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Advanced Technologies for Decontamination and Conversion of Scrap Metal

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Advanced Technologies for Decontamination and Conversion of Scrap Metal

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Introduction

The DOE has begun to decommission parts of the vast US nuclear weapons complex. This effort will include the disposition of large amounts of radioactively contaminated scrap metal including but not limited to carbon steel, stainless steel, .nickel, copper, aluminum, and Monel. Diffusion plant barrier nickel from the Oak Ridge, Paducah, and Portsmouth diffusion plants accounts for nearly 40,000 tons of the scrap and 80 percent of the total scrap value. The porous nature of the nickel barrier makes decontamination difficult. Technetium-99, which is present in the nickel barrier material, is known to be particularly troublesome to remove. Traditional chemical and mechanical means have proven in-effective at removing this contaminate from the barrier nickel.

Objectives

In October 1993, Manufacturing Sciences Corporation was awarded DOE contract DE-AC21-93MC30170 to develop and test recycling of radioactive scrap metal (RSM) to high value and intermediate and final product forms.[*] This work was conducted to help solve the problems associated with decontamination and reuse of the diffusion plant barrier nickel and other radioactively contaminated scrap metals present in the diffusion plants.

Options available for disposition of the nickel include decontamination and subsequent release or recycled product manufacture for restricted end use. Both of these options are evaluated during the course of this research effort. Work during phase I of this project successfully demonstrated the ability to make stainless steel from barrier nickel feed. This paved the way for restricted end use products made from stainless steel. Also, after repeated trials and studies, the inducto-slag nickel decontamination process was eliminated as a suitable alternative.

Approach

Phase II of the project titled "Advanced Technologies for Decontamination and Conversion of Scrap Metals" is a continuation of the Phase I work. As in Phase I, the Phase II efforts were to help solve problems associated with the disposition of large quantities of scrap metals from Department of Energy (DOE) decommissioning efforts of nuclear weapons facilities, specifically the diffusion plants.

By the end of phase I of the project, inducto-slag technology had been eliminated as a viable process for nickel decontamination. Electro-refining appeared to be a promising technology for decontamination of the diffusion plant barrier material. Goals for phase II included conducting experiments to facilitate the development of an electro-refining

[*] Research sponsored by the U.S. Department of Energy's Morgantown Energy Technology Center, under contract DE-AC21-93MC30170 with Manufacturing Sciences Corporation, 804 Kerr Hollow Rd., Oak Ridge, TN 37830; fax 423-481-3142

process to separate technetium from nickel. In parallel with those activities, phase II efforts were to include the development of the necessary processes to make useful products from radioactive scrap metal. Nickel from the diffusion plants as well as stainless steel and carbon steel could be used as feed material for these products.

Nickel decontamination

Introduction

The decommissioning of DOE uranium enrichment facilities has produced large stockpiles of radioactively-contaminated scrap metals. These metals are of considerable value if they can be economically decontaminated to the extent that they can be sold to unrestricted markets. A large stockpile (estimated as high as 40,000 tons) of high purity nickel from the decommissioning of uranium diffusion facilities has the greatest potential value of these scrap metals, since nickel has had an average commodity value of approximately $4/lb. This nickel is contaminated with technetium and uranium along with trace amounts of transuranic elements (Np, Pu, and other actinides). The criterion for release of this material to non-regulated markets in several countries in Europe is in the range of 0.4 to 1 Bq/g. In its present state, the contaminated nickel may have an activity of up to 5000 Bq/g, due to the technetium content alone.[1] At this time, no criterion exists for the unrestricted release of bulk contaminated metal in the United States. If such a criterion is established, it will probably be in the range of 1 Bq/g.

Two processes have been investigated for the decontamination of this material: melt refining, and electrorefining. In melt refining, the contaminated nickel is melted, usually in an induction furnace, in the presence of an oxidizing flux; the mechanism of the purification is that of liquid-liquid extraction. In electrorefining, the nickel is dissolved electrolytically (as an anode) in an acidic aqueous electrolyte; purified nickel is deposited in its metallic state at the cathode. The electrorefining cell potential is selected so that the impurities are left in the electrolyte and nickel is selectively deposited at the cathode. This process is used commercially on a large scale for the purification of nickel and copper.

Studies of these purification methods have shown that both electrorefining and melt refining are effective at reducing the levels of transuranic elements to below detectable levels, but are not able significantly to reduce the amount of technetium in the nickel. The Westinghouse/SEG study[2] showed that when technetium was present in the electrorefining electrolyte, technetium was always co-deposited with nickel at the cathode in a ratio equal to or greater than that which existed in the electrolyte.

Oxidation-Reduction Reactions of Technetium and Nickel in Aqueous Solution

In order to understand the electrolytic separation of nickel and technetium, the oxidation/reduction potentials of these elements must be considered carefully. In an acidic solution, Tc predominately exists in a heptavalent form as pertechnetate ions (TcO_4^-) and nickel exists as Ni^{++}. The reduction of these ions to the metallic state is governed by the following half reactions:

$$TcO_4^- + 8H^+ + 7e^- = Tc + 4H_2O \qquad e^o = 0.477\ V \quad (1)$$

$$Ni^{++} + 2e^- = Ni \qquad e^o = -0.250\ V \quad (2)$$

The magnitude of the standard potential indicates the driving force of the reaction to proceed from left to right. Therefore, the pertechnetate ions are reduced more readily to the metallic state than the nickel ions. Additionally, the standard potentials indicate that metallic nickel will act as a strong reducing agent with respect to pertechnetate ions. Combining the two previous half reaction equations yields the following reaction:

$$2TcO_4^- + 16H^+ + 7Ni = 7Ni^{++} + 2Tc + 8H_20 \quad \Delta e^o = .477 + .25 = .727V \quad (3)$$

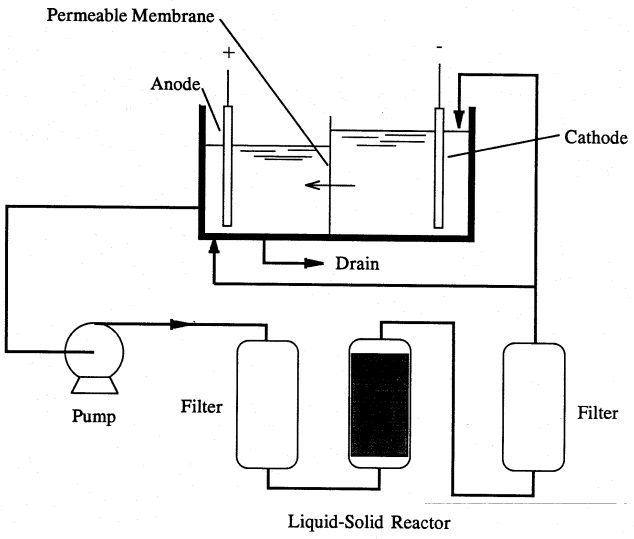
This is a metal displacement reaction, which occurs spontaneously, when metallic nickel is in contact with an acidic solution containing pertechnetate ions. The reduction of pertechnetate ions has been reported not only with nickel but also with copper, zinc, lead, mercury and tin;[3] all of which have reduction potentials less than that of technetium. This reaction is the reason why technetium is always co-deposited with nickel when it is present in the cathodic electrolyte.

The COVO Process

Covofinish has developed a process for the decontamination of radio-contaminated nickel diffusion barrier material (U.S. Patent 5,458,755) in which the technetium is removed from the electrolyte by a metal displacement reaction with nickel. Metals other than nickel could also be used to reduce the technetium. Nickel, however, is the ideal metal for this application, since the nickel ions generated by the displacement reaction are not a contaminant to the solution.

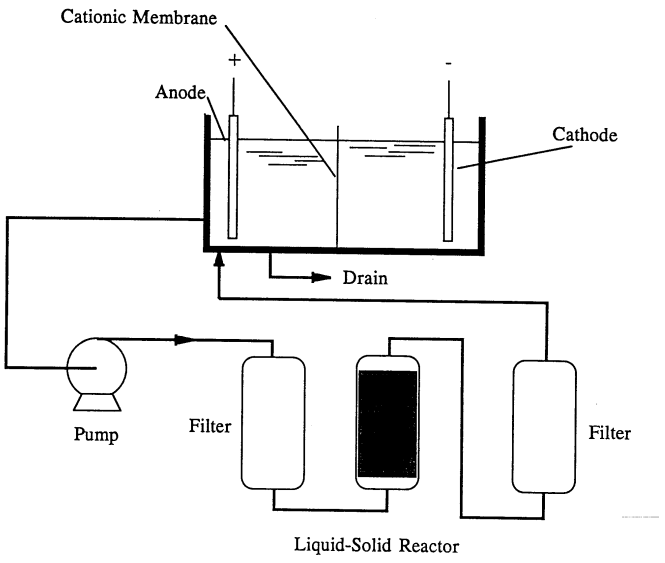
This process would eliminate the need for ion exchange or other secondary treatments which have been proposed for the removal of technetium. The use of the displacement reaction to remove pertechnetate ions from the electrolyte can be incorporated into various electrorefining processes. The electrorefining process must successfully partition the ^{99}Tc contaminated anodic electrolyte from the cathodic electrolyte, while allowing the free passage of current from cathode to anode.

Figure 1 Electrorefining Process Using a Permeable Membrane



This process is illustrated schematically in Figure 1. As can be seen, the refining cell is divided into two compartments that are separated by a permeable membrane, such as a chemically resistant cloth. In the anodic compartment, feedstock nickel anodes are dissolved electrolytically in a sulfate-based electrolyte. The electrolyte from the anodic compartment (anolyte) is continuously withdrawn via a pump and circulated through a bed of nickel powder, or some other liquid-solid contactor, where the technetium is deposited in its metallic state via the displacement reaction with nickel. The Tc free, treated anolyte is then filtered and split into two streams. One stream is returned to the anodic chamber, while the second stream is added to the cathodic chamber. The stream flow volumes are adjusted so that the catolyte liquid level is 2 to 6 inches above the anolyte liquid level. This will insure a continuous flow of electrolyte from the cathodic chamber through the permeable membrane to the anodic chamber via the hydrostatic pressure differential. This flow prevents the diffusion of pertechnetate ions from the anolyte through the membrane into the catolyte. Purified nickel is deposited from the Tc free catolyte onto thin foil "seed" nickel cathodes. The cell operates on a semi-continuous basis, needing to be interrupted only when the cathodes and anodes must be replaced. The electrolyte can be used for extended periods of time, and will only have to be treated or replaced to remove accumulated contaminants, which are present in trace amounts in the feedstock nickel.

Figure 2 Electrorefining Process Using a Cationic Membrane



The preceding electrorefining process may be modified by incorporating an ion selective membrane to partition the anodic and cathodic chambers. This process is illustrated in Figure 2. In this process, the refining cell is divided into two compartments, which are separated by a cationic membrane. In the anodic compartment, feedstock nickel anodes are dissolved electrolytically in a sulfate-based electrolyte. Anions, including pertechnetate ions, are prevented from diffusing into the cathodic compartment by the cationic membrane, however, nickel cations may pass through the membrane and be deposited at the cathode. The electrolyte from the anodic compartment is circulated continuously through a bed of nickel powder, or other liquid-solid contactor, where the technetium is deposited in its metallic state via the displacement reaction with nickel.

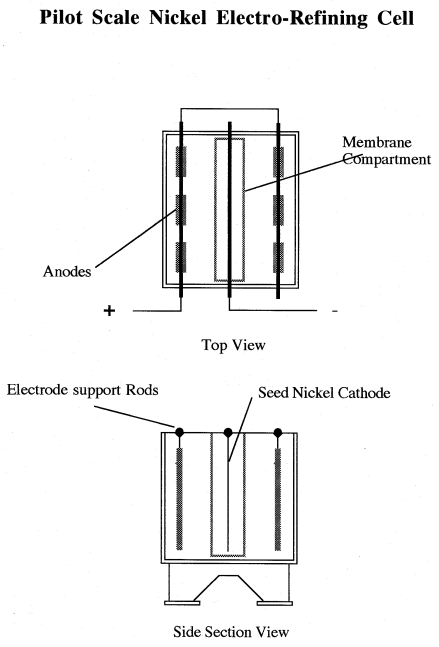
Research Objectives

Manufacturing Sciences Corporation and Covofinish Co. Inc. have collaborated to demonstrate the feasibility of the COVO process. The initial phase of the research concerned two primary tasks: the study of the Ni/Tc displacement reaction, and the small scale demonstration of the electrorefining process. This work has shown that the Ni/Tc displacement reaction is effective at removing technetium from typical electrorefining electrolytes as long as the pH is maintained at 4.5 or below. The reaction was found to exhibit first order reaction rate behavior. Experiments with the small scale electrorefining cell have confirmed that cationic membranes are effective as a barrier to pertechnetate ions and are capable of supporting nickel ion fluxes at current densities of 100 A/ft^2. Electrorefining experiments with technetium contaminated nickel anodes, in which the anolyte is continuously treated via the displacement reaction, have demonstrated the effectiveness of the process. The technetium concentration in the nickel was reduced from 460 Bq/g in the feed anode to below 1.5 Bq/g in the product cathode. These results prompted an effort to design and demonstrate a "pilot scale" system capable of processing 10 lb of nickel per day.

Apparatus

The pilot scale electrorefining system was designed using the lab scale data as a basis for scale-up. An illustration of the electrorefining cell is shown in Figure 3. The cell has one double sided cathode and two anode arrays. The cathode is 24”x24", yielding a cathodic surface area of 8 ft^2. At a current density of 20 A/ft^2, 160 Ahr is passed per hour, yielding 0.39 lbs/hr (8.4 lbs/day) of nickel, assuming 95% current efficiency. The catholyte is segregated from the anolyte via a cationic membrane enclosure. The anolyte is circulated continuously through a five micron filter and a nickel contactor which consists of a 9" diameter cylinder whose walls are lined with nickel foil. The catholyte is circulated through a heating chamber and a five micron filter. The power supply is a constant current 500 A, 24 V unit. The pH of the anolyte and catholyte is continuously monitored via pH electrodes. The anodes consist of two arrays of 5 0.75x4x28 inch nickel slabs produced by MSC from barrier material provided by the K-25 plant. The anodes contain approximately 250 Bq/g ^{99}Tc.

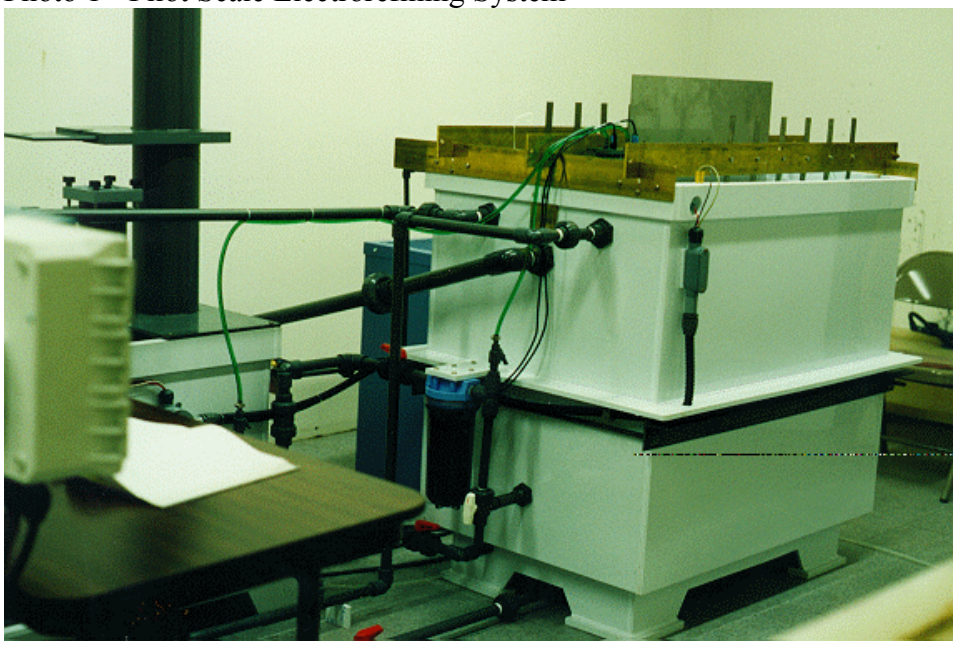
Figure 3



Experimental Procedure/Results

A Watt's type electrolyte containing nickel sulfate, nickel chloride and boric acid was used. This solution was heated to an operating temperature of 140°C. The initial pH of the electrolyte was approximately 4.0. A 24x24x1/16" Nickel starter sheet was used as the cathode, the current was adjusted to 160 A. At time intervals ranging from one to five days the anolyte and catholyte were sampled and analyzed for nickel content using ICP and technetium content using Scintillation Counting. The initial test duration was thirteen days; at the end of this test the cathode weight was approximately 130 lbs. During the test the pH of the catholyte and anolyte were stable at 4.7 and 2.8 respectively. Drillings of the cathode were taken and dissolved in 50% nitric acid to form a 1 M nickel nitrate solution; this solution was diluted 10:1 with Fisher Scintiverse scintillation cocktail and tested using a Packard Scintillation Counter. This procedure indicated a technetium content of less than 1.1 Bq/g. Subsequent trials of seven and six days duration using the pilot scale system have yielded almost identical results.

Photo 1 - Pilot Scale Electrorefining System



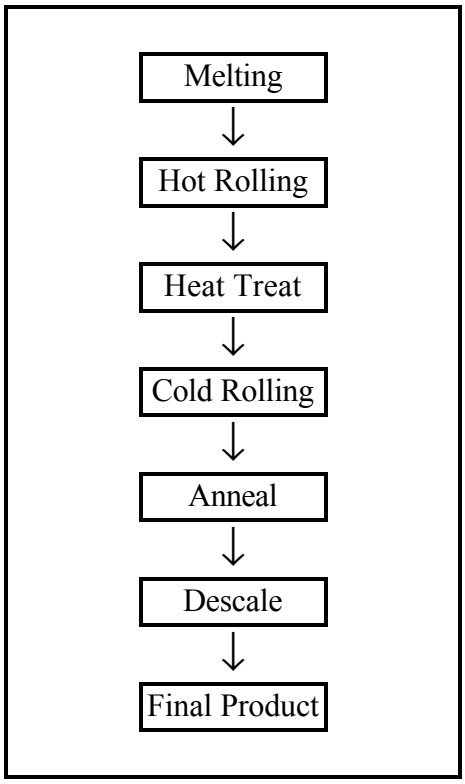
Product Manufacturing

Part of the METC phase II contract included making a restricted end use product from recycled radioactive scrap metal. Restricted end use products are those used by the nuclear industry, such as boxes, drums, and containers to store radioactively contaminated products. Research to manufacture these useful products was worked in parallel with the decontamination activities. Manufacture of useful products provides an alternative outlet for scrap metals from decommissioning activities in the DOE facilities including the approximately 40,000 tons of contaminated nickel in the diffusion plants.

Options for restricted end use products include B-25 boxes, vitrified waste containers, multi-purpose canisters, drums, and shielded containers. An investigation into a product that would best fit our production capabilities, revealed that there is a need of approximately 3,000 to 5,000 sanitary drums over the next few years for the storage of D_2O (heavy water). These stainless steel 55 gallon closed head drums are preferred for this purpose due to the smooth inner surface which allows for easier clean-up, decontamination, and survey than a standard drum with a crevice in the bottom. Production of this product proved to be a particular challenge due to the complexity of design and special tooling requirements.

The process to manufacture a product made from stainless steel can be shown in general terms by the flow chart shown in figure 4.

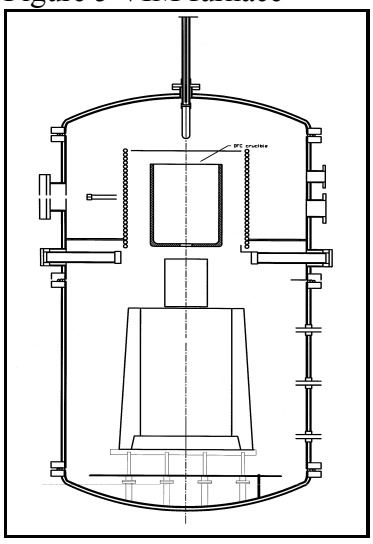
Figure 4 - Flow Chart



Melting

All RSM melting activities took place in a one ton capacity vacuum induction melting (VIM) furnace. A schematic of the VIM furnace that was used for this research is shown in figure 5. There are two main advantages to melting RSM in vacuum vessels under reduced pressure with inert gas backfill. The first is that such a vacuum environment prevents oxidation of the metal during melting, thus reducing potentially contaminated secondary wastes generated during the melting process. The second advantage is the positive containment and treatment of volatile radio-nuclides during the melt.

Figure 5 VIM furnace



The first step in making stainless steel from diffusion plant barrier nickel is to declassify or melt the material. The barrier nickel is a classified material and cannot be seen or handled by a person without a security clearance of a class "L" or better. To address this requirement, the nickel arrived in sealed thirty gallon drums. The drums were loaded directly into the VIM furnace and melted with the nickel contents. By melting the material in these sacrificial containers, declassification was accomplished without the furnace operator seeing the material.

Approximately one ton of classified nickel was melted in sacrificial drums. The material was shipped in four shipments of two to three drums per shipment. As soon as the drums arrived, they were immediately loaded into the furnace crucible. Approximately one thousand pounds of iron flake were added to the crucible to give the melt sufficient mass and density for proper operation of the induction coil. A reactive slag was added to react with radio-nuclides in the melt to declassify the material and melt refine most of the contaminates.

In order to make stainless steel sanitary drums, a one ton ingot of type 304 stainless steel was cast in the VIM furnace. A technetium contaminated iron-nickel alloy ingot from the declassification melts was used as a feed material. Low carbon electrolytic flake chromium, ferrosilicon, ferromanganese, and flake iron were added as alloy constituents.

An additional ton of nickel was required for testing of the electro-refining cell as described in the previous section. Material packaged into sacrificial carbon steel drums was not acceptable for this purpose due to the iron content in the drum. The nickel was repackaged at the K-25 plant into cardboard fiber drums and brought for declassification via melting. The nickel was melted in the VIM furnace in three heats. The resulting ingots were sliced and used as anodes in the electro-refining experiments.

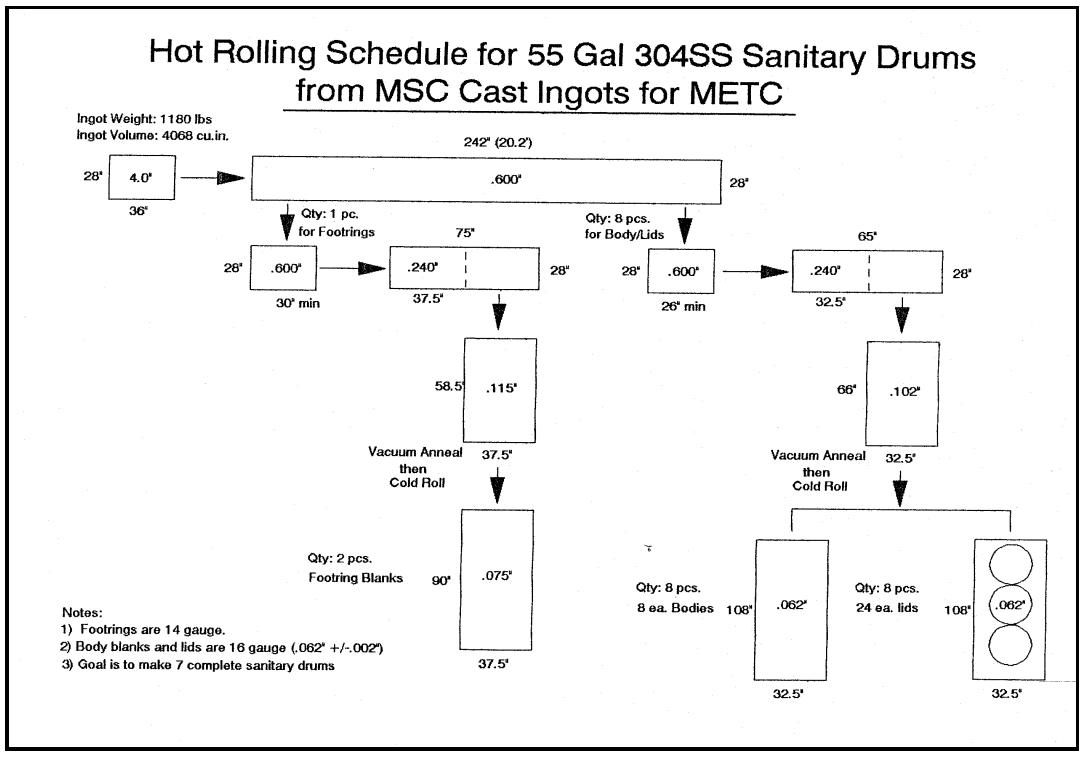
Rolling

The next step in the production of stainless steel sanitary drums involved rolling the material from an ingot thickness of approximately 5 inches down to about 0.071" (14 ga.). In a process that included both theoretical calculations and multiple rolling tests on a four high 38" wide rolling mill, a process was developed to reduce the ingot down to a sheet ready for final fabrication.

Photo 2 - Rolling mill

The final rolling procedure to produce sanitary drums is shown below.

Figure 6 - Rolling schedule for Sanitary Drums



Descaling

A stainless steel sheet requires descaling twice during fabrication activities. Once after hot rolling and once after cold rolling. After hot fabrication activities such as rolling and annealing, a heavy, adherent black scale forms on the surface of the sheet. The scale consists primarily of oxides of chromium, nickel and iron. If not removed, the scale will flake during cold rolling. The flakes become imbedded creating an uneven, unattractive surface. Descaling after cold rolling provides the clean surface to make the final product.

Mechanical, chemical, electro-chemical means and combinations thereof have been used in industry to remove scale from stainless steel sheet. Our challenge was to develop a method that is applicable to the manufacture of radioactive scrap metal while addressing the inherent problems such as radiation exposure and disposal of wastes. Other factors such as safety, cost, production rates, and environmental impact were considered.

Electrocleaning is a procedure commonly employed in the electroplating industry, where the part to be cleaned is immersed in an alkaline or acid solution under anodic, cathodic, or periodically reversing current. If anodic current is used, the passage of current from the electrolyte to the part creates a strong oxidizing environment. This allows relatively benign solutions to be used while providing a strong oxidizing action. The electrolytes used are typically dilute sulfuric acid or neutral sulfate salt solutions.

MSC subcontracted with Covofinish to perform lab scale experiments with several electrolytes, including including dilute sulfuric acid, acidic solutions of nitrate and chloride salts, citric acid, sulfuric acid/peroxide and mixtures of ferro and ferric sulfate. From the solutions tested, the most attractive formulation was found to be a 20% solution of sodium bisulfate ($NaHSO_4$). This solution has a natural pH of 1 and is self buffering. Sodium bisulfate is available as a granular powder and is commonly used as a pH adjuster in swimming pools. At a temperature of 90° to 120° F and a current density of 150 A/ft^2 this electrolyte exfoliated the scale in approximately 3 minutes. Photo 3 shows examples of the lab scale results.

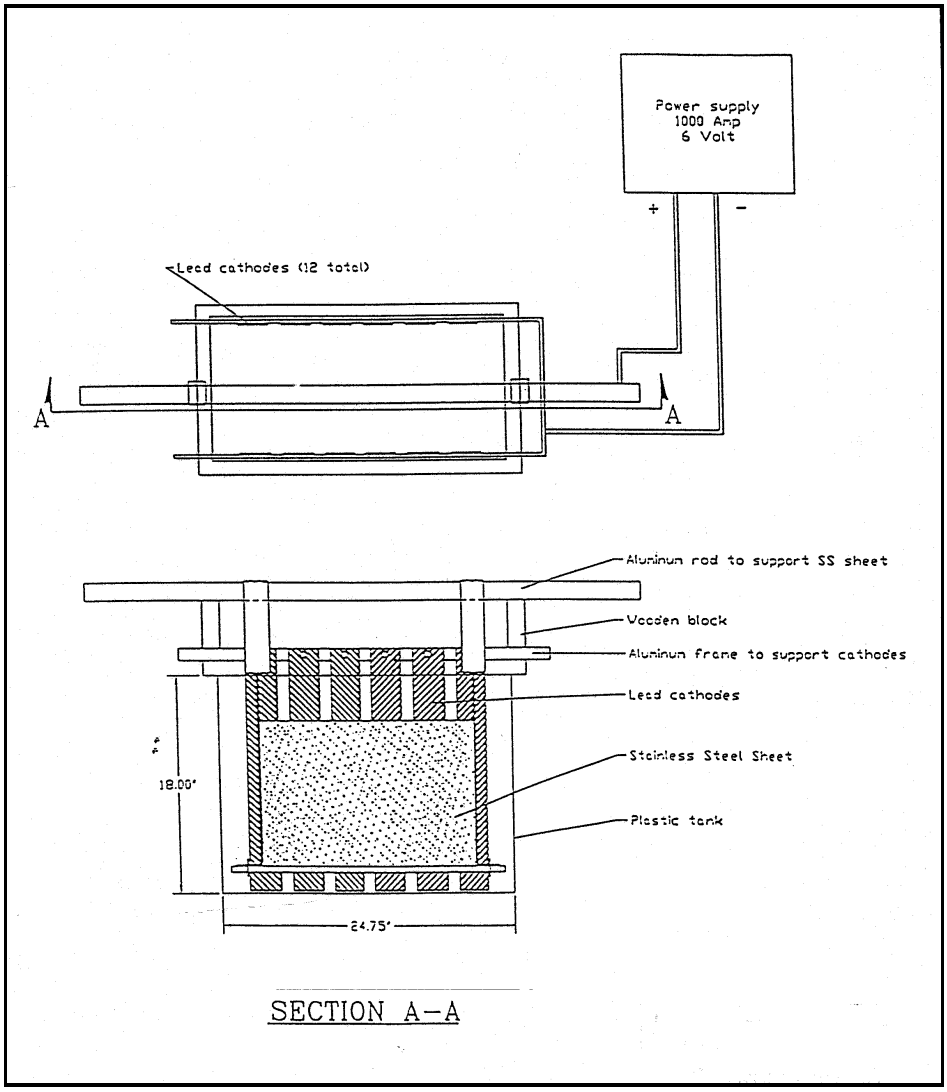
Photo 3 - Lab Scale Electro-cleaning results

Once proof of concept had been established, the system was "scaled-up" to a pilot scale model. Figure 7 shows the design of the pilot scale system.

A scaled 1/4 x 12 x 18 inch 304 stainless steel sheet was placed in an aluminum frame that served as the positive electrical contact (anode). This assembly was lowered into a 18 x 25 x 12 inch

tank filled with a 16% solution of sodium bisulfate. The outside edges of the tank included a frame of lead strips that were connected to the negative side of the power supply. These were the cathodes. The power supply used a current density of approximately 55-60 amps/ sq. ft. of stainless steel anode. After seven minutes, the stainless piece was pulled out and washed with water.

Figure 7 - Pilot scale electro-cleaning cell schematic

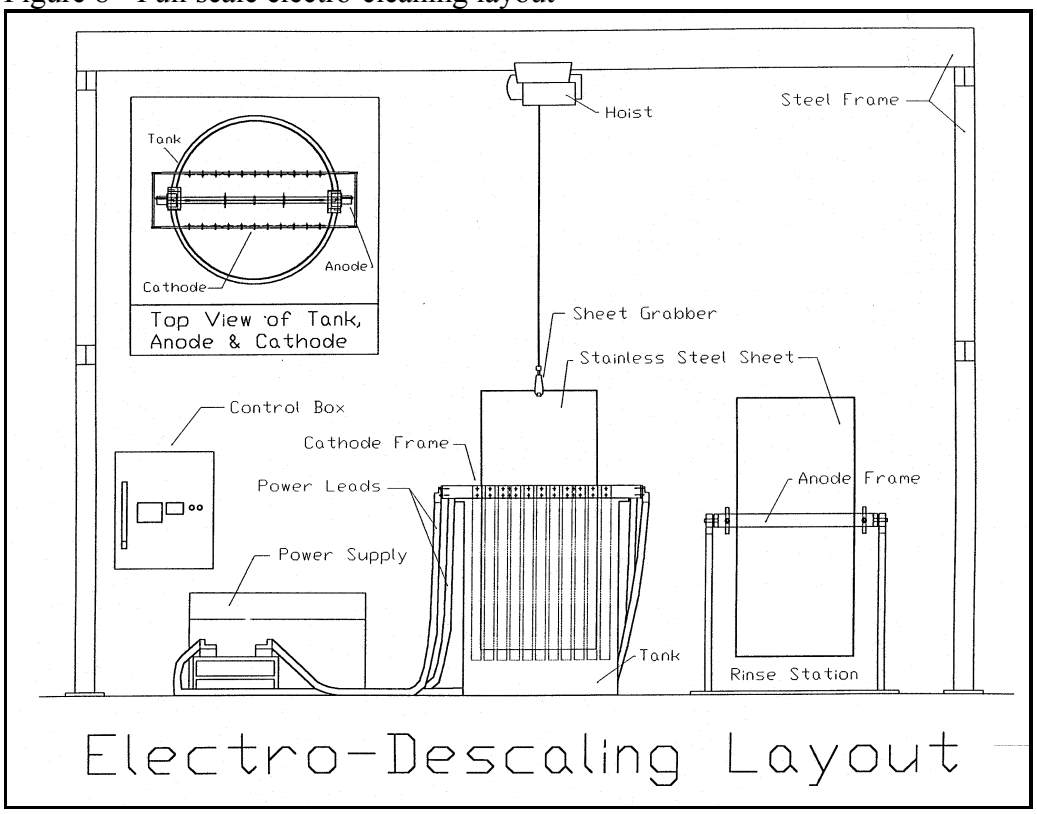


The surface appeared clean and free of the black scale that previously covered the material.

Once successful operation of the pilot scale electro-cleaning was demonstrated, a full scale system was designed and built. The full scale system had to be able to descale a stainless steel sheet up to 38" wide and 96" long. A 5 foot high by 4 foot diameter cylindrical tank was used. The sheet had to be "dipped" one half at a time in order to clean the entire length. As with the pilot scale system, the sheet was connected as the anode. The

cathodes consisted of 20 stainless steel strips 1/4 x 3 x 54 inches connected by a copper frame. The tank was filled with a 20% solution of sodium bisulfate. Polypropylene balls were placed on top to reduce fumes and a ventilation hood connected to the central ventilation system rested on top. A layout of the full scale system is shown in figure 8.

Figure 8 - Full scale electro-cleaning layout



The sheet residence time in the descaling unit was variable but each dip took between 5 and 10 minutes. The sheet did not come out of the tank completely clean. A “hotsie” steam cleaner was used to remove the remaining scale from the sheet. The resulting finishes were good and improved as the system was optimized. It was discovered that the current density of the system, which was expected to be 100 amps/ sq. ft. was operating at about 50 amps/ sq. ft. This explains the additional rinsing required to remove the remainder of the scale.

The full scale model is still in use at MSC for processes requiring descaling of stainless steel sheet.

Photo 4 - Washing the sheet after descaling

Photo 5 - Bottom half of sheet was descaled

Fabricating the final product

Once the material has been melted, rolled, annealled and descaled, it is ready to be fabricated into a sanitary drum.

Fabrication can be broken down into the following operations:

- Forming the bottom including embossing the UN number in the bottom
- Forming the top
- A press operation to emboss the serial number in the top and to punch the bung holes and form “shallow pockets” around the holes.
- Welding the flanges (bungs) onto the top
- Forming the “foot rings” that will go on the top and bottom of the drum
- Fabricating the body of the drum including installation of the “Ro-bars”
- Cleaning the drum parts
- Welding the parts together to make the drum

A drawing of the sanitary drum is shown in figure 9.

The top, bottom, and footrings on a sanitary drum are all formed using a 300 ton press along with custom designed tooling.

Figure 9 - Sanitary drum

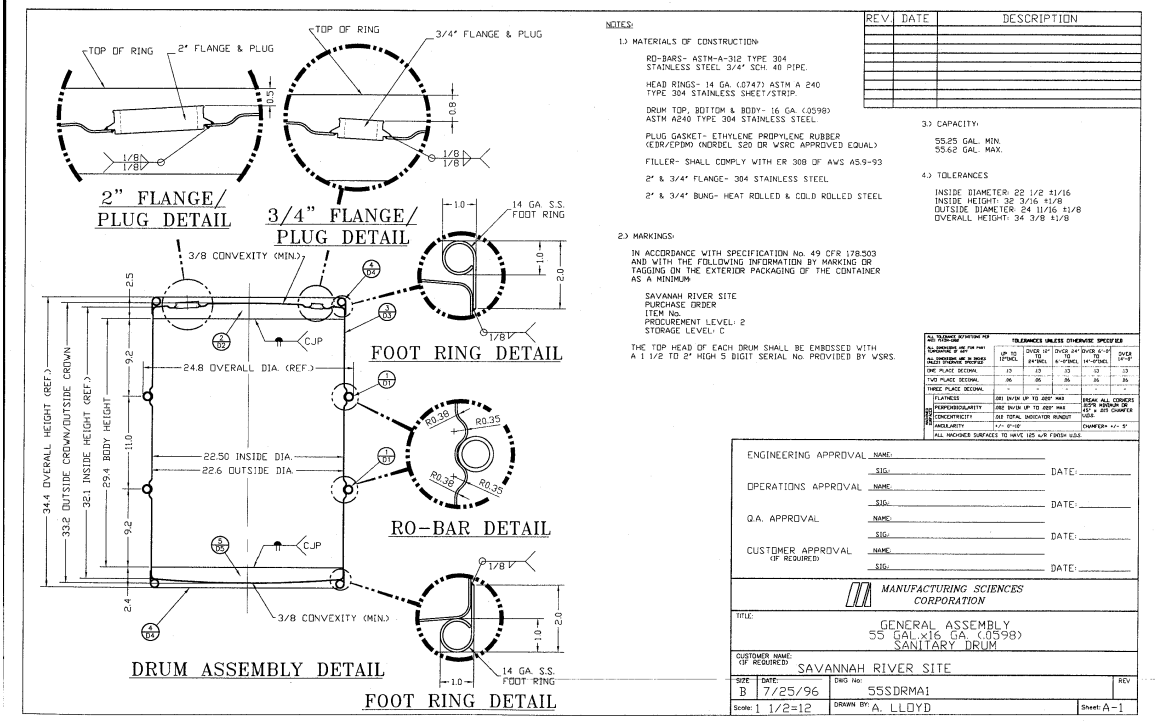
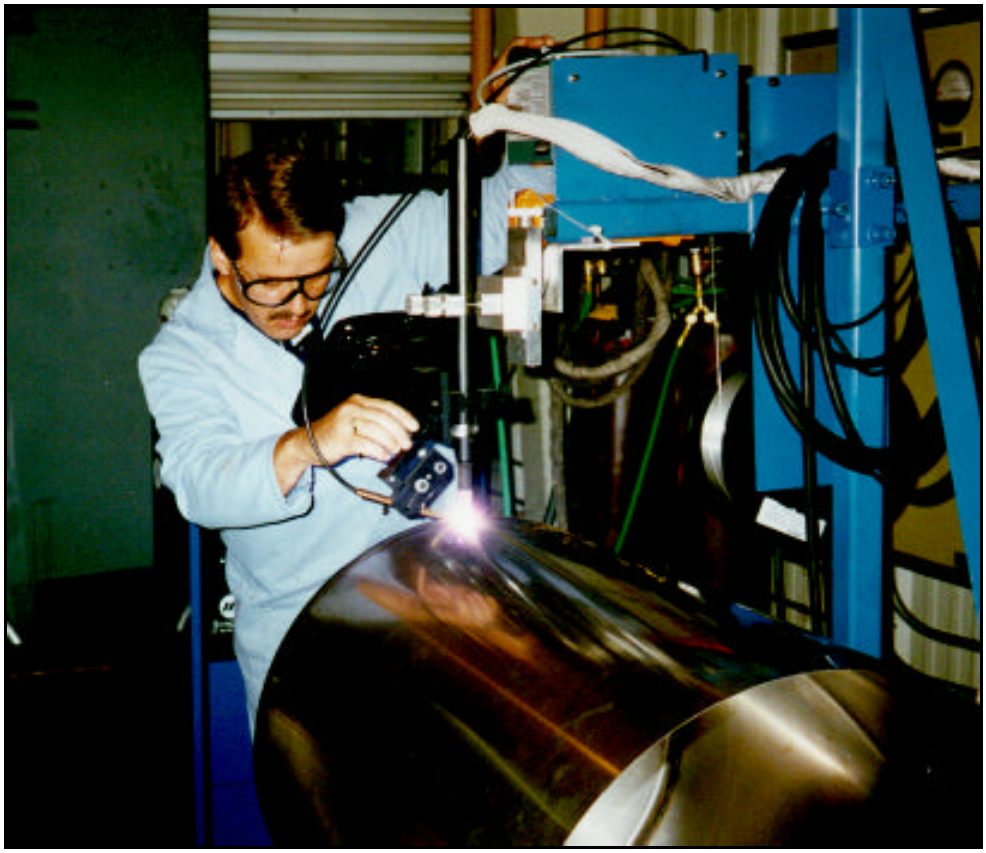


Photo 6 - Welding the sanitary drum



Fabrication of the drum body begins with a sheet of 16 gage 304 stainless steel. The sheet is then rolled using a three roll bender and seam welded to form the body. Two schedule 40 "Ro-bars" are placed around the girth of the drum. In use, these bars are used to protect the drum if the drum is placed on it's side and rolled. Four hoops are expanded into the body to hold the Ro-bars in place.

Once the parts are fabricated, they must be welded together. Special welding equipment was designed and fabricated to complete the welds. The fixture consists of a power supply, drive rolls, a wire feeder, welding torches and leads and is capable of running a full penetration butt weld around the circumference of the drum body. The welding machine has been equipped with accessories for quick changeover from automatic operation to manual TIG operations. This allows for welding of the bungs/ flanges on top of the drum. The photo 6 shows the welding equipment in operation.

A flow chart of the final process is shown in attached figures 10 and 11. This process has now been implemented for the manufacturing of sanitary drums.

Figure 10 - Flow chart for manufacturing of sanitary drums - Page 1

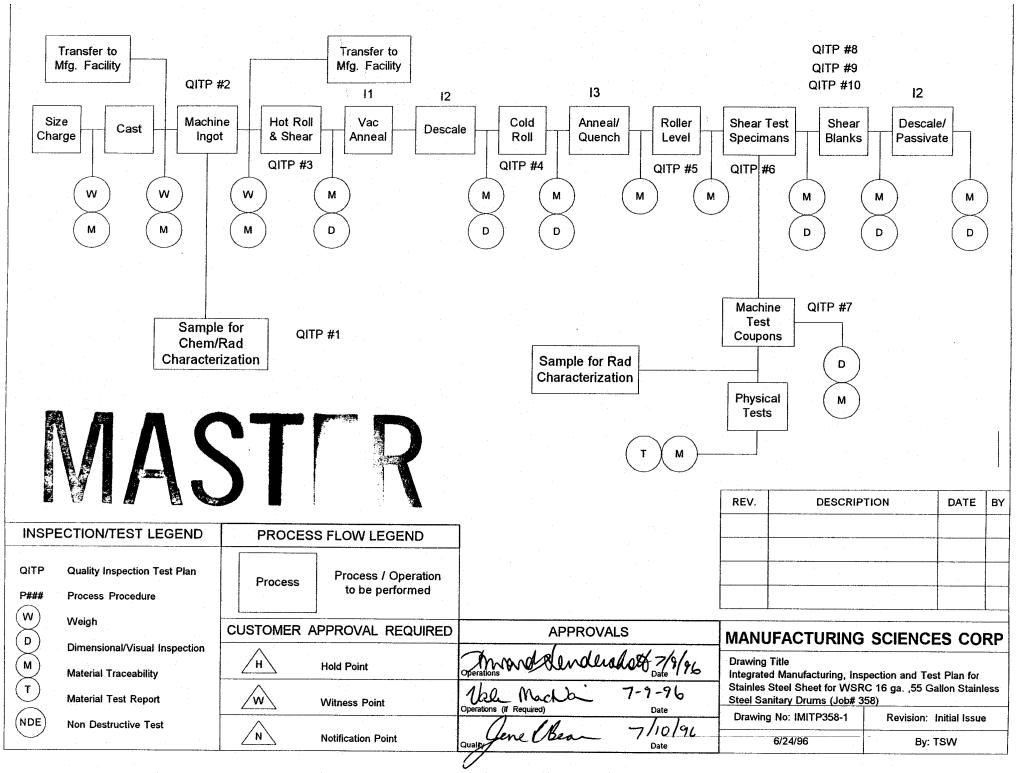
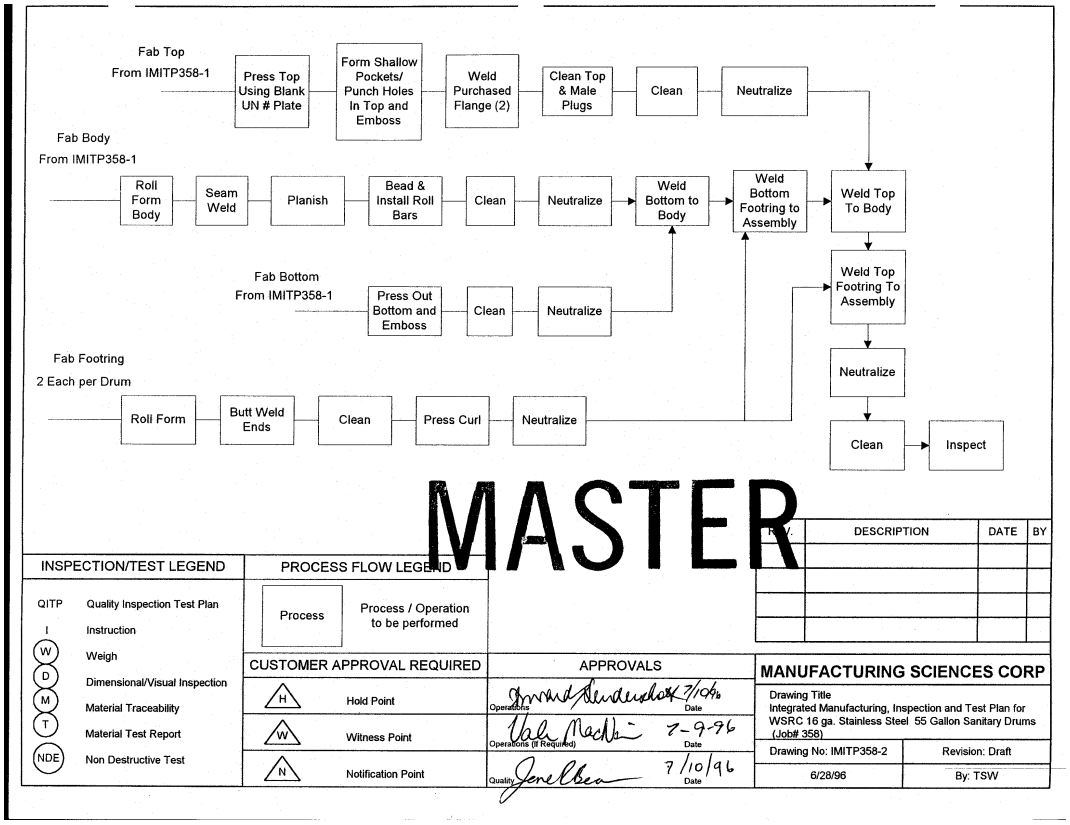


Figure 11 - Flow chart for manufacture of sanitary drums - Page 2



In making these drums, we are satisfying a customer that has a need for thousands of these containers within the next couple of years. Fabricating drums for restricted end use from recycled radioactive scrap metal reduces waste bound for landfill while providing useful products to the nuclear industry.

Development of the process to make sanitary drums paved the way for the fabrication of other products made from radioactive scrap metal. Currently, various size boxes and 55 and 85 gallon standard drums made from carbon steel and stainless steel are being fabricated for restricted end use. The feed metal is RSM from DOE facilities. This exciting development has occurred during the past two years.

Results

Research into decontamination of technetium contaminated barrier nickel resulted in the successful lab scale and pilot scale demonstrations of new electro-refining technology. The patented design, which incorporated numerous improvements over similar technologies, out performs any other process demonstrated to date. Decontamination efforts have been successful down to less than 1Bq/g of technetium.

In depth process development has resulted in restricted end use products made from radioactively contaminated scrap metals (RSM). Development of a new product during phase II, the sanitary drum made from diffusion plant nickel feed, helped to remove the production hurdles present when working with RSM. This paved the way for the melting, rolling ,and fabrication of other drums and boxes made from RSM feed material.

Application

Decontamination efforts focused on diffusion plant nickel due to the high value of the material. Even though a free release criteria does not currently exist for bulk contaminated nickel, decontamination via the new electro-refining process developed in phase II opens up options for disposition of the nickel that did not previously exist.

Within the past year, RSM from decommissioned DOE plants has been successfully converted into stainless steel and carbon steel drums and boxes. These products were shipped back to the DOE facilities for subsequent reuse. Using recycled RSM addresses the problem of disposition of piles of scrap metal while preventing the DOE from having to purchase clean metal in the form of drums or boxes that will eventually be contaminated.

Future Activities

Proposed phase III activities include developing, fabricating and testing a full scale electro-refining cell. Also, options for improving the process to declassify the barrier nickel will be explored.

Recycled RSM from the diffusion plants requires size reduction of material. Phase III will involve comparing laser and plasma technologies for cutting up of vast quantities of radioactively contaminated scrap metals.

A new product, the sanitary drum, was launched in phase II. In phase III, we plan to develop and produce another useful product made from RSM. A market survey will be conducted to match needs in the DOE to fabricating capabilities. A product will be chosen, designed, and fabricated.

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