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# A COMPENDIUM OF MECHANICAL TESTING OF AUSTENITIC STAINLESS STEELS IN HYDROGEN†

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## ABSTRACT

Archival materials test data on austenitic stainless steels for service in high pressure hydrogen gas has been reviewed. The bulk of the data were from tests conducted prior to 1983 at the Savannah River Laboratory, the predecessor to the Savannah River National Laboratory, for pressures up to 69 MPa (10,000 psi) and at temperatures within the range from 78 to 400 K (-195 to 127 °C). The data showed several prominent effects and correlations with test conditions:

- There was a significant reduction in tensile ductility as measured by reduction of area or by the total elongation with hydrogen. Hydrogen effects were observed when the specimens were tested in the hydrogen environment, or the specimens were precharged in high pressure hydrogen and tested in air or helium.
- There was a significant reduction in fracture toughness with hydrogen (and sometimes in tearing modulus which is proportional to the slope of the crack resistance curve).
- The effects of hydrogen on ductility can be correlated to the nickel content of the iron-chromium-nickel steels. The optimal nickel content to retain the high tensile ductility in these alloys was 10 to at least 20 wt. %.
- The effects of hydrogen can be correlated to the grain size. Large grain sizes exhibited a greater loss of ductility compared to small grain sizes.

The Savannah River Laboratory test data, especially those not readily available in the open literature, along with the sources of the data, are documented in this paper.

## INTRODUCTION

The Savannah River Laboratory (SRL), the predecessor to the Savannah River National Laboratory (SRNL), carried out decades of research on the effects of hydrogen and hydrogen isotopes on the mechanical properties of materials in support of high pressure hydrogen and hydrogen isotope systems materials selection and design. Caskey [1], in 1983 provided the most comprehensive SRL database, in which the stainless steels were categorized into four major groups or alloy types:

- Type I) Iron-Chromium-Nickel Alloys – 304L, 304N, 309S, 310, 316, Carpenter 20 Cb-3, Incoloy® 800H (Huntington Alloys Inc.), Nickel 200, Nickel 301, and 440 C;
- Type II) Iron-Chromium-Nickel-Manganese Alloys – Tenelon® (U. S. Steel Corp.), Nitronic®- 40 or 21-6-9 (Armco, Inc.), Nitronic®-50 or 22-13-5 (Armco, Inc.),

18-18 Plus® (Carpenter Technology), X18-3 Mn, 18-2 Mn, and 216;

- Type III) Precipitation Hardenable Alloys – A-286, JBK-75 (a modified form of A-286), 17-4PH, AM-363, CG-27, and Ni-SPAN-C (Alloy 902); and
- Type IV) High purity alloys – Alloy A (18Cr-10Ni), Alloy B (18Cr-14Ni), and Alloy C (18Cr-19Ni).

The tests and the test conditions for the above four alloy categories were first reported in Reference [1] in 1983. In addition, in 1977 Caskey and Ratliff [2] reported materials considerations in developing onboard hydrogen storage systems (and options) for vehicular use in an early initiative (1970s) for hydrogen as a replacement for hydrocarbon fuel with a key date set to 2015. The hydrogen effects on structural materials including austenitic stainless steels, embrittlement mechanisms, and fracture modes, etc. were thoroughly discussed. The stainless steel test data in Reference [2], and those published in the public domain, such as Caskey, et al. [3,4], Louthan, et al. [5,6], and Somerday, et al. [7], were carefully compared with those in Reference [1]. The test environment, conditions and data type generated at SRL and relevant to mechanical properties for hydrogen systems materials selection and design was consolidated and reported in Tables 1-4 of the technical report [8].

This present paper is a compendium of the full set of SRL mechanical test data for austenitic stainless steels and related nickel-based alloys tested to investigate the effects of high pressure hydrogen exposure. Some already-published results are included for completeness, or included after corrections were made. All the data included in this report are consistent with the datasheets in Reference [1] from pp. 81-123. In particular, the ultimate tensile strength (UTS) has been converted, as possible, to the quantity that is commonly defined as the engineering stress at the peak load. The true (plastic) strain at failure has been converted to a more familiar parameter, Reduction of Area (RA). These results are also reported in Tables 5-9 of reference 8.

The revised tensile properties of stainless steels are presented in this paper. The hydrogen effect on tensile ductility for the iron-chromium-nickel alloys can be strongly correlated to the weight percent of nickel and is graphically presented with the existing data. The grain size effect is discussed in the context of 304L stainless steel test data. Fracture testing in hydrogen environments is also discussed. Both $J_m$ (J-integral at the maximum load) and stress intensity factor (K) are reported for stainless steels under various test environments and exposure conditions. The orientation effect of the high energy rate forging (HERF) is discussed. A limited amount of specimen sensitivity study on the effect of thickness and notch/precrack was conducted.

A review of the test methods and results summarized in this paper demonstrates the importance of standardized testing. A large deviation of test data may be expected for testing materials in air with precharged hydrogen versus testing in high pressure gaseous hydrogen with precharged specimens.

## TENSILE PROPERTIES

Most of the tensile tests were carried out with smooth bar specimens with gage lengths of 12.7, 25.4, and 50.8 mm. The data are summarized in detail in a comprehensive review of these properties [8]. The test temperature ranged from 4 K (liquid helium) [9], 78 K (liquid nitrogen) to 380 K in air [1]. The test pressure was up to 69 MPa (10,000 psi) in helium or hydrogen. Some test specimens were not exposed to gases prior to testing, however, some were precharged with hydrogen or deuterium at various temperatures, durations, and pressures. The test data were reported as 0.2% offset yield stress, stress at 5% strain, UTS or UTS in true stress, uniform elongation (elongation at UTS), elongation at break or total elongation, reduction of area (RA), and/or true failure strain. Occasionally, there were tests conducted with thin sheet specimens and tube specimens [1]. The data from circumferentially notched tensile bars are not included in this paper, since they were used to enhance the hydrogen effect by stress concentration and therefore do not represent standard tensile properties. Most of the tensile tests were carried out with a crosshead speed of 0.5mm/min for specimens with 25.4 mm gage [1, 4, 10-13].

The tensile properties of the alloys from the SRL studies are listed in their entirety in reference 8; see the Introduction section of this paper for various alloys under the four categories: Type I Fe-Cr-Ni), Type II (Fe-Cr-Ni-Mn), Type III (precipitation hardenable), and Type IV (high purity). Because the conditions of hydrogen precharge would bias the test results, only the room temperature (298 K) data from noncharged specimens but were tested in air and in 69 MPa gaseous environments (hydrogen and helium) were reported. Additional tensile data for hydrogen-precharged high purity alloys (A, B, and C) tested in air at room temperature are provided to complement the data when the test pressures of the noncharged specimens are untraceable [8].

### Hydrogen Effects on Tensile Ductility

The ductility loss is the most pronounced hydrogen effect on tensile test results for stainless steels. This significant phenomenon is reflected by the data reported in Reference [1], which documented the mechanical testing conducted at SRL from 1970s to 1983 for unexposed (not precharged) specimens that were tested in high pressure hydrogen; and for specimens precharged with hydrogen with various conditions (duration and temperature) and then tested in air, helium, or hydrogen environments. The hydrogen pressure of 69 MPa was used for precharging most of the test specimens, or for the test chamber environment. No systematic studies were conducted for the effects of pressure level on tensile ductility.

Hydrogen concentrations were measured for some exposed tensile specimens and the results are listed in Table 1. In addition, the grain size and nickel content were found to be related to the degree of ductility loss (or relative reduction of area, RRA) in hydrogen environments (Fig. 1).

## Hydrogen Concentration

The hydrogen concentration in metal may be an indication of degree of hydrogen damage, which is manifested by the reduction in tensile ductility or reduction in fracture toughness. Specimens were cut from the gage or the end of the post-test tensile specimens and the hydrogen concentrations were measured with a LECO RH-1 Hydrogen Determinator [4]. Table 1, which was reproduced from Reference [4], shows the hydrogen concentration when the tensile specimens were exposed to 69 MPa deuterium ($D_2$) at 620 K for three weeks prior to testing. Some of the "retained tensile ductility" data discussed in the section of "Effects of Nickel Content," and the fracture toughness data discussed in the section of "J-integral Testing ($J_m$)" are the direct consequence of this specimen precharge condition. It is believed that the high hydrogen concentrations in Tenelon®, Nitronic® 40, and Nitronic® 50 are caused by the presence of nitrogen added as austenite stabilizer and strengthener, which trapped the excess hydrogen in the materials [4].

**Table 1: Hydrogen (Deuterium) Concentration in Austenitic Stainless Steel Tensile Specimens [4]**

| Alloy | Hydrogen Concentration (cc $D_2$/cc alloy) |
|---|---|
| 304L (bar) | 4.5 |
| 310 (plate) | 6.5 |
| 316 (bar) | 4.9 |
| 330 (bar) | 5.1 |
| A286 (bar) | 4.4 |
| I800H (bar) | 4.0 |
| **Nitronic® 40 (bar)*** | **8.7** |
| **Nitronic® 50 (bar)*** | **12.8** |
| **Tenelon® (bar)*** | **10.0** |
| A | 2.3 |
| B | 5.1 |
| C | 4.8 |

*nitrogen added in these alloys

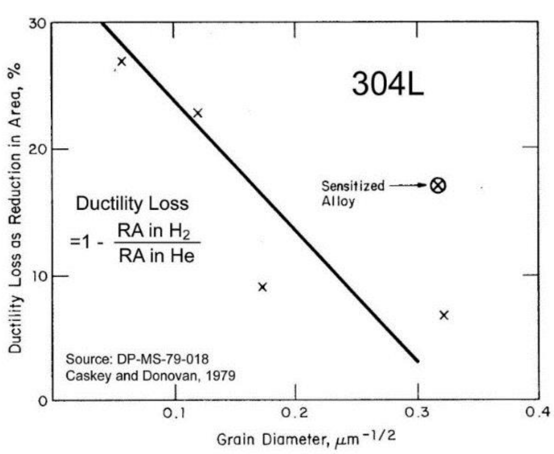



**Figure 1: Ductility loss in 69 MPa hydrogen environment for 304L with various grain sizes [14]**

## Effect of Grain Size

Stainless steel 304L was heat treated to form various grain sizes ranging from 9.5 μm (as received) to 340 μm (annealed at 1470 K for 24 hours). Unexposed tensile specimens were tested in 69 MPa helium and in 69 MPa hydrogen. It can be seen from Figure 1 that 304L with the larger gain size is more susceptible to hydrogen damage based on losing RA [14]. This is the only known study at SRL using 304L stainless steel for grain size effect.

## Effect of Nickel Content

It was first reported by Caskey [1, 3] that there is a strong correlation between the hydrogen embrittlement and the nickel content in the iron-chromium-nickel alloys based on tensile testing in 69 MPa hydrogen environment at room temperature. By plotting the RRA of the Fe-Cr-Ni alloys versus the nickel composition, low values of RRA are apparent in some low nickel alloys. This effect is even more apparent below room temperature (~200 K) [1]. It can be seen that the resistance to hydrogen damage in ductility begins to improve at nickel content between 8 to 14 wt.%. It is possible that the austenite stability was increased with respect to the transformation to α'-martensite at room temperature and to ε-martensite when the nickel content is increased (both α'-martensite and ε-martensite are detrimental to ductility). This correlation appeared to be valid for commercial grade and high purity alloys. The relationship between the RRA and the nickel content has been recently reconstructed by Morgan [17] and was modified by adding more alloy data. The resulting plot, similar to the Figure 1 in Reference [1], is shown in Figure 2. Most of the data points were obtained by room temperature testing of unexposed (not precharged) specimens in 69 MPa hydrogen, except A-286 and 17-4 for which hydrogen-precharged specimens were used in testing. Detailed conditions of the testing and the specimens in Figure 2 can be found in reference 8.

Note that the RRA in Figure 2 is defined as $RA_{H2}/RA_{air}$ or $RA_{H2}/RA_{He}$, where $RA_{H2}$, $RA_{air}$, and $RA_{He}$ are, respectively, the reduction of area (RA) for specimens tested in hydrogen, in air, and in helium. It appears that the optimal nickel content to retain the tensile ductility in wrought Fe-Cr-Ni alloys is 10 to at least 20 wt. %.

The actual values of the reduction of area for alloys in Figure 2 listed in Table 5 of reference 8. These test results indicated that the ductility of alloys 309S, 310, and HERF A-286 was actually increased in the hydrogen environment, contrary to the common observation. It should be noted that alloy 440C contains zero nickel, and exhibited a completely brittle fracture at break (no reduction of area).

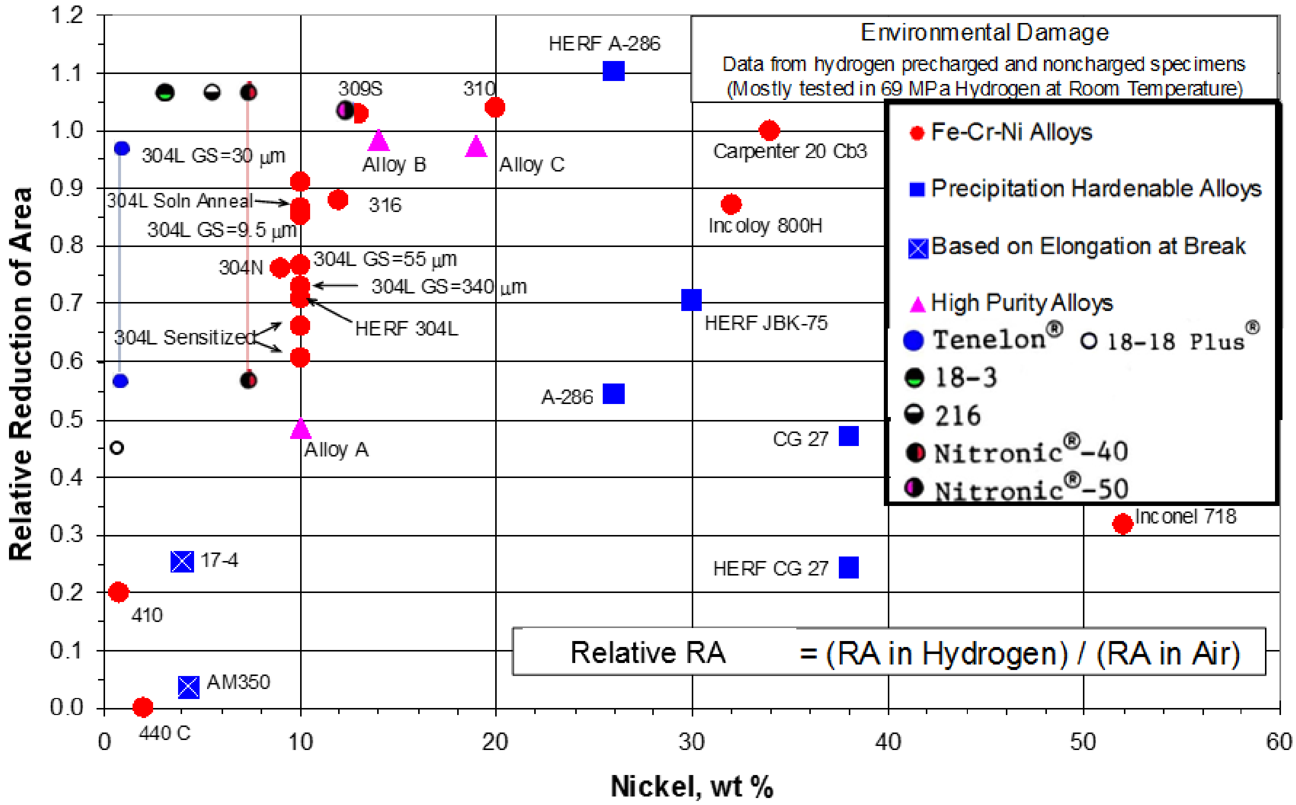



**Figure 2: Correlation between relative reduction of area (RRA) and nickel content for Fe-Cr-Ni and high purity alloys [1, 3, 17]**

The correlation between the resistance to hydrogen damage and the nickel content was not unique for iron-chromium-nickel-manganese alloys. As pointed out by Caskey [1, 3], manganese stabilizes austenite and should improve the mechanical properties of stainless steels in hydrogen service. However, as can be seen from Figure 2, the alloys with high manganese but with little or no nickel (e.g., 18-18 Plus® and Tenelon®, respectively) behave poorly in hydrogen environment. This indicates that austenite stability in itself is insufficient to minimize hydrogen damage. The susceptibility of precipitation strengthened alloys (A-286, CG 27 and Inconel 718) to hydrogen embrittlement, regardless of nickel content, can also be observed.

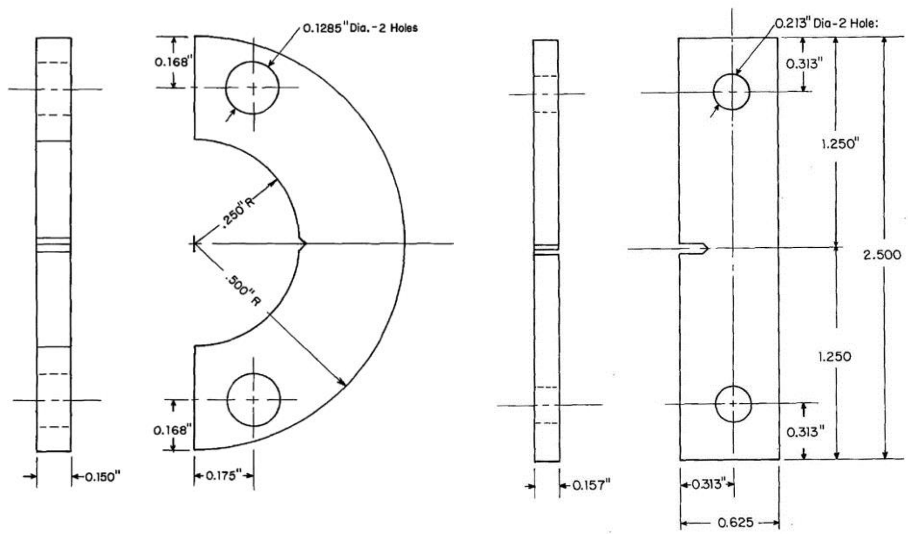
## FRACTURE PROPERTIES

The loss of fracture toughness is a pronounced hydrogen effect in stainless steels. Most of the SRL fracture testing was carried out with C-shaped specimens (Fig. 3a), which is a standard test specimen in ASTM E 399 [18] for linear elastic fracture mechanics. In addition, the single edge notched tension (SENT) specimens (Fig. 3b) were occasionally employed. The test results were summarized in Reference [1]; and for several HERF stainless steels, data can be found in Reference [19]. Because of the instrumentation difficulties for measuring fracture parameters in high pressure hydrogen environment, and the tedious test procedure for elastic-plastic fracture mechanics (ASTM E 813 [20]), SRL developed $J_m$ approach [1] as an alternative parameter for $J_{IC}$. The $J_m$ is the J-integral value calculated at the maximum load, at which the crack initiation is assumed to take place. A subsequent verification study was carried out with A-286 and 21-6-5 stainless steels [21] following ASTM E 813 procedure. It was demonstrated that the $J_m$ is about 10% higher than $J_{IC}$. However, this is considered acceptable [21] because the J-integral testing with the same material using the same technique often times contains even higher data deviation than 10%, and that is the inherent nature of material ductile failure under elastic-plastic deformation. Furthermore, considering the data deviation resulted from different fracture toughness measurement techniques or different specimen types, the variation between $J_m$ and ASTM $J_{IC}$ appears to be small.

### Fracture Data for Forged Alloys

Strong orientation effects on the mechanical properties have been noted for HERF stainless steels, especially in the determination of fracture toughness. The C-shaped specimens were fabricated such that the initial machine notch was parallel (0°), 45°, or perpendicular (90°) to the forging flow lines. The schematic specimen layout [1, 22] can be seen in

Figure 4. The actual forging flow lines in such materials can be observed through scanning electron microscopes, as shown in Figure 5. Test results indicated that the crack growth resistance was very poor when the initial notch was in parallel with the forging flow lines. Markedly higher J-integral values were obtained for notch orientation at 45° or 90° with respect to the flow lines.




(a) C-shaped specimen (b) single edge notched tension (SENT)

**Figure 3: SRL Fracture test specimens**

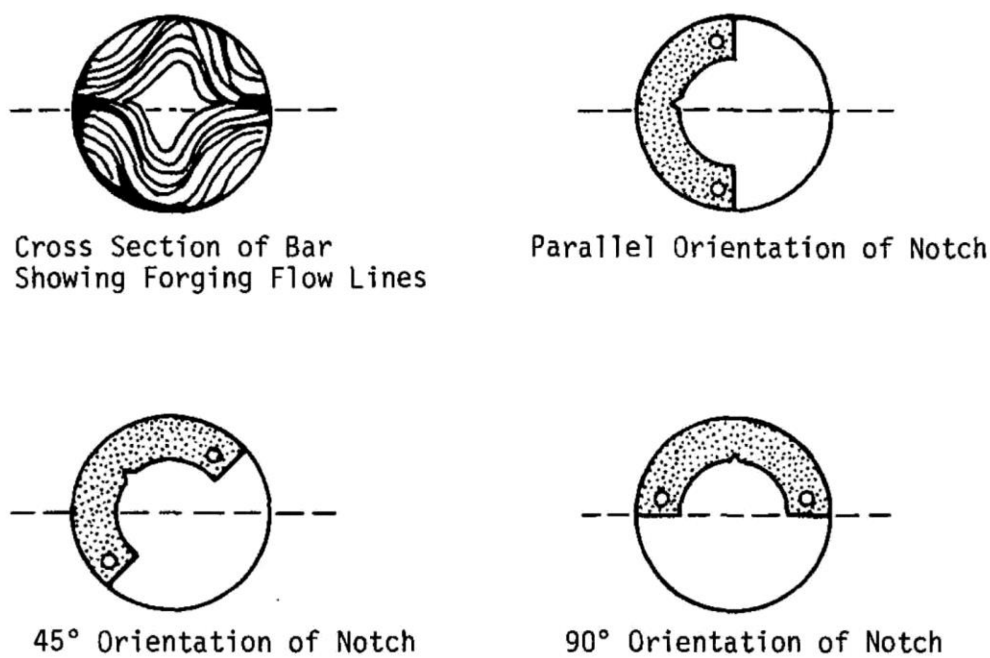
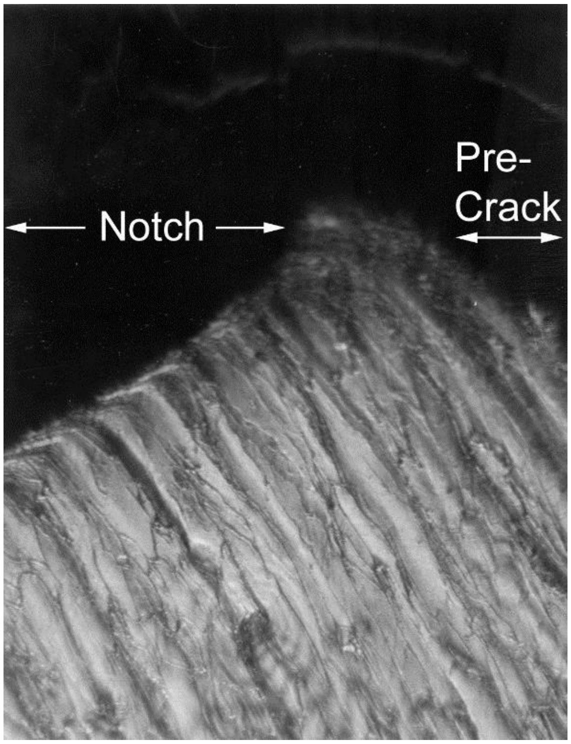



**Figure 4: Notch orientation and the forging flow lines**




(a) Specimen Cross-section (SEM 500X)

(b) Fracture surface (SEM 500X)

**Figure 5: Forging flow lines as shown by scanning electron microscope (SEM). The actual dimension was not labeled [21].**

### J-integral Testing ($J_m$)

The J-integral test data ($J_m$) for HERF 304L, Nitronic 40® (21-6-9), Nitronic 50® (22-13-5) and A-286 are shown in Figures 6 to 9, respectively. Additional test data obtained prior to June 1982 were summarized in Reference [22], which are reproduced in Table 2 and plotted in Figure 10. Note that the values of $J_m$ have been corrected for combined tension and bending in the specimen [23], and were averaged if multiple orientations were tested. In many cases, a difference between $J_m$ values measured during tests in hydrogen versus deuterium charging and testing in hydrogen is significant and can be clearly seen in Figures 6-9.

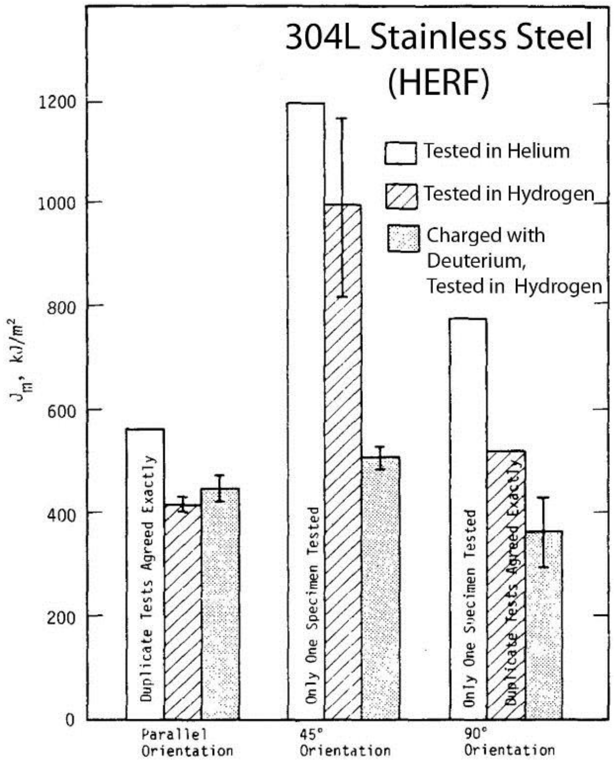



**Figure 6: J-integral test results for HERF 304L under various test environments and specimen conditions [1].**

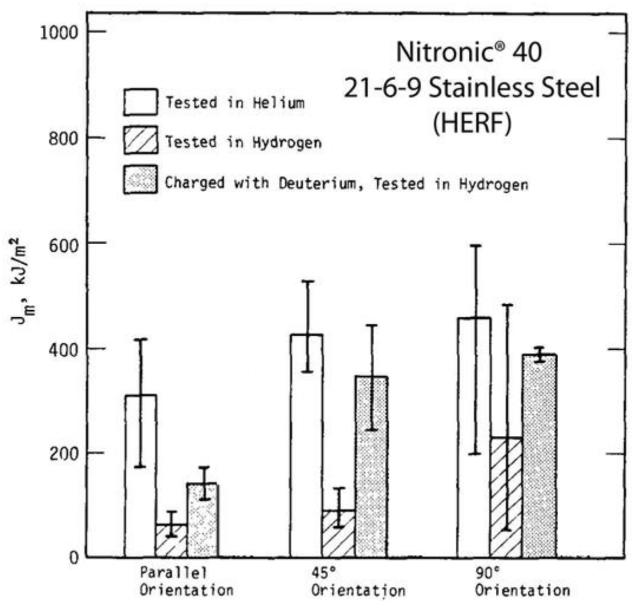



**Figure 7: J-integral test results for HERF Nitronic 40® (21-6-9) under various test environments and specimen conditions [1].**

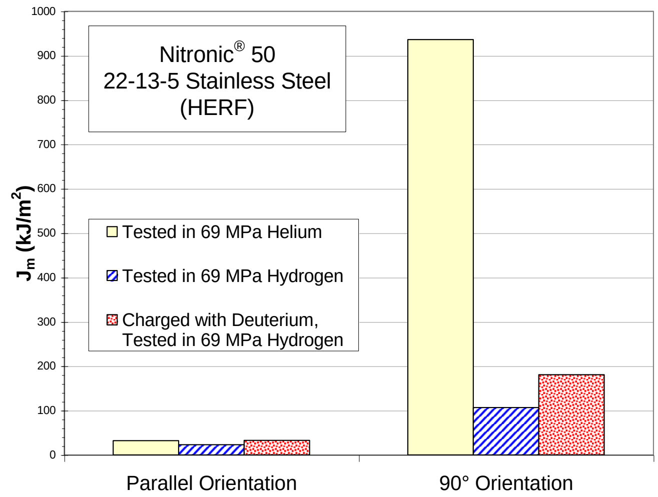



**Figure 8: J-integral test results for HERF Nitronic 50® (22-13-5) under various test environments and specimen conditions (based on Data Sheet IIC-3 in Ref. [1]).**

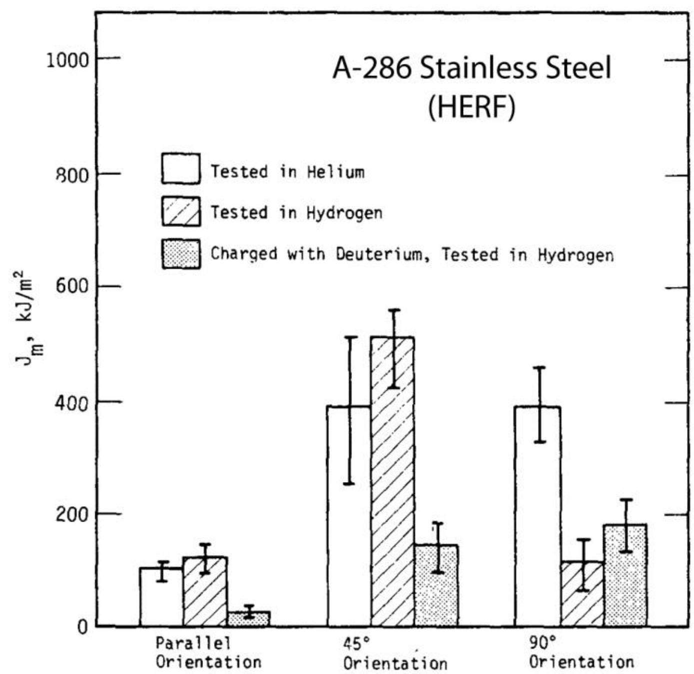



**Figure 9: J-integral test results for HERF A-286 under various test environments and specimen conditions [1].**

**Table 2: Summary of SRL fracture test results up to June 1982 [23]**

| Alloy | $J_m$[1] Tested in 69 MPa He ($kJ/m^2$) | $J_m$[1] Tested in 69 MPa $H_2$ ($kJ/m^2$) | $J_m$[1] Precharged in $D_2$ and tested in 69 MPa $H_2$ ($kJ/m^2$) | Remarks |
|---|---|---|---|---|
| 304L HERF | 701 | 573 | 489 | - |
| 316 HERF | 792 | 880 | - | - |
| 316 WR[2] | 312 | 268 | - | 1 orientation |
| 310S HERF | 537 | 417 | 291 | 6J Forging |
| 21-6-9 HERF[5] | 686 | 475 | 695 | - |
| 21-6-9 HERF[5] | | 468 | 158 | 2 orientations |
| 21-6-9 CRP[3] | 1409 | 1158 | - | Forging Step 7, 2 orientations |
| 21-6-9 WR[2] | 281 | 259 | - | 1 orientation |
| JBK-75 HERF | 560 | 377 | 201 | - |
| A-286 HERF | 539 | 497 | 132 | - |
| 22-13-5 HERF | 289 | 72 | 116 | 2 orientations |
| 17-4PH STA[4] | 80 | 4 | - | - |
| 17-4PH Annealed | 995 | 85 | - | - |

1. $J_m$: with Merkle-Corten correction [23] for the combined tension and bending in specimens. The values were averaged if multiple orientations were tested.
2. WR: Warm Rolled
3. CRP: Cross-Rolled Plate
4. STA: solution treated/aged at 783K for 1 hour
5. Alloys from different sources.

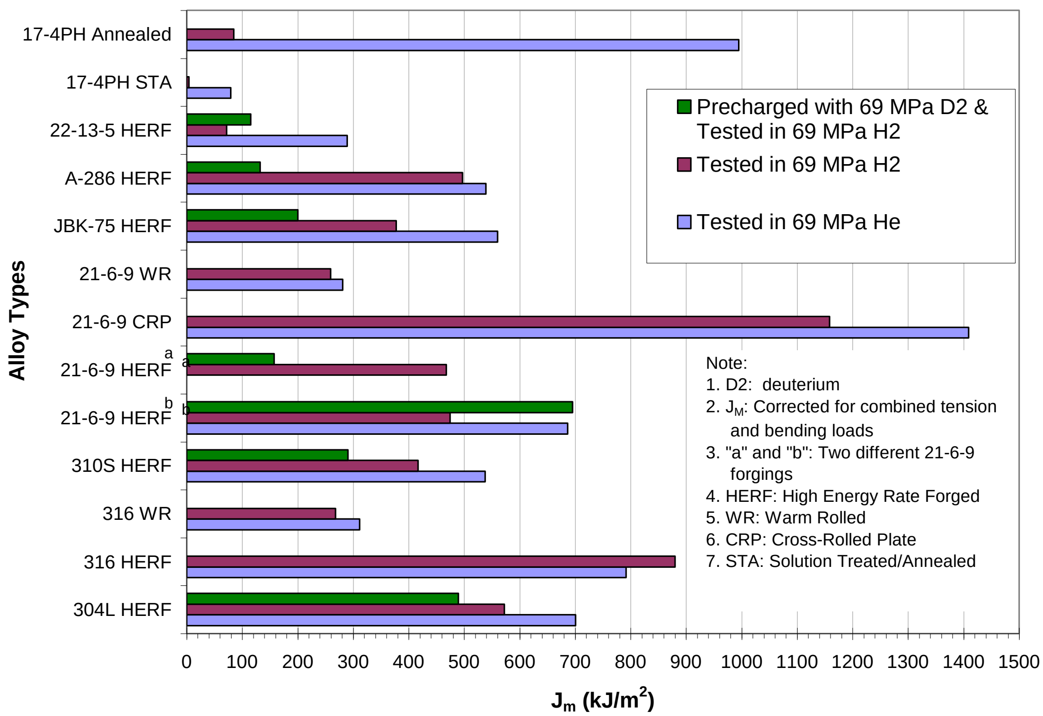



**Figure 10: Fracture toughness ($J_m$) for various types of stainless steel. Note that the values for HERF materials were averaged by the number of orientations that were tested.**

## Thickness and Notch Effects (HERF 21-6-9)

Thickness and notch effects on fracture properties with C-specimens were investigated with HERF MP35N (nickel-cobalt based alloy) and HERF 21-6-9 in 69 MPa helium and in 69 MPa hydrogen [22]. In addition to the SRL standard C-specimen thickness (3.81 mm or 0.15 in.), another thickness of 6.35 mm (¼ in.) was chosen. The initial machine notch length was 1.27 mm. Two specimens with 6.35 mm thick were not precracked (tested respectively in helium and in hydrogen). The test results for 21-6-9 are plotted in Figure 11. The data scatter is less for the standard thickness (thinner specimens).

The averaged $J_m$ values for testing in helium are higher than that in hydrogen, which is consistent with the data trend of hydrogen damage. However, the overall data scattering leads to inconclusiveness for the thickness and the notch effects. In fact, the higher averaged values of $J_m$ for thicker specimens seem to contradict the constraint theory in fracture mechanics, which predicts that, qualitatively, thinner specimens tend to have higher fracture toughness [24] because it allows much larger plastic zone to develop around the crack tip. All the discrepancies may be the result of test specimen orientation due to the anisotropy of the HERF materials (see Fig. 7 for alloy 21-6-9). A refined experiment with a carefully designed test matrix could resolve the discrepancies.

Two additional sets of test data found in Reference [22] are included in Figure 11: 1) Two specimens with different orientations precharged with deuterium in 69 MPa at 190 °C for six weeks and then tested in 69 MPa hydrogen environment (denoted by □ in Fig. 11); and 2) Two specimens tested in 69 MPa hydrogen in another experiment (denoted by Δ in Fig. 11). These data further suggest that the testing for HERF materials be conducted with careful planning and characterization.

## Stress Intensity Factor (K) Testing

Alloys Tenelon®, HERF Nitronic® 40 (21-6-9), HERF A-286, HERF JBK-75, and 17-4 PH were tested for fracture toughness in terms of stress intensity factors under various test environments (temperatures or high pressure gases) and specimen preparations (aged, annealed, or exposed to hydrogen at difference pressures). The tests were conducted with either C-shaped (Fig. 3a) or SENT (Fig. 3b) specimens. The results are listed in Tables 3 to 7.

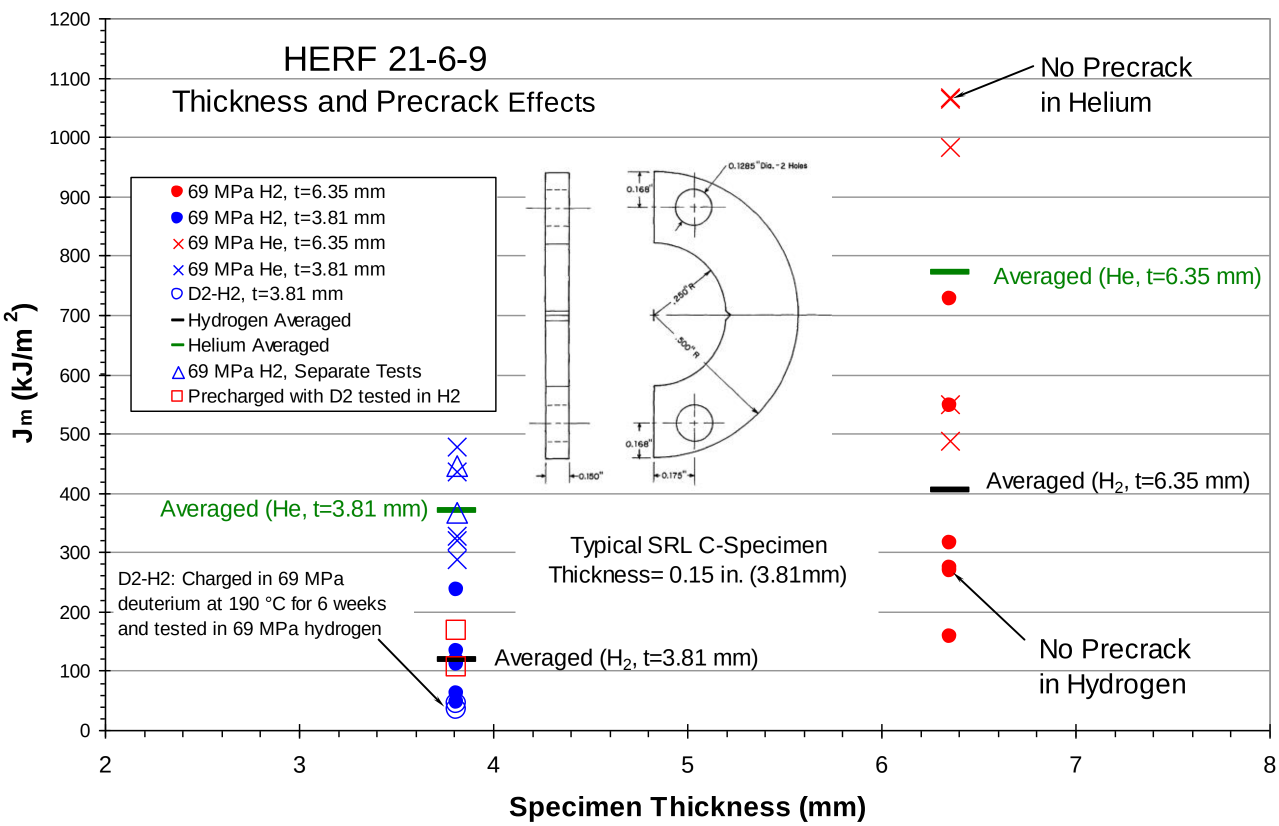



**Figure 11: Thickness and notch effects on fracture toughness ($J_m$) of HERF 21-6-9 in hydrogen environment**

**Table 3: Fracture toughness (K) for Tenelon®**

| Tenelon® (Ref.: Data Sheet IIA-3, Ref. [1], page 100) | | | | |
|---|---|---|---|---|
| Test Specimen: SENT (Fig. 3b) | | | | |
| Test Temperature (kelvin) | Test Environment | Specimen Condition | Specimen Exposure | Fracture Toughness ($MPa\sqrt{m}$) |
| 78 | - | As received | - | 68.6 |
| 78 | - | Annealed, 1170 K | - | 36.5 |
| 78 | - | Annealed, 1270 K | - | 71.4 |
| 200 | - | As received | - | 127.8 |
| 200 | - | Annealed, 1170 K | - | 99.6 |
| 200 | - | Annealed, 1270 K | - | 120.5 |

**Table 4: Fracture toughness (K) for HERF Nitronic® 40 (21-6-9)**

| Nitronic® 40 (Alloy 21-6-9) HERF (Ref.: Data Sheet IIB-10, Ref. [1], page 106) | | | | |
|---|---|---|---|---|
| Test Specimen: C-specimen (Fig. 3a) | | | | |
| Test Temperature (kelvin) | Test Environment | Specimen Condition | Hydrogen Exposure | Fracture Toughness ($MPa\sqrt{m}$) |
| 298 | 69 MPa He | - | none | 79 |
| 298 | 69 MPa $H_2$ | - | none | 81 |
| 298 | 69 MPa $H_2$ | - | 0.6 MPa $H_2$ | 62 |

Note: For independent test results for $J_m$, see Figure 7.

**Table 5: Fracture toughness (K) for HERF A-286**

| A-286 HERF (Ref.: Data Sheet IIIA-2, Ref. [1], page 114) | | | | |
|---|---|---|---|---|
| Test Specimen: SENT (Fig. 3b) | | | | |
| Test Temperature (kelvin) | Test Environment | Specimen Condition | Hydrogen Exposure | Fracture Toughness ($MPa\sqrt{m}$) |
| 298 | 69 MPa He | Aged 4 hrs 990 K (Heat 1) | none | 76 |
| 298 | 69 MPa $H_2$ | Aged 4 hrs 990 K (Heat 1) | none | 89 |
| 298 | 69 MPa He | Aged 8 hrs 990 K (Heat 1) | none | 71 |
| 298 | 69 MPa $H_2$ | Aged 8 hrs 990 K (Heat 1) | none | 90 |
| 298 | 69 MPa He | Aged 16 hrs 990 K (Heat 1) | none | 81 |
| 298 | 69 MPa $H_2$ | Aged 16 hrs 990 K (Heat 1) | none | 82 |
| 298 | 69 MPa He | Aged 8 hrs 990 K (Heat 2) | none | 93 |
| 298 | 69 MPa $H_2$ | Aged 8 hrs 990 K (Heat 2) | none | 89 |
| 298 | 69 MPa He | Aged 8 hrs 990 K (Heat 2) | 1.6 MPa ($D_2$) | 88 |
| 298 | 69 MPa $H_2$ | Aged 8 hrs 990 K (Heat 2) | 1.6 MPa ($D_2$) | 97 |
| 298 | 69 MPa He | HERF, not aged, $R_c$-11 | none | 52 |
| 298 | 69 MPa $H_2$ | HERF, not aged, $R_c$-11 | none | 56 |
| 298 | 69 MPa $H_2$ | HERF, not aged, $R_c$-11 | 1.5 MPa ($D_2$) | 59 |
| 298 | 69 MPa He | Aged 8 hrs 990 K $R_c$-11 | none | 93 |
| 298 | 69 MPa $H_2$ | Aged 8 hrs 990 K $R_c$-11 | None | 90 |
| 298 | 69 MPa $H_2$ | Aged 8 hrs 990 K $R_c$-11 | 1.5 MPa ($D_2$) | 97 |

Note: $D_2$ denotes deuterium

**Table 6: Fracture toughness (K) for HERF JBK-75**

| JBK-75 HERF (Ref.: Data Sheet IIIB-2, Ref. [1], page 118) | | | | |
|---|---|---|---|---|
| Test Specimen: C-specimen (Fig. 3a) | | | | |
| Test Temperature (kelvin) | Test Environment | Specimen Condition | Hydrogen Exposure | Fracture Toughness ($MPa\sqrt{m}$) |
| 298 | 69 MPa He | - | None | 80 |
| 298 | 69 MPa $H_2$ | - | None | 80 |
| 298 | 69 MPa $H_2$ | | 0.7 MPa $D_2$ at 625 K | 81 |

**Table 7: Fracture toughness (K) for17-4 PH**

| 17-4 PH (Ref.: Data Sheet IIIC-2, Ref. [1], page 119) | | | | |
|---|---|---|---|---|
| Test Specimen: C-specimen (Fig. 3a) | | | | |
| Test Temperature (kelvin) | Test Environment | Specimen Condition | Hydrogen Exposure | Fracture Toughness ($MPa\sqrt{m}$) |
| - | 69 MPa He | Underaged[1] | - | 104 |
| - | 3.5 MPa $H_2$ | Underaged | - | 31 |
| - | 69 MPa $H_2$ | Underaged | - | 20 |
| - | 69 MPa He | Peak aged[2] | - | 97 |
| - | 3.5 MPa $H_2$ | Peak aged | - | 29 |
| - | 69 MPa $H_2$ | Peak aged | - | 13 |
| - | 69 MPa He | Overaged[3] | - | - |
| - | 3.5 MPa $H_2$ | Overaged | - | 57 |
| - | 69 MPa $H_2$ | Overaged | - | 34 |
| - | 69 MPa He | Solution annealed[4] | - | 97 |
| - | 3.5 MPa $H_2$ | Solution annealed | - | 71 |
| - | 69 MPa $H_2$ | Solution annealed | - | 31 |

Condition of Heat Treatments: 1 Solution annealed 2 hours at 1339 K and aged at 709 K, Hardness $R_c$= 38
2. Solution annealed 2 hours at 1339 K and aged at 783 K, Hardness $R_c$= 42
3. Solution annealed 2 hours at 1339 K and aged at 866 K, Hardness $R_c$= 35
4. Hardness $R_c$= 28

## CONCLUDING REMARKS

A range of austenitic stainless steels were tested for hydrogen compatibility for service condition up to 69 MPa (10,000 psi) hydrogen and temperatures from 78 to 400 K (some tests were carried out at 4 K in liquid helium) at the Savannah River Laboratory (the predecessor to the Savannah River National Laboratory) to support materials selections and designs for systems in high pressure hydrogen service. These steels included the iron-chromium-nickel alloys (304L, 304N, 309S, 310, 316, Carpenter 20 Cb-3, Incoloy® 800H, Nickel 200, Nickel 301, and 440C), iron-chromium-nickel-manganese alloys (Tenelon®, Nitronic®-40 or 21-6-9, Nitronic®-50 or 22-13-5, 18-18 Plus®, X18-3 Mn, 18-2 Mn, and 216), precipitation hardenable alloys (A-286, JBK-75, 17-4PH, AM-363, CG-27, and Ni-SPAN-C or Alloy 902), and high purity alloys (18Cr-10Ni, 18Cr-14Ni, and 18Cr-19Ni). An in-depth summary of the hydrogen transport in these alloys (permeation) and the hydrogen effects on the mechanical properties (tensile and fracture) was provided by Caskey [1]. This present report reviewed the SRL test data which are, in general, not readily available in the open literature. The following conclusions can be made:

- Hydrogen has a minor influence on the yield stress and the ultimate tensile strength of the austenitic stainless steels. However, the tensile ductility suffers significant loss when the hydrogen is present, either externally as the service environment, or internally resulting from extended exposure or precharging. This material behavior (hydrogen embrittlement) is similar in carbon steels [25, 26].
- The ductility loss increases as the grain size increases, as shown by 304L testing on the heat treatment effects [14] (Fig. 1).
- The retain ductility [1, 17], defined by the ratio of reduction of area in hydrogen to the reduction of area in helium, correlates well with the nickel content in Fe-Cr-Ni alloys. The optimal nickel content to retain the tensile ductility in wrought Fe-Cr-Ni alloys is 10 to at least 20 wt. % (Fig. 2).
- The fracture toughness testing shows a strong orientation effect with respect to the forging flow lines in the high energy rate forged (HERF) stainless steels (Figs. 6-10).

- The fracture toughness (J-integral or stress intensity factor) is reduced significantly when the hydrogen is present in the test environment or internally in the metal by extended exposure to hydrogen (Figs. 6-10). Similar behavior has been observed for carbon steels [25, 27].

The SRL test data also indicated that the specimen condition has a significant influence on the mechanical property measurement, such as the surface polishing or plating, and the orientations in the HERF stainless steels. Previous testing attempted to explore the effects of specimen geometry (such as the sample thickness and precracking), but only inconclusive results were obtained. A refined experiment with advanced fracture mechanics analysis of the constraint effect may be employed to resolve the discrepancy and uncertainty.

More recent SRNL test data are mostly related to tritium exposure and aging, which results in helium-3, a radioactive decay product, and has a different mechanism for mechanical property degradation. Limited hydrogen-only (tritium-only) effects are reported and the information is available in open literature (e.g., [28-32]). The general trend is consistent with the earlier data which have been covered in this paper. The quantitative comparison is not possible because the alloy composition, specimen fabrication, exposure condition, and test environment may be different.

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