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Laura A. Worl^a; David Devlin^a; Dallas Hill^a; Dennis Padilla^a; F. Coyne Prenger^a

^a Los Alamos National Laboratory, Los Alamos, New Mexico, U.S.A.

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PARTICULATE CAPTURE OF PLUTONIUM BY HIGH GRADIENT MAGNETIC SEPARATION WITH ADVANCED MATRICES

**Laura A. Worl,* David Devlin, Dallas Hill,
Dennis Padilla, and F. Coyne Prenger**

Los Alamos National Laboratory, Los Alamos,
New Mexico 87545

ABSTRACT

A high performance superconducting magnetic separator is being developed for near single particle retrieval from low concentration field collected samples. Results show that maximum separation is obtained when the effective matrix element diameter approaches the diameter of the particles to be captured. Experimentally, we were able to capture very dilute levels of 0.2 to 0.8 μm PuO_2 particles with dodecane as a carrier fluid. The development of new matrix materials is being pursued through the deposition of nickel dendrites on an existing stainless steel matrix material. The new materials are promising for the submicron collection of paramagnetic particles. Results indicate that these new matrices contain a high number of capture sites for the paramagnetic particles. We have also derived a force-balance model that uses empirically determined capture cross section values. The model can be used to optimize the capture cross section and thus increase the capture efficiency. This enables the prediction of high gradient magnetic separator performance for a variety of materials and applications.

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INTRODUCTION

Magnetic separation has been introduced and developed for industrial,¹⁻³ medical^{4,5} and governmental⁶⁻⁸ applications as a selective separation technology. The application of magnetic forces for actinide separations has been developed for nuclear wastewater filtration,^{9,10} nuclear material recovery,^{11,12} high level waste treatment,¹³ and soil remediation.^{6,15} Currently, the Department of Energy (DOE) is developing an enhanced magnetic separation technology for the capture of sub-micron actinide particles from forensic samples of interest.¹⁶

The generation of high magnetic field gradients is critical in the development of an enhanced magnetic separation and is usually termed high gradient magnetic separation (HGMS). HGMS utilizes high magnetic field gradients generated from the magnetic induction of small matrix fibers within a magnetized volume. The progression of HGMS with advent of superconducting magnets and advanced matrix materials has made it feasible to effectively separate sub-micron sized weakly paramagnetic particles. Because actinide compounds are paramagnetic, Table 1, HGMS extraction of micron sized actinides and actinide contain-

Table 1. The Volume Magnetic Susceptibility of a Number of Compounds and Elements of Interest

Compound / Element	Volume Susceptibility x 10 ⁶ , (SI)
Paramagnetic	
FeO	7200
Pr ₂ O ₃	2422
Fe ₂ O ₃	1478
UO ₂	1204
Cr ₂ O ₃	844
NiO	740
Am	707
Pu	636
PuF ₄	488
PuO ₂	384
CuO	242
Th	83
UO ₃	41
Diamagnetic	
CaO	-1
ZrO ₂	-11
ThO ₂	-8
MgO	-11
SiO ₂	-14
Al ₂ O ₃	-18



ing particulates from a variety of host materials is now possible. Typically, HGMS separators consist of a high field solenoid magnet, the bore of which contains a fine-structured ferromagnetic matrix material. The matrix fibers locally distort the magnetic field generating high field gradients at the surface of the filaments. These areas then become trapping sites for paramagnetic particles and are the basis for the magnetic separation.

Because of the commercial availability of 45-micron diameter stainless steel wool, HGMS of micron sized materials has been demonstrated in the kaolin clay industry,¹⁶ for soil remediation,^{6,14,17} and for water treatment.¹⁸⁻²⁰ The magnetic capture of smaller particles (submicron) can be obtained by increasing the magnetic force between the fibers and the paramagnetic particles. One approach towards increasing the magnetic force is to reduce the size of the matrix filament, which is believed to be the most effective method of improving separator efficiency. Others²¹⁻²³ have examined submicron sized spheres for the collection of nanometer sized particles. In addition, fractionation methods for HGMS of nanometer particles are underdevelopment.^{24,25} We are investigating dendritic growth on surfaces to augment matrix surface topography in an effort to reduce the effective matrix element diameter.

The interaction of the magnetized matrix with the paramagnetic particles has been modeled by various approaches.²⁶⁻³² Our approach is semi-empirical combining a mechanistic analytical model with empirical input from controlled experiments. We have described a rate model^{14,33} that is derived from a force balance on individual paramagnetic particles in the immediate vicinity of a matrix fiber. The model assumes that if the magnetic capture forces are greater than the competing viscous drag and gravity forces, the particle is captured and removed from the flow stream. The rate model depends on the magnitude of the capture cross section (separation coefficient) as determined by the force balance on the particle. The model also includes the loading behavior of particles on the matrix fibers using the continuity equation²⁶ and we utilize capture rate data from the experimental results. The analytical model predicts the critical separation parameters for submicron particle collection presented here. It thus provides a predictive tool for HGMS tests conducted here, and can be used for designing both prototype and full-scale units for specific applications.

EXPERIMENTAL

The separator used for the studies described here was a six inch warm-bore superconducting high gradient magnetic separator (manufactured by Cyromagnetics, Inc.) with an 8 tesla maximum field strength. The baseline matrix material of extruded stainless steel wool was obtained from MEMTEC, Inc. The matrix body canister was designed and machined in our laboratories. The matrix body was packed to achieve a 94% void fraction. The experiments described here were



conducted with PuO_2 and were used to define the significant separation and magnet design parameters. PuO_2 was obtained from ongoing work in our laboratories. Specific ranges of micron and submicron particles of PuO_2 were obtained by sedimentation and decantation procedures, relying on the relationship that exists between settling velocity and particle diameter in Stokes Law.^{34,35} Particle sizes were verified by dynamic light scattering for particle size analysis.

The HGMS experiments have been described previously.¹⁵ For the HGMS experiments, a fixed volume of solution was pumped through the magnetic matrix using a peristaltic pump at constant flowrate. Typically, one pass through the matrix was performed using 100 ml of feed solution. Particle collection from the matrix was done with varied matrix rinsing procedures. Typically, back flushing of the magnetic matrix was done at zero magnetic field, with the flow direction reversed. Feed, effluent, and backwash samples were analyzed for contaminant concentration. Plutonium samples were analyzed by liquid scintillation with a Packard Instrument. In most cases, duplicate samples were analyzed to check consistency. Separation efficiencies were determined using the masses of the paramagnetic species determined by the total activity values from the feed and effluent solutions.

Nickel dendrites were deposited on the extruded steel wool matrix by chemical vapor deposition (CVD) of $\text{Ni}(\text{CO})_4$ described previously.³⁶ The reactor is a hot-wall reactor as opposed to the previous cold-wall system. This type of system is easier to scale for deposition on large mats. The extreme toxicity of nickel carbonyl required special precautions in the vapor deposition system. All the valves were pneumatically controlled so the reactor could be activated or shut down remotely. Vacuum lines and gas flush systems were also remotely controlled. The system was operated in a hood within a room, which was also operated on its own exhaust system at a negative pressure with respect to the building. Mass flow controllers regulated argon carrier gases. The nickel carbonyl flow was regulated using a calibrated rotometer. Rotometers are necessary because the reactive carbonyl tends to plate on the thermal sensor of the mass flow controller resulting in erroneous readings. The exhaust gases were passed through a hot scrubber at 500°C to consume unused nickel carbonyl leaving the reaction zone. During operation the lines were heated to 40°C to avoid condensation of carbonyl. The sample was placed in the middle of the reaction chamber. Reactant gases were injected through 0.25" stainless steel tube, which reaches near the center of the reactor. This maintains a high reactant gas velocity in the heated zone avoiding premature reaction. The reactor pressure was maintained with a throttle valve and controller. Processing parameters were adjusted to optimize the nickel dendrite distribution. The material was characterized by scanning electron microscopy. Magnetization data was collected using a Quantum Design MPMS-7 SQUID Magnetometer, making DC magnetization measurements.



RESULTS AND DISCUSSION

A high efficiency HGMS method for actinide collection is being developed for use in the analysis of nuclear materials. Because all actinide compounds and many fission products are paramagnetic, magnetic fields with high magnetic field gradients can be used to extract trace quantities of these materials from environmental sources or collected samples. Trace quantities of actinides can hydrolyze under environmental conditions to yield colloidal or submicron hydrous oxide particles.³⁷ Much of the colloidal material will remain suspended in solutions due to the near neutral pH of the water and small particle size of the hydrolyzed metals. In addition, small particulates can be electrostatically or mechanically retained on debris and soils, which can be liberated for collection. The challenge here is to effectively collect tracer quantities of actinide submicron paramagnetic particles from a variety of materials. This challenge demands improvement of HGMS and we have focused our efforts on the design and testing of new matrix materials to meet the stringent collection requirements.

To develop HGMS for the collection of minute levels of plutonium particles, we were interested in determining the number of particles in a given solution volume. Assuming a spherical particle with a known density, the concentration of a specific diameter particle can be calculated. The single particle concentrations for plutonium oxide at various particle diameters are shown in Table 2. For a concentration of 4 parts per billion (ppb) of a 0.8 micron particle, there would be approximately 1300 plutonium oxide particles in 1 ml of solution. In the initial feed concentrations for our experiments, we typically use starting plutonium concentrations in the range of 2-15 ppb and an initial volume of 100 ml. This would translate to approximately 6500 particles with a 0.8 micron diameter in the feed solution. For this application, we attempt to extract the majority of the 6500 particles by an enhanced HGMS method.

Separation Variables

In the HGMS process, there are at least a dozen variables affecting the performance of the magnetic separation. The variables are described as either mate-

Table 2. The Table Depicts the Calculated Number of PuO₂ Particles in Concentrations of 4 Parts Per Billion to 3 Parts Per Trillion

[PuO ₂] (ppb)	5 mm Particle Count	0.8 mm Particle Count	0.2 mm Particle Count
4	5	1300	83330
0.006	0.008	2	125
0.003	0.004	1	62



rial characteristics (particle size distribution, paramagnetic particle or diamagnetic solids concentration, magnetic susceptibility, and carrier fluid pH and viscosity) or separator parameters (matrix element size and spacing, applied magnetic field strength, matrix material, residence time and superficial flow velocity). The significance of these parameters has been discussed elsewhere.^{15,33} For the application described here, the particle size and magnetic susceptibility of the species of interest appear to be the most important material variables. The magnetic force on the particle is proportional to the particle magnetic susceptibility and is given by: $F_m = \gamma B \text{ grad} B V / \mu_0$, where γ is the volume magnetic susceptibility, B is the magnetic induction, V is the volume of the particle and μ_0 is the permeability of free space. A particle with a high paramagnetic susceptibility has a high attractive magnetic force and should be readily separated in the proper magnetic environment. We have found that the separation effectiveness is optimized when the effective matrix element size approaches the paramagnetic particle size. This is the primary motivation to reduce matrix element size in the interest of capturing smaller particles. For complex matrix geometries, this can be expressed as an effective matrix diameter that describes the area of the matrix capture. In our baseline tests, we have used MEMTEC extruded stainless steel wool with a 20-25 μm diameter. Residence time in the matrix and superficial velocity of the slurry has also been determined to be significant separation variables. For these studies, our testing shows a minimum residence time of 15 seconds and a superficial velocity near 1 cm/s is required for effective performance.

Model Results

Previously, we have described an analytical model based on geometry comprised of a highly porous bed of ferromagnetic parallel cylinders describing the matrix elements. The model was developed and validated with a systematic series of soil remediation tests.³³ The model is formulated from a force balance on the paramagnetic particle and an expression for the capture rate. The latter formulation describes the distribution of particles in the magnetic matrix and can be used to generate breakthrough curves for the matrix. In the experiments described here, the concentration of the plutonium particles is so dilute that breakthrough is not an issue as matrix capacity far exceeds the particle count. The rate of particle capture is related to the capture cross section of the magnetic separator. We determine the capture cross section empirically from HGMS experiments. From the previous work, we found that the capture cross section is significantly less than one, which implies that the particles can be swept around the matrix elements by the fluid flow even if the particles occupy an upstream capture zone defined by the projected area of the matrix element.³³ The capture cross section was found to be proportional to the magnetic force and the particle diameter and inversely propor-



tional to the effective matrix element diameter and the effective matrix element spacing. This indicates that the improved capture of submicron particles can be achieved by decreasing the effective matrix element diameter and the effective spacing between the matrix elements.

Two variables in an HGMS separation were compared with results from the model. Figure 1 shows the predicted PuO_2 HGMS effectiveness for matrix wire diameters of 5, 15 and 25 μm . For PuO_2 particles below 0.2 μm , HGMS separation is much more effective with smaller wire diameters. For the same matrix size the effect of changing the applied magnetic field from 7 T to 2.5 T is also shown in the figure. This change is not nearly as significant as the effect of reducing the matrix size. At 2.5 T the matrix material is nearly saturated magnetically, and any further increase in the applied field appears to have a small effect on the separation. Increasing the matrix residence time will also increase the separation effectiveness, yet at extremely low superficial velocities the resulting particle settling can be a significant problem. To greatly enhance the HGMS performance and to achieve a single particle collection threshold, it has become apparent through model predictions and experimental data that the matrix fiber diameter is a crucial separation variable.

HGMS Matrix Material

Tests have been initiated to develop new HGMS matrix materials with sub-micron sized fiber diameters. One approach, described here, is the controlled

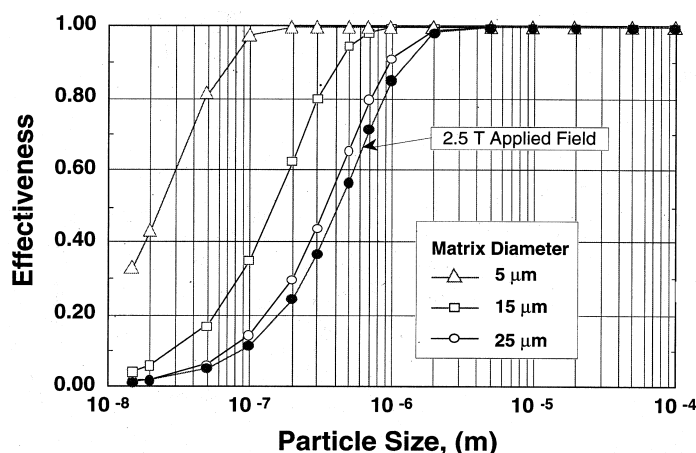


Figure 1. Model results for HGMS separation effectiveness for three matrix fiber diameters. The prediction is based on an applied field of 7.0 T (open points) or 2.5 T (closed points); the superficial velocity is 1 cm/s; the carrier fluid is dodecane.



growth of micron sized nickel dendrites on stainless steel wool fibers by chemical vapor deposition. The foundation matrix material consists of magnetic stainless steel fibers that contain multiple ridges formed from an extrusion process. These ridges form a continuum of sharp axial edges that reduce the effective matrix element diameter by generating large magnetic field gradients that serve as trapping sites for the submicron particles. This extruded material is the baseline matrix both for nickel dendrite deposition and for the HGMS experiments described below.

High aspect ratio dendrite nickel, believed to further reduce the effective matrix element diameter, was grown by chemical vapor deposition (CVD) via the decomposition of nickel tetracarbonyl³⁶ on the extruded stainless steel matrix material. The desired outcome is a controlled CVD of dendrites on the base material. The basis of the technique is the reversible reaction between nickel and carbon monoxide: $\text{Ni(s)} + 4\text{CO(g)} = \text{Ni(CO)}_4\text{(g)}$. Several groups have defined the kinetics that controls the complex decomposition reaction to generate nickel metal.³⁸⁻⁴⁰ The growth of dendrites is typically the result of boundary layer effects or mass transport limitations. The diffusion limited transport of reactants and products through the boundary layer leads to a lower concentration of reactants and a build up of products at the surface. The factors affecting boundary layer formation and growth rate include pressure, concentration of reactants, temperature, flow rate, reactor geometry and substrate geometry. The nucleation density is also important in determining microstructure. Unfortunately many of the same parameters that control growth rates also control nucleation rates and it can be difficult to independently control the two processes. In our present application we would like to reduce the nucleation rate and possibly determine the temperature regimes where either growth or nucleation dominates.

We have explored a number of parameters related to the deposition of nickel dendrites with the aim of developing some degree of control over the microstructure. We have preliminarily explored variations in temperature, pressure, deposition time and surface condition. High temperature results in a higher growth rate, and in the case of Ni(CO)_4 reduced pressures favor decomposition. Figure 2 shows the weight gain at two different pressures 5 and 10 Torr and 130°C for 120 min. The growth curves are exponential as expected for dendritic growth and the rate constant at 10 Torr is about twice the value of the 5 Torr run. This is consistent with the apparent first order kinetics as observed previously.³⁹

The difference in morphology obtained with different pressure regimes is shown in Figure 3. The lower pressure appears to show larger dendritic features than that obtained at the higher pressure. This may suggest that lower pressures result in a lower nucleation density. Variations in morphology also occur with deposition temperature. Higher temperature depositions (200°C - 60°C) result in finer, smoothed microstructure not characteristic of the dendrite needles. Our preliminary results suggest that controlling the deposition time allow for the growth of short dendrites desired for HGMS experiments.

The magnetic properties of the matrix material influence the magnetic field



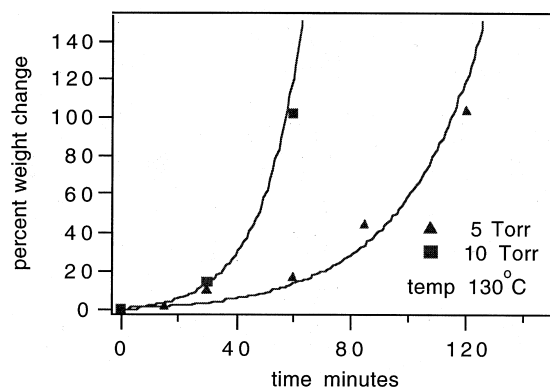


Figure 2. The effect of pressure on the deposition of nickel on steel wool matrix material.

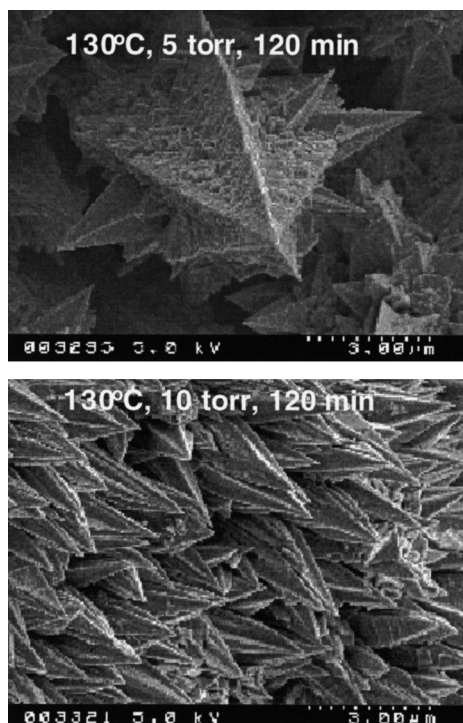


Figure 3. The variation in dendrite morphology for materials processed at different CVD pressures.

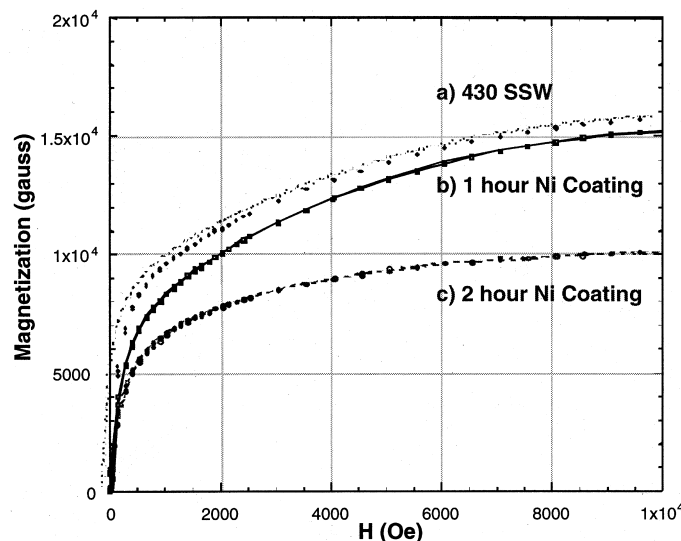


Figure 4. Magnetization curves for three HGMS matrix materials: a) baseline extruded stainless steel wool – 430 series, b) nickel dendrite coating at a one hour deposition and c) nickel dendrite coating at a two hour deposition time. Chemical vapor deposition was done at 130 °C and 5 mtorr.

and, therefore, affect HGMS experimental design. Thus, magnetization measurements of the matrix materials were obtained to determine saturation magnetization values for the nickel-coated materials, see Figure 4. Ferromagnetic stainless steel wool (400 series) typically saturates around 2.0 tesla,¹ and nickel is reported to saturate at 0.6 tesla. The concern with a nickel dendrite coated matrix was that the saturation magnetization would be reduced from the baseline ferromagnetic extruded stainless steel wool matrix value of 2.1 tesla. From Figure 4, a one-hour deposition time for the growth of nickel dendrites generated a minor decrease in the total magnetization behavior of the stainless steel matrix foundation. As the dendrite deposition time increases the saturation magnetization occurs at a lower magnetization. This seems consistent with the larger fraction of Ni present. This indicates that it may be desirable to limit the total mass of nickel dendrites on the surface of the ferromagnetic matrix materials so that the saturation magnetization of the magnetic stainless steel base is not compromised.

Typically, the nickel dendrite structures were uniformly deposited on the surface throughout the wool structure. The length of the dendrites is 1 – 2 orders of magnitude smaller than the diameter of the wool fiber substrate. The resulting matrix material has a very high surface area containing fine points that should



greatly enhance the high magnetic field gradients, reducing the effective matrix diameter, for better HGMS performance.

HGMS Results

The capture of plutonium oxide particles from dilute samples is a difficult challenge. For example, one ml concentration of a $0.8\ \mu\text{m}$ particle of plutonium oxide is 3 ppt. We must be below this limit for submicron single particle collection. To obtain a concentration of less than $3 \times 10^{-11}\ \text{M}$ (~ 3 ppt) of plutonium oxide, the small but finite solubility of PuO_2 in water demands the use of dodecane as a carrier fluid because of the oxide insolubility in long chain hydrocarbons.

HGMS experiments were conducted with the baseline extruded steel matrix material and with the nickel dendrite coated materials described above. The HGMS results are illustrated in Figure 5. The bar graph depicts the

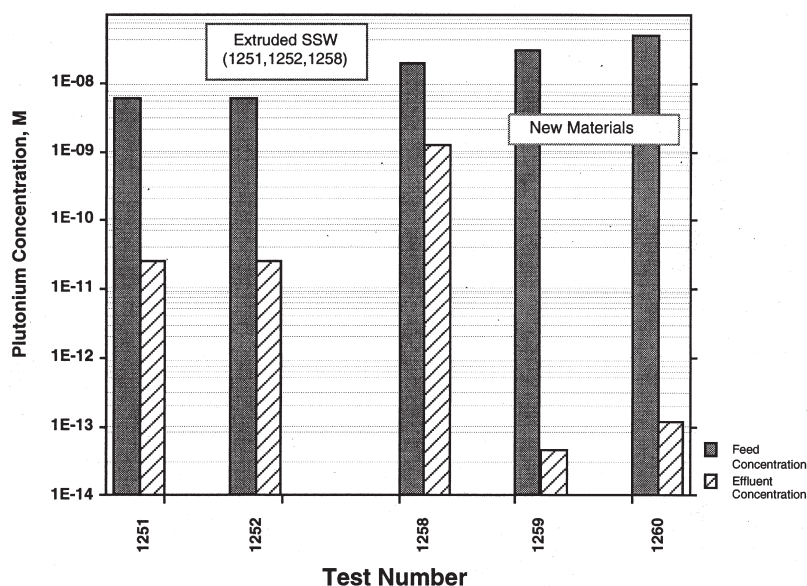


Figure 5. The bar graph depicts the plutonium concentration in feed and effluent for 5 HGMS experiments with varied matrix materials. All experiments were conducted in dodecane as a carrier fluid with a 0.2 to $0.8\ \mu\text{m}$ PuO_2 particle size range. Experiment numbers 1251, 1252 and 1259 used the baseline extruded stainless steel matrix material. Experiment numbers 1260 and 1261 used the nickel dendrite coated matrix material. Runs 1251 and 1252 used a 7.0 tesla magnetic field strength and runs 1258-1260 were conducted at 2.0 tesla field strength.



plutonium concentration in the HGMS feed and effluent from one pass through the magnetic separator. All experiments were conducted with a 0.2 to 0.8 μm PuO_2 particle size range. Experiment numbers 1251, 1252 and 1259 used the baseline extruded stainless steel matrix material. Experiment numbers 1260 and 1261 used the nickel dendrite coated matrix material. The applied magnetic field strength was also varied in the experimental series. Runs 1251 and 1252 used a 7.0 tesla magnetic field strength and runs 1258 - 1260 were conducted at a 2.0 tesla field strength.

Plutonium oxide particles sized from 0.2 – 0.8 microns were removed below 10^{-13} M in the effluent solutions from the new matrix materials. The separation efficiencies for tests 1260 and 1261 were over 99.99%. The nickel coated matrix material performed significantly better than the baseline material, highly enhancing the HGMS performance. In the baseline tests with the extruded stainless steel material the final solutions were $\sim 3 \times 10^{-11}$ M or 6 ppt. From Table 2, there are predicted to be 2 particles in one ml of solution at this concentration. Yet under these conditions for the smaller 0.2 μm particles, over 125 particles still remain in one ml of the effluent liquid. On the other hand, the nickel coated matrix material is able to effectively collect all of the submicron particles from the initial solution.

The effect of applied magnetic field strength on the HGMS performance is also apparent with the new materials. It can be concluded from the data that the new materials can efficiently collect submicron particles at a lower applied field (2.0 tesla) compared to the baseline matrix material. This also agrees with the analytical model results, which indicate that the matrix element size and shape play a more significant role in HGMS than the applied field strength, Figure 1.

SUMMARY

We have derived an analytical HGMS model based on a particle force balance and matrix loading that uses empirically determined capture cross section values. The model can be used to optimize the capture cross section and thus increase the capture efficiency in HGMS experiments. Model results show that maximum separation effectiveness is obtained when the effective matrix element diameter approaches the diameter of the particles to be captured.

The development of new matrix materials is being pursued through the deposition of nickel dendrites on the existing magnetic stainless steel matrix material. Our examination of the deposition parameters has been preliminary, and a correlation between deposition parameters and the resulting microstructure has not yet been attained. Despite the inconclusive results we have observed that the microstructure is relatively insensitive to surface condition, and furthermore that larger dendrites or a lower density of dendrites seems to be favored by lower pressures. The new materials are highly promising for the collection of submicron



paramagnetic particles. Experimentally, we obtained an enhanced HGMS performance with the nickel coated matrix materials, approaching the removal of all minute particles in a solution of 0.2 μm PuO_2 particles and dodecane as a carrier fluid.

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PARTICULATE CAPTURE OF PLUTONIUM

1349

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