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Evaluation of Hydrogen Embrittlement of SAFKEG 3940A Package in KAMS

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1. EXECUTIVE SUMMARY

This report documents the evaluation of the potential for hydrogen embrittlement of the stainless steel of the inner containment vessel (ICV) and the outer containment vessel (OCV) components of the SAFKEG 3940A shipping package under transportation conditions and storage conditions in the K-Area Monitored Storage (KAMS). The components are evaluated under a bounding scenario for hydrogen exposure.

The bounding hydrogen exposure scenario was a hydrogen pressure of 116 psia which is the design pressure for the ICV. Hydrogen ingress into the ICV was assumed at the bounding temperature of 392°F, which is the maximum temperature of the ICV including normal and accident conditions. A total of 550 appm would be the total estimated hydrogen absorbed at these conditions. This amount would be expected to cause only a small reduction in the ICV ductility. Slow crack growth in the ICV is also not predicted for the ICV for the predicted hydrogen concentration. The integrity of the SAFKEG 3940A package, specifically the ICV and OCV components, would not be adversely impacted by this bounding hydrogen exposure scenario. These results are based on the assumption that the SAFKEG shipping package is maintained during shipping and storage within the normal and accident conditions described in the Safety Analysis Report for the Package and the KAMS Documented Safety Analysis.

2. INTRODUCTION

Plutonium materials currently stored at several DOE sites may be shipped to SRS in Model SAFKEG 3940A packages for interim storage in the K-Area Material Storage (KAMS). Concerns associated with hydrogen embrittlement have been identified for the shipment of plutonium oxide materials. These materials can contain chemisorbed water that, due to radiolysis, can lead to hydrogen production.

The concern of hydrogen embrittlement the 9975 package, a package similar to SAFKEG, was raised previously in Reference 1 and was assessed in the technical report [2] in reference to the 9975 package. In that report, the phenomena leading to the build up of hydrogen in plutonium packages were addressed; among which radiolysis of water was the primary mechanism of hydrogen generation in the package. Hydrogen embrittlement can occur in austenitic stainless steels in the form of reduced ductility or through slow crack growth under loads that, under normal conditions, would not produce cracking.

The SAFKEG shipping package is described in Reference 3. It consists of an outer drum assembly, insulation, an outer containment vessel (OCV), and a primary containment vessel (ICV). The plutonium oxide material is contained within the ICV in its own convenience packaging. For the purposes of this investigation, it is assumed that the convenience packaging does not prevent hydrogen from reaching the ICV.

The SAFKEG shipping package is qualified to carry up to 5.0 kg of plutonium oxide, containing up to 0.5% moisture and some organic materials (bagging) [3, 4]. This would provide a maximum of 25 g of water. Radiolysis of the water and organic materials provide the hydrogen source under investigation. Although the hydrogen is assumed to escape the convenience packaging, the escape route is assumed to be sufficiently long that the radiolyzed atomic hydrogen recombines and becomes molecular hydrogen before it reaches the ICV wall. After hydrolysis, the hydrogen may recombine with oxygen, it may be absorbed by the convenience packaging (carbon steel), or it may be absorbed by the ICV.

The ICV and OCV are constructed of 304L stainless steel with the exception of the lid nut, which is annealed S21800 stainless steel (a.k.a. Nitronic 60). Both vessels are constructed from Schedule 40S pipe, standard weight pipe caps, and closure components machined from bar stock. The components are welded per the ASME Boiler and Pressure Vessel Code, with no post-weld heat treatment. It is conservatively assumed that no hydrogen diffuses into the gap between the ICV and OCV. This assumption neglects the OCV as a virtual sink for hydrogen and maximizes the amount of hydrogen in the ICV. With this approach, any conclusions regarding the integrity of the ICV will bound the OCV.

The stainless steels of the ICV and OCV are subject to a maximum temperature of 392 °at the hypothetical accident conditions in transport [3]. This bounding temperature applies to all transportation and storage conditions. The temperature in storage in KAMS will be lower [5].

3. ANALYSIS

3.1 General Description of Hydrogen Embrittlement

Steels show a tendency for reduced strength and ductility following exposure to hydrogen. The magnitude of this embrittlement varies with the type of steel, its microstructure and stress state, and details of the hydrogenous environment. The effects of greatest importance to austenitic stainless steels include loss of ductility and hydrogen stress cracking [6].

The most noticeable change in 304L stainless steel following hydrogen exposure is a decrease in ductility, measured as elongation and/or reduction in area in a tensile test. In addition, the tensile strength is observed to decrease and the yield strength increases slightly. The change in yield strength is not consistently observed in all cases. In addition, slow crack growth is observed under stresses lower than required to produce crack growth in the absence of hydrogen. Most researchers studying hydrogen embrittlement of austenitic stainless steels express the degree of embrittlement by reporting the decrease in ductility, while some also report the changes in strength and other parameters. [References 7-12, for example]

3.2 Calculation of Hydrogen Embrittlement Effects

Since the literature data is based on testing designed to produce hydrogen embrittlement, the conditions tested are typically more severe than those forecast for the SAFKEG shipping container. Specifically, the hydrogen concentrations are higher, and in some cases, the stress states and temperatures are also more extreme. In order to accurately predict the influence of hydrogen on the shipping package, a common ground is required to allow comparison of the shipping package exposure conditions and the effects described in the more aggressive tests. Reference 2 developed a correlation between the equilibrium hydrogen concentration and its partial pressure during exposure to the determined the effect of hydrogen environments on properties for the 9975 package. This was accomplished by calculating the concentration of hydrogen absorbed within the vessel for comparison with the hydrogen concentrations used in the literature tests. Since many of the references do not report the actual hydrogen concentrations in their test specimens, these concentrations are also calculated in some cases.

Using the same approach, the impact of a bounding hydrogen environment on properties can be determined for the ICV and OCV of the SAFKEG package. Accordingly, the following steps will be followed to calculate the impact of hydrogen on the SAFKEG shipping package.

(1) Utilize the previous mathematical model describing the hydrogen concentration in test specimens for specific hydrogen conditions (temperature, pressure) [2].

(2) Using this model, calculate the steady state hydrogen concentration in the ICV.

(3) Compile literature data on the degree of hydrogen embrittlement vs. hydrogen concentration and extrapolate the data to the conditions of the ICV.

(4) Estimate the impact of hydrogen exposure on the SAFKEG ICV.

3.2.1 Mathematical Model

The literature identifies two research techniques used to study the effects of hydrogen exposure in stainless steels. Both techniques are designed to produce measurable uptake of hydrogen. In one approach, the test specimen is exposed to a high pressure hydrogen atmosphere for a period of time to allow hydrogen to diffuse into the specimen. The second approach establishes the test specimen as the cathode in an electrolytic reaction. As atomic hydrogen is liberated in the reaction it is absorbed by the test specimen. The first of these approaches closely simulates the exposure conditions of the ICV. References that used this approach were used to develop a correlation to calculate the hydrogen concentration in the 9975 package [2]. The data from these references are summarized in Table 1.

Some of the exposures listed in Table 1 may not have been long enough to reach a steady state. In particular, those concentrations listed as surface values were significantly higher than the concentration deeper inside the specimen. However, the surface concentrations are assumed to be steady state values. The consistency of the correlation below in matching most of the data supports this assumption.

Table 1. Hydrogen absorption data

Material	Exposure Time (days)	Exposure Temperature (K)	H_2 Pressure (psi)	Hydrogen Conc. In Specimen (appm) *	Reference
Aust. SS	(long time)	348	145(tritium)	340 (surface conc.)	5
304L	1	300	10000	55	6
304L	14	846	10000	1400 (constant profile)	12
304L	(long time)	700	2000	2000	Cited in 13
304L	10	473	3480	2500 (surface conc.)	14
308L weld	10	473	3480	2500 (surface conc.)	15
308 weld	4	673	3556	2780 (average)	13
"similar" to 308/347	4	673	3556	2775-3330	Cited in 13
304L	10	473	3495	3050 (surface conc.)	16
304L	30	373	10000	3122	17
304L	10	473	10000	4000(surface conc.)	14
304L	(long time)	470	10000	4000 (constant profile)	22

* The hydrogen concentration is reported as appm, wt ppm, or ccNTP/cc metal. I ccNTP/C_e metal = 11.25 wt ppm [14].

Ignoring trace elements, the conversion from wt ppm to appm for 304 stainless steel is given by: At.wt. SS / At.wt. H = (56(0.73)+52(0.18)+59(0.08)+55(0.01))/(1) = 55.5 and 1 wt ppm = 55.5 appm. This value is consistent with conversions reported in reference 8.

Two references identify correlation for the steady state concentration of hydrogen absorbed in 304L stainless steel [14, 18]. Both correlations are of the same form, but use different coefficients. Sieverts' law describes the pressure dependence, and an Arhennius relationship describes the temperature variation. Combining these relationships gives the following correlation:

$$C_e = C_o \sqrt{P} \exp(-E/T)$$

Where C_e = equilibrium hydrogen concentration (appm) C_o = empirical constant
P = hydrogen gas pressure (psi)
E = empirical constant (=activation energy / gas constant) T = absolute temperature of gas (K)

Specific correlations described in References 14 and 18, were converted to render them in the present format [2]. Comparing each of these correlations to the Table 1 data gives a reasonable match to the specific data reported in that particular reference. However, neither correlation gives a good match to the collected data of other researchers. Accordingly, a third correlation was developed to provide a better match to the larger body of data in Table 1 [2]. Table 2 shows all three correlations and their predictions for the conditions listed in Table 1.

The predicted hydrogen concentrations from the nominal correlation previously developed [2] compare well to most of the data. The few data points that are not closely predicted are conservatively over-predicted. In order to provide a calculation, which conservatively bounds all the data, the correlation coefficient is increased to produce a bounding correlation of:

$$C_e = 70\ P^{1/2}\ \exp(-150/T)$$

Table 2. Steady State hydrogen concentration correlation predictions (as reported Reference 2)

	I	Hydrogen Concentration (appm)			
Temp. (K)	H_2 Pressure (psi)	Measured	Prediction of Ref. 14 correlation[1]	Prediction based on Ref. 18 correlation[2]	Prediction of correlations[3]
298	14	11	24	11	124 / 160
348	145	340	130	190	430/550
300	10000	55	660	320	3300/4200
846	10000	1400	5700	580000	4600/5900
700	2000	2000	2000	110000	2000/2500
473	3480	2500	1300	13000	2400/3000
473	3480	2500	1300	13000	2400/3000
673	3556	2800	2500	120000	2600/3300
673	3556	2800-3300	2500	120000	2600/3300
473	3495	3000	1300	13000	2400/3000
373	10000	3100	1300	3100	3700/4700
473	10000	4000	2300	23000	4000/5100
470	10000	4000	2200	21500	4000/5100

[1] $C_e = 188\ P^{1/2}\ \exp(-1002/T)$

[2] $C_e = 356500\ P^{1/2}\ \exp(-3485/T)$

[3] Nominal case: $C_e = 54.9\ P^{1/2} \exp(-150/T)$ / Bounding case: $C_e = 70\ P^{1/2}\ \exp(-150/T)$

3.2.2 Steady State Hydrogen Concentration in the ICV

A bounding calculation estimates the hydrogen partial pressure within the SAFKEG shipping package to be the design pressure for the ICV of 116 psia [3]. The amount of hydrogen expected to be generated from the maximum entrained water for the plutonium materials in the 3013 canister would be considerable less (i.e., 15 psia) [3]. Using the gross conservative assumption of hydrogen at the ICV design pressure as a bounding case, and calculating the level of hydrogen from the correlation developed in the previous section, the steady state hydrogen concentration in the shipping package is 550 appm.

3.2.3 Correlation of Hydrogen Concentration with Embrittlement Effects

Hydrogen embrittlement of 304L stainless steel is manifest in two ways that could impact the structural integrity of the SAFKEG shipping package. Regardless of the stress state of the package, the absorption of hydrogen can reduce the ductility. This ductility reduction is dependent on temperature; a minimum in ductility occurs between 220 and 280 K (-64 to 44 F) [24]. However, it is unlikely that the temperatures of the ICV and OCV would drop below room temperature for extended periods of time. Changes in yield and tensile strength are also observed. In addition, depending on the stress levels, the presence of hydrogen can lead to slow crack growth, propagating to failure over time. These two phenomena are examined separately.

A number of studies have examined the effect of hydrogen concentration on the mechanical properties of 304L stainless steel. In general, the ductility (measured as reduction in area or elongation) decreases with increasing hydrogen concentration and the strength can either increase or decrease with increasing hydrogen. Figure 1 illustrates these trends. The literature data are summarized in Table 3 and Figure 2.

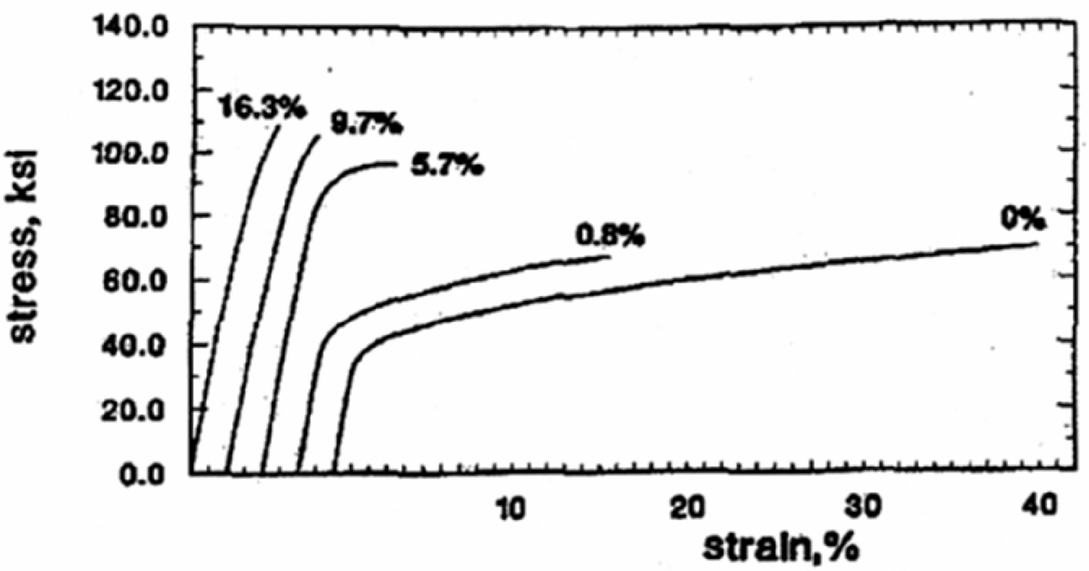

Figure 1. Stress-strain curves for 304 stainless steel at varying hydrogen concentrations (given in wt%). [8]

Table 3. Change in mechanical properties due to hydrogen absorption

H conc.	UTS (ksi)			Yield		(ksi)	Elongation (%)			
(appm)	no H	with H	% change	no H	with H	% change	no H	with H	% change	Referenc
33	97	99	2	64	64	0	--	--	--	19
110	108	99	-8	--	--	--	--	--	--	7
2500 *	87	91	5	52	59	13	53	44	-17	16
2500 *	90	89	-1	57	62	9	23	16	-30	15
2780 *	85	97	14	--	--	--	36	37	3	14
3050	88	77	-12	31	32	3	77	32	-58	17
3100	87	77	-11	31	32	3	73	33	-55	18
3300	95	80	-16	--	--	--	57	49	-14	20
3420	87	77	-11	--	--	--	--	--	--	21
4000 *	90	97	8	57	69	21	23	12	-48	15
8000	70	67	-4	41	36	-12	40	18	-55	8
22200	67	58	-13	26	34	31	34	3	-91	10
57000	70	97	39	41	80	95	40	7	-82	8

* Weld metal samples

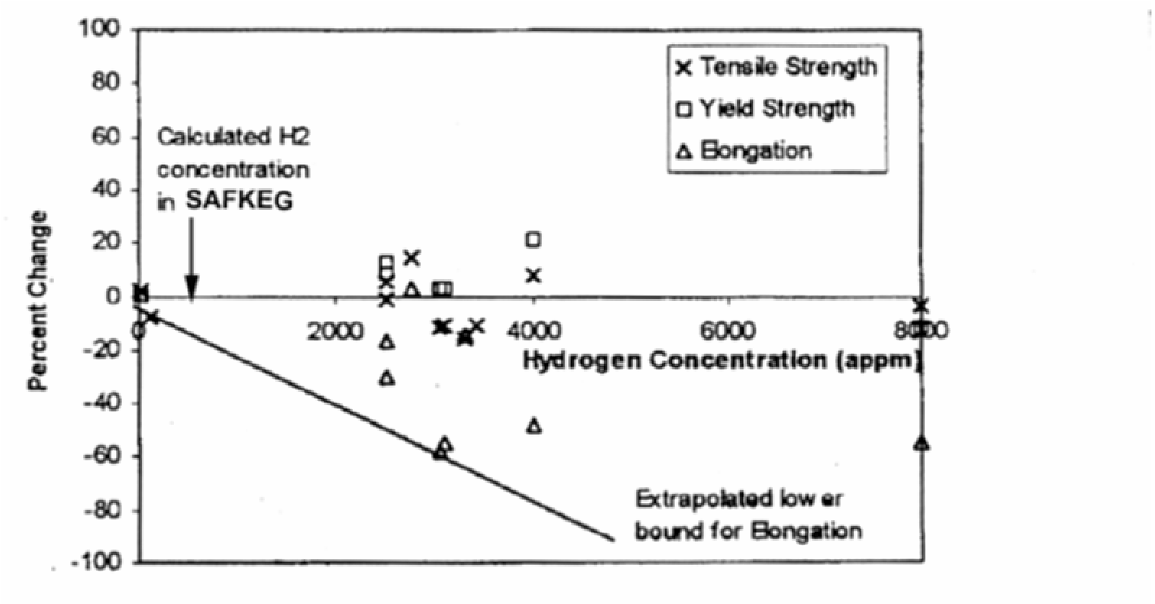

Figure 2. Change in mechanical property parameters with hydrogen concentration, using data from Table 3.

Table 3 includes data for both wrought 304L and 308L weld metal. Both material forms show a distinct drop in elongation with hydrogen levels of 2500 appm or more. The changes in tensile and yield strength are less consistent. The wrought material tensile strength generally decreases with hydrogen absorption, while the weld metal tensile strength generally increases with hydrogen absorption. The yield strength of both materials increased with hydrogen absorption in all but one case, although the magnitude of the increase was variable.

Slow crack growth can occur in hydrogenated stainless steels if the local stress intensity is great enough. The stress intensity is a function of the applied stress and the geometry and orientation of defects in the material. Such defects could include cracks, slag, and lack of fusion in welds. The ICV and OCV receive non-destructive testing in accordance with Section III of the ASME Boiler & Pressure Vessel Code [3] to ensure the absence of such defects; however, such inspections can only assure the absence of defects which are above a detectable size. The threshold stress intensity for slow crack growth is shown in Figure 3 for two heats of 304 stainless steel.

Using the lower of the Figure 3 curves, the SAFKEG shipping package hydrogen concentration is expected to cause slow crack growth at a threshold stress intensity of about 90 MPa-√m, or 81 ksi√in. In practice, this threshold is not an absolute limit, but a value below which the crack growth rate drops rapidly with decreasing stress intensity. In addition, the threshold stress intensity might be somewhat lower than this value for other heats of material. Over the extended period of time the SAFKEG shipping package will be in transit and storage, some amount of crack growth might occur if stress intensities approached this threshold value.

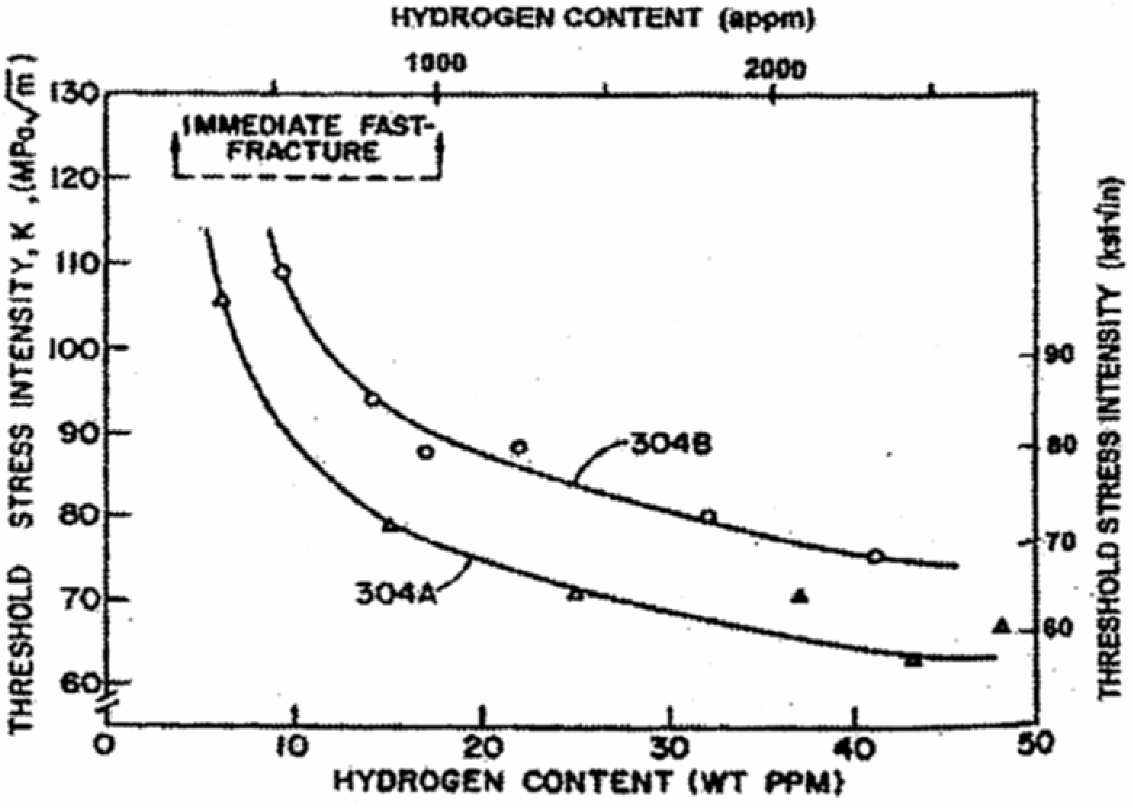

Figure 3. Threshold stress intensity for slow crack growth vs. hydrogen concentration. (Data from Reference 25 for two heats of 304 stainless steel)

3.2.4 Impact of Hydrogen Exposure on the SAFKEG ICV

A maximum equilibrium concentration of about 550 appm hydrogen is estimated for the ICV of the SAFKEG shipping package. No literature data were identified for material at this specific concentration.

However, numerous data were identified at higher concentrations and the effects recorded for those higher concentrations will serve to characterize the behavior at this lower concentration.

The ICV and OCV are constructed of 304L stainless steel components meeting the requirements of ASTM A-240, A-312 and A-479. In addition, the lid nut is constructed of S21800 (a.k.a. nitronic-60) meeting the requirements of ASTM A-479. The requirement in the safety analysis [3] was that the yield stress would not be exceeded, but additional concern would be raised if the elongation was significantly reduced [3]. A line added to Figure 2 provides a bounding linear extrapolation of the elongation data. This line shows a ~15% relative decrease in elongation at 550 appm hydrogen concentration. Reference 21 reports that the strain to fracture (i.e. elongation) decreases linearly with increasing hydrogen concentration until a saturation level is reached. This validates using the linear extrapolation of Figure 2.

The weld metal has a minimum specified elongation of 35% [22]. Applying the same relative ductility loss to this value produces a minimum elongation of 30% following absorption of 550 appm hydrogen. The weld metal strength is at least as high as that of the base metal, and the effects of hydrogen on strength are bounded by the effects on the base metal. Accordingly, the moderate loss in ductility of the weld metal will not compromise the structural integrity of the ICV and OCV.

The SAFKEG shipping package is designed to withstand normal conditions of transport and hypothetical accident conditions without the stresses in the ICV and OCV exceeding specified allowable values. For normal operation, the maximum allowable stress for the ICV and OCV is 16.7 ksi [3], which is below the minimum specified yield strength of 19.2 - 25 ksi (depending on temperature). Based on design analyses, the applied stress will never reach the material yield strength for normal operating conditions, especially since the yield strength will likely increase upon absorption of hydrogen. The yield strength might be briefly exceeded during postulated accidents that lead to deformation of the OCV or ICV. In these extreme cases, the accident analyses will demonstrate margin to failure is maintained.

4.0 DISCUSSION

Austenitic stainless steels (including 304L stainless steel) exhibit a high degree of toughness and ductility in a wide range on environments, making them the preferred material for many demanding applications. Compared to other types of stainless steel (i.e. ferritic and martensitic types) and other steels in general, the austenitic stainless steels are relatively insensitive to hydrogen environments. Laboratory testing in hydrogenous environments has shown some degradation in mechanical properties. Since hydrogen absorption generally tends to increase the yield strength of austenitic stainless steel, the absorption of hydrogen does not impact the margin to yielding under service loads deleteriously. It is noted on the other hand that the presence of hydrogen might accelerate failure of a component if other defects or failure mechanisms are active.

The hydrogen concentration calculated in this report for the ICV is an equilibrium concentration for the maximum package temperature of 392 ºF. This assumes that the package remains at this temperature sufficiently long for equilibrium to be reached. In practice, the actual temperature will be lower than this for most of the time, and the hydrogen concentration will be correspondingly lower. This evaluation did not attempt to estimate the time required for the hydrogen to reach equilibrium in the ICV. It is conservatively assumed that this concentration is reached immediately and lasts for the lifetime of the package. The ductility loss predicted for the ICV will accrue as the equilibrium hydrogen concentration builds up. No further degradation will occur from the hydrogen after that point. Therefore, this evaluation is time-independent. Hydrogen embrittlement is not predicted to occur during the projected service period of 12 years, or for any foreseeable period afterwards. Note, however, that the available test data address hydrogen embrittlement of austenitic stainless steels only for specific periods of exposure duration.

No literature data was found specific to hydrogen embrittlement of S21800. While the PCV lid nut is not exposed to the environment inside the PCV, any hydrogen present will eventually diffuse throughout all components. However, in this case a different material must be used for the lid nut to prevent galling. The hydrogen embrittlement potential of S21800 may differ from 304L stainless steel, but even if the potential of S21800 is greater than 304L, this component would be exposed to much less hydrogen that of the PCV or the lid. Accordingly, the potential for hydrogen embrittlement of the S21800 lid nut is less than that of the rest of the PCV, and no impairment of the lid nut structural integrity is predicted.

5.0 CONCLUSIONS

Hydrogen embrittlement of austenitic stainless steels has been documented in the form of reduced ductility and slow crack growth under loads that normally would not produce cracking. Bounding calculations show the amount of hydrogen absorbed into the SAFKEG ICV is sufficient to produce a modest reduction in the ICV ductility. This reduction is not sufficient to compromise the vessel structural integrity. Minor changes in strength can also occur, with a slight increase in yield strength being the most likely change. Slow crack growth in the ICV is not predicted for the calculated hydrogen concentration. Specifically, these results are based on an assumed maximum vessel temperature of 392 ºF which is the maximum temperature of the ICV, including normal and accident conditions as a transportation package, and 116 psia which is the design pressure for the ICV. The steel of the ICV exposed to hydrogen at these conditions would lead to a hydrogen content of 550 appm in the ICV steel.

6.0 QUALITY ASSURANCE

The work in this report involved the compilation and evaluation of information from literature sources. Execution of this technical work and its documentation were performed in accordance with the requirements in the WSRC E7 Manual, procedure 3.60. Internal technical review of this report is governed by the WSRC E7 Manual, procedure 2.40.

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