# Characterization of Structural Materials for Nuclear Hydrogen Generation

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## Introduction

A zirconium alloy, namely Zr705 has been tested for evaluation of its metallurgical and corrosion behavior for application as a structural material in heat exchangers for nuclear hydrogen generation. The results of tensile testing at different temperatures indicate that the magnitude of the yield strength (YS) and ultimate tensile strength was gradually reduced with increasing temperature, as expected. However the ductility in terms of percent elongation was enhanced at temperatures up to 200°C followed by its reduction beyond this temperature. No failure was observed with this alloy when tested in a 90°C simulated acidic solution at a constant load equivalent to 98% of its YS value. The results of stress corrosion cracking (SCC) tests using the slow-strain-rate (SSR) technique indicate that Zr705 may experience increased ductility at elevated temperatures when tested in a similar environment. The results of localized corrosion study using polarization technique indicate that this alloy did not exhibit any pits or crevices. SCC testing in an autoclave indicates that the C-ring specimens did not undergo cracking in a similar environment at higher temperatures showing insignificant weight-loss as a function of the exposure period. Fractographic evaluation of the tested specimens by scanning electron microscopy (SEM) indicates that the primary fracture surface was characterized by dimpled microstructures, indicating ductile failures.

**Keywords** Zr-705, Cracking, Constant-Load, Slow-strain-rate, Tensile Testing, Fractography

## Work Description

The United States Department of Energy has initiated an ambitious program to generate hydrogen as an alternate source of energy using nuclear power. The structural materials to be used in the heat exchanger for nuclear hydrogen generation must withstand unusually high temperatures (up to 400°C) and aggressive chemical species (pH≈1) associated with the application of a thermochemical cycle known as sulfur-iodine (S-I) process. While many metals and alloys are being considered as structural materials for such applications, this paper is focused on the metallurgical and corrosion characterization of a zirconium (Zr) alloy known as Zr705. The chemical composition and the metallurgical microstructure of the solution-annealed Zr705 are shown in Table 1 and Figure 1, respectively.

**Table 1. Chemical Composition of Zr705**

| Material/Heat No. | Element (Wt %) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Zr | Hf | Fe + Cr | H | N | C | Nb | O |
| **Zr705/ 845558 Zr** | 95.5 (min) | 0.9 | 0.1 | 0.0003 | 0.006 | 0.009 | 2.6 | 0.12 |

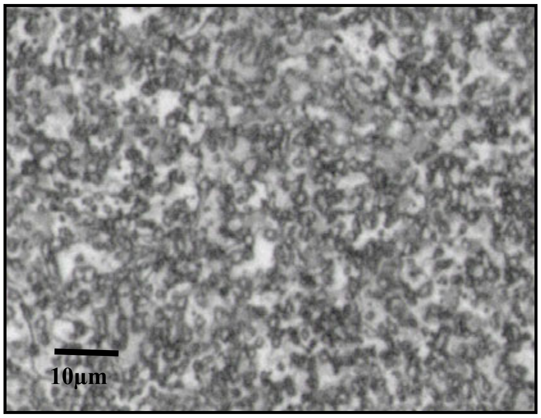


**Figure 1. Optical Micrograph of Zr705, HF 4-5%, $HNO_3$ 30-35% and $H_2O$ balance, 100X**

The tensile properties of the Zr705 have been determined incorporating cylindrical specimens at temperature ranging from ambient to 400°C using a conventional testing machine. The susceptibility of this alloy to SCC has been determined under constant-load (CL) and slow-strain-rate (SSR) conditions in an environment relevant to the S-I cycle at ambient and elevated temperatures. SCC testing has also been performed using self-loaded C-ring specimens in an autoclave containing an acidic solution at 150°C. Since the cracking susceptibility of a structural material can be influenced by an external potential, SCC testing has also been performed using the SSR technique at potentials, anodic or cathodic with respect to the critical potentials determined by the cyclic potentiodynamic polarization (CPP) technique. The extent and morphology of failure in cylindrical specimens have been determined by SEM. The configuration of the cylindrical specimen is shown in Figure 2. The comprehensive test results are given below.

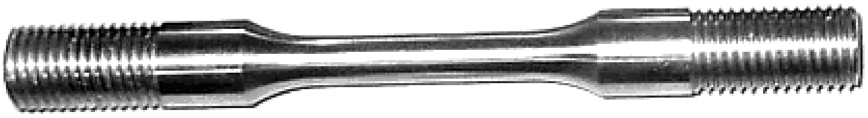


**Figure 2. Cylindrical Specimen Configuration**

## Results

1. The comparisons of the engineering stress versus strain (s-e) diagrams for Zr705 at different testing temperatures are shown in Figure 3, indicating reduced yield strength (YS) and ultimate tensile strength (UTS) values with increasing temperature, as expected. The variations of YS and UTS at different temperatures are illustrated in Figure 4.

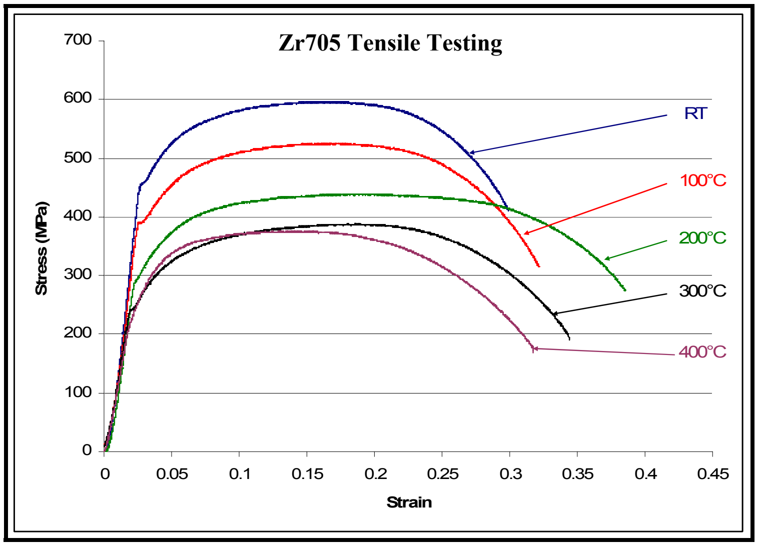
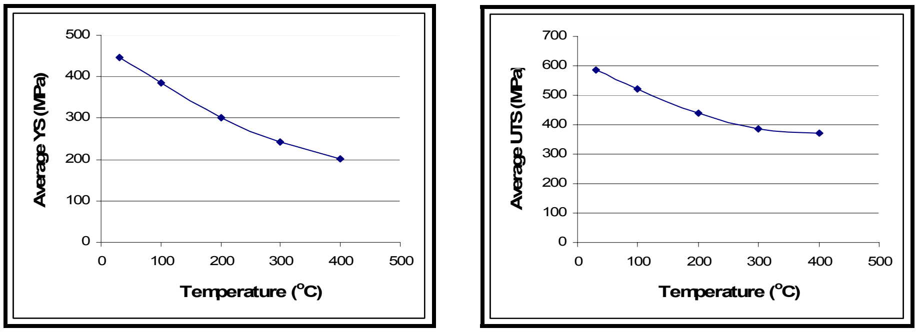


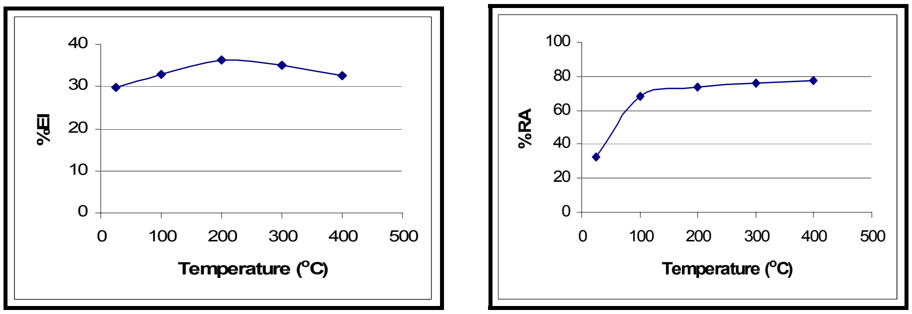
Figure 3. Stress-Strain Diagram vs. Temperature



(a) (b)

Figure 4. Comparison of YS and UTS vs. Temperature

2. The ductility in terms of %RA was enhanced at higher temperatures (Figure 5a). However, the magnitude of %El was enhanced only at temperatures up to 200°C, followed by a gradual decline at temperatures beyond 200°C (Figure 5b). The reduced failure strain at temperatures above 200°C may be attributed to dynamic strain ageing of Zr705. A similar behavior has been reported for Zr alloys in the open literature [1,2,3].



(a) (b)

Figure 5. Comparison of %El and %RA vs. Temperature

3. No failures were observed in the SCC testing at CL even at an applied stress corresponding to the 98 percent of the room temperature YS value of Zr705.

4. The results of SSR testing revealed enhanced ductility in terms of failure strain at higher temperature (Figure 6), showing no significant effect of the testing environment. The SSR test results are given in Table 2, showing reduced true failure stress ($\sigma_f$) but increased ductility (%El, %RA) and TTF with increasing temperature.

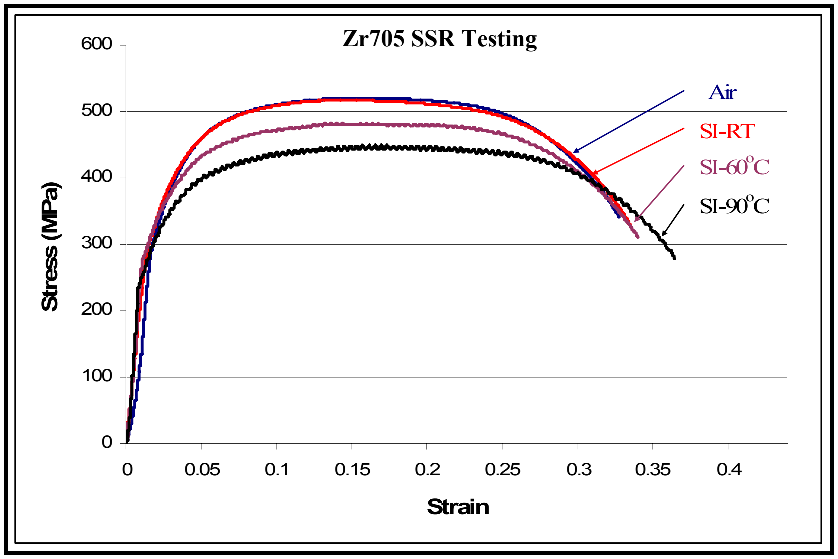


Figure 6. Stress-Strain Diagram vs. Temperature

Table 2. Average SSR test data

| Temperature (°C)/ Environment | (f (ksi) | %El | %RA | TTF (hrs) |
|---|---|---|---|---|
| Air-Ambient | 125.47 | 31.84 | 60.85 | 28.73 |
| S-I-Ambient | 120.62 | 32.1 | 60.32 | 29.535 |
| S-I-60 | 115.08 | 34.23 | 60.75 | 30.68 |
| S-I-90 | 105.82 | 34.65 | 61.41 | 31.99 |

5. No localized attack was observed in the Zr705 specimens during the CPP testing. The CPP diagram showed negative hysteresis loop during the reverse potential scan, as shown in figure 7. The magnitude of the corrosion potential (Ecorr) determined from the CPP diagram, became more active (negative) with increasing temperature as illustrated in Figure 8.

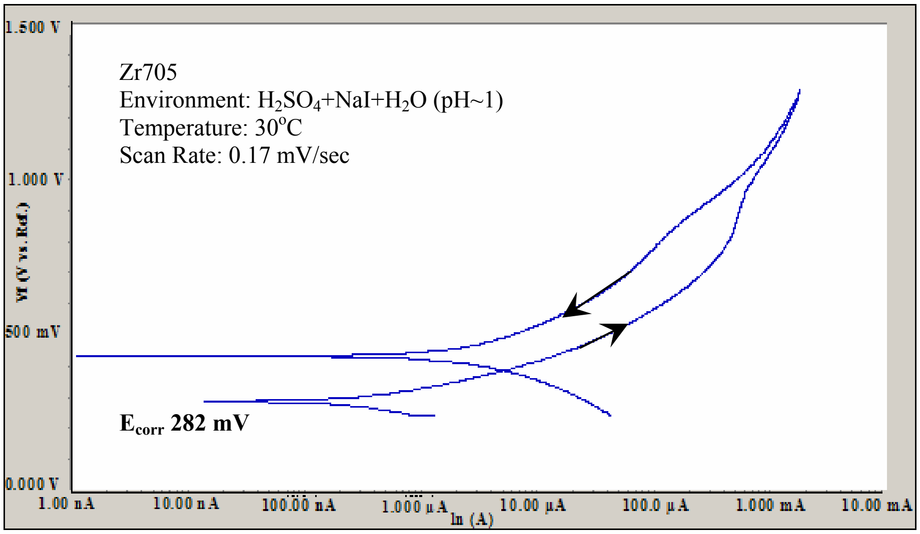


Figure 7. Results of CPP Testing.

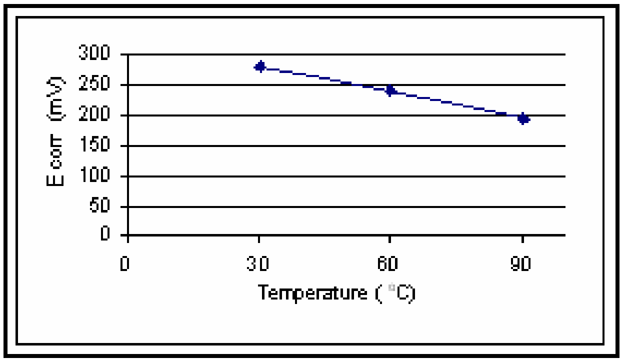


Figure 8. Corrosion Potential vs. Temperature

6. The fractographic evaluations of the primary fracture surface of the specimens used in tensile and SSR testing by SEM revealed ductile failures, characterized by dimpled microstructures. (Figure 9).

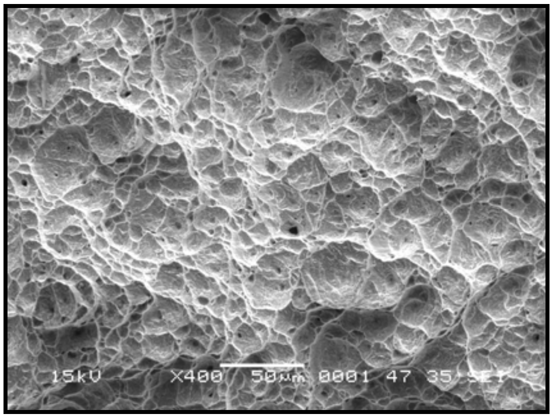


Figure 9. SEM Micrograph of Zr705 Tested at 400°C, 400X

## References

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