_2, 30% CaO, 10% Al_2O_3.

* *Double refined with fresh slag.*

Table 6. Experimental results on removal of uranium from copper.(14)

Uranium, ppm in Metal	Urainum, ppm in slag	W_{Slag}/W_{Metal}	Slag Composition, wt.%
0.13	934	10/100	25 CaO, 25 Al_2O_3, 50 SiO_2
0.37	341	10/100	20 CaO, 20 Al_2O_3, 60 SiO_2
0.11	4110	10/100	15 CaO, 15 Al_2O_3, 70 SiO_2
0.14	213	10/100	35 CaO, 65 SiO_2, 15 CuO
0.54	265	10/100	20 CaO, 65 SiO_2, 5 CuO, 10 Al_2O_3
0.45	390	10/100	30 CaO, 55 SiO_2, 5 CuO, 10 Al_2O_3
0.83	1813	10/100	10 CaO, 75 SiO_2, 10 Al_2O_3, 5 Fe_2O_3
0.04	1273	10/100	10 CaO, 65 SiO_2, 10 Al_2O_3, 5 Fe_2O_3
0.25	943	10/100	30 CaO, 55 SiO_2, 10 Al_2O_3, 5 Fe_2O_3

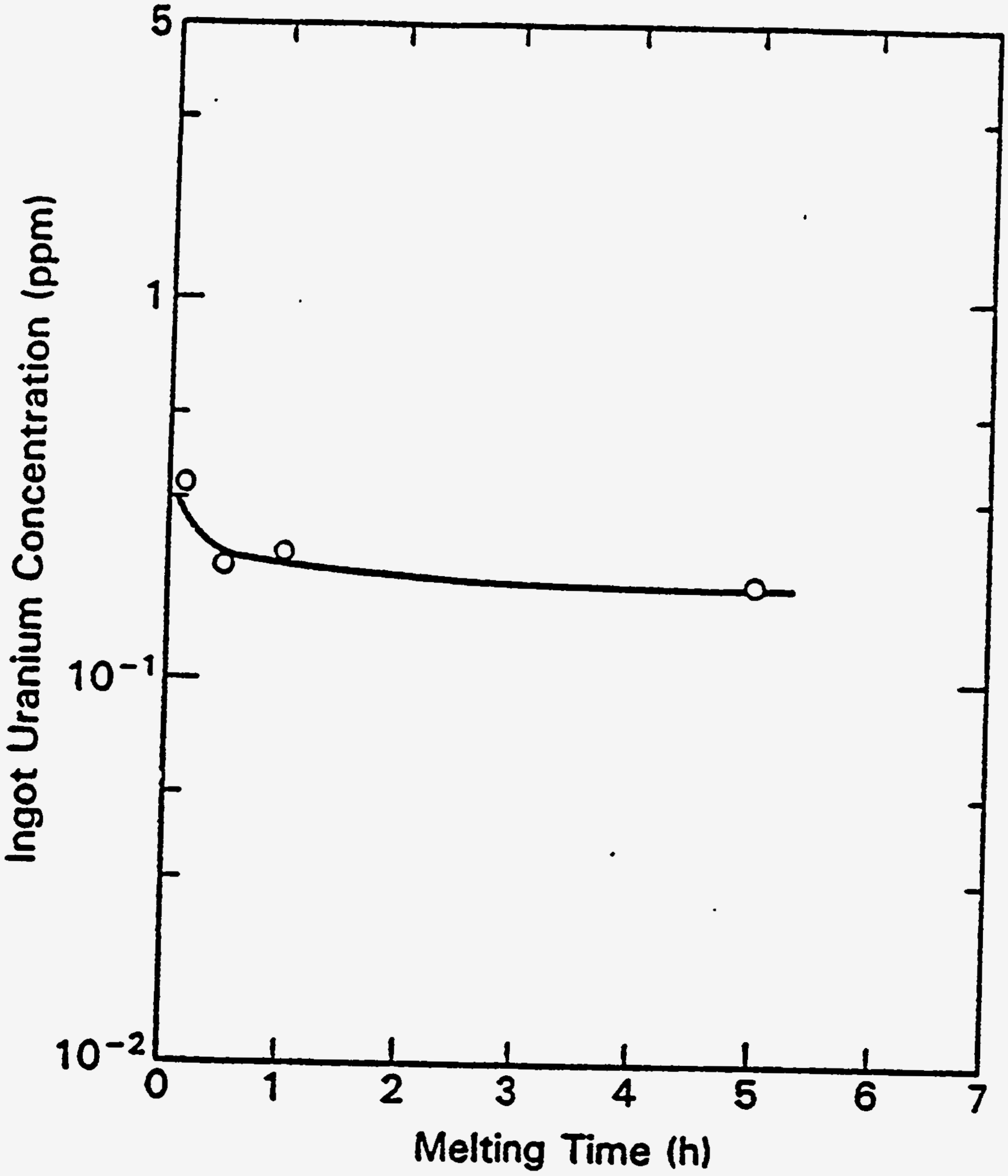

Figure 1. Uranium concentration as a function of time. Temperature = 1640°C. Slag contains 40% SiO_2, 40% CaO and 20% Al_2O_3.(11)

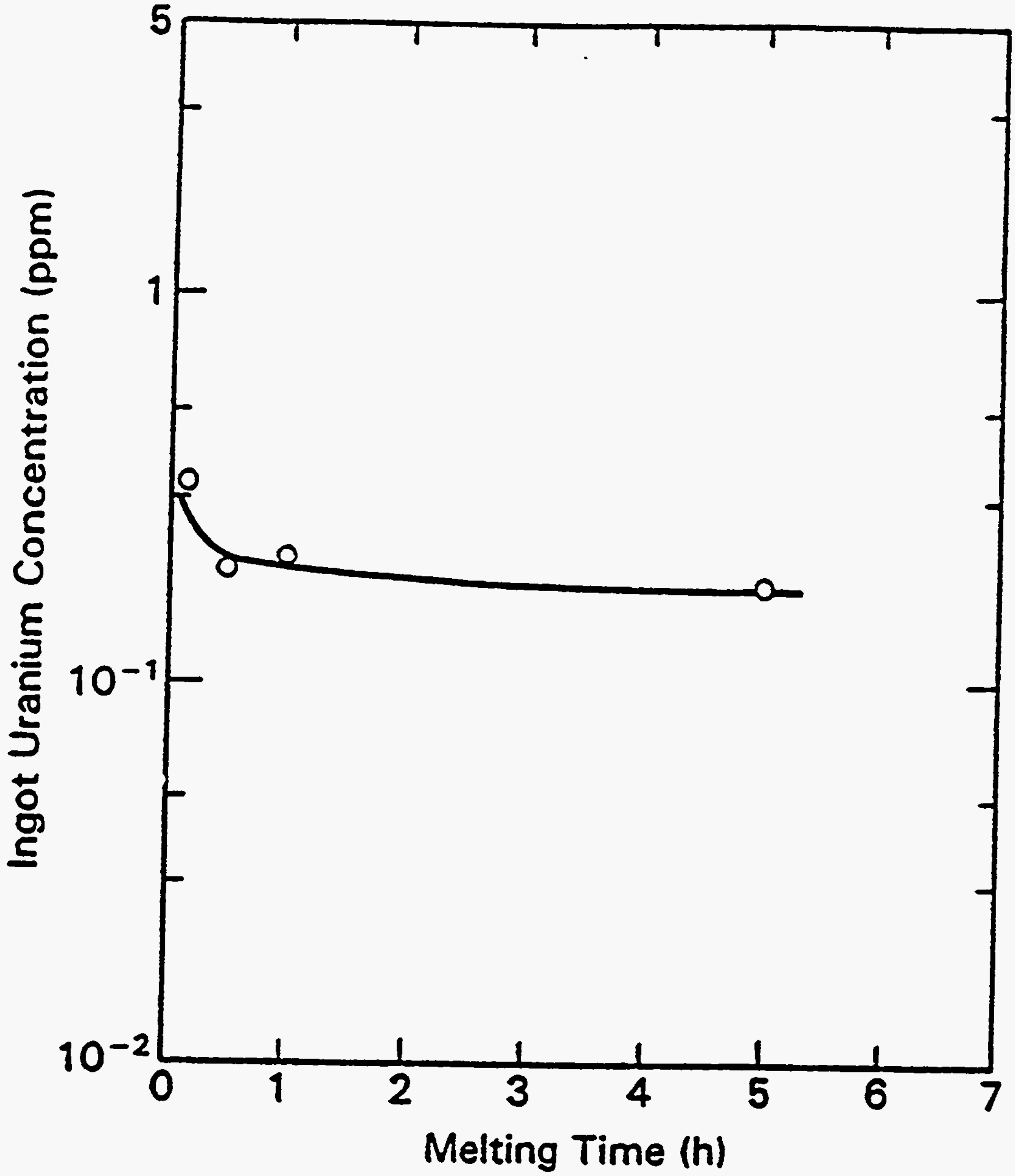

Figure 2. Effect of slag basicity (CaO + NiO or $CaF_2/SiO_2 + Al_2O_3$) on uranium concentration in a mild steel ingot.(11)

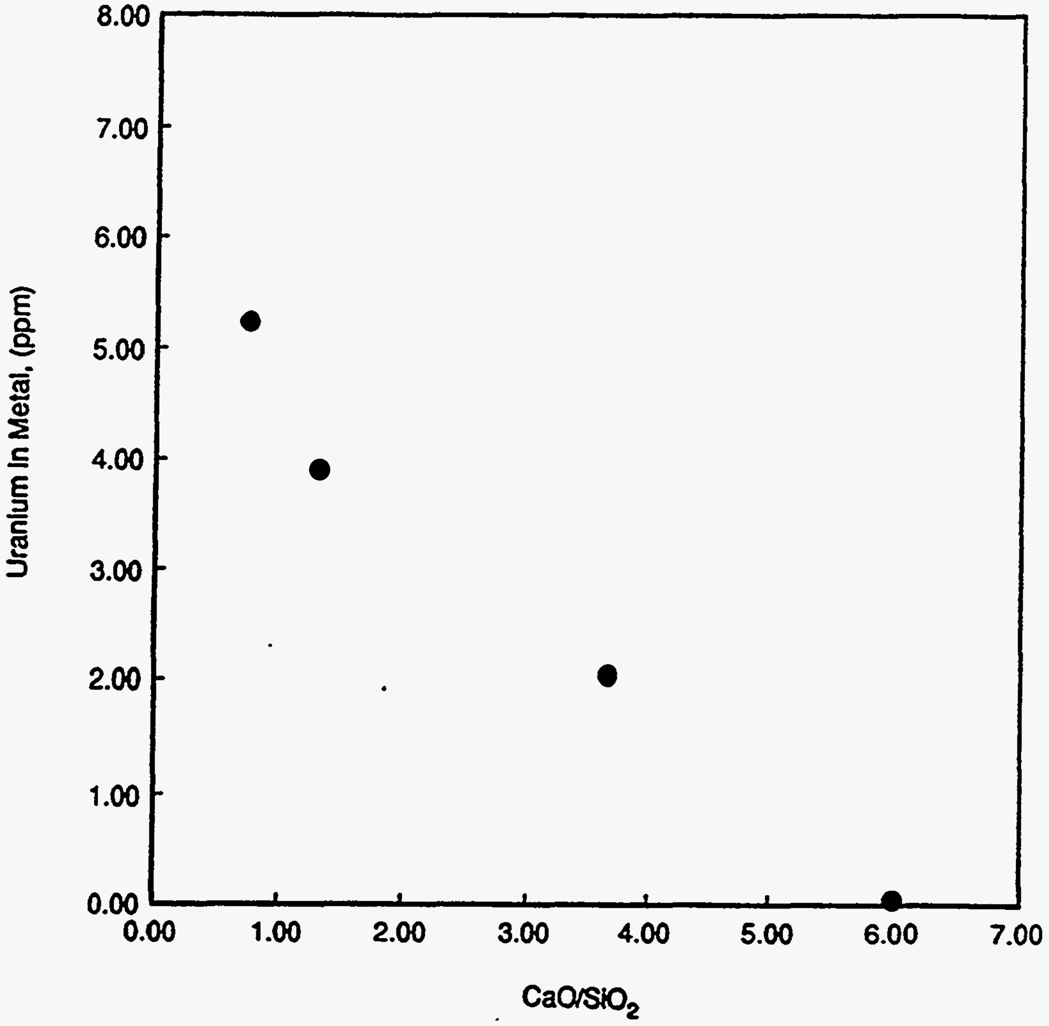

Figure 3. Effect of CaO/SiO$_2$ on uranium concentration in mild steel. Slag = CaO + SiO$_2$ + 30% Fe$_2$O$_3$ (initially).(14)