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**DECONTAMINATING AND MELT RECYCLING
TRITIUM CONTAMINATED STAINLESS STEEL (U)**

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Executive Summary

A review of the literature associated with tritium contamination of austenitic stainless steels, and subsequent analysis, has provided valuable technical information and direction to the Beneficial Reuse Program at the Savannah River Site (SRS). Tritium contaminated stainless steel components are found mainly in tritium gas handling facilities, heavy water moderated nuclear reactors, and fusion power reactors. Research and experience with contaminated stainless steels in these applications is being increasingly documented in the literature. Most of the tritium in contaminated stainless steel components is bound in the surface oxide film, although at high pressure or temperature tritium can permeate and create a significant amount of tritium in the bulk of the component. The contamination level of potential SRS recycle material must be determined by appropriate radiological analysis, since no theory currently exists to accurately estimate tritium concentration in oxide films. Several decontamination methods have been used to achieve significant tritium decontamination of stainless steel surfaces. Most methods involve moderate heating in the presence of high humidity, or heating to higher temperatures.

Two important approaches to refine molten stainless steel are vacuum methods and slag/melt equilibrium methods. Vacuum refining, originally developed to remove hydrogen from steels, may be appropriate for tritium removal if the tritium contamination level is sufficient. Hydrogen isotope removal is possible in slag-melt refining; however, the thermodynamics and kinetics of removal is more complicated and can lead to increased tritium levels under certain circumstances. Since significant tritium outgassing from stainless steel occurs at 600°-800°C, a preheat step in this temperature range prior to melting the stainless steel will significantly decontaminate the charge and avoid introducing excess tritium into the melt. If enough tritium evolves to warrant removal from the gas waste stream of the chosen melt process, the melt process must employ an inert atmosphere, to protect available hydrogen getter materials. The acceptable tritium level of the product should be specified and could be a factor in defining the selected melt process. Tritium contamination and decontamination of the melt facility itself should also be considered.

Introduction

The Westinghouse Savannah River Company, Idaho National Engineering Laboratory, and several university and industrial partners are evaluating recycling radioactively contaminated stainless steel. The goal of this program is to recycle contaminated stainless steel scrap from US Department of Energy national defense facilities. There is a large quantity of stainless steel at the DOE Savannah River Site from retired heavy water moderated nuclear material production reactors (for example heat exchangers and process water piping), that will be used in pilot studies of potential recycle processes. These parts are contaminated by fission products, activated species, and tritium generated by neutron irradiation of the primary reactor coolant, which is heavy (deuterated) water. This report reviews current understanding of tritium contamination of stainless steel and previous studies of decontaminating tritium exposed stainless steel. It also outlines stainless steel refining methods, and proposes recommendations based on this review.

Literature Review

Materials from the family of austenitic stainless steels are the preferred structural and confinement material for both nuclear reactors and tritium handling facilities, because of their strength, ductility, and relative resistance to environmentally induced degradation. Tritium handling facilities expose stainless steel to both molecular tritium gas, tritiated water and water vapor, and (less commonly) tritium compounds. (Tritium facilities routinely handle tritiated water because tritium getter systems commonly oxidize molecular tritium and absorb the water on various types of getter bed materials.) Heavy water reactor moderator water contains tritiated water (~ 9 Curies/liter in SRS reactors while operating) created by neutron irradiation of the moderator while in the reactor core. In addition, atomic and ionic tritium accelerated toward stainless steel is studied to determine the performance of stainless steels in the interior of fusion power reactors. Increasing numbers of tritium facilities and reactors are being decontaminated and decommissioned, resulting in a growing literature documenting research and experience in decontaminating tritium from stainless steel [1, 2, 3, 4, 5, 6]. An overview of molecular tritium and tritiated water contamination of stainless steel is presented in this report; accelerated and ionized tritium effects are not covered.

Tritium Location in Exposed Stainless Steel Parts

Tritium in Surface Oxide

Many studies have concluded that the majority of tritium in contaminated stainless steel parts exposed near ambient temperature to either tritiated water or low pressure tritium gas is bound in the surface oxide film [1, 2, 3, 4, 5, 6, 7]. The amount of tritium bound in the surface varies, of course, based on the exposure conditions. The ability to decontaminate stainless steel in one experiment changed as the exposure time increased [6]; the longer parts were exposed, the less tritium was removed by the various decontamination methods used. This led the authors to postulate two forms of bound tritium in the oxide, that they termed "weakly bound" and "strongly bound".

Several retired storage tanks made of stainless steel in a tritium facility in the United Kingdom have been analyzed to determine the tritium burden [1]. A tank that stored tritium gas for about 20 years had a total tritium burden of about 6300 Ci/m². The amount of tritium that permeated into the tank wall was a factor of about 80 below the observed total wall. Clearly a significant amount of tritium was absorbed into the oxide surface.

Several independent thermal desorption experiments, in which the quantity of tritium evolved from stainless steel oxide is measured while heating the sample, revealed similar characteristic peak temperatures of increased tritium evolution as the temperature of tritium exposed stainless steel samples increased [2, 3]. Samples were exposed to tritium gas in these studies, and became water by combining with oxygen from the oxide film. The first peak, at about 200-300°C, accounted for most of the tritium in contaminated samples and was tritiated water (HTO or T₂O, where T stands for tritium, ³H). The tritium resulting in this peak is interpreted as residing in the stainless steel surface oxide [2, 3]. The tritium is believed to be bound in hydroxyl groups (OH⁻). Peaks observed at higher temperatures are molecular peaks (HT or T₂), and the total amount of tritium off gassing at high temperatures is much less than that during the first low temperature peak.

Further evidence that tritium strongly interacts with stainless steel surface oxides is found in experiments that reveal stainless steel surfaces interact with molecular tritium and catalyze the formation of tritiated water vapor [8, 9]. This phenomenon has been investigated for potential use in magnetically confined fusion systems. Some of

the water vapor created on the surface reacts and remains bound to the surface, illustrating the affinity tritium has for stainless steel.

Tritium in the Bulk

The concentration of tritium in a stainless steel part exposed to gaseous tritium can be calculated as a function of location in the part if the history of exposure temperature and tritium partial pressure is known [10]. These calculations provide accurate estimates of total tritium burden when parts are exposed to tritium at elevated temperature or pressure. A finite difference computer program can be used [10], as well as various steady state approximations, if warranted. Reliable permeation data for stainless steel have been compiled [11]. Permeation of tritium from tritiated heavy water into stainless steel cannot be calculated, even when exposure conditions are known, because the thermodynamic activity of tritium in tritiated water interacting with the stainless steel is unknown. In addition, permeation calculations do not account for tritium absorbed by the stainless steel surface oxide.

Tritium Decontamination Techniques

Studies of decontaminating tritium exposed stainless steel show that several techniques can provide effective decontamination. Passing water saturated air over metal parts at ambient temperature ("wet outgassing") removed about 90% of the tritium activity in 10 days and 99% after 20 days [1]. Outgassing treatments by heating tritium exposed parts encapsulated in quartz ampoules to temperatures between 50°C and 400°C resulted in a maximum decontamination factor (DF) of 37 at 400°C [1]. The decontamination factor can be defined by:

$$DF = \frac{\text{Total Contamination Before Decontamination}}{\text{Total Contamination After Decontamination}} \quad 1$$

Another series of outgassing experiments between 600°C and 1500°C revealed a maximum decontamination factor of almost 10^4 at 800°C when the argon or argon/5% hydrogen furnace atmosphere was circulated; static atmospheres having the same compositions resulted in a maximum decontamination factor of 10^3 at 800°C [1]. The decontamination factor at the melting point, ~1500°C., was reduced to 4×10^3 with a circulating atmosphere and to 167 with a static atmosphere. (This result may be related to the increase solubility of tritium in liquid stainless steel as discussed in the next section.)

Repeatedly exposing tritium contaminated stainless steel samples to a helium glow discharge at 13 MHz in a special system resulted in a decontamination factor of about 250 [2]. In another study, vacuum outgassing at 500°C resulted in a decontamination factor of 22, and this treatment followed by an acid dissolution using 5% hydrochloric acid resulted in a decontamination factor of about 200 [7].

Stainless Steel Refining Methods and Hydrogen Isotopes

Hydrogen Solution in Stainless Steel

Refining hydrogen from molten stainless steel has long been an important process [12]. Excessive amounts of dissolved hydrogen causes "flakes" or flat shaped pores in billets made for large forgings. Hydrogen dissolves in both solid and liquid stainless steel according to Sievert's law

$$c_H = c_H^\circ \cdot \sqrt{p_H} \cdot e^{-\Delta H/RT} \quad 2$$

which relates the concentration of hydrogen in the steel c_H to the hydrogen partial pressure p_H above the steel, temperature T , gas constant R and material constants ΔH and c_H° . The quantities ΔH and c_H° differ for liquid and solid stainless steel. The solubility of hydrogen in liquid stainless steel just above the melting point is about twice that of solid steel just below melting, and increases further with increasing temperature. The increase of hydrogen solubility with temperature in liquid stainless steel, about 0.032 wppm/°C [13], is much greater than that in solid stainless steel, 0.0034 wppm/°C [11]. In steel refining plants significant quantities of hydrogen accumulate in molten steel if the molten steel is exposed to air moisture. Moist air was the largest source of dissolved hydrogen in steel refining until protective covers and atmospheres were developed [12].

Vacuum Refining

By applying a vacuum above liquid stainless steel, dissolved hydrogen can be refined from the steel, since as p_H approaches 0 (under vacuum) c_H approaches 0 (Eq. 2). On the other hand, without a vacuum or inert cover gas, water vapor in the air decomposes and is a source of dissolved hydrogen in the liquid steel. Various types of furnaces are used for vacuum refining stainless steel [12]. A novel method for producing steel having very low concentrations of carbon, nitrogen, hydrogen, and

sulfur by injecting iron ore powder into the melting during vacuum refining steel has been developed [14].

Slag/Melt Refining

Another type of stainless steel refining utilizes slag-melt equilibrium. (When the stainless steel is formed into consumable electrodes for an electric arc furnace, the technique is termed electro-slag remelting, or ESR.) Slags are mixtures of oxides, typically silica, calcia, alumina, and magnesia, and other compounds such as fluorspar, and serve as fluxing agents in melting operations. Hydrogen isotopes can dissolve in the slag from the common hydrogen forms, including water vapor, molecular or atomic hydrogen [15]. In all cases, the hydrogen probably exists as hydroxyl group (OH^-) in the slag. Depending on conditions, hydrogen can be either added to or removed from stainless steel during slag-melt refining. The hydrogen concentration can increase by absorption of water vapor above the melt as discussed above for vacuum refining. However, this absorption can be avoided by using a covered ladle with a flowing inert cover gas above the melt [16]. The type of flux used for the slag also influences the hydrogen content of the steel [16]. A detailed model of hydrogen behavior during electro-slag remelting shows that in addition to hydrogen in the atmosphere, hydrogen in the electrode can be transferred to the melt, depending on the flux chemistry, hydrogen content of the electrode and melt, and temperature [17].

Recycle of Tritiated Stainless Steel at Savannah River

Anticipated Conditions

The experience of handling and decontaminating tritium exposed stainless steel outlined above allows assessment of tritium behavior in a stainless steel recycle process. For components exposed to tritiated heavy water at the Savannah River Site, most of the tritium probably resides in the surface oxide. The total tritium burden can be determined by dissolution and scintillation counting of samples removed from the recycle feed material. This assay is the vital first step in designing a stainless steel recycling process having effective tritium decontamination.

As indicated above, most of the tritium will evolve at relatively low temperatures ($<600\text{-}800^\circ\text{C}$). Even at 300°C , much of the tritium in the surface oxide will likely be released. Most of the tritium will be released as tritiated water vapor.

Savannah River Stainless Steel Considerations

The characteristics of Savannah River Site recycle program feed materials, such as reactor process water piping, differs from that used in decontamination studies documented in this report. Although the conclusions found in the literature for contaminated stainless steel should be qualitatively valid for Savannah River Site pipe exposed to tritium contaminated heavy water, the effectiveness of tritium decontamination in any proposed recycle process must be characterized by experiment for the particular recycle feed material.

Water vapor saturated air has been observed to decontaminate tritium contaminated parts (above). Parts can be decontaminated before melting by simply exposing them to the atmosphere, which is an advantage for some of the very large pipe sections at Savannah River. Parts having a large tritium burden may have to be partially decontaminated in a separate facility, if the contamination is high enough and the part is small enough.

As time passes the amount of tritium will also decrease by radioactive decay. The half life of tritium is about 12.3 years. Radioactive decay occurs according to

$$\frac{C}{C_0} = e^{-\ln 2 \cdot t / T_{1/2}} \quad 3$$

where the original concentration C_0 decays to concentration C after time t for a radioisotope having half life $T_{1/2}$. The concentration ratio is related to the decontamination factor (Eq. 1) by

$$\frac{C}{C_0} = DF^{-1} \quad 4$$

and this leads to a natural tritium decontamination factor of 10 after 41 years and 100 after 82 years (Eqs. 3, 4).

Most of the tritium will likely be released from stainless steel at relatively low temperatures early in the melting operation. As discussed earlier, the hydrogen solubility in stainless steel increases with increasing temperature. Furthermore, when the steel melts, the hydrogen solubility in molten steel increases at an even higher rate with increasing temperature. This solubility behavior implies that to achieve the

minimum amount of dissolved tritium after melting, as much decontamination should be performed before melting as practical. (Evidence for stainless steel retaining tritium at temperatures near the melting point was observed in a previous study (discussed earlier) [1].) A low temperature hold prior to melting could enable effective decontamination while minimizing pickup of tritium by the melt. Holding the recycle feed material at 800°C in flowing inert gas would be very similar to decontamination practices performed in the past [1] and might achieve a decontamination factor of up to 10^4 .

Vacuum refining methods, such as vacuum arc remelting, appear to be more appropriate for tritium decontamination than slag-melt equilibrium methods. Vacuum refining is commonly used to remove hydrogen from liquid stainless steel. If a slag-melt process is considered, the complicated thermodynamic and kinetic issues with hydrogen/flux/slag/melt system must be addressed. However, it is certainly possible that a slag-melt furnace could be used for effective melt recycling of tritium contaminated stainless steel. Radioisotopes other than tritium must also be decontaminated from contaminated feed materials, and a slag-melt process might be required for the non-tritium isotopes. If this is the case, it would be desirable to include tritium decontamination in the design of the slag, to enable a single process cycle to decontaminate all radioisotopes.

The acceptable concentration of residual tritium in the stainless steel product must be determined. The acceptable level may influence the choice of a melt decontamination process and the need for a separate decontamination procedure before melting. Although this acceptable level is a regulatory issue, the selected process may have a technical limit for the decontamination factor.

Melt Facility Considerations

The amount of tritium released to the environment during melting must be evaluated. If the amount of tritium offgassing exceeds environmental limits for the facility, some method of gettering the tritium must be developed. Various types of gettering systems are currently being developed [18]. The choice of getter materials is much greater and their performance is vastly improved if the gas stream is inert, since nitrogen and oxygen quickly saturate most getter materials. Also, the waste stream of most hydrogen isotope getter systems is water, so handling tritiated water must be taken into account in the overall design. If a vacuum melt recycling process is chosen,

gas from the pump discharge could be either sent directly to a getter system or to a hold tank for subsequent tritium clean up.

Several surface modification techniques have been developed that could reduce the tritium contamination of the metal surfaces of a recycle facility, for example in the exhaust system and vacuum chamber. One study evaluated the tritium interaction with stainless steel surfaces prepared using several surface treatments, including thermal oxidation, chemical passivation, chromium electrodeposition combined with thermal oxidation and a chromium diffusion coating [19]. The chromium diffusion coating significantly reduced the amount of tritium absorbed during exposure to molecular tritium gas [19]. Several types of aluminum coatings have been found to effectively reduce tritium permeation in other studies: an aluminum ion implanted layer [20], and a proprietary aluminization coating [21, 22]. All of these surface modification techniques alter the stainless steel surface to reduce the ability of molecular tritium to react with and dissociate on the normal austenitic stainless steel oxide surface. The design of a recycle facility should consider these methods, to mitigate contamination of equipment.

Although tritium has a very low solubility in most ceramic materials, concrete has been observed to absorb tritium readily [5]. Concrete is porous, and contains significant amounts of hydrogen, mainly in hydroxyl group form. Since concrete is a common material of construction of large facilities, the contamination of a tritium melt recycle facility should be considered. Although paints and other organic coatings also absorb large amounts of tritium, coating the porous concrete with a paint or epoxy sealant may reduce the contamination of the concrete. Any refractory brick used in a furnace could similarly absorb tritium. The question of whether this occurs under the conditions in a melt furnace can only be addressed by experiment.

Final Assessments and Recommendations

A literature survey of research and experience in tritium contamination and decontamination of stainless steel allows the following general assessments and recommendations to be made about tritium contaminated stainless steel melt recycling.

- 1) Most of the tritium contamination of stainless steel reactor components resides in the surface oxide film of the stainless steel. The contamination level must be measured- it cannot be calculated with any accuracy.

- 2) The absorbed tritium will likely evolve from the surface oxide during moderate heating, beginning at about 300°C. Heating to temperatures up to 800°C will yield more rapid and more complete decontamination.
- 3) Significant tritium decontamination occurs when contaminated components are exposed to humid air, even at ambient temperature. This may be the only practical decontamination method for large structures. Also, since the half life of tritium is about 12.3 years, tritium naturally decays rapidly, compared to some long-lived radioisotopes.
- 4) Vacuum refining may be the most effective way of removing hydrogen isotopes from liquid stainless steel. Slag-melt methods can also remove hydrogen isotopes, however the requirement of tritium decontamination will influence the design of the slag.
- 5) Because the solubility of hydrogen isotopes in stainless steel increases with increasing temperature, and becomes much greater at the melting point and above, a preheat stage during melt recycling is recommended to minimize re-absorption of tritium by liquid stainless steel. This stage would consist of holding the charge at about 600-800°C to allow as much tritium decontamination as possible. A decontamination factor of 10^4 may be achievable during the preheat stage.
- 6) If material to be melt recycled contains a large enough quantity of tritium, a getter system may be required to reduce emissions. Current getter materials require either an inert atmosphere or a "vacuum" with tritium a dilute impurity, because most getters will saturate and become useless if exposed to significant quantities of nitrogen or oxygen.
- 7) Materials of construction of a stainless steel melt facility may influence the contamination level of the facility after processing tritiated stainless steel. Several types of surface treatments of metals have been shown to minimize tritium contamination. Concrete will absorb significant amounts of tritium. Paints and coatings may protect concrete and metal surfaces from tritium. Refractory materials, commonly used in melt furnaces, may also absorb tritium.

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