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TRITIUM PERMEATION IN PASSIVATED 304L  
STAINLESS STEELS by T.L. NISBING

1. Desc: "Tritium Permeation"

Since tritium permeation can lead to hydrogen and helium 3 embrittlement it is important to know about the processes that are involved with tritium permeation. Type 304L stainless steel is frequently used as engineering material for containing tritium. Therefore, studies were performed using 304L stainless steel coupons. However, other commonly available stainless steel such as 316 should perform similarly.

From the literature we have found that surface permeation is rate controlling. When the surface is sputtered to remove oxides and then coated with palladium, tritium permeation increases one to three orders of magnitude above that for a normal oxide coated stainless steels. The catalytic <sup>for</sup> nature of palladium is responsible for some of the permeation increase. However experiments have demonstrated that a "clean" stainless steel surface is much more permeable than one with a passive oxide film.

2. Desc: Schematic showing tritium permeating through a metal with surface films

This schematic shows some of the reactions that are associated with tritium permeation. When tritium comes in contact with a surface film some of it disassociates into nascent tritium or a monatomic ionic species. When palladium is the film, this reaction is accelerated and permeation is increased.

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Once the tritium is available in a monatomic form it permeates through the surface film; then through the metal; through the surface film on the exit side to the surface where it must again recombine to  $T_2$  in order to exit. The rate controlling reactions are either the disassociation reactions on the entrance side and/or permeation through the film.

3. Desc: Triangle (Treatments,  $T_2$  Studies, Characterization)

In previous studies it was very difficult to characterize the oxide films that were being exposed to tritium. Auger and ESCA were used but are too expensive and cumbersome to be practical. In the current study Auger and ESCA are used to evaluate the films but the primary characterization methods are electrochemical and chemical which have been shown to have an excellent correlation with tritium permeation. The bulk of the characterization work was performed at the Rockwell Corporate Science Center, Thousand Oaks. The tritium permeation studies were performed at LANL.

4. Desc: "Previous Work"

In order to compare and contrast this work to those studies reported in the literature we will take a closer look at the studies. Most of these studies were performed at high temperature through a thin foil "diaphragm". These studies demonstrated that the least permeable treatment was a dichromate treatment... Progressing through these treatments to the most permeable treatment, palladium. As mentioned before, characterization was generally XPS and Auger. However, in all

cases the films were highly variable i.e., were whatever result a given treatment gave.

5. Desc: Permeation Schematic

The diaphragm method gives a measure of the overall flux of tritium between the foil surface (1 & 7). Los Alamos performed a different type of analysis in the present work. They used ion implantation spectroscopy where they bombarded the sample with deuterons and monitored fission products. This method has several advantage over the diaphragm method:

- 1) real thick samples can be used
- 2) complications involving permeation through the exit surface (5, 6, 7) are avoided.
- 3) The actual tritium profiles are measured (1, 2, 3, 4, 5).

In practice approximately a month transpired between tritium permeation and profile measurement so that the resulting profile looked like this (aged).

6. Desc: "Project History"

At the beginning of the project we treated samples by several different treatments. We performed static electropotential measurements on the samples and could easily differentiate between them.

7. Desc: "Rockwell Corporate Science Center"

Following the rather crude electropotential measurements at RFP, we submitted samples to the Science Center in order to identify an inexpensive, nondestructive, rapid characterization method(s) that would correlate well with permeation.

8. Desc: "Treatments"

The two facilities evaluated samples given these treatments.

9. Desc: "LANL Studies"

Read slide.

10. Desc: RCSC - LANL Permeability order.

Both RCSC'S electrochemical and chemical studies and LANL tritium permeation studies resulted in an ordering of the passivation treatments with regard to their permeation resistance. In RCSC'S case the least permeable treatment was assumed to be the one with the most coherent, defect free, chemically stable surface. You can see that the characterization agrees well with observed tritium permeation. The EP+ nitric/nitrad and EP+ steam are transposed between the two methods... However both methods found that both treatments were "poor" and approximately the same amount of "poor"

11. Desc: Permeability Order

Since steam is not a candidate process for any projected production use, this is the order of the treatments; from most permeable to least.

12. Desc: "Conclusion"

Read slide

13. Desc: "Future Work"

In the future treatments will be chosen to closely match production condition. Machined surfaces will be used instead of electropolished. Other austenitic stainless steels such as 316 will be tested. Again the samples will be characterized by the Science Center and evaluated for tritium permeation by LANL. Because it is financially feasible, more samples will be run at the Science Center.

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