ABSTRACT

ALSMADI, ZEINAB YOUSEF. High Temperature Creep-Fatigue Behavior of Alloy 709 Austenitic Stainless Steel in Sodium-Cooled Fast Reactors. (Under the direction of Dr. K.L. Murty).

Generation IV (GEN IV) nuclear reactors are an important source of base load power in the middle–long term (2030–2050) and are designed to be safer, more reliable, more efficient and have longer lifetimes (60-80 years) than current nuclear reactors. The only possible mid-term available fast reactor is Sodium-Cooled Fast Reactor (SFR) with an acceleration in research and development along with reduction in economic competitiveness. However, the design and operation of SFR exhibiting cyclic thermal stresses during start-ups and shut-downs introduce time-dependent effects like creep during on-load periods leading to creep-fatigue interaction in many of the reactor components. Therefore, the structural material of SFRs should have excellent mechanical properties that can withstand the harsh operating environments. Fe-25Ni-20Cr austenitic stainless steel (Alloy 709) is developed for boiler tubing and high-temperature applications such as SFRs and ultra-supercritical (USC) power plants due to its adequate mechanical properties. Thus, it is important to understand the creep-fatigue interaction of Alloy 709 in order to generate enough data in a reasonable time and extrapolate them to the service conditions expected in SFRs.

To establish fatigue characteristics of Alloy 709, strain-controlled low-cycle fatigue (LCF) tests were performed at strain ranges from 0.3% to 2.5% at 750 °C and 2×10^{-3} s^{-1} strain rate (0.1 Hz). It is found that the predicted fatigue life of Alloy 709 shows a better correlation with the characteristic slopes method rather than the universal slopes method.

To investigate the effect of hold time on high-temperature creep-fatigue behavior of Alloy 709, strain-controlled creep-fatigue tests were performed with hold times of 60, 600, 1,800 and
3,600 seconds introduced at the maximum tensile strain at 1% strain range at $2 \times 10^{-3} \text{ s}^{-1}$ strain rate (0.1 Hz) and 750 °C. The creep-fatigue life of Alloy 709 and the number of cycles to macro-crack initiation are found to decrease with increasing hold time indicating faster crack initiation and higher growth rates. The fractographs of the deformed samples at 1% strain range with no hold time indicated that fatigue is the dominant mode of deformation whereas for the samples deformed at the same strain range with different hold times, both fatigue and creep have contributed to the overall deformation of the alloy.

The effect of strain range on high-temperature creep-fatigue interaction of Alloy 709 is investigated by conducting strain-controlled creep-fatigue tests with tensile hold times of 0, 600, 1,800 and 3,600 seconds at strain ranges varying from 0.6% to 1.2% at 750 °C and $2 \times 10^{-3} \text{ s}^{-1}$ (0.1 Hz) strain rate. In general, with increasing strain range at a given hold time, the number of cycles to failure decreases until saturation. The fractographs of the deformed samples exhibited increased number of cracks with increasing strain range along with Cr$_{23}$C$_6$ precipitates and high dislocation density.

Finally, the effect of temperature on creep-fatigue behavior of Alloy 709 is investigated by conducting strain-controlled creep-fatigue tests with tensile hold times of 0, 60, 600, 1,800 and 3,600 seconds at 1% strain range at 650 °C and $2 \times 10^{-3} \text{ s}^{-1}$ (0.1 Hz) strain rate. It is found that the number of cycles to failure at 650 °C is higher than that at 750 °C, and the material exhibits fluctuations in the number of cycles to failure at 650 °C due to the effect of Dynamic Strain Aging (DSA). The fractographs of the deformed samples exhibited increased density of cracks and creep cavities at higher temperatures along with Cr$_{23}$C$_6$ precipitates and high dislocation density. Furthermore, creep-fatigue life is evaluated by linear damage summation (LDS) of cyclic and
creep damage fractions according to ASME code and creep-fatigue interaction diagram of Alloy 709 is constructed at different strain ranges, temperatures and hold times.
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High Temperature Creep-Fatigue Behavior of Alloy 709 Austenitic Stainless Steel in Sodium-Cooled Fast Reactors

by
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A dissertation submitted to the Graduate Faculty of North Carolina State University in partial fulfillment of the requirements for the degree of Doctor of Philosophy

Nuclear Engineering

Raleigh, North Carolina
2020

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DEDICATION

To

My Mother: Sameera

My Father: Yousef
BIOGRAPHY

Zeinab Alsmadi was born in Az Zarqa, Jordan in 1993. She had her schoolings until the 12th grade in Az Zarqa, Jordan. Zeinab graduated from Jordan University of Science and Technology (JUST) with Bachelor of Science degree in Nuclear Engineering in 2016 with very good academic status. In 2017, Zeinab joined the department of Nuclear Engineering at North Carolina State University (NCSU) in Raleigh, NC, USA to fulfil higher education in Nuclear Engineering Ph.D. program. Then, she joined the nuclear material research group under the supervision of Dr. K. L. Murty and worked towards her Ph.D. on high temperature mechanical properties for structural materials of next-generation nuclear reactors. During her Ph.D., Zeinab also worked towards the non-thesis Master degree in Materials Science and Engineering (MMSE) from the department of Materials Science and Engineering at NCSU.
ACKNOWLEDGMENTS

All thanks and praises to God (ALLAH) for providing me with the power, determination and patience to have this work accomplished.

I wish to express my extreme gratitude to my PhD advisor Prof. K. L. Murty for the time he spent reviewing my career goals and recommending strategies for achieving them. His advice was very helpful and gave me a new perspective on available opportunities. I am deeply grateful for his support, encouragement and guidance throughout the years. It was an honor to pursue my PhD under the direction of Prof. Murty.

My sincere gratitude and appreciation go to the committee members: Prof. Mohamed Bourham, Prof. Ge Yang, and Prof. Ronald Scattergood for their invaluable time, feedback and discussion.

Special thanks to Dr. Nilesh Kumar, Dr. Abdullah Alomari and Dr. Boopathy Kombaiah for their helpful guidance and assistance in getting this work accomplished. I also would like to thank the staff who were very supportive and helpful throughout my time at NCSU; Ervin Miller, Chris Sanford, Emad Tawardrous, Stefanie Keto, Lisa Marshall, Robert Green, Hua Wheeler, Chintan Kanani and Mario Milev. Thanks are also given to my colleagues and friends for being kind, caring and supportive during my PhD study.

This work would have never been possible without the support, sacrifice and patience of my parents; Yousef and Sameera and my siblings; Ahmad, Fatmeh, Sarah and Ibrahim. No words can express how lucky I am to have them in my life.
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>List of Tables</td>
<td>ix</td>
</tr>
<tr>
<td>List of Figures</td>
<td>x</td>
</tr>
<tr>
<td><strong>1. INTRODUCTION</strong></td>
<td>1</td>
</tr>
<tr>
<td>1.1. Motivation</td>
<td>1</td>
</tr>
<tr>
<td>1.2. Austenitic Stainless Steels</td>
<td>3</td>
</tr>
<tr>
<td>1.3. References</td>
<td>7</td>
</tr>
<tr>
<td><strong>2. BACKGROUND AND LITERATURE REVIEW</strong></td>
<td>9</td>
</tr>
<tr>
<td>2.1. Creep Damage</td>
<td>9</td>
</tr>
<tr>
<td>2.1.1. Creep Testing</td>
<td>9</td>
</tr>
<tr>
<td>2.1.2. Creep Properties</td>
<td>10</td>
</tr>
<tr>
<td>2.1.3. Creep Mechanisms</td>
<td>14</td>
</tr>
<tr>
<td>2.1.4. Creep Damage Assessment</td>
<td>19</td>
</tr>
<tr>
<td>2.2. Fatigue Damage</td>
<td>20</td>
</tr>
<tr>
<td>2.2.1. Fatigue Testing</td>
<td>21</td>
</tr>
<tr>
<td>2.2.2. Fatigue Properties</td>
<td>21</td>
</tr>
<tr>
<td>2.2.3. Crack Growth</td>
<td>31</td>
</tr>
<tr>
<td>2.2.4. Fatigue Life</td>
<td>34</td>
</tr>
<tr>
<td>2.2.5. Fatigue Damage Assessment</td>
<td>35</td>
</tr>
<tr>
<td>2.3. Creep-Fatigue Interaction</td>
<td>36</td>
</tr>
<tr>
<td>2.3.1. Creep-Fatigue Damage Properties</td>
<td>36</td>
</tr>
<tr>
<td>2.3.2. Creep-Fatigue Damage Mechanisms</td>
<td>40</td>
</tr>
<tr>
<td>2.3.3. Factors Affecting Creep-Fatigue Life</td>
<td>43</td>
</tr>
<tr>
<td>2.3.3.1. Effect of Hold Time</td>
<td>43</td>
</tr>
<tr>
<td>2.3.3.2. Environmental Effect</td>
<td>48</td>
</tr>
<tr>
<td>2.3.3.3. Effect of Grain Size and Strain Range</td>
<td>49</td>
</tr>
<tr>
<td>2.3.3.4. Effect of Creep Ductility</td>
<td>49</td>
</tr>
<tr>
<td>2.3.3.5. Effect of Unequal Strain Rate</td>
<td>50</td>
</tr>
<tr>
<td>2.3.4. Mechanical Analysis of Creep-Fatigue Damage</td>
<td>55</td>
</tr>
<tr>
<td>2.3.4.1. Crack Initiation Endurance</td>
<td>55</td>
</tr>
<tr>
<td>2.3.4.2. Crack Growth</td>
<td>58</td>
</tr>
<tr>
<td>2.3.4.3. Post-Test Examination</td>
<td>60</td>
</tr>
</tbody>
</table>
2.3.5. Creep-Fatigue Damage Assessment .........................................................60
2.4. References .................................................................................................66

3. EXPERIMENTAL SETUP ...............................................................................69
3.1. Material .......................................................................................................69
3.2. Method .........................................................................................................71
3.3. Microstructure Characterization .................................................................72
    3.3.1. Optical Microscopy ..............................................................................72
    3.3.2. Scanning Electron Microscopy .............................................................73
    3.3.3. Transmission Electron Microscopy .......................................................74

4. EFFECT OF HOLD TIME ON HIGH TEMPERATURE CREEP-FATIGUE
   BEHAVIOR OF Fe-25Ni-20Cr (WT.%) AUSTENITIC STAINLESS STEEL
   (ALLOY 709) ...................................................................................................76
4.1. Introduction ..................................................................................................76
4.2. Experimental Details ..................................................................................78
    4.2.1. Material ...............................................................................................78
    4.2.2. Method ................................................................................................79
4.3. Results and Discussion ..............................................................................81
    4.3.1. Low-Cycle Fatigue Tests ....................................................................81
    4.3.2. Creep-Fatigue Tests ............................................................................83
        4.3.2.1. Creep-Fatigue Properties ............................................................83
        4.3.2.2. Stress Relaxation Behavior .........................................................85
        4.3.2.3. Linear Damage Summation .........................................................88
        4.3.2.4. Effect of Creep-Fatigue on Crack Properties ...............................91
        4.3.2.5. Crack Morphology .....................................................................93
4.4. Conclusions ..................................................................................................99
4.5. References ..................................................................................................101

5. EFFECT OF STRAIN RANGE ON HIGH TEMPERATURE CREEP-FATIGUE
   BEHAVIOR OF Fe-25Ni-20Cr (WT.%) AUSTENITIC STAINLESS STEEL
   (ALLOY 709) ..................................................................................................104
5.1. Introduction ..................................................................................................105
5.2. Experimental Details ..................................................................................106
    5.2.1. Material ...............................................................................................106
    5.2.2. Experimental Method .........................................................................107
5.3. Results and Discussion .................................................................108
   5.3.1. Low-Cycle Fatigue Properties ..............................................108
   5.3.2. Creep-Fatigue Life of Alloy 709 ..........................................109
   5.3.3. Hysteresis Loop Behavior ..................................................110
   5.3.4. Cyclic Stress Response ......................................................114
   5.3.5. Determination of Crack Initiation .......................................115
   5.3.6. The Effect of Strain Range on Time to Fracture .....................116
   5.3.7. Stress Relaxation Behavior ...............................................118
   5.3.8. Creep-Fatigue Interaction Diagram ....................................118
   5.3.9. Crack Morphology ...........................................................123
   5.3.10. Microstructural Evolution ................................................124
   5.3.11. Dislocation Structure and Precipitation Behavior ..................126
5.4. Conclusions ..............................................................................132
5.5. References ..............................................................................134
6. EFFECT OF TEMPERATURE ON CREEP-FATIGUE BEHAVIOR OF Fe-25Ni-20Cr (WT.%) AUSTENITIC STAINLESS STEEL (ALLOY 709) .........................139
   6.1. Introduction ...........................................................................140
   6.2. Experimental Details .............................................................141
      6.2.1. Material ........................................................................141
      6.2.2. Method .........................................................................142
   6.3. Results and Discussion ..........................................................143
      6.3.1. Effect of DSA on Creep-Fatigue Behavior ...........................143
      6.3.2. Effect of Temperature on Time to Fracture, \( t_f \) ..............145
      6.3.3. Stress-Strain Behavior ....................................................146
      6.3.4. Crack Initiation Properties ..............................................148
      6.3.5. Effect of Temperature on Degree of Work Hardening ...........149
      6.3.6. Creep-Fatigue Interaction Diagram of Alloy 709 ...............150
      6.3.7. Microstructural Damage ..................................................152
   6.4. Conclusions ...........................................................................159
   6.5. References ...........................................................................161
7. CONCLUSIONS AND SUGGESTED FUTURE WORK ..............................164
   7.1. Conclusions ..........................................................................164
   7.2. Suggested Future Work ..........................................................168
7.3. References .......................................................................................................................... 173

ACKNOWLEDGMENTS ........................................................................................................... 174

APPENDICES ........................................................................................................................... 175

APPENDIX A ............................................................................................................................. 176

List of Publications and Presentations .................................................................................... 176

APPENDIX B ............................................................................................................................. 178

Simulation and Experiments of Multi-Layer Dry Cask Shielding
Materials ................................................................................................................................... 178
LIST OF TABLES

Table 2.1  A summary of creep mechanisms with relevant parametric dependencies [1] ....... 19
Table 2.2  Influence of unequal ramp rates on endurance of alloy 800H at 850°C [4, 31] ....... 52
Table 3.1  Chemical composition (wt%) of Alloy 709 ................................................................. 70
Table 4.1  Chemical composition (wt%) of Alloy 709 ................................................................. 78
Table 4.2  The fatigue life of Alloy 709 at 750 ºC at different strain ranges ......................... 83
Table 4.3  Values of fitting parameters of power-law equation ................................................. 86
Table 5.1  Chemical composition (wt%) of Alloy 709 ................................................................. 107
Table 6.1  Chemical composition (wt%) of Alloy 709 ................................................................. 142
LIST OF FIGURES

Figure 1.1. A schematic of a typical Sodium-cooled Fast Reactor (SFR) [2].................................2

Figure 1.2. Schaeffler diagram showing the effect of the nickel and chromium content on the stability of austenite [16] .................................................................................................................................4

Figure 2.1. Experimental setup of a lever-arm creep tester [3].........................................................10

Figure 2.2. A typical creep curve showing different stages of creep [1].........................................11

Figure 2.3. The effect of (a) temperature and (b) stress on creep curves [1]...............................13

Figure 2.4. Schematic of dislocation glide–climb event [1]..........................................................16

Figure 2.5. Mass transport through the lattice as in N–H model (a) and through grain boundaries (Coble creep) [1] ..........................................................................................................................17

Figure 2.6. Tension–compression (a) and tension–tension (b) cycles [1]....................................21

Figure 2.7. A cantilever fatigue beam testing facility [1]............................................................21

Figure 2.8. S–N curves for mild steel and an aluminum alloy [1]...............................................23

Figure 2.9. Typical fatigue life plot along with corresponding stress–strain loops [1, 4, 10]....25

Figure 2.10. (a) Cyclic stress–strain curve depicting hysteresis loop. (b) Hysteresis loops during cyclic hardening (i) and cyclic softening (ii) [1]..........................................................26

Figure 2.11. Typical waveforms for strain-controlled fatigue testing [4, 12]...............................27

Figure 2.12. Parameters associated with hysteresis loop [4, 12].............................................28

Figure 2.13. The crack growth rate as a function of the stress intensity factor range [1]..........30

Figure 2.14. Schematic of fatigue crack propagation across a specimen section depicting three stages of fatigue failure [4, 13].................................................................31

Figure 2.15. (a) A schematic fatigue fracture surface showing clamshell markings. (b) An SEM image showing the striations in a fatigued 304L austenitic stainless steel [1]...........................................................................................................32

Figure 2.16. Shear decohesion mechanism for stage II fatigue crack growth [9, 14]..............33

Figure 2.17. Plastic blunting mechanism for stage II fatigue crack growth [14].......................34
Figure 2.18. Schematic representation of fatigue crack development [7].................................35

Figure 2.19. Creep-fatigue cracking mechanisms (a) fatigue dominated; (b) creep dominated; (c) creep-fatigue interaction (due to “consequential” creep damage accumulation); (d) creep-fatigue interaction (due to “simultaneous” creep damage accumulation) [7].................................................................37

Figure 2.20. Influence of hold time on the cyclic/hold creep-fatigue endurance of 1%CrMoV steel at 550 °C where crack development is identified as being pure creep (C) pure fatigue (F) or creep-fatigue (CF) [7].................................................................37

Figure 2.21. Examples of stress-strain hysteresis diagrams without hold time (a), with hold time (strain control) (b) and with hold time (force control) (c) [15] .........................39

Figure 2.22. Test specimen configurations [15]...........................................................................40

Figure 2.23. Example of Creep-Fatigue Cycle Shapes [15].................................................................41

Figure 2.24. The integrated stress-time area is shown smaller for cyclic loading (b), than it is for monotonic loading (a), thus, the damage is greater for cyclic loading [4, 16]....42

Figure 2.25. Schematic representation of how the three mechanisms of creep-fatigue interactions may lead to deviation from the linear damage rule [4, 16]..................43

Figure 2.26. Influence of hold time (10 minutes) on fatigue life of type 304 stainless steel at 923 K [4, 17]........................................................................................................44

Figure 2.27. The accumulation of creep strain calculated from a stress relaxation model as a function (a) number of cycles and (b) time \((N \times t_h)\) in 316 SS at 873 K [4, 24]........47

Figure 2.28. Partitioning of typical cyclic hold hysteresis loop \((\Delta \varepsilon_{pp} = \text{pure fatigue inelastic strain range}; \Delta \varepsilon_p = \text{plastic strain component at half-life}; \Delta \varepsilon_c = \text{true creep strain component at half-life}; \dot{\varepsilon}_{m} = \text{strain rate depicting the change in the dominant mode of damage between matrix and triple point wedge type cracking}) [4, 25].....48

Figure 2.29. Influence of creep ductility on the cyclic/hold creep-fatigue endurance of 1%CrMoV steel at 550 °C [7]........................................................................................................50

Figure 2.30. Influence of creep-ductility on creep-fatigue cracking mechanisms [7] ...............51

Figure 2.31. A schematic figure describing how a slow-fast cycle can introduce a net sliding offset in each cycle, producing of W-type cavity damage [4, 16].................................53

Figure 2.32. A fatigue damage map for austenitic stainless steel [4, 16]....................................54
Figure 2.33. Generic flow diagram representing creep-fatigue crack initiation assessment procedure [7, 33]..................................................................................................................................................57

Figure 2.34. Creep-fatigue interaction diagram for ASME Subsection NH materials. The coordinates of the intersections, I, of the bi-linear curves are shown in the legend [34]........................................................................................................................................................58

Figure 2.35. Schematic representation of (a) short-crack growth within a cyclic plastic zone; and (b) long crack growth beyond the boundary of a cyclic plastic zone [7]........58

Figure 2.36. Creep-fatigue damage diagram showing regions of different failure modes for type 316 stainless steel [4, 35]..........................................................................................................................................................61

Figure 2.37. The four types of partitioned strain-range cycles [36].................................................................63

Figure 2.38. A typical creep-fatigue strain hysteresis cycle [36]........................................................................64

Figure 2.39. Creep-fatigue crack development in a 1%CrMoV rotor steel at 565 °C [7]........65

Figure 3.1. The hot-rolled plate of Alloy 709 ........................................................................................................69

Figure 3.2. Alloy 709 sample geometry ................................................................................................................70

Figure 3.3. The Experimental set-up for LCF and creep-fatigue tests ....................................................................72

Figure 3.4. HIROX microscope (Model C7214-9015-1) .......................................................................................73

Figure 3.5. FEI Quanta 3D FEG SEM (a) and SEM Verios 460L (b) .................................................................74

Figure 3.6. JEOL 2000FX TEM (a) and FEI Talos F200X TEM (b) .................................................................75

Figure 4.1. The specimen geometry (a) and the experimental set-up (b)..............................................................80

Figure 4.2. Schematic of loading cycle of the strain-controlled creep-fatigue test depicting strain–time, stress–time and stress–strain curves without hold time (a) and with hold time imposed at the peak tensile strain (b) ..............................................................80

Figure 4.3. Hysteresis loops of the LCF tests of the Alloy 709 at 0.5% and 1% strain ranges showing the second cycle..............................................................................................................................82

Figure 4.4. Strain amplitude, $\Delta\varepsilon/2$, vs. number of reversals, $2N_f$, in log-log scale showing both LCF and HCF parts (a) and experimental life vs. predicted life of Alloy 709 under LCF and HCF (b)..........................................................................................................................83

Figure 4.5. Second cycle hysteresis loops for the creep-fatigue tests of the Alloy 709 at 1% strain range and different hold times ..........................................................................................................................85
Figure 4.6. Maximum and minimum stresses vs. number of cycles to failure at different hold times (a), maximum stress with time at different hold times (b) and number of cycles to failure vs. hold time (c) (all at 1% strain range) ...........................................87

Figure 4.7. Stress relaxation of Alloy 709 with time at 1% strain range and different hold times at mid-life cycle.................................................................................................................88

Figure 4.8. Creep-fatigue interaction diagram using linear damage summation; the red dashed line shows the ideal failure criterion........................................................................................................90

Figure 4.9. Peak tensile and compressive stress ratio for creep-fatigue tests of the Alloy 709 at 1% strain range and different hold times (a) and the effect of hold time on number of cycles to crack initiation and failure (b) .........................................................92

Figure 4.10. Effect of hold time, \( t_h \), on the time to failure, \( t_f \), (a) and one cycle time, \( c_t \), (b) under creep-fatigue .................................................................................................................................93

Figure 4.11. Fractographs of the free surface of Alloy 709 after creep-fatigue tests at 1% strain range at hold times of 600 second (a) and 3,600 second (b,c). (Stress axis perpendicular to the page)..............................................................................................................95

Figure 4.12. SEM micrograph of the as-received Alloy 709 taken in backscattered electron mode (a) and bright-field TEM micrographs at zone axis [112] with dislocations and Nb(C,N) precipitates [25] (b) and (c).........................................................................................................................96

Figure 4.13. Fractographs of the fracture surface of Alloy 709 after LCF test at 1% strain range depicting different microscopic features representative of different modes of failure.........................................................................................................................97

Figure 4.14. Fractographs of the fracture surface of Alloy 709 after creep-fatigue tests at 1% strain range and hold time of 60 second .........................................................................................................................99

Figure 4.15. Fractographs of the fracture surface of Alloy 709 after creep-fatigue tests at 1% strain range and hold time of 1,800 second .................................................................................................................99

Figure 5.1. The hot-rolled plate of Alloy 709 (a) and the specimen geometry (b) ..............107

Figure 5.2. Strain range, \( \Delta \varepsilon \), vs. number of cycles to failure, \( N_f \), in log-log scale showing both experimental data along with the predictions using characteristic and universal slopes...............................................................................................................109

Figure 5.3. The strain range vs. number of cycles to failure at different hold times at 750 \( ^\circ \)C.........................................................................................................................................................110

Figure 5.4. Half-cycle hysteresis loops for the creep-fatigue tests at 750 \( ^\circ \)C and different strain ranges and hold times of 600 seconds (a) and 3,600 seconds (b). Area
under the curve vs. strain range at same hold times at half-cycle (c) and (d)........112

Figure 5.5. Half-cycle peak stress amplitude vs. strain range at different hold times at 750 ºC..................................................................................................................................................113

Figure 5.6. Relationship between the plastic strain range, $\Delta \varepsilon_p$, and number of cycles to failure, $N_f$,..................................................................................................................................................................................113

Figure 5.7. Cyclic stress response with respect to the number of cycles in semi-log scale at different strain ranges and hold times of 600 seconds (a) and 1,800 seconds (b) ..115

Figure 5.8. The effect of strain range on the number of cycles to crack initiation at different hold times..................................................................................................................................................................116

Figure 5.9. The effect of strain range, $\Delta \varepsilon$, on the time to fracture, $t_f - t_o$, at different hold times and a strain rate of 2x10^{-3} s^{-1} ........................................................................................................................................................................117

Figure 5.10. Half-cycle stress relaxation with time at different strain ranges and hold times ....120

Figure 5.11. Creep-fatigue interaction diagram of Alloy 709 at 750 ºC; the black line shows the ideal failure criterion according to linear damage summation.........................122

Figure 5.12. Fractographs of the free surface of Alloy 709 after creep-fatigue tests at 0.6% strain range and hold times of 1,800 seconds (a) and at 1.2% strain range and hold times of 600 seconds (b). (Stress axis perpendicular to the page)..............124

Figure 5.13. SEM fractographs of the fracture surface after creep-fatigue test at 0.8% strain range and hold time of 1,800 seconds.........................................................................................................................126

Figure 5.14. SEM fractographs of the fracture surface after creep-fatigue test at 1% strain range and hold time of 1,800 seconds [7].................................................................................................................................126

Figure 5.15. Bright-field STEM image of as-received Alloy 709 at zone axis [110] showing dislocations and precipitates (a,b).........................................................................................................................129

Figure 5.16. Bright-field STEM image of Alloy 709 subjected to creep-fatigue test at 0.8% strain range and 600 seconds hold time at zone axis [110] showing dislocations and precipitates (a,b) along with EDS maps (c,d) .................................................................130

Figure 5.17. Bright-field STEM image of Alloy 709 subjected to creep-fatigue at 1% strain range and 600 seconds hold time at zone axis [110] showing dislocations and precipitates (a,c) along with EDS maps of annealing twins (c).................................131

Figure 6.1. The specimen geometry (a) and the experimental set-up (b)..............................143
Figure 6.2. Number of cycles to failure vs. hold time at 1% strain range and temperatures of 650 ºC and 750 ºC [6] .................................................................145

Figure 6.3. Maximum stress with respect to time at 650 ºC (a) and effect of hold time, \( t_h \), on the time to failure, \( t_f \), at 650 ºC and 750 ºC (b) (all at 1% strain range and different hold times) .................................................................146

Figure 6.4. Second-cycle hysteresis loops for the creep-fatigue tests at 1% strain range and 650 ºC at hold times of 0, 1,800 and 3,600 seconds (a), 60 seconds (b) and 600 seconds (c) .................................................................147

Figure 6.5. Half-cycle hysteresis loops for the creep-fatigue tests at 1% strain range and 650 ºC at hold times of 0, 1,800 and 3,600 seconds (a), 60 seconds (b) and 600 seconds (c) .................................................................148

Figure 6.6. The effect of hold time on number of cycles to crack initiation and failure under creep-fatigue tests of Alloy 709 at 1% strain range and 650 ºC .........................................................149

Figure 6.7. The effect of temperature on degree of work hardening under creep-fatigue tests of the Alloy 709 at 1% strain range and different hold times .........................................................150

Figure 6.8. Creep-fatigue interaction diagram of Alloy 709 at different strain ranges, temperatures and hold times; the green dashed line shows the ideal failure criterion according to linear damage summation \( (D=1) \) .........................................................152

Figure 6.9. Fractographs of the free surface of Alloy 709 after creep-fatigue tests at 1% strain range and 650 ºC at hold times of 0 seconds (a), 1,800 seconds (b) and 3,600 seconds (c) (Stress axis perpendicular to the page) .........................................................154

Figure 6.10. Fractographs of the fracture surface of the Alloy 709 after creep-fatigue tests at 1% strain range and 650 ºC at hold times of 0 seconds (a), 60 seconds (b), 600 seconds (c), 1,800 seconds (d) and 3,600 seconds (e) .........................................................155

Figure 6.11. Bright-field STEM image of the Alloy 709 under creep-fatigue tests at 1% strain range and 1,800 seconds hold time at 650 ºC at zone axis [110] showing dislocations (a) and precipitates (b) .........................................................157

Figure 6.12. Bright-field STEM image of the Alloy 709 under creep-fatigue tests at 1% strain range and 1,800 seconds hold time at 750 ºC at zone axis [110] showing dislocations and precipitates (a) along with EDS maps (b) .........................................................158
1. INTRODUCTION

1.1. Motivation

With increasing demand on energy these days and for the near future, there should be a continuous development of lifetime and safety of the energy systems to satisfy the future needs for energy. Generation IV (GEN IV) nuclear reactors are designed to be safer, more reliable, more efficient and have a longer lifetime (over 60 years) than current nuclear reactors that are reaching their life expectancy. An example of GEN IV nuclear reactors is Sodium-Cooled Fast Reactor (SFR) that operates at temperatures as high as 550 °C and uses sodium as coolant and moderator due to its low specific heat, high thermodynamic efficiency, high melting point and heavy atoms compared to water (Figure 1.1) [1-3]. The structural material of SFR should have superior mechanical properties that can withstand high temperatures, high corrosive environments and high radiation doses such as austenitic stainless steels with FCC structure, which are corrosion-resistant and have good creep properties due to their high nickel and chromium contents. For example, Fe-25Ni-20Cr austenitic stainless steel (known as Alloy 709) originally developed by Nippon Steel (Tokyo, Japan) contains 20% chromium and 25% nickel; it is stabilized by nitrogen and strengthened by niobium. Preliminary data suggests that Alloy 709 is an excellent candidate as structural material for SFR because it is corrosion-resistant, has high strength, sodium compatibility, thermal stability and good creep properties [4]. Alloy 709 (NF709) is also applicable in ultra-supercritical (USC) power plants as superheater and reheater for coal fired boilers [5]. Therefore, it is of importance to understand the plastic deformation of this alloy and predict how it will behave under typical SFR operating conditions. In order to achieve this goal, different mechanical testing methods should be performed to understand and thus explain the damage mechanisms of alloy 709 at similar operating conditions expected in SFR.
Creep-fatigue interaction is expected to be a contributor to the damage mode for many reactor components such as reactor cladding, pressure vessels and gas turbines operating at high temperatures in next-generation nuclear reactors [6-10], in which intergranular creep cavitation damage initiates due to applying hold times whether in tension or/compression part(s) of the fatigue cycle during startups and shutdowns. Therefore, simultaneous creep-fatigue interactions cause increased crack growth rate thereby decreasing the crack initiation endurance.

The diagnosis of mechanical failure considers the associated material condition and the mechanical characteristics prior to operating history. Material condition refers to the chemical composition and mechanical properties relative to those applied for the failed component, and the appearance and extent of microstructural and physical damage responsible for failures [11]. The American Society for Testing of Materials (ASTM) has developed specific standard procedures for testing damage mechanisms of materials and data requirements from creep, fatigue, and creep-fatigue interaction as discussed later.
High-temperature component assessment from creep-fatigue data is classified into: **a)** defect-free component assessment procedures which are used for design purposes, and **b)** defect component assessment procedures that are used for life evaluations and inspection management. Component assessments and especially defect-free assessments depend on the data obtained from creep-fatigue tests, in which creep and fatigue damages occur simultaneously or consequentially [11, 12]. There has been no standard procedure that shows how creep-fatigue tests should be performed under appropriate loading conditions, and thus data requirement for the determination of failure and crack initiation endurance were not reliable and dependent on various procedures in different test laboratories. However, this obstacle was overcome when ASTM published a new standard, E2714-09 procedure based on the Electrical Power Research Institute (EPRI) worldwide survey results on conducting a new standard creep-fatigue testing in which creep-fatigue data requirements for component assessments were identified [12]. Eventually, the results obtained from creep-fatigue tests should provide a clear indication of the influence of cyclic loading on creep deformation characteristics and the influence of creep deformation on cyclic plastic response.

**1.2. Austenitic Stainless Steels**

Austenitic stainless steels are widely used in many engineering applications including conventional and nuclear power plants because they are corrosion-resistant, have high-temperature strength, and economic feasibility of the alloys. They are primarily composed of a Fe-Cr-Ni alloys, in which the chromium content is generally over 12 wt% to form a passive chromium oxide film providing absence of staining, rusting and corroding in various aqueous or corrosive chemical environments, and nickel is added to improve the corrosion resistance as well as the high-
temperature strength of the material. Austenitic stainless steels have face-centered-cubic (FCC) crystal structures, meaning they have a close packed structure compared to ferritic steels with more open BCC lattice structure and thus, provide a reduced diffusivity in the lattice [13-15].

Austenite, ferrite and martensite are the primary phases found in stainless steels, which depend on the composition and the thermal processes involved in preparing the material. For example, martensite forms when austenite is quenched rapidly leaving the material in a metastable phase leading to increased hardness of the material. Austenite is stabilized by N, C, Ni, Mn, and Cu, while ferrite is stabilized with addition of Cr, Si, Mo, and Nb. The Schaeffler diagram shown in Figure 1.2 is an empirical guide to determine what phase(s) to expect at a certain composition in stainless steels as described by nickel equivalent and chromium equivalent. As shown, Alloy 709 composition falls within the austenitic phase domain on the Schaeffler diagram [14, 16].

Figure 1.2. Schaeffler diagram showing the effect of the nickel and chromium content on the stability of austenite [16].
Conventional and advanced austenitic stainless steels are alloyed with varying combinations
of elements which directly influence the mechanical properties over a wide range of temperatures [13, 14]:

- **Chromium**: is a crucial alloying element that improves hardenability, strength, wear resistance and the corrosion resistance in the alloy [17, 18]. It forms precipitates like $Cr_2N$, $M_6C$, $M_{23}C_6$ which enhance high-temperature creep strength and have relatively high coarsening rates leading to the disappearance of precipitate strengthening. Also, $M_{23}C_6$ carbides have detrimental effects on corrosion resistance, toughness and ductility. Other precipitates like sigma phase and Z-phase are also formed. Z-phase precipitates are highly stable, making them attractive in the advanced austenitic stainless steels for enhancing the mechanical properties at high temperatures.

- **Nickel**: is an essential element in most of the austenitic stainless steels, in which it is added for stabilizing austenite and contributing to solid solution strengthening. Nickel also plays a role in increasing the corrosion resistance of the alloy.

- **Manganese**: is an austenite stabilizer and added to increase the hardenability, ductility and wear resistance of the stainless steel. Manganese is a cheaper austenite stabilizer than Nickel. Therefore, it can reduce the cost of the stainless steel [19]. It should be noted that the composition of manganese in high-temperature stainless steels must be low because it can decrease the creep rupture life and corrosion resistance.

- **Silicon, Phosphor, Sulphur and Boron**: are added to promote oxidation, increase machinability, improve the hot workability and increase the corrosion resistance [20]. However, very small amounts of such elements could significantly affect the ductility and creep strength.
• **Molybdenum:** is added because it increases the high-temperature strength, improves creep resistance due to solid solution hardening, and can help in corrosion resistance as well [21]. In some stainless steels, molybdenum is replaced with tungsten because tungsten forms precipitates that can resist creep damage [17].

• **Niobium and Titanium:** are both ferrite stabilizer elements, and strong carbide and nitride formers which can enhance the corrosion properties [22, 23]. Niobium forms a very stable \( Nb(CN) \) precipitate that can remove carbon from the matrix helping the corrosion resistance and helps for pinning mobile dislocations [17]. The \( Nb(CN) \) precipitates are very stable at high temperatures so they will often be found in 20Cr-25Ni-Nb alloys after solution treating and quenching [18].

• **Carbon and Nitrogen:** both contribute to the strength of the material through the formation of precipitates that help in precipitate strengthening of the material. However, carbon stabilizes the austenite phase and improves the formation of chromium carbides [24]. Also, it can provide higher creep strength by solid solution strengthening and precipitation strengthening [23]. It is detrimental on the corrosion resistance and it is usually kept below about 0.03% to improve stress corrosion cracking resistance [25]. Nitrogen also assists in strengthening the material by forming nitrides like \( NbN \) and \( TiN \). However, high nitrogen content can lead to intergranular stress corrosion cracking in austenitic stainless steels [26].

The other alloying elements, such as \( Al, V, Cu, W \), etc. may be added in austenitic stainless steels depending on the applications and the desired properties.
1.3. References


2. BACKGROUND AND LITERATURE REVIEW

2.1. Creep Damage

Creep is time-dependent plastic deformation that occurs at constant stress and temperature above \( \sim 0.4T_m \) where \( T_m \) is the melting point of the material. It often occurs at elevated temperature, but some materials creep at room temperature such as lead (Pb) unlike iron (Fe) because for lead, room temperature represents a higher homologous temperature (i.e., \( T/T_m \approx 0.5 \)) than that of iron (\( \approx 0.16 \)) [1, 2]. Creep takes place in all types of materials: metals, polymers and ceramics. For metals, creep occurs at moderate stresses and temperatures, but for polymers, it occurs at relatively low stresses and temperatures. While for ceramics, it occurs at high stresses and temperatures. A typical example of creep is in the materials used in turbine blades of a gas turbine engine [2].

2.1.1. Creep Testing

In a conventional creep test, the specimen is loaded to constant stress and temperature. The main purpose of performing the creep test is to determine the maximum stress and temperature up to which the material can sustain for a long time. That is, evaluating the creep resistance of the material and the resultant plastic deformation (strain) is measured and plotted as a function of time. For brittle materials, creep tests are conducted under compression loading, where the specimens used are right cylinders with an aspect ratio (length to diameter) ranging from 2 to 4. Creep tests can be performed at a given temperature at different stresses or at different temperatures for a given stress level. Eventually, all data are collected to obtain the creep behavior of the material [2]. Figure 2.1 shows the experimental setup of a creep tester with specimen loading [3].
2.1.2. Creep Properties

Creep curve is plotted as creep strain versus time at a constant stress and temperature as shown in Figure 2.2, which has three stages: (i) primary stage (transient creep) in which work hardening during plastic deformation is progressed more than softening and thus, undergoing a reduction in strain rate with time because of rearrangement of dislocations in the specimen to maintain equilibrium with the applied load [2], (ii) secondary stage (steady-state creep rate) in which the rate of work hardening and softening balance each other and the creep rate reaches its minimum, and (iii) tertiary stage which is characterized by an accelerating creep rate where softening behavior dominates. The third stage of tertiary creep is often considered as fracture rather than deformation [1]. Sometimes the test specimen does not undergo all three stages of creep deformation. In some cases, fracture may occur in the secondary stage and therefore, the final stage (stage III) will not be observed. Also, the duration of the three stages can be different depending
on the material, the applied load and temperature [2]. In Figure 2.2, the strain represented by \( \varepsilon_o \), occurs instantaneously on application of the load which is not due to creep and it is subtracted from the total strain in the creep specimen to give the strain only due to creep [4]. The strains in the three stages are given by [2]:

(i) **Stage I:** \( \varepsilon = At^{1/3} \)  

(ii) **Stage II:** \( \varepsilon = \dot{\varepsilon}_s + \beta t \)  

(iii) **Stage III:** \( \varepsilon = B + C \exp (\gamma t) \)

where \( \varepsilon \) is the creep strain, \( t \) is the time and \( A \) is a material constant.

where \( \dot{\varepsilon}_s \) is the steady-state creep rate and \( \beta \) is a material constant.

where \( B, C \) and \( \gamma \) are material constants.

![Figure 2.2](image)

**Figure 2.2.** A typical creep curve showing different stages of creep [1].

To describe the dependence of strain rate on stress at a given temperature, Norton’s law is given as follows [1, 2]:

11
\[ \dot{\varepsilon} = A_1 \sigma^n \]  

(2.4)

where \( A_1 \) is a constant dependent on the test temperature and \( n \) is the stress exponent. The effect of temperature and stress on creep curve is shown in Figure 2.3. The increase of the steady-state creep rate with test temperature under a given stress (Figure 2.3(a)) follows an Arrhenius equation with a characteristic activation energy for creep, \( Q_c \), \([1, 2]\):

\[ \dot{\varepsilon} = A_2 e^{-Q_c/RT} \]  

(2.5)

where \( A_2 \) is a constant dependent on the applied stress. Therefore, the temperature and stress variations of creep rate can be combined into one equation, known as the power law equation \([1, 2]\):

\[ \dot{\varepsilon} = A_3 e^{-Q_c/RT} \sigma^n \]  

(2.6)

where \( A_3 \) is a material constant and \( R \) is the gas constant (1.987 cal mol\(^{-1}\) K\(^{-1}\)). Also, steady-state creep rate is proportional to \( D_L \) (self-diffusion), so that the creep rate equation becomes \([1]\):

\[ \dot{\varepsilon} = A_4 D \sigma^n = A_5 D \left( \frac{\sigma}{E} \right)^n \]  

(2.7)

where \( A_4 \) and \( A_5 \) are constants dependent on the material and microstructure such as the grain size.

The Bird–Mukherjee–Dorn (BMD) equation is used to express the constitutive behavior during creep deformation \([1]\):

\[ \dot{\varepsilon} = \frac{ADEb}{K T} \left( \frac{\sigma}{E} \right)^n \left( \frac{b}{d} \right)^p \]  

(2.8)
\[
\frac{\dot{\varepsilon} K T}{DEb} = A \left( \frac{\sigma}{E} \right)^n \left( \frac{b}{d} \right)^p
\]  

(2.9)

where \( A \) is a material constant, \( d \) is the grain size, and \( p \) is known as the grain size exponent.

Figure 2.3. The effect of (a) temperature and (b) stress on creep curves [1].

During creep deformation, the higher the rupture time, the longer the life of structures in service. Stress-rupture tests are conducted up to fracture and times to fracture are collected at different temperatures and stresses. Larson–Miller parameter (LMP) is one of the common empirical relations to describe stress–rupture behavior of a material [1, 2]:

\[
LMP = T \log (C+tr)
\]  

(2.10)

where \( C \) is the LMP constant (~20), \( T \) is the temperature in Kelvin, and \( t_r \) is the rupture time in hours and thus, LMP has a unit of \( K\cdot h \). Another common and important empirical relation is the Monkman–Grant (MG) relation in which the time to rupture decreases with strain rate [1]:

13
\[
\dot{\varepsilon}_s \cdot t_c = \text{constant}
\] (2.11)

where \(\dot{\varepsilon}_s\) is the steady-state creep rate. Another known parameter is the Zener–Holloman parameter given as follows [1]:

\[
Z = \dot{\varepsilon}e^{Q/RT}
\] (2.12)

Also, Sherby–Dorn parameter can describe stress–rupture behavior of a material and involves normalization with temperature compensated time [1]:

\[
P_{SD} = t e^{-Q/RT}
\] (2.13)

2.1.3. Creep Mechanisms

Creep fracture can be characterized as bulk cavitation damage associated with intergranular failure occurring at low and medium stresses, whereas the necking failure is associated with transgranular ductile fracture occurring at very high stresses. The intergranular failure criterion in creep damage consists of: (a) Cavity nucleation, (b) Cavity growth, (c) Interlinkage of cavities, (d) Crack propagation and (e) Final failure. Nucleation of cavities at grain boundaries can occur in many different ways [4]:

- Slip bands collision on grain boundary creating a stress concentration which might nucleate a cavity, in which edge dislocations piled-up against grain boundary will coalesce to form a crack nucleus [4, 5, 6].
- Nucleation of cavities at triple junctions and on grain boundary, in which wedge cracks (W-type) form at triple junctions because of the accelerated growth of the round cavities
(R-type) at grain boundary and their interlinkage in the vicinity of the triple junctions [4, 6].

- Nucleation of cavities at grain boundary ledges (tensile ledges).
- Nucleation of cavities at grain boundary particles.

On the other hand, the growth of creep cavities can occur by the following mechanisms [4]:

- Condensation of vacancies into the cavities.
- Advancement of the tip of the cavity by the continued sliding.
- Combination of sliding and vacancy condensation.

Creep by dislocation climb (referred to as Weertman dislocation climb model) is a result of the glide and climb of dislocations with climb being the rate-controlling process. Dislocation glide is hindered by long range stresses due to dislocation interactions, in which the stresses are relieved by dislocation climb and annihilation. Creep strain increases due to the glide of dislocations. In the glide–climb model, dislocations produced by the Frank–Read source (FR) glide a distance \( L \) till the lead dislocation comes across a barrier of height \( h \) at which it has to climb so that another dislocation can be generated by the source as shown in Figure 2.4. The creep strain is given by [1]:

\[
\Delta \gamma = \Delta \gamma_g + \Delta \gamma_c = \Delta \gamma_g = \rho b L
\]  

(2.14)

where \( \rho \) is the dislocation density, \( b \) is burgers vector and \( L \) is the glide distance. The time of dislocation glide–climb event is given by [1]:

\[
t = t_g + t_c \approx t_c = h/v_c
\]  

(2.15)

where \( v_c \) is the climb velocity. Therefore, strain rate is given by [1]:

\[
\frac{\partial \gamma}{\partial t} = \rho b v_c
\]
\[ \dot{\gamma} = \frac{\Delta \gamma}{t} = \frac{\rho b L}{h / v_c} \]  

(2.16)

where \( v_c \propto \Delta \gamma e^{-Em/RT} \) with \( E_m \) being the activation energy for vacancy migration. Natural creep law is given by [1]:

\[ \dot{\varepsilon} = AD_L \sigma^3 \]  

(2.17)

Assuming that the dislocation density (\( \rho \)) varies as the stress raised to power 2 (\( \sigma^2 \)).

Figure 2.4. Schematic of dislocation glide–climb event [1].

In Weertman pill-box model, dislocation loops are generated from Frank–Read sources on parallel slip planes. In many alloys, the alloying elements migrate to the dislocations and lock them at high temperatures causing the glide of dislocations to slow down. In these alloys, dislocation glide and climb take place as in pure metals, but since the glide becomes slower controlled by the diffusivity of alloying elements compared to the climb, creep will be controlled by the dislocation glide. Thus, creep rate becomes [1]:

\[ \dot{\varepsilon} = AD_s \sigma^3 \]  

(2.18)

where \( D_s \) is the solute atom diffusivity and equals to \( D_s = D_s^o \ e^{-Q_s/RT} \), \( Q_s \) is the activation energy for solute atom diffusion.
At relatively high temperatures and/or low stresses, creep can take place by diffusional mass transport through the lattice in which point defects (vacancies) diffuse from tensile boundaries to compressive boundaries for small grain size samples. This phenomenon was first considered by Nabarro in 1948 and Herring in 1950. A few years later, Coble proposed that grain boundaries could also provide an alternative path for stress assisted diffusional mass transport. As shown in Figure 2.5, grain boundaries normal to the applied stress will develop a higher concentration of vacancies. On the other hand, grain boundaries parallel to the applied stress will undergo compressive stresses with reduced vacancy concentration. Therefore, it will cause a vacancy concentration gradient between the two boundaries leading to a flux of vacancies diffusing from the normal grain boundaries to the parallel grain boundaries. The diffusion of vacancies will depend on the grain size and temperature. The diffusion of vacancies from one grain boundary to the other leads to crystal strain, thus, causing the deformation of the grains and therefore, the deformation of material [1].

Figure 2.5. Mass transport through the lattice as in N–H model (a) and through grain boundaries (Coble creep) [1].
With decreasing grain size, Coble creep dominates N–H creep and vice versa. However, these two mechanisms operate in parallel so that the strain rate is given by [1]:

$$\dot{\varepsilon} = \frac{B\varepsilon\Omega}{d^{2}kt}D_{\text{eff}}^{2}$$

(2.19)

where $B$ is constant, $\Omega$ is the atomic volume, $k$ is the Boltzmann’s constant, $d$ is the grain size and $D_{\text{eff}}$ is the effective diffusion coefficient given by [1]:

$$D_{\text{eff}} = D_{L}(1 + \frac{\pi D_{B}\delta_{B}}{dD_{L}})$$

(2.20)

where $\delta_{B}$ is the grain boundary thickness, $D_{L}$ is the lattice diffusivity and $D_{B}$ is the grain boundary diffusivity. These models assume that the grain boundaries are perfect sources and sinks of vacancies and the initial dislocation density of the crystal is low.

When the strain rate is proportional to the applied stress, the creep mechanisms are known as viscous creep mechanisms. Large-grained polycrystalline and bulk single-crystalline materials undergo viscous creep but are insensitive to the grain size in contrast to N–H and Coble creep mechanisms and it is known as Harper–Dorn creep (H–D). H-D creep which takes place at stresses below the critical stress needed for Frank Read (FR) source operation. At intermediate stresses and relatively small grain sizes, grain boundary sliding (GBS) mechanism is observed as in superplastic materials. Another creep mechanism known as low-temperature climb which becomes important when diffusion through dislocation pipes becomes dominant, where the climb creep rate is proportional to dislocation pipe diffusivity that increases with the dislocation density. Table 2.1 shows the various creep mechanisms with corresponding $n$, $p$, and $Q$ parameters [1].

18
Table 2.1. A summary of creep mechanisms with relevant parametric dependencies [1].

<table>
<thead>
<tr>
<th>Mechanism</th>
<th>( Q^a )</th>
<th>( n )</th>
<th>( p )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Climb of edge dislocations</td>
<td>( Q_L )</td>
<td>5</td>
<td>0</td>
</tr>
<tr>
<td>Viscous glide (solute drag creep)</td>
<td>( Q_S )</td>
<td>3</td>
<td>0</td>
</tr>
<tr>
<td>Grain boundary sliding</td>
<td>( Q_{GB} )</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Low-temperature climb</td>
<td>( Q_C )</td>
<td>7</td>
<td>0</td>
</tr>
<tr>
<td>Harper–Dorn</td>
<td>( Q_L )</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>Nabarro–Herring</td>
<td>( Q_L )</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>Coble</td>
<td>( Q_{GB} )</td>
<td>1</td>
<td>3</td>
</tr>
</tbody>
</table>

a) \( Q_L \): activation energy for lattice diffusion, \( Q_S \): activation energy for solute diffusion, \( Q_{GB} \): activation energy for grain boundary diffusion, \( Q_C \): activation energy for dislocation core diffusion.

Finally, a creep-resistant material should have the following properties [1]:

- A high melting point.
- Solid solution strengthening and dispersion strengthening can be used as strengthening mechanisms at high temperatures.
- Precipitation hardening can be useful when there is no particle coarsening, consequent softening or dissolution.
- Grain refinement is invalid under creep conditions and the opposite is required to minimize the grain boundary sliding effect.

2.1.4. Creep Damage Assessment

Creep damage can be represented in terms of feature type, size or development state:

- Creep damage can be characterized as either cavities on grain boundaries or voids due to particle/matrix decohesion. The particles are usually inclusions and carbides at intragranular sites.
• Creep damage can be characterized in terms of a quantity representing its extent, for example, by a classification based on reference micrographs or a density measurement of cavities/mm².

• The characterization of damage size can involve measuring the size of the damage feature (e.g., cavity diameter), but because of the high sensitivity of damage feature diameter and its dependent parameters on metallographic surface preparation, the measurement of cavity/void size is not widely practiced [7].

2.2. Fatigue Damage

Fatigue failures occur when the material is subjected to cyclic loading either tension-tension or tension-compression cycles, where at each given stress, there is a certain number of cycles for failure and the fracture surface turns out to be perpendicular to the applied stress. It can occur with less plastic deformation at elevated temperatures less than melting point ($T_m$). Also, even when the cyclic stress is less than the yield strength, microscopic plastic deformation can occur. Figure 2.6 shows various types of fatigue loading: a fully reversed tension–compression stress cycle (Figure 2.6(a)) and a tension–tension cycle (Figure 2.6(b)). During the tensile cycle, slip occurs on the plane of maximum shear stress. During the compression cycle, slip occurs on a nearby parallel slip plane with the slip displacement on the opposite direction, acting thus as the nucleation sites for the fatigue cracks. Since no fatigue failure is possible under compression–compression cycle, fatigue cracks will not initiate [1].
2.2.1. Fatigue Testing

One of the fatigue testing techniques is the cantilever beam fatigue tester as shown in Figure 2.7, in which the specimen is in line with a cantilever loaded at one end and rotated at the same time by a high-speed motor leading to the upper surface of the specimen in tension and the lower surface in compression. During each revolution, the surface layers pass through a full cycle of tension and compression [1, 2].

2.2.2. Fatigue Properties

Some of the common terms used in fatigue for stress or strain [1, 2]:

- Mean stress ($\sigma_m$) is the average of the maximum and minimum stresses in the cycle:
\[ \sigma_m = (\sigma_{\text{min}} + \sigma_{\text{max}})/2 \]  \hspace{1cm} (2.21)

- Stress range \((\sigma_r)\) is the difference between \(\sigma_{\text{max}}\) and \(\sigma_{\text{min}}\):

\[ \sigma_r = \sigma_{\text{max}} - \sigma_{\text{min}} \]  \hspace{1cm} (2.22)

- Stress amplitude \((\sigma_a)\) is half of the stress range:

\[ \sigma_a = \sigma_r/2 \]  \hspace{1cm} (2.23)

- The stress ratio \((r)\) is the ratio of minimum and maximum stresses:

\[ r = \sigma_{\text{min}}/ \sigma_{\text{max}} \]  \hspace{1cm} (2.24)

There are ways in which a cyclic stress can be applied to the material, one where the \(\sigma_{\text{min}}\) and \(\sigma_{\text{max}}\) are equal and opposite in magnitude and it is referred to as *completely reversed stress cycle*, or when \(\sigma_{\text{min}}\) and \(\sigma_{\text{max}}\) are asymmetrical and relative to zero stress, that is referred to as *repeated stress cycle* [2].

When the specimen is loaded at a given stress \((S)\), the number of cycles to failure \((N_f)\) is determined. The test can be repeated at different stress levels and the number of cycles to failure \((N_f)\) is again determined at each level. The S-N curve is obtained where the stress is plotted against the number of cycles to failure \((N_f)\) indicating that the higher the stress, the smaller the fatigue life of the material and vice versa. Fatigue limit or endurance limit is the largest value of stress that will not cause failure regardless of the number of cycles [1, 2]. Figure 2.8 shows the S-N curve for mild steel and an aluminum alloy [1]. As shown, fatigue limits range between 35\% and 60\% of the tensile strength for steels.
Figure 2.8. S–N curves for mild steel and an aluminum alloy [1].

The S-N curve is classified into low cycle fatigue (LCF) controlled by ductility for \( N < 5 \times 10^5 \) cycles and high cycle fatigue (HCF) controlled by strength for \( N > 5 \times 10^5 \) cycles. When Fatigue test is performed in strain-controlled mode, the total strain range is divided into elastic and plastic regions [1]:

\[
(\Delta \varepsilon = \Delta \varepsilon_p + \Delta \varepsilon_e) / 2
\]  

(2.25)

HCF is characterized by Basquin equation [1]:

\[
\Delta \varepsilon_e / 2 = \sigma_j (2N)^b
\]  

(2.26)

LCF is characterized by Coffin–Manson equation [1]:

\[
\Delta \varepsilon_p / 2 = \varepsilon_j (2N)^c
\]  

(2.27)
where \( N_f \) is the number of cycles to failure at a given strain amplitude, \( \Delta \varepsilon_E / 2 \) is the elastic strain amplitude, \( \sigma_f' \) is the fatigue strength coefficient, \( b \) is the fatigue strength exponent ranging from -0.05 to -0.12, \( \Delta \varepsilon_p / 2 \) is the plastic strain amplitude, \( c \) ranging from -0.5 to -0.7 and \( \varepsilon_f' \) is the fatigue ductility coefficient. Therefore, Eq. (25) can be represented as [8, 9]:

\[
(\Delta \varepsilon = \Delta \varepsilon_p + \Delta \varepsilon_E) / 2 = \varepsilon_f' (2N_f)^c + \sigma_f'(2N_f)^b
\] (2.28)

LCF is an important aspect in the design and operation of high-temperature systems that exhibit temperature gradients from being subjected to repeated thermal stresses during start-ups and shut-downs. Also, at high temperatures, LCF life is affected by the frequency, strain rate, hold position, hold duration and strain range [4].

The process of fatigue failure consists of three steps: (a) crack initiation, (b) crack propagation, and (c) final failure [1, 2]. Therefore, the fatigue life \( (N_f) \) is the summation of the number of cycles for crack initiation \( (N_i) \) and the number of cycles for crack propagation \( (N_p) \) [1]:

\[
N_f = N_i + N_p
\] (2.29)

A typical fatigue life plot as strain range \( (\Delta \varepsilon) \) against number of cycles to failure \( (N_f) \) along with the corresponding stress–strain loops (broad in LCF and narrow in HCF) is shown in Figure 2.9 [1, 4, 10]. Fatigue life at which the transition from low cycle (plastic) to high cycle (elastic) occurs is determined by [1]:

\[
2N_tr = (\varepsilon_f E / \sigma_f)^{1/b-c}
\] (2.30)
where $\varepsilon_f$ is the true fracture strain, $\sigma_f$ is the true fracture stress, $E$ is Young's modulus and $b$ and $c$ are material constants. As shown in Figure 2.9, the LCF (plastic) curve appears at larger strain amplitudes than the HCF (elastic) curve, which means that a high ductility of the material is required for resistance to large strain ranges, while a high tensile strength is required for resistance to low strain ranges [4].

![Figure 2.9](image)

Figure 2.9. Typical fatigue life plot along with corresponding stress–strain loops [1, 4, 10].

In strain-controlled fatigue tests, cyclic stress–strain curve generates a hysteresis loop as shown in Figure 2.10(a), where O–A–B is the initial loading curve and on unloading the yielding occurs at lower stress (point C) which is known as Bauschinger Effect. A shown in Figure 2.10(b), cyclic hardening causes the peak strain to decrease, while cyclic softening causes it to increase with increasing number of cycles. The stress–strain follows a power-law relationship [1, 4]:

$$\Delta\sigma = K'(\Delta\varepsilon)^{n'}$$

(2.31)
where $K'$ is the strength coefficient and the slope of stress-strain curve on log-log scale gives the slope $n'$, which is known as cyclic strain hardening exponent that ranges from 0.1 to 0.2 for many metals and is given by the ratio of the parameters $(b/c)$. Also, it is an indicator of the material's fatigue resistance [4]. It has been shown that $c = -1/(1 + 5n')$, so that materials with larger values of $n'$ have larger LCF fatigue lives and $b = -n'/(1 + 5n')$, so that lower values of $n'$ have larger LCF fatigue lives [11].

![Diagram](image)

Figure 2.10. (a) Cyclic stress–strain curve depicting hysteresis loop. (b) Hysteresis loops during cyclic hardening (i) and cyclic softening (ii) [1].

During a strain-controlled fatigue test, the stress required to reach the strain limit gradually increases in case of hardening and decreases in case of softening with increasing number of cycles as shown in Figure 2.10. The values of stress can change in the plastic strain range $(Δε_p)$ and elastic strain range $(Δε_E)$ depending on the testing conditions employed. Therefore, the stress range $(Δσ)$ and the plastic and elastic strain ranges are monitored.
Figure 2.11 shows typical waveforms for strain-controlled fatigue testing, where a symmetrical and a continuous fatigue cycle in tension and compression with a hold time at a constant peak strain is generated during creep-fatigue interaction which will be discussed later. Slow-fast and fast-slow strain-time waveforms are used to study the creep-fatigue interaction effects [4, 12]. In the slow-fast cycle, the tensile strain rate is less than the compression strain rate, while in the fast-slow cycle, the compression strain rate is less than the tension strain rate [4].

Figure 2.11. Typical waveforms for strain-controlled fatigue testing [4, 12].

Figure 2.12 shows a schematic of the hysteresis loop along with some important parameters associated with the LCF testing [4, 12]:
• $\sigma_a$ is the stress amplitude = $\Delta \sigma / 2$
• $\Delta \varepsilon_E$ is the elastic strain range.
• $\Delta \varepsilon_p$ is the plastic strain range.
• $\Delta \varepsilon_t$ is the total strain range = $\Delta \varepsilon_E + \Delta \varepsilon_p$.
• $\Delta \sigma$ is the stress range.
• $E$ is the elastic modulus.
• $\Delta \varepsilon_E / 2$ is the elastic strain amplitude = $\Delta \sigma / 2E$.
• $\Delta \varepsilon_p / 2$ is the plastic strain amplitude.

Figure 2.12. Parameters associated with hysteresis loop [4, 12].

The percentage of life consumed in one cyclic loading depends on the magnitude of stress in subsequent cycles. The linear cumulative damage rule, known as Miner’s rule, assumes that the
fatigue life of the material can be estimated by adding up the life fraction consumed in each loading cycle. If \( N_i \) is the number of cycles to failure at \( i^{th} \) cyclic loading and \( n_i \) is the number of cycles experienced by the structure, then [1]:

\[
\sum \frac{n_i}{N_i} = 1
\] (2.32)

However, Miner’s rule can fail when notches are present in the material or when the mean stress and temperature are high enough or cyclic frequency is low such that creep deformation dominates over fatigue damage [1].

One of the empirical equations used to determine the crack growth rate per cycle, \( da/dN \), with the range of the stress intensity factor [1, 2]:

\[
\frac{da}{dN} = A(\Delta K)^m
\] (2.33)

where \( A \) and \( m \) (known as Paris exponent \( \sim 2-4 \)) depend on the material, environment and frequency, and \( \Delta K \) is the range of stress intensity factors \( (=\Delta \sigma \sqrt{\pi a}) \). Therefore, critical crack length is given in terms of the plane strain critical fracture toughness \( (K_{IC}) \) [1]:

\[
a_f = \frac{K_{IC}}{Y^2 \pi^{\frac{3}{2}} \sigma_{max}}
\] (2.34)

Also, the number of fatigue cycles \( (N_f) \) using Paris law is determined by [1, 2]:

\[
N_f = \frac{1}{AY^m (\Delta \sigma)^m \frac{a_f}{\pi^{m/2}}} \int_{a_c}^{a_f} \frac{da}{a^{m/2}}
\] (2.35)

The time to failure \( (t_f) \) is given in terms of the cycling frequency \( (f) \) [1]:
\[ t_f = \frac{N_f}{f} \] (2.36)

A log–log plot of \( \frac{da}{dN} \) versus \( \Delta K \) is shown in Figure 2.13 in which a threshold stress intensity factor, a regime of subcritical crack growth and a fast fracture regime are depicted. Region II is known as Paris region, where crack undergoes stable propagation until it reaches a critical crack length (\( a_c \)) at the beginning of Region III during which unstable crack propagation occurs until final failure. In case of corrosion and radiation, the variation of \( \frac{da}{dN} \) with \( \Delta K \) would decrease the threshold stress intensity range and the critical crack length at fracture which then is indicated in terms of \( K_{SCC} \) instead of \( K_{IC} \) [1].

![Figure 2.13. The crack growth rate as a function of the stress intensity factor range [1].](image)
2.2.3. Crack Growth

During Fatigue deformation, crack nucleation usually takes place at a free surface and crack growth occurs in three stages, as shown in Figure 2.14 [1, 4, 13]:

- **Stage I:** After initiation of a crack (10µm), the crack propagates slowly along crystallographic planes of high shear stresses and oriented at approximately 45° with the stress axis [1, 4].

- **Stage II:** Crack growth rate increases rapidly with a change in the propagation direction that it is perpendicular to the stress axis [1, 4].

- **Stage III:** The critical crack length is reached and the final failure starts [1, 4].

The fractional area of fracture surface associated with each of the three above stages depends on the applied strain level. At low strain levels, stage I dominates the fracture surface while at high strain levels, stage II dominates. However, stage III dominates only for very high strain level tests [4].

Figure 2.14. Schematic of fatigue crack propagation across a specimen section depicting three stages of fatigue failure [4, 13].
The fracture surface can be characterized in two ways [2]: beach marks/clamshell marks observed macroscopically as shown in Figure 2.15(a), and striations observed microscopically as shown in Figure 2.15(b) [1]. Usually these marks appear in the region where crack propagated. However, these markings are not observed during the last stage of fatigue failure [2].

![Figure 2.15. (a) A schematic fatigue fracture surface showing clamshell markings. (b) An SEM image showing the striations in a fatigued 304L austenitic stainless steel [1].](image)

There have been models proposed to explain crack growth during fatigue deformation classified into two categories: (i) Crack growth based on relative shear process and (ii) Crack growth based on slip process [4].

(i) **Shear Decohesion Model**: Applying a low tensile stress leads to formation of localized flow bands within ±45° of the crack plane as shown in Figure 2.16(b). As tensile crack propagation occurs, a new crack surface is formed by *shear decohesion* as depicted in Figure 2.16(c), while for larger crack openings, new flow bands can be formed (Figure 2.16(f)). Reducing the stress causes a reversal of the
flow processes, but the geometrical changes at the crack tip are not fully reversible. Therefore, a striation is formed as shown in Figure 2.16(e) [4].

![Diagram of crack growth mechanisms](image)

**Figure 2.16.** Shear decohesion mechanism for stage II fatigue crack growth [9, 14].

**Plastic Blunting Model:** It is assumed in this model that the crack tip is sharp at the start of loading cycle (Figure 2.17(a)). When the tensile stress is applied, the small double notch at the crack tip concentrates the slip along the planes orientated at 45° to the crack plane as displayed in Figure 2.17(b). As the crack opens to its maximum extension (Figure 2.17(c)), it grows longer by plastic shearing and at the same time its tip becomes blunted. Under stress compression, the slip direction in the end zones is reversed (Figure 2.17(d)). The crack faces are crushed and the new crack surface formed in the plane of the crack (Figure 2.17(e)) which
undergoes buckling to form a resharpened crack tip. Then it is ready to grow and be blunted in the next stress cycle [4].

![Plastic blunting mechanism for stage II fatigue crack growth](image)

Figure 2.17. Plastic blunting mechanism for stage II fatigue crack growth [14].

### 2.2.4. Fatigue Life

Fatigue life can be affected differently by several factors. Fatigue strength is affected slightly by the frequency of cyclic loading. On the other hand, fatigue life is not affected by the waveform of stress cycle [1]. The environment is one of the factors that strongly affects fatigue life. For example, the fatigue life in vacuum could be about 10 times more than that in air. Also, stress concentrators such as keyways enhance crack initiation and thus decrease the fatigue life. The thickness of the test specimen and surface smoothness or roughness affect the fatigue properties; thinner samples and polished surfaces decrease the crack growth rate and thus enhance the fatigue life [1, 2].
Escalation of fatigue damage however, can be attributed to cyclic creep, microstructural inhomogeneities such as in welded joints and the presence of residual stress [1].

2.2.5. Fatigue Damage Assessment

As shown in Figure 2.18 during stage I of crack growth, fatigue cracks propagate to a depth of 1–2 grain diameters along persistent slip bands before entering stage II, in which cracks grow in a transgranular mode perpendicular to the maximum stress applied. On the surface, many short fatigue cracks are formed. Depending on the applied strain amplitude, one or more short crack(s) will dominate and grow to a long crack that is responsible for final failure [7].

Figure 2.18. Schematic representation of fatigue crack development [7].
2.3. Creep-Fatigue Interaction

2.3.1. Creep-Fatigue Damage Properties

Alloys that are found to be creep resistant are also found to be fatigue resistant. However, when a material has the best creep strength does not mean that it will have the best fatigue strength. Therefore, it is important to study the material’s response under both creep and fatigue by performing mechanical testing where both creep and fatigue loading conditions are applied. In the absence of hold time and/or at relatively high strain rates at high temperatures, material deformation is attributed to fatigue as shown in Figure 2.19(a). However, with increasing hold time and/or decreasing strain rate at high temperatures, the creep damage dominates (Figure 2.19(b)). At intermediate hold times and/or strain rates, transgranular fatigue cracking interacts with intergranular creep cavitation consequentially or simultaneously, leading to accelerated crack propagation and reduced crack initiation endurance as shown in Figures 2.19(c), 2.19(d) and 2.20 [7].

According to ASTM standard E2714-13 [15], the purpose of conducting creep-fatigue tests is to determine the material-property data for the (a) assessment of input data for the damage condition analysis of engineering structures operating at high temperatures, (b) the verification of constitutive damage model effectiveness, (c) material characterization, or (d) verification and development of new rules for construction and life assessment of high-temperature components subjected to cyclic loading with steady-state operation conditions.
Figure 2.19. Creep-fatigue cracking mechanisms (a) fatigue dominated; (b) creep dominated; (c) creep-fatigue interaction (due to “consequential” creep damage accumulation); (d) creep-fatigue interaction (due to “simultaneous” creep damage accumulation) [7].

Figure 2.20. Influence of hold time on the cyclic/hold creep-fatigue endurance of 1%CrMoV steel at 550 °C where crack development is identified as being pure creep (C) pure fatigue (F) or creep-fatigue (CF) [7].
Data that can be determined from creep-fatigue tests in either stress or strain control at elevated temperatures involve: (a) cyclic stress-strain hysteresis loops, (b) stress relaxation (cyclic creep), (c) cyclic hardening and cyclic softening response or (d) cycles to crack formation. Figure 2.21 shows examples of stress-strain hysteresis diagrams with hold time (strain-control and force-control) and without hold time. On the other hand, Figure 2.22 shows examples of creep-fatigue test specimen configurations in which uniform gage section test specimen is adopted (Figure 2.22(a)). In Figure 2.22(b), the hour-glass test specimen is usually used for high strain range tests due to the risk of buckling of parallel gage section test specimens [15].

There are some standard definitions related to creep-fatigue testing method according to the ASTM standard [15]:

- **Cycle, N**, in fatigue testing, is a sequence of force (strain) that is repeated under constant amplitude loading (straining).
- **Hysteresis diagram**, is the stress-strain path per one cycle.
- **Hold-time, \( \tau_h \)**, is the amount of time in the cycle where the controlled test variable (force, strain, displacement) is held constant with time. They are usually placed at tensile and/or compressive peak stress or strain.
- **Total cycle period, \( \tau_t \)**, is the time for completion of one cycle.
- **Initial modulus of elasticity, \( E_o \), \([FL^{-2}]\)**, is the modulus of elasticity determined during the loading part of the first cycle.
- **Modulus of elasticity at cycle N, \( E_N \), \([FL^{-2}]\)**, is the average of the modulus of elasticity determined during the elastic tension and compression portions of the hysteresis diagram for the \( N_{th} \) cycle.
- **Stress range, \( \Delta \sigma \), \([FL^{-2}]\)**, is the difference between the maximum and minimum stresses.
Figure 2.23 shows the cycle shapes that may be used for creep-fatigue testing; (a) low frequency triangular waveforms with low ramp rates, (b) saw-tooth waveforms in which the ramp rate of the tensile transient is significantly different than that of the compression, and (c) Cyclic/hold waveforms in which hold times of the control variable are applied [15].

Figure 2.21. Examples of stress-strain hysteresis diagrams without hold time (a), with hold time (strain control) (b) and with hold time (force control) (c) [15].
2.3.2. Creep-Fatigue Damage Mechanisms

The creep-fatigue interaction failure can be described in two ways [4, 16]:

- Influence of cyclic loading on cavitation damage.
- Influence of cavitation on cyclic crack initiation and propagation.

Creep-fatigue damage can be hidden by extensive oxidation, where crack path details are shown in such a way that the actual damage mechanism is not visible anymore and valuable evidence can be lost by its removal. However, creep-fatigue damage development depends on the material condition, being influenced by temperature, strain range, strain rate, hold time, creep...
ductility, creep strength and creep strengthening mechanism; whether by precipitation strengthening or by solid solution strengthening [7].

![Diagram of creep-fatigue cycle shapes](image)

Figure 2.23. Example of Creep-Fatigue Cycle Shapes [15].

Creep-fatigue damage can be either dominated by cavitation-damage, or by crack-damage. In the case of cavitation damage (Figure 2.24), applying cyclic loading enhances either crack initiation or propagation. As shown, the area under stress-time curve is smaller for cyclic loading than it is for monotonic loading, therefore, the cavitation damage is greater [4].

On the other hand, experimental observations show that in case of pure fatigue damage, one or two cracks are initiated, whereas in creep-fatigue damage, many grain boundary cracks are
initiated, and the largest among them propagates as the main crack leading to failure. Therefore, there are three mechanisms of creep-fatigue interaction as shown in Figure 2.25 [4, 16]:

- Cavitation damage enhanced by cyclic loading.
- Crack initiation enhanced by cavitation damage.
- Crack propagation enhanced by cavitation damage.

Figure 2.24. The integrated stress-time area is shown smaller for cyclic loading (b), than it is for monotonic loading (a), thus, the damage is greater for cyclic loading [4, 16].
Figure 2.25. Schematic representation of how the three mechanisms of creep-fatigue interactions may lead to deviation from the linear damage rule [4, 16].

2.3.3. Factors Affecting Creep-Fatigue Life

2.3.3.1. Effect of Hold Time

Hold time has an effect on fatigue life that depends on the strain rate, microstructure, position and duration of hold during a cycle. In case of austenitic stainless steels, it has been shown that at relatively high temperatures, fatigue life decreases when a hold time is applied in the cycle [4, 17]. Also, the imposition of hold times in the tension part of the cycle has been shown to be more detrimental than those imposed in the compression part of the cycle as shown in Figure 2.26 [9].

In the work done by IGCAR, Kalpakkam and KFA, Julich on Alloy 617 at 1173 K in simulated reactor helium [4, 18, 19], observations showed that hold times at the tensile peak strain led to a significant decrease in life in comparison with the hold time during compressive peak strain. Also,
symmetrical hold times in both tension and compression led to reduction factors that were very close to the pure fatigue data. However, during continuous cycling or pure fatigue tests at all the strain rates, final failure involved transgranular cracks with no creep or oxidation damage. In the one-minute tension hold time test on the other hand, cracks initiated transgranularly whereas they propagated by a mixed mode of transgranular and intergranular cracks. When hold time was greater than 10 minutes, intergranular cracks initiated and mixed mode propagation occurred. Hold times in the compression part of the cycle resulted in dimple fracture similar to a tensile fracture accompanied by necking.

The reduction in fatigue life during tensile hold times is due to the interaction between surface-initiated fatigue cracks and interior creep cavitation damage developed at grain boundaries [4, 18, 19].

![Figure 2.26. Influence of hold time (10 minutes) on fatigue life of type 304 stainless steel at 923 K [4, 17].](image-url)
During the tensile hold time, damage behavior is characterized by the tensile stress relaxation as a function of time indicating the build-up of tensile inelastic strain which leads to R-type cavitation damage in the material. R-type creep cavities form by clustering of vacancies at the junction between grain boundaries and second phase particles under the applied tensile stress. Also, these cavities can grow by diffusional transport of vacancies or by deformation of grain matrix material [4, 20].

Furthermore, it has been shown that increasing the duration of hold periods results in many grain boundary cracks in the surface regions of the material. Also, the formation of oxidation at the surface during longer hold times can lead to chromium depleted zones in which the carbide precipitates will be dissolved. The loss of grain boundary carbides therefore, causes grain boundary sliding enabling the formation of wedge cracks [4, 18, 19].

During compression holds, interior creep cavities were not observed in the case of Alloy 617 [4, 18, 19] but fatigue life decreases with hold time because compression hold produces geometric instabilities and consequently tensile necking failure. Bulk damage is unlikely to occur during compressive holds because the process of initiation and growth of interior creep cavities require both shear and normal tensile stresses across the grain boundary [4, 21].

For symmetrical hold times during creep-fatigue tests, cavitation damage has been observed close to the fracture surface with no wedge cracks unlike the observation in tensile hold tests. Therefore, symmetrical holds exhibited better fatigue resistance than compression-only holds as the deformation is not increasing during the cycle [4, 18, 19]. In the work done by Majumdar and Maiya [4, 22] on 304 austenitic stainless steel, symmetrical holds produced only transgranular failure unlike the above observations by IGCAR, Kalpakkam and KFA, Julich [4, 18, 19].
It has been suggested that, when creep cavities are formed during the tensile hold time, they will undergo sintering during the following compression hold [4, 23]. However, they nucleate and grow by irreversible shear deformation when no compression hold is applied [4, 18, 19]. Hales investigated creep-fatigue damage on 316 stainless steel at 873 K [4, 24] with tensile hold times up to 1,000 minutes along with a strain amplitude of ±0.25%. As observed, the total length of damaged grain boundaries per unit area increased linearly with the number of cycles. Also, the rate of growth per cycle increased with hold time. The accumulation of creep strain from a stress relaxation model is given by [4, 24]:

$$\varepsilon = \frac{\sigma_{\text{max}} - \sigma_i}{E} = \frac{1}{E} \left\{ \left( \sigma_{\text{max}} \right) - \left[ + AE(n-1)(t-1)^m \sigma_{\text{max}}^{-1} \right]^{1/n} \right\}$$

(2.37)

where, $E$ is Young’s modulus, $\sigma_{\text{max}}$ is the stress at the beginning of relaxation process and $\sigma_i$ is the stress after hold time, $t$. The constants $A$, $m$ and $n$ are given by the creep equation [4, 24]:

$$\dot{\varepsilon} = A \sigma^n t^m$$

(2.38)

where $A$, $m$ and $n$ were obtained from creep tests. Figure 2.27 shows a plot of creep strain predicted by the stress relaxation on 316 stainless steel at 873 K as conducted by Hales [4, 24], in which creep strain increases with hold time.

It should be mentioned that a creep strain rate of less than $10^{-4}$ s$^{-1}$ is necessary for causing grain boundary damage. Furthermore, only a fraction of relaxation strain in creep-fatigue tests with tensile hold times contributes to creep damage. This fraction can be determined by a transition strain rate above which matrix deformation dominates and contributes to fatigue damage and below which grain boundary damage accumulates leading to creep damage. These fractions can be obtained by partitioning of a typical cyclic hold hysteresis loop as shown in Figure 2.28 [4, 25].
Figure 2.27. The accumulation of creep strain calculated from a stress relaxation model as a function (a) number of cycles and (b) time ($N \times t_h$) in 316 SS at 873 K [4, 24].

Although it has been shown that the effect of tensile hold time is more detrimental than compression hold, many nickel-based alloys exhibit a more detrimental compressive strain hold than the tensile one [4, 26]. Compressive hold times contribute to the development of high levels of tensile mean stresses during asymmetric cycling, in which they have been suggested to enhance fatigue crack growth by increasing the growth rate of round cavities. However, under tensile hold, compressive mean stresses cause the cavities to grow into elongated flat cracks. Since the work required to fracture a grain boundary is greater when the boundary contains round cavities, it was argued that compressive holds should be the most detrimental [4, 27].
Figure 2.28. Partitioning of typical cyclic hold hysteresis loop ($\Delta e_{pp}$ = pure fatigue inelastic strain range; $\Delta e_p$ = plastic strain component at half-life; $\Delta e_c$ = true creep strain component at half-life; $\dot{e}_{mw}$ = strain rate depicting the change in the dominant mode of damage between matrix and triple point wedge type cracking) [4, 25].

2.3.3.2. Environmental Effect

The environmental effect on the creep-fatigue endurance can be studied by conducting creep-fatigue tests at different conditions like in air or vacuum. For pure fatigue and creep-fatigue tests performed on 304 stainless steel at 593 °C, data exhibited large differences in cyclic life between pure fatigue data ($\dot{\varepsilon} = 4 \times 10^{-3}$ s$^{-1}$) generated in air and high vacuum (1.3 µPa). However, these differences decrease as the hold time increases [4, 28]. Continuous cycling test in air and vacuum resulted in transgranular failure while intergranular failure when applying hold times. Therefore, the degradation in cyclic life due to intergranular damage results from creep damage rather than from environmental interactions [4].
2.3.3.3. Effect of Grain Size and Strain Range

Grain size dependence of fatigue life is more significant under test conditions that result in intergranular failure. For example, fatigue lives of various stainless steels decreased with increasing grain size at high temperatures due to the formation of wedge cracks and cavities at the grain boundaries in the material [4, 29].

Creep-fatigue failure occurs at relatively high strain ranges and ductilities, while creep failure occurs at low strain ranges and ductilities. At low strain ranges, the surface crack initiation becomes very slow compared to the initiation of internal creep damage and thus the failure becomes creep dominant. However, fatigue failure becomes dominated at both high strain ranges and rapid cycling conditions because there is not enough time for the occurrence of creep damage [4, 30].

2.3.3.4. Effect of Creep Ductility

The extent of creep-fatigue interaction is strongly dependent on creep ductility as shown in Figure 2.29 and Figure 2.30; when creep ductility is high, creep voids form at inclusions due to particle-matrix decohesion, and creep dominated cracking tends to be transgranular. Therefore, creep-fatigue failure is due to damage summation. On the other hand, when creep ductility is low, creep voids form at grain boundaries and creep dominated cracking tends to be intergranular. Therefore, the extent of creep-fatigue interaction can be high [7].
Figure 2.29. Influence of creep ductility on the cyclic/hold creep-fatigue endurance of 1%CrMoV steel at 550 °C [7].

### 2.3.3.5. Effect of Unequal Strain Rate

Applying unequal strain rates in creep-fatigue tests has major advantages such as [4]:

- Ease of testing, including a single control mode.
- Results can be directly interpreted than constant stress or relaxation tests.
- Greater proportion of time-dependent strain range to total strain range is available for damage in each cycle.

Creep-fatigue testing using unequal strain rates was conducted on Alloy 800H iron-based austenitic stainless steel at 850 °C [4, 31]. As shown in Table 2.2, a balanced fast-fast test results in a greater endurance, while the shortest life was obtained during unbalanced slow-fast test. The fast-slow and slow-slow tests have detrimental effect on creep-fatigue life but not as severe as that of the slow-fast test. Furthermore, microstructure characterization revealed transgranular failure...
in fast-fast tests, intergranular failure in slow-slow tests, tensile necking failure in fast-slow tests and purely intergranular failure in slow-fast tests. As a result, the final failure mechanism and thus endurance was strongly affected by wave-form shape. In the slow-slow tests, tensile and compressive strain rates are equal, meaning that any bulk damage produced in tension may equal the damage recovered in compression and thus little or no bulk damage accumulation is observed. On the other hand, fast-slow tests are equally damaging as slow-slow tests because of shape instability resulting from deformation associated with tensile mean stress.

Figure 2.30. Influence of creep-ductility on creep-fatigue cracking mechanisms [7].
Applying a slow tensile strain rate into continuous cycling enabled creep cavitation damage to accumulate at grain boundary particles. Bulk intergranular damage took place due to creep damage produced during slow strain-rate part, which was not sintered by subsequent compressive straining. Also, the environmental effects on cavitation near the surface are severe and the diffusion of environmental species hinder the collapse of cavity leading to intergranular crack initiation. However, environmental damage would be smaller in fast-slow tests than in slow-slow and slow-fast tests [4]. It is worth mentioning that in case of existing oxidation in slow-fast tests, creep and oxidation contribute together to the degradation of fatigue life [4].

Sliding mechanism is necessary for the formation of wedge cracking; and since sliding is a shear process, it takes place in both tension and compression. In slow-fast tests, the unsymmetrical strain rate produces more sliding in tension than in compression which leaves an offset sliding in each cycle as shown in Figure 2.31. On the other hand, in equal strain rates tests, sliding is symmetrical and little damage is obtained in each cycle and thus crack propagates transgranularly [4, 16].

Raj has proposed fatigue maps depicting different damage mechanisms in strain rate-temperature regime for an austenitic stainless steel as shown in Figure 2.32 [4, 16]. As is clear, fatigue damage can occur at any strain rate, but it is likely to be a major contribution to final fracture in the high strain-rate regime, where the applied strain rate, $\dot{\varepsilon}$, is greater than transition
strain rate, $\dot{\varepsilon}_w^*$, for the occurrence of wedge cracking. However, when $\dot{\varepsilon}_e < \dot{\varepsilon}_w^*$, an interaction between cavitation damage and fatigue crack damage can occur, leading to creep-fatigue interaction.

Figure 2.31. A schematic figure describing how a slow-fast cycle can introduce a net sliding offset in each cycle, producing of W-type cavity damage [4, 16].

Transition strain rate, $\dot{\varepsilon}_w^*$, for wedge cracking, separating the homogeneous deformation at high strain rates from the inhomogeneous deformation at low strain rates, is given by [4, 16]:

$$\dot{\varepsilon}_w^* = 0.27 \frac{Y\Omega}{KT} \frac{\delta D_b}{f_b Lp^3} 
$$  \hspace{1cm} (2.39)

where $\dot{\varepsilon}_w^*$ = tensile strain rate, $Y$ = effective tensile yield stress of the material, $L$ = grain size, $\Omega =$ atomic volume, $p$ = particle size, $f_b =$ area fraction of the particles on the boundary, $\delta D_b =$
boundary width multiplied by the diffusivity and $T =$ temperature. On the other hand, the transition strain rate, $\dot{\varepsilon}_r^*$, for the occurrence of R-cavities is given by [4, 16]:

$$
\dot{\varepsilon}_r^* = 0.27 \frac{8Y}{KT} \frac{\delta D_b}{\lambda^3}
$$

where $\lambda =$ interparticle spacing. $\dot{\varepsilon}_r^*$ is the boundary between constrained and unconstrained cavity growth.

Figure 2.32. A fatigue damage map for austenitic stainless steel [4, 16].
2.3.4. Mechanical Analysis of Creep-Fatigue Damage

2.3.4.1. Crack Initiation Endurance

The generic flow diagram shown in Figure 2.33 is used to assess the risk of creep-fatigue crack initiation in high-temperature defect-free components. In all creep-fatigue assessments, determination of the state of stress and strain at critical locations in the component is an important step. Thus, it is important to know the thermal transients and external forces experienced by the structure during service operation, and representations of creep and fatigue deformation properties of the materials in terms of model constitutive equations. After determination of the state of stress and strain at critical locations, the fatigue and creep damage fractions accumulated per cycle at the component critical location can be determined and therefore, it is possible to determine the creep-fatigue crack initiation endurance [7, 32].

Creep and fatigue damage fractions are calculated based on how the creep damage is calculated, either due to self-equilibrating loading (secondary), i.e., by strain-fraction or time-fraction methods. Time-fraction method calculates creep damage due to directly-applied loading (primary) as a function of creep rupture time, $t_R(\sigma)$, as shown in Eq. (2.41) [7]:

$$
D_C = \sum_j \left\{ N_j \left[ \frac{t_h}{t_R(\sigma_p)} \right] \right\} + \sum_j \left\{ N_j \left[ \int_0^{t_h} \frac{dt}{t_R(\sigma_s)} \right] \right\} 
$$

(2.41)

In which the above two terms account for creep damage accumulated due to primary and secondary loading, and the creep rupture times, $t_R(\sigma_p)$ and $t_R(\sigma_s)$ are determined from creep rupture strength data for a given material. $N_j$ is the cycle number for $j$th cycle type, $t_h$ is the hold time and $\sigma_p$, $\sigma_s$ are the stresses due to primary (directly-applied) and secondary (self-equilibrating) loading. On the other hand, creep damage in terms of strain-fraction method is given by [7]:
\[ D_C = \sum_j \left\{ N_j \left[ \frac{t_h}{t_R(\sigma_p)} \right] \right\} + \sum_j \left\{ N_j Z_j \left[ \frac{t_R}{\varepsilon_R(\dot{\varepsilon}_C)} \right] \right\} \]  

(2.42)

where \( Z_j \) is the elastic follow-up factor for the \( j \)th cycle type, \( N_j \) is the cycle number for \( j \)th cycle type, \( t_h \) is the hold time, \( t_R \) is the time to creep rupture, \( \sigma_p \) is the stress due to primary (directly-applied) loading, \( \dot{\varepsilon}_C \) is creep strain rate and \( \varepsilon_R \) is creep rupture strain (ductility) determined from creep rupture tests.

Fatigue damage fraction is determined from a model data of smooth specimen and Low-Cycle Fatigue (LCF) crack initiation endurance is given by [7]:

\[ D_f = \sum_j \left[ \frac{N_j}{N_i(\Delta\varepsilon_i)} \right] \]  

(2.43)

where \( N_j \) is the cycle number for \( j \)th cycle type, \( N_i \) is the number of cycles to crack initiation and \( \Delta\varepsilon_i \) is the total strain range.

Finally, creep and fatigue damage fractions are summed and compared to a reference of creep-fatigue damage summation diagram as shown in Figure 2.33. In the ASME Code, creep-fatigue life is evaluated by a linear summation of fractions of fatigue and creep damages. The creep-fatigue criterion is given by [33]:

\[ \sum_j \left[ \frac{n}{N_d} \right] + \sum_k \left[ \frac{\Delta t}{T_d} \right] \leq D \]  

(2.44)

where \( n \) and \( N_d \) are the number of cycles of type \( j \) and the allowable number of cycles of the same cycle type, respectively; and \( \Delta t \) and \( T_d \) are the actual time at stress level \( k \) and the allowable time at that stress level, respectively; \( D \) is the allowable combined damage fraction. This can be
represented graphically by the creep-fatigue interaction diagram shown in Figure 2.34 for different materials according to the ASME Division 5 [33].

Figure 2.33. Generic flow diagram representing creep-fatigue crack initiation assessment procedure [7, 32].
2.3.4.2. Crack Growth

Crack propagation due to creep-fatigue interaction may occur within the boundaries of plastic zone when the crack is small (i.e., < ~2 mm) or beyond the limits of the size of the plastic zone, $r_p$ when the crack is long as shown in Figure 2.35 [7].

Figure 2.35. Schematic representation of (a) short-crack growth within a cyclic plastic zone; and (b) long crack growth beyond the boundary of a cyclic plastic zone [7].
In case of long-crack propagation, creep-fatigue crack growth is represented by fatigue and creep crack growth rate characteristics as following [7]:

\[
\frac{da}{dN}_{CF} = \frac{da}{dN}_C + \frac{da}{dN}_F
\]  
(2.45)

\[
\frac{da}{dN}_F = A(T, v, t_h). (\Delta K_{eq})^m
\]  
(2.46)

\[
\frac{da}{dN}_C = \int_0^t D(\varepsilon_R). (C^*)^\gamma. dt/\upsilon
\]  
(2.47)

where \( \Delta K_{eq} \) is the range of stress intensity factor, \( m \) and \( \gamma \) are exponents, \( t_h \) is the hold time, \( D(\varepsilon_R) \) is a constant and \( \upsilon \) is the frequency. The function, \( A(T, v, t_h) \), accounts for any influence of prior creep and oxidation damage at the crack tip, which can be determined experimentally. The creep rate dependent parameter, \( C^* \), provides a geometry-independent function for correlating creep crack growth rates for long cracks under steady-state creep conditions and ahead of the crack tip [7].

In case of short-crack propagation, creep-fatigue crack growth rate can be expressed as a function of total strain range [7]:

\[
\frac{da}{dN} = B'. (\Delta \varepsilon)^b.a^0.(1-D_C)^2
\]  
(2.48)

where \( Q \) is the crack size exponent which equals \( \sim 1 \), \( a \) is the crack length, \( b \) is the strain range exponent, \( B' \) is constant and \( D_C \) is the total creep damage fraction. For advanced martensitic steels, the total creep damage fraction, \( D_C \), can be replaced by a microstructural condition parameter (\( \phi \)), in which it is a function of the sub-grain size [7].
2.3.4.3. Post-Test Examination

Post-test examination of the test specimens is necessary to obtain an effective quantitative interpretation of the final damage, which requires knowledge of the microstructural and damage conditions of the materials under controlled loading conditions. For example, the classification of fatigue damage appearance in terms of $K_{mean}$, $\Delta K$, and temperature allows to indicate the loading conditions responsible for final failure.

At high temperatures, the presence of oxidation can camouflage important features for damage justification. However, oxide thickness measurements provide a useful indication of the time of exposure and thus, the basis for oxide dating [7]:

$$x^2 = k_p t$$

(2.49)

where $k_p$ is a function of material, temperature and surface condition that represent the oxide growth law constant, $x$ is the oxide thickness and $t$ is the time.

2.3.5. Creep-Fatigue Damage Assessment

Creep-fatigue damage diagrams facilitate the prediction of lower bound high-temperature cyclic endurance of a material as shown in Figure 2.36. As shown, creep-fatigue interaction occurs if sufficient creep relaxation strain is accumulated to create intergranular cavitation. Also, accumulated creep strain initiation requires a minimum number of cycles, and the failure is transgranular below this number. This region is shown as "ab" in Figure 2.36 where the extent of "ab" depends on microstructure, hold time and temperature. However, at strain ranges below the value given by "d", failure is dominated by creep damage and the endurance is given by "df". The creep-fatigue interaction regime is described by the field bounded by "bcde", of which the line
"bed" is a lower limit. Furthermore, if the hold time is reduced from $t_1$ to $t_2$, a new creep failure line "c'd" must be constructed [4, 34].

Figure 2.36. Creep-fatigue damage diagram showing regions of different failure modes for type 316 stainless steel [4, 34].

The main suggested criteria for design under conditions of creep-fatigue interaction are [35]:

- **Linear damage accumulation rule**, in which it is used to determine the total combined damage of creep and fatigue [35]:

\[
\sum_{j=1}^{j-k} \left( \frac{n}{N_d} \right) j + \sum_{l=1}^{l-m} \left( \frac{t}{t_d} \right) l \leq D
\]  

(2.50)

where $n = \text{number of fatigue cycles applied at loading condition } j$

$N_d = \text{number of design allowable cycles at loading condition } j$

$t = \text{time under the applied creep load condition } l$

$t_d = \text{allowable time at load condition } l$
$D =$ total allowable creep-fatigue damage. Often $D = 1$, but sometimes $D < 1.0$

- **Modification of fatigue relationships**, by introducing a frequency term that depends on time at high-temperature fatigue deformation through [35]:

  $$v^k t_f = v^k (N/n) = Nv^{k-1}$$

  (2.51)

  where $t_f$ is the time to failure, $k$ is a constant that depends on temperature and $Nv^{k-1}$ is known as the *frequency modified fatigue life*.

- **Strain-range partitioning**, which is based on the idea that any reversed inelastic strain cycle can be broken down into four terms: completely reversed plasticity or fatigue, $\Delta \varepsilon_{pp}$, tensile fatigue reversed by compressive creep, $\Delta \varepsilon_{cp}$, tensile creep reversed by compressive fatigue, $\Delta \varepsilon_{pc}$; and completely reversed creep, $\Delta \varepsilon_{cc}$. Two types of deformation are considered (Figure 2.37); time-dependent creep deformation ($c$) and time-independent plastic deformation ($p$). The first subscript letter refers to the type of deformation in the tensile part of the cycle, while the second one refers to the type of deformation in the compression part of the cycle. In any hysteresis loop, only one of $\Delta \varepsilon_{pc}$ and $\Delta \varepsilon_{cp}$ will exist depending on whether creep or fatigue is the larger component as shown in Figure 2.38. Also, the difference between the two plastic flow components must equal the difference between the creep components, i.e., $AC - DB = BA - CD$. This difference is either $\Delta \varepsilon_{pc}$ or $\Delta \varepsilon_{cp}$. In addition, the sum of all the strain ranges terms equals the total inelastic strain range, i.e., $\Delta \varepsilon_i = \Delta \varepsilon_{cc} + \Delta \varepsilon_{pp} + \Delta \varepsilon_{pc}$. At a given value of $\Delta \varepsilon_i$, the components can be written as fractions [35]:
\[ F_{pp} = \frac{\Delta \varepsilon_{pp}}{\Delta \varepsilon_i} \quad F_{pc} = \frac{\Delta \varepsilon_{pc}}{\Delta \varepsilon_i} \quad F_{cc} = \frac{\Delta \varepsilon_{cc}}{\Delta \varepsilon_i} \quad F_{cp} = \frac{\Delta \varepsilon_{cp}}{\Delta \varepsilon_i} \] (2.52)

Therefore, the predicted life for the combined creep and fatigue damages, \( N_{pred} \) is given by the interaction damage rule [18]:

\[
\frac{1}{N_{pred}} = \frac{F_{pp}}{N_{pp}} + \frac{F_{pc}}{N_{pc}} + \frac{F_{cc}}{N_{cc}} + \frac{F_{cp}}{N_{cp}}
\] (2.53)

The assessment of creep-fatigue damage is applicable for materials in which intergranular creep damage may be quantified. Measurements are performed away from the main crack in order to estimate the homogeneous damage due to creep without interaction with the main crack. Creep-fatigue damage may then be quantified as a function of cracked grain boundary fraction and surface crack density [7].

Figure 2.37. The four types of partitioned strain-range cycles [35].

Creep-fatigue damage involves the development of transgranular fatigue cracking from the surface and intergranular creep damage from sub-surface to the point where the intensity of grain
boundary damage is sufficient to deflect crack propagation onto the grain boundaries. Figure 2.39 shows an example of the development of creep-fatigue damage in 1%CrMoV rotor steel at 565 °C. Multiple short fatigue crack development occurs at the surface, while creep damage evolves from the center of the specimen. When the fatigue crack meets the creep damage, interaction occurs [7].

Figure 2.38. A typical creep-fatigue strain hysteresis cycle [35].
Figure 2.39. Creep-fatigue crack development in a 1%CrMoV rotor steel at 565 °C [7].
2.4. References


3. EXPERIMENTAL SETUP

3.1. Material

The material investigated in this work is Fe-25Ni-20Cr advanced austenitic stainless steel (known as Alloy 709) manufactured by G.O. Carlson heat 58776-4 (GOC ID: 58776-4-B1) and received in the form of a rectangular plate with dimensions of about 735 mm (length) × 189 mm (width) × 30.48 mm (thickness). As-received Alloy 709 is shown in Figure 3.1. The G.O. Carlson plate had an argon-oxygen-decarburization (AOD) melt condition. The chemical composition of the alloy is given in Table 3.1. The history of the material started from an ingot of the Alloy 709 that was fabricated by hot-rolling and then solution-annealing at 1100 °C for 75 minutes (4,500 seconds) followed by subsequent water-quenching.

Figure 3.1. The hot-rolled plate of Alloy 709.
Parallel-gage test specimens (Figure 3.2) were machined from the as-received plate with 3 mm diameter gage section and 12 mm length along the rolling direction for low-cycle fatigue (LCF) and creep-fatigue tests.

Table 3.1. Chemical composition (wt%) of Alloy 709.

<table>
<thead>
<tr>
<th>Element</th>
<th>Wt %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>52.0288</td>
</tr>
<tr>
<td>Ni</td>
<td>25</td>
</tr>
<tr>
<td>Cr</td>
<td>19.9</td>
</tr>
<tr>
<td>Mo</td>
<td>1.46</td>
</tr>
<tr>
<td>Mn</td>
<td>0.88</td>
</tr>
<tr>
<td>Si</td>
<td>0.28</td>
</tr>
<tr>
<td>Nb</td>
<td>0.23</td>
</tr>
<tr>
<td>N</td>
<td>0.14</td>
</tr>
<tr>
<td>C</td>
<td>0.063</td>
</tr>
<tr>
<td>Ti</td>
<td>0.01</td>
</tr>
<tr>
<td>P</td>
<td>0.005</td>
</tr>
<tr>
<td>B</td>
<td>0.0022</td>
</tr>
<tr>
<td>S</td>
<td>0.001</td>
</tr>
</tbody>
</table>

Figure 3.2. Alloy 709 sample geometry.
3.2. Method

Low-cycle fatigue (LCF) tests have been carried out on the Alloy 709 at total strain ranges varying between 0.3%-2.5% and creep-fatigue tests at total strain ranges varying between 0.6%-1.2% with tensile hold times of 0, 60, 600, 18,00 and 3,600 seconds at a strain rate of $2 \times 10^{-3}$ s$^{-1}$ (0.1 Hz) and temperatures varying between 650 °C-750 °C.

The tests were carried out using an electro-dynamic creep-fatigue machine (Model 810LE3) from TestResources, Inc. The strain was measured and controlled using a Linear Variable Differential Transducer (LVDT) with an accuracy of 2 μm displacement (or 0.008% strain) attached to a high-temperature extensometer mounted on the grip-section of the samples. The LVDT was connected to a PC through a LVDT control box and a data acquisition box. To enable high-temperature testing, a three-zone resistive-heating furnace from Applied Testing Systems was used during the tests, and the temperature was controlled using a K-type thermocouple attached to the gage section of the sample and maintained within ±2 °C during the tests. When the desired temperature is reached before starting the test, each sample is soaked at that temperature for 30 minutes to ensure homogeneous distribution of the temperature over the entire gage length. Following the completion of each test, the sample is rapidly cooled to room temperature by turning the furnace off and using an air fan to preserve the deformation microstructure. The experimental set-up for both LCF and creep-fatigue tests is shown in Figure 3.3, in which the load and extension were recorded using LCF software connected to the testing machine through MTL programming via a PC.
3.3. Microstructure Characterization

3.3.1. Optical Microscopy

Post-test microstructural examination of the free surfaces of Alloy 709 was performed using HIROX optical microscope (Model C7214-9015-1) to study the crack and grain morphologies as shown in Figure 3.4. The free surfaces were sectioned to about 2 mm thickness, then mechanically polished using 600–1200 SiC papers followed by 9–1 μm water-based diamond suspension. Subsequently, 50 nm colloidal silica solution was utilized followed by ultrasonic cleaning in acetone. Finally, the samples were air dried before placing them on the microscope sample holder.
3.3.2. Scanning Electron Microscopy

Microstructural examination of the as-received and deformed samples of Alloy 709 was performed using scanning electron microscopy (SEM) to study the grain and crack morphologies before and after creep-fatigue interaction. SEM fractography was carried out in FEI Quanta 3D FEG and SEM Verios 460L at Advanced Instrumentation Facility (AIF) at NC State University (Figure 3.5). The fractured samples were sectioned to about 2 mm thickness and cleaned in acetone before being placed inside the SEM sample holder, while the free surfaces (~ 2 mm thick) were mechanically polished using 600–1200 SiC papers followed by 9–1 μm water-based diamond suspension. Subsequently, 50 nm colloidal silica solution was utilized followed by ultrasonic cleaning in acetone. Finally, the samples were air dried before placing them on the microscope sample holder.
Transmission Electron Microscopy

Transmission electron microscopy (TEM) characterization of the as-received and deformed samples of Alloy 709 was used to study the microstructure in terms of phase transformation, defect structure and phase and chemical analyses of precipitates prior to and after creep-fatigue damage using JEOL 2000FX TEM at Advanced Instrumentation Facility (AIF) at NC State University, operated at an accelerating voltage of 200 kV (Figure 3.6(a)). Also, FEI Talos F200X TEM in the Irradiation Microstructure Examination Laboratory (IMCL) at Materials and Fuels Complex (MFC) at Idaho National Laboratory (INL) was used (Figure 3.6(b)).

The preparation of TEM thin specimens of Alloy 709 started from cutting samples with thickness of about 0.9 – 1.2 mm then slowly polishing them using 320 grit and 400 grit silicon carbide (SiC) papers down to 500 μm followed by using 600 grit and 800 grit papers down to 200
μm. After that, the sections were polished down to ~ 70 μm thick using Aluminum Oxide Lapping film of 30 – 1 micron grades. Then, the TEM specimens were electro-polished using twin-jet electro-polishing technique which uses an electrolyte of 10 vol% perchloric acid and 90 vol% methanol solution at ~ -35 °C at a voltage of 30 V and a current of about ~ 30 mA, flowing onto the specimen to produce an electron-transparent region within the discs. Finally, the TEM specimens were air dried before being placed inside the TEM grid.

Figure 3.6. JEOL 2000FX TEM (a) and FEI Talos F200X TEM (b).
4. EFFECT OF HOLD TIME ON HIGH TEMPERATURE CREEP-FATIGUE BEHAVIOR OF Fe-25Ni-20Cr (WT.%) AUSTENITIC STAINLESS STEEL (ALLOY 709)

Abstract

To understand high temperature creep-fatigue interaction of the Alloy 709, strain-controlled low-cycle fatigue (LCF) tests were performed at strain ranges varying from 0.3% to 1.2% with fully reversible cycle of triangular waveform at 750 °C. In addition, different hold times of 60, 600, 1,800 and 3,600 seconds were introduced at the maximum tensile strain to investigate the effect of creep damage on the fatigue-life at strain range of 1% at 750 °C. The creep-fatigue life and the number of cycles to macro-crack initiation and failure are found to decrease with increasing hold time indicating higher crack initiation and growth rates. Creep-fatigue life is evaluated by a linear summation of fractions of cyclic and creep damages according to ASME code. The fractographs of the samples deformed at 1% strain range indicated that fatigue might have been the dominant mode of deformation whereas, for the samples deformed at the same strain range with different hold times, both fatigue and creep have contributed to the overall deformation and fracture of the alloy.

4.1. Introduction

With increasing demand on energy these days and for the near future, there should be a continuous improvement in lifetime and safety of the energy systems to satisfy the future needs.

---------------------------------------------------------------------------------------------------------------------

Generation IV nuclear reactors are being designed to be safer, more reliable and more efficient with a longer lifetime than the fleet of current nuclear reactors that are reaching their life expectancy. An example of Generation IV nuclear reactors is Sodium-cooled Fast Reactor (SFR) that is being developed to operate at temperatures as high as 550 °C and uses sodium as coolant and moderator due to its high melting point, low specific heat, high thermodynamic efficiency and heavy atoms compared to water. The structural material of SFR should have superior mechanical properties that can withstand high temperatures, high corrosive environments and high radiation doses. Preliminary data suggests that a newly developed Fe-25Ni-20Cr austenitic stainless steel stabilized by nitrogen and strengthened by niobium (known as Alloy 709) is an excellent candidate as structural material for SFR because it is corrosion-resistant, has high strength, thermal stability, sodium compatibility and good creep properties [1]. Alloy 709 has been developed based on the NF709 in ultra-supercritical (USC) power plants as superheater and reheater for coal fired boilers [2].

Creep-fatigue interaction is expected to be a contributor to the damage mode for many reactor components such as reactor cladding, gas turbines and pressure vessels operating at high temperatures in next-generation nuclear reactors [3-7]. In the past decades, many efforts have been made to study the effect of hold time, strain range, strain rate and temperature on the creep-fatigue life of austenitic stainless steels such as 304 and 316 stainless steels which are selected for the primary components in liquid metal-cooled fast reactors. However, most of these studies were performed at temperature range of 550-650 °C with different strain ranges and strain rates which were found to exhibit decreasing in the number of cycles to failure and increasing in the time to failure with applying tensile hold times during continuous cycling [4, 8]. Nevertheless, the service temperature for structural components of SFRs can operate at temperatures as high as 550 °C, other
components can be exposed to higher service temperatures and thus, exhibit shorter lifetimes such as boiler tubes. Therefore, it is important to investigate and understand the creep-fatigue interaction of an advanced austenitic stainless steel such as Alloy 709 at higher temperatures as 750 °C to generate enough amount of data in a reasonable time and then extrapolate it to the service conditions expected in SFRs. In previous publications [1], a general comparison between Alloy 709 and 316H stainless steel showed that Alloy 709 has outstanding properties over other austenitic stainless steels under creep-fatigue tests.

In this chapter, the creep-fatigue interaction of the Alloy 709 is investigated by conducting a series of strain-controlled low-cycle fatigue (LCF) tests and creep-fatigue tests with hold times of 60, 600, 1,800 and 3,600 seconds at 750 °C followed by microstructural evaluations. The results are reported and discussed in terms of creep-fatigue life predictions using the linear summation rule and the effect of creep-fatigue interaction on the crack properties and morphologies.

4.2. Experimental Details

4.2.1. Material

The investigated material is Fe-25Ni-20Cr austenitic stainless steel (Alloy 709) in the form of a plate that had undergone hot-rolling followed by solution-annealing heat-treatment at 1100 °C and the chemical composition of the alloy is given in Table 4.1. Parallel-gage test specimens (Figure 4.1(a)) were machined from the as-received plate with 3 mm diameter gage section and 10 mm length oriented along rolling direction for low-cycle fatigue and creep-fatigue tests.

<table>
<thead>
<tr>
<th>Element</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>N</th>
<th>Ti</th>
<th>Nb</th>
<th>B</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wt%</td>
<td>0.07</td>
<td>0.91</td>
<td>0.44</td>
<td>&lt;0.014</td>
<td>&lt;0.000</td>
<td>19.93</td>
<td>24.98</td>
<td>1.51</td>
<td>0.148</td>
<td>0.04</td>
<td>0.26</td>
<td>0.0045</td>
<td>Bal.</td>
</tr>
</tbody>
</table>
4.2.2. Method

The low-cycle fatigue and creep-fatigue tests were performed using an electro-dynamic creep-fatigue machine (Model 810LE3) from TestResources, Inc (Figure 4.1(b)). The strain was measured and controlled using a Linear Variable Differential Transducer (LVDT) attached to a high-temperature extensometer mounted on the grip-section of the test specimen. For high-temperature testing, a furnace from Applied Testing Systems was used during the tests, and the temperature was controlled using a K-type thermocouple attached to the gage section of the sample and monitored within ±2 °C during the tests. The experimental set-up is shown in Figure 4.1(b). Microstructural examination was performed using optical microscopy, scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The fractured samples were cleaned in acetone before performing microstructural characterization and optical microscopy was made using HIROX microscope (Model C7214-9015-1). SEM fractography was carried out in FEI Quanta 3D FEG. The TEM samples were prepared using twin-jet electro-polishing in 10 vol% perchloric acid and 90 vol% methanol solution at ~ -35 °C at a voltage of 20 V. The TEM examination was done using JEOL 2000FX.

Strain-controlled fatigue tests were conducted at 0.3% to 1.2% strain ranges and creep-fatigue tests were performed with hold times of 60, 600, 1,800 and 3,600 seconds at 1% stain range at 750 °C and 2×10^{-3} s^{-1} strain rate (0.1 Hz). As shown in Figure 4.2(a), a triangular waveform is produced when no hold time is introduced while a trapezoidal waveform is produced with imposed hold times (Figure 4.2(b)). Also shown in Figure 4.2 are the corresponding variations of the stress and hysteresis loops during loading cycles.
Figure 4.1. The specimen geometry (a) and the experimental set-up (b).

Figure 4.2. Schematic of loading cycle of the strain-controlled creep-fatigue test depicting strain–time, stress–time and stress–strain curves without hold time (a) and with hold time imposed at the peak tensile strain (b).
4.3. Results and Discussion

4.3.1. Low-Cycle Fatigue Tests

Strain-controlled fatigue tests for the Alloy 709 were performed at 750 °C and 2×10^{-3} s^{-1} strain rate (0.1 Hz) with strain ranging from 0.3% to 1.2%. Typical hysteresis loops corresponding to the second cycle at 0.5% and 1% strain ranges shown in Figure 4.3 exhibiting similar shapes at different strain ranges. The number of cycles to failure of Alloy 709, \(N_f\), at each tested strain range, \(\Delta \varepsilon\), is listed in Table 4.2 in which the number of cycles to failure decreases with increasing strain range. Figure 4.4(a) shows the strain amplitude, \(\Delta \varepsilon/2\), vs. number of reversals, \(2N_f\), on a log-log scale with the low-cycle fatigue (LCF) and high-cycle fatigue (HCF) portions clearly delineated along with fatigue life \((N_t)\) at which the transition from LCF to HCF occurs. LCF is characterized by Coffin–Manson equation [3]:

\[
\frac{\Delta \varepsilon_p}{2} = A (2N_f)^c
\]  

(4.1)

where \(\Delta \varepsilon_p\) is the plastic strain range, \(c\) ranging from -0.5 to -0.7 and \(A\) is a material constant that is proportional to the tensile ductility. On the other hand, HCF is characterized by Basquin equation [3]:

\[
\frac{\Delta \varepsilon_E}{2} = B (2N_f)^b
\]  

(4.2)

where \(\Delta \varepsilon_E\) is the elastic strain range, \(b\) ranging from -0.05 to -0.12 and \(B\) is a material constant that is proportional to the ultimate tensile strength. LCF is controlled by ductility and HCF is controlled by strength. Therefore, the LCF (plastic) curve appears at higher strain amplitudes than the LCF (elastic) curve. This means that good resistance to high strain amplitudes requires high
material ductility during LCF, while resistance to low strain amplitudes requires high tensile strength during HCF [9]. The fatigue data correlated with the following equation:

\[
\frac{\Delta \varepsilon}{2} = 0.5968 \ (2N_f)^{0.1234} + 8.2558 \ (2N_f)^{-0.5232}
\]  

(4.3)

where \( \Delta \varepsilon / 2 \) is the strain amplitude. Figure 4.4(b) shows the experimental life vs. the predicted life obtained using Eq. (4.3) on a log-log scale, where a linear correlation is observed between the experimental and predicted values.

Figure 4.3. Hysteresis loops of the LCF tests of the Alloy 709 at 0.5% and 1% strain ranges showing the second cycle.
Figure 4.4. Strain amplitude, $\Delta \varepsilon / 2$, vs. number of reversals, $2N_f$, in log-log scale showing both LCF and HCF parts (a) and experimental life vs. predicted life of Alloy 709 under LCF and HCF (b).

Table 4.2. The fatigue life of Alloy 709 at 750 °C at different strain ranges.

<table>
<thead>
<tr>
<th>Strain Range (%)</th>
<th>0.3</th>
<th>0.4</th>
<th>0.5</th>
<th>0.6</th>
<th>0.8</th>
<th>1.0</th>
<th>1.2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of Cycles to Failure, $N_f$</td>
<td>109395</td>
<td>17417</td>
<td>3745</td>
<td>2495</td>
<td>892</td>
<td>526</td>
<td>453</td>
</tr>
</tbody>
</table>

4.3.2. Creep-Fatigue Tests

4.3.2.1. Creep-Fatigue Properties

The second cycle of creep-fatigue hysteresis loops for the Alloy 709 at different hold times at 1% strain range are shown in Figure 4.5. The width of the hysteresis loops is found to increase with hold time indicating the rise of the inelastic strain [6]. It is found that the increase in the inelastic strain was due to transformation from elastic strain during stress relaxation. Therefore, this behavior suggests the occurrence of creep damage at high temperatures [5, 10, 11]. Creep...
damage here means that the material will soften due to recovery processes during the hold time under strain-controlled creep-fatigue tests. Also, this rise in the inelastic strain during strain-controlled hold time, corresponds to the fact that stresses do not relax completely as will be discussed later [5]. To understand the softening behavior of the alloy during cyclic deformation at 1% strain range, peak stresses during tension and compression parts of the loading cycles were plotted as a function of number of cycles on a log scale at different hold times (Figure 4.6(a)). In continuous cyclic loading without imposing hold time, the alloy exhibits rapid rate of cyclic softening while cyclic hardening is observed when hold times are incorporated. Moreover, the rate of cyclic hardening becomes more obvious with increasing hold time. The change of the cyclic behavior is related to the microstructure and can be due to: (i) dislocation-dislocation, (ii) dislocation–precipitate and (iii) dislocation–solute atom interactions [12]. These observations are comparable with the creep-fatigue test results obtained by Porter, et. al., [13] on Alloy 709 at 550 °C and 650 °C, at 0 and 1,800 seconds hold times and $10^{-3} \text{ s}^{-1}$ strain rate. Time variations of the maximum tensile stresses is shown in Figure 4.6(b) on a log scale at each investigated hold time that indicate small initial increase followed by distinct decrease at longer hold times. The creep-fatigue life of the alloy was found to decrease with increasing hold time (Figure 4.6(c)) until it reached saturation where number of cycles to failure is essentially independent of the hold time [5]. Similar saturation behavior has been reported by Dewa, et. al., [6] and Totemeier [14] in Alloy 617 where the evolution of carbide precipitation at high temperatures was suggested to be responsible for this saturation. In the Alloy 709, Alomari et al. have found different types of carbide and nitride precipitates during creep deformation at 750 °C [15] and thus the saturation noted here might be due to the strength of carbide precipitation.
Figure 4.5. Second cycle hysteresis loops for the creep-fatigue tests of the Alloy 709 at 1% strain range and different hold times.

4.3.2.2. Stress Relaxation Behavior

Stress relaxation occurs during the tensile hold time as shown in Figure 4.7 for typical mid-life stress relaxation response curves of the Alloy 709 for different hold times. The decrease in the tensile stress during hold time is related to the recovery process [6], and all the stress relaxation curves are quite similar with the stress decreasing rapidly at the beginning followed by a relatively stable state of stress relaxation. However, the minimum stress during relaxation is found to decrease significantly with increasing hold time and the stress at the end of the hold time continued to decrease with increasing hold duration [5]. During stress relaxation, creep damage occurs which is indicated by the stress reduction. The stress relaxation curve during hold time can be fitted to a power-law equation [5] as shown in Figure 4.7:
\[ \sigma = b_0 (t + t_o)^h \]  \hspace{2cm} (4.4)

where \( b_o, t_o \) and \( b_1 \) are fitting parameters in which their values for each hold time are shown in Table 4.3. The fitted-curves are plotted as solid lines in Figure 4.7. Therefore, the creep rate can be calculated from stress relaxation curves using the Maxwell model as follows [6, 16, 17]:

\[ \sigma_r = E \dot{\varepsilon} \]  \hspace{2cm} (4.5)

where \( \sigma_r \) is the measured relaxed stress, \( E \) is the elastic modulus and \( \dot{\varepsilon} \) is the creep rate; \( \sigma_r \) values were found to be 204.5, 147.4, 119.9 and 27.4 MPa for hold times of 60, 600, 1,800 and 3,600 seconds, respectively. The creep rate during stress relaxation was found to be in the range of \( 4.2 \times 10^{-6} - 5 \times 10^{-4} \) s\(^{-1}\) for hold times of 60 and 600 seconds, whereas it was found to be in the range \( 2.8 \times 10^{-8} - 5.9 \times 10^{-8} \) s\(^{-1}\) for hold times of 1,800 and 3,600 seconds. The creep rate depends on the stress following Norton's power law with stress exponent \( (n) \) between 5.9–10.5 which is comparable to the value obtained for this alloy \( (n = 7) \) [15].

**Table 4.3. Values of fitting parameters of power-law equation.**

<table>
<thead>
<tr>
<th>Hold Time (second)</th>
<th>Cycle Number</th>
<th>( b_o )</th>
<th>( t_o )</th>
<th>( b_1 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>60</td>
<td>270</td>
<td>226.009</td>
<td>4.746</td>
<td>-0.054</td>
</tr>
<tr>
<td>600</td>
<td>207</td>
<td>203.422</td>
<td>5.008</td>
<td>-0.058</td>
</tr>
<tr>
<td>1,800</td>
<td>135</td>
<td>256.162</td>
<td>2.068</td>
<td>-0.125</td>
</tr>
<tr>
<td>3,600</td>
<td>105</td>
<td>400.276</td>
<td>5.590</td>
<td>-0.416</td>
</tr>
</tbody>
</table>
Figure 4.6. Maximum and minimum stresses vs. number of cycles to failure at different hold times (a), maximum stress with time at different hold times (b) and number of cycles to failure vs. hold time (c) (all at 1% strain range).
Figure 4.7. Stress relaxation of Alloy 709 with time at 1% strain range and different hold times at mid-life cycle.

4.3.2.3. Linear Damage Summation

According to the ASME Code, Section III, Subsection NH, creep-fatigue life is determined by a linear summation of cyclic and creep damage fractions. The linear damage summation rule is used because it is applicable over a wide variety of conditions with the availability of application data, and also to recognize which damage developed more rapidly until fracture. The creep-fatigue criterion is given by [5, 6, 18]:

\[
\sum_j \left[ \frac{n_j}{N_{d_j}} \right] + \sum_k \left[ \frac{\Delta t_k}{T_{d_k}} \right] \leq D
\]  

(4.6)

where \( n_j \) and \( N_{d_j} \) are the number of cycles of type \( j \) and the allowable number of cycles of type \( j \), respectively; and \( \Delta t \) and \( T_{d} \) are the actual time at stress level \( k \) and the allowable time at same stress
level, respectively; $D$ is the allowable combined damage fraction which is usually assumed to be equal to one. The first term accounts for fatigue damage, $D_f$, in terms of number of cycles and the second term accounts for creep damage, $D_c$, in terms of time-fraction. Fatigue damage fraction is defined in terms of the ratio of the cycles to failure under creep–fatigue condition, $n$, to the number of cycles to failure under continuous cycling loading, $N_d$, at the same conditions applied in the creep–fatigue test. On the other hand, creep damage fraction is calculated using time-fraction method based on a linear correlation between the rupture time ($t$), temperature ($T$), and applied stress ($\sigma$) using Larson-Miller Parameter (LMP) as employed by Wright, et al., [5]:

$$LMP = T(\log(t) + C) \tag{4.7}$$

where $C$ is the Larson-Miller constant (~20), $t$ is rupture time in hours. A linear equation in semi-log scale describes the creep data using LMP:

$$LMP = a_o + a_1 \log(\sigma) \tag{4.8}$$

where $a_o$ and $a_1$ are the fitting parameters which are found to be 38357.14 and -7142.86, respectively. Therefore, creep damage can be calculated as follow [5]:

$$D^c_k = \frac{b_o^{-m}}{A(1-b_m)} \left( (t_h + t_o)^{1-b_m} - (t_o)^{1-b_m} \right) \tag{4.9}$$

where $t_h$ is the stress relaxation hold time in seconds, $A$ and $m$ are fitting parameters in the Larson-Miller parameter as a function of stress which are found to be $1.125 \times 10^{20}$ and -6.98, respectively. The creep damage in Eq. (4.6) is calculated at the half-life cycle and then multiplied by the corresponding creep-fatigue life. Figure 4.8 shows the creep-fatigue interaction diagram for the Alloy 709 where creep damage is plotted as a function of the fatigue damage at different hold
times; the ideal failure criterion of linear damage summation is also shown. As shown, both the fatigue and creep damages decrease with increasing the hold time, where data points are shown to lie above and below the ideal failure criterion. Decreasing the creep damage can be attributed to decreasing the relaxed stress during stress relaxation with hold time, in which it was fitted to a power law equation as shown earlier according to the approach followed by Wright, et. al., [5]. Generally, the creep–fatigue damage envelope represents the average trend of the interaction between the creep and fatigue damages depending on the material and microstructure [5]. In this study of the Alloy 709, however, more data is needed to help us establish a better understanding of the creep-fatigue behavior of the Alloy 709 with increasing hold time, and, therefore, determine the creep-fatigue damage envelope.

Figure 4.8. Creep-fatigue interaction diagram using linear damage summation; the red dashed line shows the ideal failure criterion.
4.3.2.4. Effect of Creep-Fatigue on Crack Properties

According to ASTM standard E2714-13 [19], the number of cycles to crack formation are determined by the number of cycles when a specific crack size is formed based on: (a) a specific percentage drop in the peak tensile stress with respect to the level during the test, (b) a specific percentage drop in the tensile modulus of elasticity to the compressive modulus of elasticity of the hysteresis loops, (c) a specific percentage drop in the peak tensile stress with respect to the compressive stress, or (d) a specific increment in crack size as measured by an electrical potential drop monitoring crack propagation. Therefore, to determine the point of macro-crack initiation and failure following the approach applied by Totemeier, et. al., [20], the absolute value of the ratio of maximum tensile stress to compressive stress is plotted as a function of cycle number (Figure 4.9(a)); macro-crack initiation is defined as the point at which the stress ratio deviates from linearity (indicated by black circles) while failure is defined by a 20% drop in stress ratio from the point of deviation (indicated by black squares). Determining the creep-fatigue life from this ratio distinguishes between changes in peak stresses due to cyclic hardening or softening and those due to crack initiation and propagation [5, 13, 20]. Figure 4.9(b) shows the effect of hold time on the numbers of cycles to crack initiation and failure where we note that the number of cycles decreases with increasing hold time. Also, with increasing the hold time, the difference between number of cycles to crack initiation and failure decreases indicating higher crack initiation and growth rates for longer hold times.
Figure 4.9. Peak tensile and compressive stress ratio for creep-fatigue tests of the Alloy 709 at 1% strain range and different hold times (a) and the effect of hold time on number of cycles to crack initiation and failure (b).

Both the number of cycles to failure, \( N_f \), and time to fracture, \( t_f \), can be used to describe the creep-fatigue lifetimes. The time to fracture, \( t_f \), can be calculated by using the strain rate, \( \dot{\varepsilon} \), or ramping frequency, \( f \), as following [7]:

\[
t_f = \frac{N_f}{f} + N_f t_h
\]  \hspace{1cm} (4.10)

where \( f \) is given by,

\[
f = \frac{\dot{\varepsilon}}{2 \cdot \Delta \varepsilon_f}
\]  \hspace{1cm} (4.11)

so that,

\[
t_f = N_f \left( \frac{2 \cdot \Delta \varepsilon_f}{\dot{\varepsilon}} + t_h \right)
\]  \hspace{1cm} (4.12)
where \( t_h \) is the hold time and \( \Delta \varepsilon_T \) is the total strain range. The effect of hold time in one cycle on the time to failure in Alloy 709 under creep-fatigue is shown in Figure 4.10(a) on a log-log scale where we note increased failure time with increasing hold time; the term \((2 \cdot \Delta \varepsilon_T / \dot{\varepsilon}) + t_h\) in Eq. (4.12) is defined as one cycle time, \( c_t \). Figure 4.10(b) shows the relationship between time to failure, \( t_f \), and one cycle time, \( c_t \), on a log-log scale, in which the same trend as in Figure 4.10(a) is noted.

![Figure 4.10](image)

Figure 4.10. Effect of hold time, \( t_h \), on the time to failure, \( t_f \), (a) and one cycle time, \( c_t \), (b) under creep-fatigue.

### 4.3.2.5. Crack Morphology

Creep-fatigue deformation is a combination of creep deformation and fatigue deformation at high temperatures. Generally, the creep-fatigue interaction is described as a mixed mode crack growth of pure fatigue and creep cavities developing independently on the grain boundaries [6, 16, 17, 21]. In order to investigate the damage mechanisms responsible for the final fracture of Alloy 709 subjected to creep-fatigue tests, detailed observations were carried out of specimen free
surfaces and fracture surfaces using both optical and scanning/transmission electronic microscopy (SEM/TEM).

For optical microscopy, the free surfaces of Alloy 709 after creep-fatigue tests were characterized at different magnifications and Figure 4.11 shows these fractographs taken at the transverse cross section perpendicular to the stress axis at 1% strain range and different hold times. Highly branched and almost closed cracks propagate in the specimen during low hold time (600 second) where oxygen is shown to penetrate along these cracks as revealed by a thin oxide layer at the crack tip. However, the cracks observed during longer hold time (3,600 second) are widely opened and almost straight filled with oxide (Figure 4.11(b,c)) than those observed at shorter hold times (Figure 4.11(a)). The straight morphology of the cracks in Figure 4.11(b,c) suggests a faster propagation than that of the branched cracks observed at shorter hold times [22]. The oxidation process is time-dependent in which the penetration depth of oxidation will be higher with increasing hold time. Therefore, the presence of cracks filled with oxide might exhibit a faster propagation (slip irreversibility [23], adjustment of the crack tip deformation behavior [24], etc) or a slower propagation (total “healing” of the crack [23]).
Figure 4.11. Fractographs of the free surface of Alloy 709 after creep-fatigue tests at 1% strain range at hold times of 600 seconds (a) and 3,600 seconds (b,c). (Stress axis perpendicular to the page).

In general, two types of crack phenomena are observed during a tensile hold in creep-fatigue tests that decrease the number of cycles to failure than those observed for low-cycle fatigue (LCF): earlier crack formation and faster crack growth [12]. Figure 4.12(a) shows an SEM image of as-
received material taken in backscattered electron imaging mode where microstructural features such as grain-boundaries and annealing twins are observed. Also, grain morphology is irregular and some of the grains seen in the micrographs are larger than 50 μm. Figure 4.12(b) and Figure 4.12(c) show two bright-field TEM images of as-received material taken from two different regions of the TEM sample in two-beam condition, in which a very low density of dislocations is observed within the matrix and a pile-up of dislocations at the grain boundary along with micro-sized precipitates. Different types of precipitates including Nb rich and Cr rich particles have been reported in the Alloy 709 [15, 25]. The fractographs of the fracture surface of Alloy 709 after low-cycle fatigue (LCF) test at 1% strain range using the SEM are shown in Figure 4.13. Crack nucleation sites are observed as shown in the high magnification fractograph corresponding to region 1 in Figure 4.13(b). On the other hand, the high magnification fractograph of region 2 (Figure 4.13(c)) shows fatigue striations due to crack propagation. Finally, the dimple features of the fractograph of region 3 in Figure 4.13(d) are usually observed during the last stages of pure fatigue tests and representing the overload region.

Figure 4.12. SEM micrograph of the as-received Alloy 709 taken in backscattered electron mode (a) and bright-field TEM micrographs at zone axis [112] with dislocations and Nb(C,N) precipitates [25] (b) and (c).
Figure 4.13. Fractographs of the fracture surface of Alloy 709 after LCF test at 1% strain range depicting different microscopic features representative of different modes of failure.

Also, the fracture surfaces of Alloy 709 after creep-fatigue tests were characterized using the SEM at different magnifications; Figure 4.14 and Figure 4.15 include fractographs at 1% strain range and different hold times of 60 and 1,800 seconds, respectively. As shown in these micrographs, striations start to disappear with increasing hold time indicating that the dominant damage mode is comprised of both creep and fatigue damages unlike the case of LCF where the
density of striations was higher due to dominant fatigue damage. Also, density of creep cavities is shown to increase with hold time as well, where large round creep cavities at the grain boundaries are noted (some are indicated by dotted red circles in Figure 4.14(b) and Figure 4.15(b,c)). The fractographs show ductile failure with higher crack density as hold time increases, where typical intergranular cracks (some are indicated by white arrows in Figure 4.14(b) and Figure 4.15(b,c)) and transgranular cracks (some are indicated by red arrows in Figure 4.14(b,c)) are observed. It is clear that cracking mode changes from mixed mode (intergranular and transgranular) to intergranular mode along with dimple features with increasing hold time where intergranular cracks become more dominant than the transgranular ones.

As mentioned by Hecht [26], during high temperature creep-fatigue interaction, multiple processes occur either simultaneously or in parallel: microstructural evolution and environmental interactions at high temperatures, fatigue damage and softening effect due to cyclic loading, accumulation of creep damage during tensile hold durations. In general, crack nucleates due to forward or reverse plastic flow of a slip band. In pure cyclic deformation, dislocations pile up and form persistent slip bands due to the movement of material along slip planes and thus, transgranular crack initiation and propagation is observed. In creep deformation, on the other hand, extensive creep cavitation occurs during deformation (specially in tertiary creep region) leading to intergranular crack initiation and propagation with dimple features. In this study, the fracture morphology showed crack nucleation sites, striations, ductile failure under shear stress and dimple features suggesting the influence of creep and fatigue interaction. Thus, the simultaneous interaction between intergranular creep cavities and transgranular fatigue cracks increases the crack growth rate and decreases the crack initiation endurance.
4.4. Conclusions

To understand high temperature creep-fatigue interaction of the Alloy 709; a candidate structural material for next-generation Sodium-Cooled Fast Reactor (SFR), strain-controlled low-cycle fatigue (LCF) tests were performed at strain ranges ranging from 0.3% to 1.2% with fully reversible cycle of triangular waveform at 750 °C in air. In addition, different hold times of 60, 600, 1,800 and 3,600 seconds were introduced at the maximum tensile strain to investigate the
effect of the creep damage on the fatigue-life at a strain range of 1% at 750 °C. The concluding remarks are:

- During creep-fatigue tests, the width of the hysteresis loops is found to increase with hold time suggesting the rise of inelastic strain with hold time indicating the occurrence of creep damage at high temperatures.
- The creep-fatigue life of the alloy decreases with increasing hold time until it saturates at longer hold times.
- With increasing hold time, the number of cycles to macro-crack initiation and failure decrease; indicating higher crack initiation and growth rates.
- The optical microscope revealed highly branched and almost closed crack growth in the specimen during low hold time (600 seconds) while almost straight, filled with oxide and widely opened cracks were observed during longer hold time (3,600 seconds).
- The fractographs of the fracture surface of Alloy 709 after low-cycle fatigue (LCF) test at 1% strain range using the SEM, showed crack nucleation sites, striations due to crack propagation and dimple features representing the region of overload fracture. On the other hand, the fracture surfaces of Alloy 709 after creep-fatigue tests showed much lower density of striations with increasing hold time indicating that the dominant damage mode is related to both creep and fatigue damages. In addition, the density of creep cavities and cracks is shown to increase with hold time, where typical intergranular and transgranular cracks are observed.
- Creep-fatigue life is evaluated by a linear summation of fractions of cyclic and creep damages according to ASME code. However, more studies are needed to confirm the creep-fatigue damage envelope for this alloy at different strain ranges and hold times.
4.5. References


5. EFFECT OF STRAIN RANGE ON HIGH TEMPERATURE CREEP-FATIGUE BEHAVIOR OF Fe-25Ni-20Cr (WT.%) AUSTENITIC STAINLESS STEEL (ALLOY 709)

Abstract

Since the preliminary data suggest that Fe-25Ni-20Cr austenitic stainless steel (Alloy 709) is an excellent candidate as structural material for high-temperature applications such as Sodium-Cooled Fast Reactors (SFRs) and Ultra-Supercritical (USC) power plants, the effect of strain range on creep-fatigue interaction of the Alloy 709 is investigated by conducting strain-controlled creep-fatigue tests with tensile hold times of 0, 600, 1,800 and 3,600 seconds at strain ranges varying from 0.6% to 1.2% at a temperature of 750 °C and $2 \times 10^{-3}$ s$^{-1}$ (0.1 Hz) strain rate. To establish fatigue characteristics of Alloy 709, strain-controlled fatigue tests were performed at strain ranges from 0.3% to 2.5% at 750 °C and $2 \times 10^{-3}$ s$^{-1}$ strain rate (0.1 Hz). It is found that the predicted fatigue life of Alloy 709 shows a better correlation with the characteristic slopes predictive method rather than the universal slopes method. In general, with increasing strain range at a given hold time, the number of cycles to failure decreases until saturation. Creep-fatigue interaction diagram of Alloy 709 at different strain ranges and hold times is presented according to linear damage summation (LDS). Finally, the fractographs of the deformed samples exhibited increased number of cracks with strain range along with $M_{23}C_6$ precipitates and high dislocation density.

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2 This chapter is submitted for publication in Journal of Materials at High Temperatures.
5.1. Introduction

Fe-25Ni-20Cr austenitic stainless steel (Alloy 709) is derived from NF709 (Fe-20Cr-25Ni-1.5Mo-Nb,B,N), which is a commercial heat- and corrosion- resistant austenitic stainless steel developed by Nippon Steel Corporation in Japan for boiler tubing applications [1]. Alloy 709 is an excellent high-performance candidate as structural material for high-temperature applications such as Sodium-Cooled Fast Reactors (SFRs) and Ultra-Supercritical (USC) power plants due to its adequate mechanical properties such as creep, and corrosion resistance that can tolerate the operating conditions in the plant [2, 3]. However, the design and operation of high-temperature nuclear reactors exhibiting cyclic thermal stresses during start-ups and shut-downs introduce time-dependent effects like creep during on-load periods at high temperatures. This phenomenon known as creep-fatigue introduces additional damage. Next-generation nuclear reactors such as Generation IV nuclear reactors are designed to operate as graphite-moderated, helium-cooled and thermal neutron spectrum reactors for the purpose of producing electricity and hydrogen without producing greenhouse gases, and they are also designed to have better efficiency greater than 40% compared to the current nuclear reactors that exhibit an efficiency of about 30% [4-6]. Therefore, creep-fatigue interaction is an important damage mode in next generation nuclear reactors such as SFR as a result of power transients in many reactor components like gas turbines, reactor cladding, pressure vessels, core supports, primary and secondary piping, and possibly intermediate and compact heat exchangers [1, 7-12]. Creep-fatigue behavior of Nickel-based alloys, such as 617, Hastelloy X and 230, have been studied at different conditions of hold times, strain ranges, strain rates and temperatures since these high-temperature metallic alloys are selected as the primary candidate materials for the Intermediate Heat Exchangers (IHXs) and other components like Hot Gas Ducts (HGDs) operating at temperature range of 800-1000 °C in the Very High Temperature
Reactors (VHTRs) [4, 10, 12, 13]. Also, creep-fatigue life has been investigated in austenitic stainless steels such as 304 and 316 which are selected for the primary components in liquid metal-cooled fast reactors at a temperature range of 550-650 °C and different strain ranges, which were found to exhibit decreasing number of cycles to failure at higher temperatures and higher strain ranges [10, 14-16]. As SFR is being developed to operate at temperatures as high as 550 °C, other components like boiler tubes exhibit higher operating temperatures and shorter lifetimes. Therefore, detailed investigations of creep-fatigue interaction need to be carried out at higher temperatures to examine the mechanical behavior of the structural materials of SFRs such as Alloy 709, to understand and thus explain the effects of strain range, hold time and temperature on its creep-fatigue life at expected operating conditions [7].

This work is part of an ongoing study of the effect of hold time [6] and strain range on creep-fatigue damage of the Alloy 709. In this part, the effect of strain range on creep-fatigue interaction of the Alloy 709 is investigated by conducting strain-controlled creep-fatigue tests with tensile hold times of 0, 600, 1,800 and 3,600 seconds at strain ranges varying from 0.6% to 1.2% at 750 °C. Also, strain-controlled fatigue tests were performed at strain ranges from 0.3% to 2.5% at 750 °C and 2×10⁻³ s⁻¹ strain rate (0.1 Hz). The results are reported and the fractographs of the deformed samples are presented and discussed.

5.2. Experimental Details

5.2.1. Material

Alloy 709 contains 20% chromium and 25% nickel, and the chemical composition of the alloy is included in Table 5.1 [7]. Figure 5.1(a) shows the as-received hot-rolled plate of Alloy 709 from which test specimens (Figure 5.1(b)) were machined with 3 mm diameter gage section and 12 mm gage length oriented along rolling direction.
Table 5.1. Chemical composition (wt%) of Alloy 709 [7].

| Element | C     | Mn   | Si   | P     | S     | Cr   | Ni   | Mo   | N    | Ti   | Nb   | B    | Fe   |
|---------|-------|------|------|-------|-------|------|------|------|------|------|------|------|------|------|
| Wt %    | 0.07  | 0.91 | 0.44 | <0.014| <0.000| 19.93| 24.98| 1.51 | 0.148| 0.04 | 0.26 | 0.0045| Bal. |

Figure 5.1. The hot-rolled plate of Alloy 709 (a) and the specimen geometry (b).

5.2.2. Experimental Method

Creep-fatigue tests on Alloy 709 were performed using an electro-dynamic creep-fatigue machine from TestResources, Inc. equipped with a high-temperature furnace from Applied Testing Systems (ATS) in which the temperature was monitored using k-type thermocouples attached to the gage section of the sample and connected to a high-temperature readout unit. High-temperature extensometer equipped with a Linear Variable Differential Transducer (LVDT) was used to measure and record strain data [7]. Microstructural characterization was performed using optical microscopy (HIROX microscope, Model C7214-9015-1), scanning electron microscopy (SEM) using FEI Quanta 3D FEG and SEM Verios. In addition, transmission electron microscopy (TEM) was carried out using JEOL 2000FX and FEI Talos F200X at Idaho National Laboratory (INL). The experimental setup along with details on sample preparation for microstructural
characterization have been reported earlier [7]. Strain-controlled creep-fatigue tests were conducted at 0.6% to 1.2% strain ranges with tensile hold times of 0, 600, 1,800 and 3,600 seconds at 750 °C and at a strain rate of 2×10^{-3} s^{-1} (0.1 Hz). Also, strain-controlled fatigue tests were performed at strain ranges from 0.3% to 2.5% at 750 °C and 2×10^{-3} s^{-1} strain rate (0.1 Hz) to characterize the fatigue behavior of Alloy 709.

5.3. Results and Discussion

5.3.1. Low-Cycle Fatigue Properties

Figure 5.2 shows the strain range, Δ\varepsilon, vs. number of cycles to failure, Nf, in a log-log scale under both low-cycle fatigue (LCF) and high-cycle fatigue (HCF) regions from which the parameters associated with Basquin (HCF) and Coffin-Manson (LCF) equations are determined. The fatigue life data of the alloy are analyzed in terms of both predictive methods of characteristic and universal slopes [17-20]. The fatigue life of the alloy calculated using the characteristic slopes correlated well with the experimental fatigue data with b = -0.15 and c = -0.90 (Figure 5.2) so that the strain range is related to the number of cycles to failure as in the following equation:

\[
\Delta \varepsilon = 1.73 \ (N_f)^{-0.15} + 157.55 \ (N_f)^{-0.90}
\]

On the other hand, the universal slopes equation is given by:

\[
\Delta \varepsilon = 0.50 \ (N_f)^{-0.12} + 45.00 \ (N_f)^{-0.60}
\]

As noted in Figure 5.2, the predicted fatigue life of Alloy 709 shows a better correlation with the characteristic slopes method.
Figure 5.2. Strain range, $\Delta \varepsilon$, vs. number of cycles to failure, $N_f$, in log-log scale showing both experimental data along with the predictions using characteristic and universal slopes.

5.3.2. Creep-Fatigue Life of Alloy 709

Creep-fatigue interaction occurs at relatively high strain ranges while creep failure dominates at low strain ranges [21, 22]. Figure 5.3 shows the applied strain range with respect to the number of cycles to failure, $N_f$, at different hold times indicating that the number of cycles to failure decreases with increasing strain range at a given hold time. At 0.8% strain range, it is found that the number of cycles to failure at 3,600 seconds hold time is slightly higher than that at 1,800 seconds hold time but still lower than that at 600 seconds hold time. Also, number of cycles to failure becomes relatively independent of the strain range at increased strain range and/or hold time. Such saturation behavior at high strain ranges and hold times have been observed in earlier study [7]. The saturation behavior is important for design considerations since hold times that follow it do not contribute to further damage. This observation was also noted in high chromium
steels [12], nickel-based superalloys (Inconel 718 and GH4169) [23] and 304 stainless steel [9]. It is known that the reduction in fatigue life during tensile hold times is due to the interaction between surface-initiated fatigue cracks and interior creep cavitation damage developed at grain boundaries [21, 24, 25]. Therefore, increasing the strain range results in reduction of creep-fatigue life due to the increase in corresponding stress amplitude, during which the critical crack length for failure decreases [26].

![Graph showing strain range vs. number of cycles to failure at different hold times at 750 °C.](image)

Figure 5.3. The strain range vs. number of cycles to failure at different hold times at 750 °C.

5.3.3. **Hysteresis Loop Behavior**

The half-cycle hysteresis loops for the creep-fatigue tests of the Alloy 709 at different strain ranges and hold times of 600 seconds and 3,600 seconds are shown in Figure 5.4, and the width of the hysteresis loop increases with increasing strain range at both hold times exhibiting more inelastic strain. This observation is also noted in Figure 5.4(c) and Figure 5.4(d) as the area under
the curve of the half-cycle hysteresis loop increasing with increasing strain range. However, the extent of the width-increase decreases with increased strain range at both hold times. Figure 5.5 shows the half-cycle peak stress amplitude, $\Delta \sigma /2$, increasing when strain range is increased at different hold times indicating a higher degree of cyclic hardening. Also, a linear relationship between the plastic strain range, $\Delta \varepsilon_p$, and the number of cycles to failure at a given hold time and strain rate of $2 \times 10^{-2}$ s$^{-1}$ is shown in Figure 5.6 in a log-log scale where this linearity follows the Coffin-Manson relation given by [7-9, 27]:

$$\frac{\Delta \varepsilon_p}{2} = A(2N_f)^c$$  \hspace{1cm} (5.3)

where $A$ is the fatigue-ductility coefficient and $c$ is the fatigue ductility exponent, respectively. As shown in Figure 5.6, the fatigue ductility coefficient increases with hold time while the fatigue ductility exponent decreases. Coffin-Manson perfectly predicts the lifetimes of low-cycle fatigue (LCF) at high strain ranges unlike at lower values because at low strain ranges, the plastic strain is very low. Thus most of the grains are plastically deformed (probably not all of them) and also, the crack growth is slow enough to allow healing of the cracks due perhaps to the growth of the oxide layer [28].
Figure 5.4. Half-cycle hysteresis loops for the creep-fatigue tests at 750 °C and different strain ranges and hold times of 600 seconds (a) and 3,600 seconds (b). Area under the curve vs. strain range at same hold times at half-cycle (c) and (d).
Figure 5.5. Half-cycle peak stress amplitude vs. strain range at different hold times at 750 °C.

Figure 5.6. Relationship between the plastic strain range, $\Delta \varepsilon_p$, and number of cycles to failure, $N_f$. 

$\Delta \varepsilon_p/2 = A(2N_f)^c$

$\dot{\varepsilon} = 2\times 10^{-3} \text{ s}^{-1}$
$T = 750 ^\circ \text{C}$

$34.30(2N_f)^{-0.64}$
$67.02(2N_f)^{-0.87}$
5.3.4. Cyclic Stress Response

The cyclic stress response of the Alloy 709 is shown in Figure 5.7 at 600 seconds (Figure 5.7(a)) and 1,800 seconds (Figure 5.7(b)) hold times at 750 °C in semi-log scale. As shown, the profiles of creep-fatigue peak tensile stresses with respect to failure cycles were similar regardless of the applied strain ranges, in which the cyclic stress response exhibits a region in which there is a plateau in stress extending to the failure point (defined by 90% drop in peak tensile stress [7]). At 600 seconds hold time, the peak tensile stresses at strain ranges from 0.6% - 1% are almost the same except at 1.2% strain range, where the material exhibits a higher peak stress and a lower degree of cyclic hardening followed by a rapid rate of cyclic softening until the stress drops off at the end of the test due to initiation and propagation of cracks. At 1,800 seconds on the other hand (Figure 5.7(b)), the material hardens up rapidly at all strain ranges followed by almost a steady state rate of cyclic hardening and then finally a rapid rate of cyclic softening. Cyclic hardening occurs due to the generation and interactions of dislocations whereas cyclic softening occurs due to the dynamic recovery of the dislocation substructure [7, 29, 30].
Figure 5.7. Cyclic stress response with respect to the number of cycles in semi-log scale at different strain ranges and hold times of 600 seconds (a) and 1,800 seconds (b).

5.3.5. Determination of Crack Initiation

The point of macro-crack initiation was determined following the approach applied by Totemeier, et. al. [4] and Alsmadi, et al. [7] in which the macro-crack initiation is defined as the point at which the absolute value of the ratio of maximum tensile stress to compressive stress deviates from linearity. Figure 5.8 shows the number of cycles to crack initiation at strain ranges varying from 0.6% to 1.2% and hold times of 600, 1,800 and 3,600 seconds. In general, the number of cycles to crack initiation decreases with increasing strain range at a given hold time. Regarding the initiation data at 0.8% strain range, the number of cycles to crack initiation at 1,800 seconds hold time is slightly higher than that at 0.6%. Crack initiation at 0.6% strain range occurred approximately at 72% of total life at 600 second hold time and 54% of total life at 1,800 seconds hold time. At 0.8% strain range on the other hand, crack initiation occurred approximately at 60%-76% of total life at all hold times. On the other hand, crack initiation occurred approximately at 65%-75% of total life at all hold times at 1% strain range. At 1.2% strain range, crack initiation
occurred approximately at 55%-62% of total life at all hold times. This is consistent with the initiation trends commonly observed for cycling loading, namely crack initiation occurs near the end of life for high-cycle fatigue (HCF) at low strain ranges, while crack initiation occurs early for low-cycle fatigue (LCF) at high strain ranges where the fatigue life is dominated by crack propagation [4, 31].

Figure 5.8. The effect of strain range on the number of cycles to crack initiation at different hold times.

### 5.3.6. The Effect of Strain Range on Time to Fracture

The effect of strain range, $\Delta \varepsilon$, on time to fracture, $t_f - t_o$, is shown in Figure 5.9 in a log-log scale, where $t_o$ is time to fracture with no hold time. Both fracture times are determined as mentioned by Alsmadi et al. [7] and Wang et al. [12]. It can be seen that time to fracture under creep-fatigue decreases with increasing strain range at a given hold time in a linear correlation.
Time to fracture at 0.6% strain range and 600 seconds hold time is very close to the one at the same strain range and 1,800 seconds hold time. As observed in the previous study on the effect of hold time on time to fracture in Alloy 709 [7], time to fracture increases with hold time whereas number of cycles to failure decreases. Both trends are correct and no contradiction exists, since introducing a hold time into the strain cycle increases the cycle time by a factor that is greater than that corresponding to the reduction in the number of cycles to failure. Therefore, the introduction of the tensile hold time will lead to the decrement of number of cycles to failure and the increment of time to fracture [9, 32]. This phenomenon was also observed in other steels like 304 and 316 stainless steels and Cr-Mo steel [9, 33, 34].

Figure 5.9. The effect of strain range, Δε, on the time to fracture, t_f - t_o, at different hold times and a strain rate of 2x10^{-3} s^{-1}. 
5.3.7. Stress Relaxation Behavior

During tensile hold time in strain-controlled creep-fatigue tests, the material exhibits stress relaxation in which creep damage occurs, indicated by stress reduction. Figure 5.10 shows the half-cycle stress relaxation of Alloy 709 at different strain ranges and hold times, in which the stress relaxes at the same rate regardless of the strain range applied at each hold time [7]. However, the shape of the relaxation curve is strongly dependent on strain range as also observed in 304 stainless steel at high temperatures [9]. At 0.8% strain range, the stress drops significantly at 600 and 1,800 seconds hold times and exhibits the minimum reduction at 3,600 seconds hold time. Almost all curves are found to exhibit serrations during stress relaxation. As mentioned by Sarkar, et al. [35], during a particular serration, an increase in the stress corresponds to the locking of dislocations in the solute environment. As reported earlier, different types of precipitates including Nb rich and Cr rich particles have been observed in the Alloy 709 [7, 36, 37]. Therefore, the time for stress increase corresponds to the waiting time of dislocations at the obstacle which is also proportional to the dislocation density at the obstacle. When these dislocations are released from the obstacle via thermal activation, the stress drops proportionally to the strength of the obstacle or the number of solute atoms locking the dislocations. During tensile hold time, the creep mechanism prevents the rearrangement of the dislocation cells, coalesces the subgrain, decreases the dislocation density and forms precipitates. Thus, chromium carbides can evolve at the creep temperature range, leading to an adverse effect of softening behavior [11, 29].

5.3.8. Creep-Fatigue Interaction Diagram

Creep-fatigue life is determined by a linear damage summation (LDS) of cyclic and creep damage fractions independently according to ASME [7]:

118
where \( n \) and \( N_d \) are the number of cycles of type \( j \) and the allowable number of cycles of type \( j \), respectively, and \( \Delta t \) and \( T_d \) are the actual time at stress level \( k \) and the allowable time at the same stress level, respectively, and \( D \) is the allowable combined damage fraction which is usually assumed to be equal to one [7, 10, 11, 38]. Therefore, fatigue damage fraction is determined based on the ratio of the cycles to failure under creep–fatigue condition, \( n \), to the number of cycles to failure under continuous cycling loading, \( N_d \), at the same conditions applied in the creep–fatigue test. Creep damage fraction is calculated using time-fraction method based on a linear correlation between the rupture time \( t \), temperature \( T \), and applied stress \( \sigma \) using Larson-Miller Parameter (LMP) and stress relaxation curve which is fitted to a power-law equation.
Creep damage is thus determined from the following formula and the detailed derivation of which is found in Alsmadi, et al. [7] and Wright, et al. [10]:

\[
D^*_h = \frac{b_o^m}{A(1-b_l m)} \left( (t_h + t_o)^{1-h_m} - (t_o)^{1-h_m} \right),
\]

(5.5)

where \( t_h \) is the hold time in seconds, \( A, m \) are fitting parameters in the LMP as a function of stress, whereas \( b_o, b_l \) and \( t_o \) are fitting parameters in power-law equation fitted to stress relaxation curves.
at different strain ranges and hold times (Figure 5.10). The creep damage is calculated at the half-life cycle and then multiplied by the corresponding creep-fatigue life [7, 10]. Creep-fatigue interaction diagram of Alloy 709 at different strain ranges and hold times is depicted in Figure 5.11 along with the ideal failure criterion according to LDS. Interaction diagrams are given to limit the combination of creep and fatigue damages below unity in the current design codes because failure is usually observed at a damage summation less than unity [23]. As shown, rather than following the ideal failure criterion, all data points lie below or above the line with damage values at 1,800 seconds and 3,600 seconds hold times close or below the $D=I$ curve. Data points lying below the line represent a strong creep-fatigue interaction, while the ones lying above it may represent the opposite trend [21, 39]. Also, high values of creep damage indicate that cavitation damage is enhanced by cyclic loading. Low values of fatigue damage indicate that crack initiation is enhanced by cavitation damage while high values indicate that crack propagation is enhanced by cavitation damage [21, 39]. As shown in Figure 5.11, at a given hold time, creep damage decreases with increasing strain range while fatigue damage increases. It was found that the value of $D$ is in the range of 0.6 to 1.0 for types 304 stainless steels [9, 40], which is comparable to what is observed in Alloy 709 with $D$ ranging from 0.2 to 1.2. However, LDS was not sufficient to determine the creep-fatigue life of Alloy 617 because the creep-fatigue damage data points lie above the draft code case line or the bilinear damage envelope determined by ASME [11].

It was reported that the LDS method generates nonconservative results for loading case of continuous cycling while conservative results for loading case of compression-hold-only creep-fatigue tests. Although LDS is used because it is applicable over a wide variety of conditions with the availability of application data, it still has some limitations [9]:
- The assumption of load-path independence to each of the fatigue and creep damage processes.
- The strain rate dependence of creep damage accumulation is not incorporated in this approach.
- LDS assumes a tensile hold damage similar to the compressive hold damage, while compression holds are found to have a healing effect in austenitic steels [41].
- This method cannot be used for waveforms without tensile hold times.

More data are needed to help establish the creep-fatigue bilinear damage envelope of Alloy 709 at different strain ranges, hold times and temperatures.

![Creep-fatigue interaction diagram of Alloy 709 at 750 °C](image)

Figure 5.11. Creep-fatigue interaction diagram of Alloy 709 at 750 °C; the black line shows the ideal failure criterion according to linear damage summation.
5.3.9. Crack Morphology

The free surfaces of Alloy 709 subjected to creep-fatigue tests at different strain ranges and hold times were characterized using optical microscopy (Figure 5.12) where these fractographs were taken at the transverse cross section perpendicular to the stress axis. As shown at 0.6% strain range and 1,800 seconds hold time (Figure 5.12(a)), transgranular mode of cracks are dominant on the surface while at higher strain ranges such as 1.2% at 600 seconds hold time (Figure 5.12(b)), mixed mode of cracks is observed where transgranular fatigue cracking is noted near the edge of the free surface followed by intergranular creep cavitation [30]. Crack nucleates due to forward or reverse plastic flow of slip bands in which persistent slip bands are formed due to the movement of material along slip planes and thus, transgranular crack initiation and propagation is observed. When applying hold time, creep cavitation occurs leading to intergranular crack initiation and propagation. Therefore, simultaneous interaction between intergranular creep cavities and transgranular fatigue cracks increases the crack growth rate and decreases the crack initiation endurance. Furthermore, very fine carbide stringers are observed at both strain ranges with higher density at 0.6% strain range as shown in Figure 5.12(a).

Creep-fatigue damage in air is observed to be influenced by the oxidizing environment, in which cracks initiated at oxidized regions will exhibit a faster propagation and failure. The degree of oxidation is higher at 0.6% strain range than at 1.2% strain range.
Figure 5.12. Fractographs of the free surface of Alloy 709 after creep-fatigue tests at 0.6% strain range and hold time of 1,800 seconds (a) and at 1.2% strain range and hold time of 600 seconds (b). (Stress axis perpendicular to the page).

5.3.10. Microstructural Evolution

As reported by Rodriguez et al. [21, 22], at low strain ranges, the surface crack initiation becomes very slow compared to the initiation of internal creep damage and thus the failure becomes creep dominated. At high strain ranges on the other hand, fatigue failure becomes dominant at rapid cycling conditions because there is not enough time for the occurrence of creep damage. SEM images of the as-received Alloy 709 are presented in Alsmadi, et al. [7]. Figure 5.13 and Figure 5.14 show an SEM images of the fracture surfaces of Alloy 709 under creep-fatigue tests at strain ranges of 0.8% and 1% at 1,800 seconds hold time, respectively. As shown at 0.8% strain range (Figure 5.13(a)), three observed secondary cracks propagate and finally coalesce in addition to the main crack that leads to failure while at 1% strain range (Figure 5.14(a)), more secondary cracks are observed along with the main crack which overall increase the crack density at the fracture surface. Also, creep damage dominates at low strain range of 0.8% with creep
cavities developed on the grain boundary (indicated by red circles) and a smaller number of cracks (Figure 5.13(b)). On the other hand, with increasing the strain range to 1% (Figure 5.14(b)), the number of cracks increases to a mixed mode of intergranular cracks (indicated by yellow arrows) and interior grain boundary cracking (indicated by blue arrows). Interior grain boundary cracks have little impact on the initiation and propagation of short intergranular surface cracks [13]. Striations are not observed at any of the strain ranges here due to the long hold time of 1,800 seconds. Also, at 0.8% strain range (Figure 5.14), the fracture surface is featured with rough dimples due to the hard inclusions in the material that do not deform at the same rate as the matrix, thus leading to the formation and interlinkage of voids where each dimple represents a void.

It is suggested that, when creep cavities are formed during tensile hold time, they will not undergo sintering since no compression hold is applied. Instead, they will nucleate and grow by irreversible shear deformation and thus cause intergranular failure [21, 25, 32, 42]. Furthermore, intergranular crack initiation can occur due to the diffusion of environmental species that hinder the collapse of cavities on the surface [21]. Under tensile hold time, compressive mean stresses cause the cavities on the grain boundaries to grow into elongated flat cracks [21, 43]. Crack nucleation and propagation of Alloy 709 was discussed in detail in Alsmadi, et al. [7]. The transition from crack nucleation to crack propagation has been reported as the transition from governing microcracks by cyclic plastic strain to governing crack propagation by fracture mechanics [44, 45]. It is worth mentioning that the rate controlling step for creep–fatigue failure can be either (a) the propagation of a short-oxidized cracks (fatigue component), (b) the growth and linkage of separated interior grain boundaries (creep component) or (c) a complex combination of both (a) and (b) [13].
Figure 5.13. SEM fractographs of the fracture surface after creep-fatigue test at 0.8% strain range and hold time of 1,800 seconds.

Figure 5.14. SEM fractographs of the fracture surface after creep-fatigue test at 1% strain range and hold time of 1,800 seconds [7].

5.3.11. Dislocation Structure and Precipitation Behavior

Bright-field STEM images of the as-received Alloy 709 and under creep-fatigue at 0.8% and 1% strain ranges at 600 seconds hold time are presented in Figure 5.15, Figure 5.16 and Figure
5.17, respectively at a zone axis [110] along with EDS maps of some of the observed features. As shown in Figure 5.15 in the as-received sample, the precipitates appear in a nodular shape that were uniformly distributed with no precipitation on the grain boundaries. It was confirmed that these precipitates are NbN or cementite (Fe3C) and that no M23C6 precipitates were noted [46]; Some of the precipitates shown were formed on dislocations (Figure 5.15).

At 0.8% strain range and 600 seconds hold time (Figure 5.16), precipitates with different shapes such as diamond, hexagonal, rectangular and some nodular are randomly distributed in the matrix as well as on subgrain boundaries. EDS analyses in Figure 5.16(c) indicate that these particles are S/P-rich and mostly Cr-rich (Cr23C6) which have detrimental effects on corrosion resistance, toughness and ductility [47, 48]. EDS maps of some of the diamond shaped precipitates (Figure 5.16(d)) indicate they are rich of aluminum oxide (AlO2) and some silicon nitride (Si3N4) in the center while nickel carbides (Ni3C) with some S/P carbides at the periphery. Furthermore, high dislocation density is observed at 0.8% strain range where tangled dislocations together with some free dislocation segments and dislocation pileups indicate that the recrystallization was not complete after the deformation and annealing processes [49].

At 1% strain range on the other hand (Figure 5.17), the observed precipitates are mainly nodular and rectangular-shaped distributed with a higher density of precipitates at the grain boundaries compared to those noted at 0.8% strain range. Lamella-shaped M23C6 precipitates are formed near incoherent twin boundaries (Figure 5.16(c) and Figure 5.17(c)) with the growing direction parallel to the coherent twin boundary [46]. EDS analyses indicate that these particles are mainly Cr-rich along with some Nb/P-carbides and silicon nitride (Si3N4). The frequency of the intergranular M23C6 and the lamella-shaped M23C6 becomes higher with increasing aging time in the alloy [46]. Figure 5.17(b) shows the process of precipitate fragmentation and diffusion to the interface. It is
suggested that Cr diffusion from the incoherent matrix to the interface contributed to formation of these precipitates. Also, the high interfacial energy of the incoherent interface between the intergranular M\textsubscript{23}C\textsubscript{6} precipitate and the matrix was the driving force for the growth of intergranular M\textsubscript{23}C\textsubscript{6}. Therefore, the loss of ductility near the intergranular precipitates weakens the grain boundaries and accelerates the micro-voids formation [46]. At 1% strain range, high density of dislocation networks is observed along with dislocation pileups at the grain boundaries. The intrinsic dislocations are known to provide the nucleation sites for the intragranular precipitates when the material undergoes aging [46, 50]. Also, the elongated shape of M\textsubscript{23}C\textsubscript{6} along the grain boundaries suggests that the growing speed alongside the grain boundaries is higher than that towards the austenite matrix [46]. The very small particles shown in Figure 5.16(a,b) at 0.8% strain range and Figure 5.17(a,b) at 1% strain range are mainly Cr-rich.

Under creep–fatigue interaction, subgrains are formed with well-organized boundaries consisting of arrays or networks of dislocations, unlike the case of continuous cycling where the material exhibits a cellular structure with dislocations tangled in a disorganized manner, because creep–fatigue deformation at high temperatures results in dislocation climb and recovery, thereby allowing cellular network boundaries or subgrains to form [13, 51-53]. Eventually, the microstructural evolution will influence the mechanical properties of the material; Alloy 709 will exhibit shorter elongation and higher ultimate tensile strength (UTS) due to precipitation hardening. Also, both the Yield strength and the UTS increase with increasing volume fraction of the M\textsubscript{23}C\textsubscript{6} precipitates [46] thereby contributing to creep-fatigue life degradation of the material.
Figure 5.15. Bright-field STEM image of as-received Alloy 709 at zone axis [110] showing dislocations and precipitates (a,b).
Figure 5.16. Bright-field STEM image of Alloy 709 subjected to creep-fatigue test at 0.8% strain range and 600 seconds hold time at zone axis [110] showing dislocations and precipitates (a,b) along with EDS maps (c,d).
Figure 5.17. Bright-field STEM image of Alloy 709 subjected to creep-fatigue test at 1% strain range and 600 seconds hold time at zone axis [110] showing dislocations and precipitates (a,c) along with EDS maps of annealing twins (c).
5.4. Conclusions

The preliminary data suggest that Fe-25Ni-20Cr austenitic stainless steel (Alloy 709) is an excellent candidate as structural material for SFR, in which high temperature creep-fatigue interaction is an important damage mode due to power transients. Therefore, the effect of strain range on creep-fatigue interaction of the Alloy 709 is investigated by conducting strain-controlled creep-fatigue tests with tensile hold times of 0, 600, 1,800 and 3,600 seconds at strain ranges varying from 0.6% to 1.2% at a temperature of 750 °C and 2x10⁻³ s⁻¹ (0.1 Hz) strain rate while strain-controlled fatigue tests were performed at strain ranges from 0.3% to 2.5% at same temperature and strain rate to understand fatigue characteristics of Alloy 709. The concluding remarks are:

- The predicted fatigue life of Alloy 709 shows a better correlation with the characteristic slopes predictive method rather than the universal slopes method.
- In general, the number of cycles to failure decreases with increasing strain range at a given hold time.
- The half-cycle peak stress amplitude, Δσ / 2, increases when strain range is increased at different hold times indicating a higher degree of cyclic hardening.
- The plastic strain range, Δε_p, and the number of cycles to failure at a given hold time follow Coffin-Manson relation.
- At all strain ranges, the cyclic stress response exhibits a region in which there is a plateau in stress extending to the failure point defined by 90% drop in peak tensile stress.
- For high-cycle fatigue (HCF) at low strain ranges, crack initiation occurs near the end of life, while for low-cycle fatigue (LCF) at high strain ranges, crack initiation occurs early and the fatigue life is dominated by crack propagation.
• In stress relaxation during hold time, serrations are formed due to increase in the stress corresponding to dislocations locking by the solute atoms and decrease in the stress due to the release of these dislocations from the obstacles via thermal activation.

• Creep-fatigue interaction diagram of Alloy 709 at different strain ranges and hold times is constructed according to linear damage summation (LDS). Although LDS is used because it is applicable over a wide variety of conditions with the availability of application data, it still has some limitations as discussed earlier.

• The fractographs of the deformed samples show increased number of cracks and decreased number of creep cavitation with increasing strain range.

• TEM images show that the microstructure is rich of $\text{M}_{23}\text{C}_6$ precipitates which have detrimental effects on corrosion resistance, toughness and ductility, and high dislocation density at different strain ranges. The intrinsic dislocations provide nucleation sites for the intragranular precipitates when the material undergoes aging.

• Under creep–fatigue interaction, subgrains are formed with well-organized boundaries consisting of arrays or networks of dislocations while in continuous cycling, the material exhibits a cellular structure with dislocations tangled in a disorganized manner.
5.5. References


6. EFFECT OF TEMPERATURE ON CREEP-FATIGUE BEHAVIOR OF Fe-25Ni-20Cr
(WT.%) AUSTENITIC STAINLESS STEEL (ALLOY 709)³

Abstract

Creep-fatigue is a major consideration in the design of high-temperature systems subjected to thermal transients under strain-controlled conditions during startups and shutdowns, in which on-load periods at elevated temperatures, creep becomes important. This creep-fatigue interaction is expected to be a contributor to the damage mode for many reactor components operating at high temperatures in next-generation nuclear reactors such as Sodium-Cooled Fast Reactors (SFRs). Preliminary data suggest that Fe-25Ni-20Cr (wt%), an advanced austenitic stainless steel (known as Alloy 709) is an excellent candidate as structural material for SFR due to its good mechanical properties. Therefore, the effect of temperature on creep-fatigue interaction of the Alloy 709 is investigated by conducting strain-controlled creep-fatigue tests with tensile hold times of 0, 60, 600, 1,800 and 3,600 seconds at 1% strain range and 2x10⁻³ s⁻¹ (0.1 Hz) strain rate at 650 °C and 750 °C. It is found that the number of cycles to failure at 650 °C is higher than that at 750 °C and the material exhibits fluctuations in the number of cycles to crack initiation and failure at different hold times due to the effect of Dynamic Strain Aging (DSA). Also, work hardening is found to increase with decreasing temperature. Creep-fatigue interaction diagram of Alloy 709 is constructed according to linear damage summation (LDS) at different strain ranges, temperatures and hold times. The fractographs of the deformed samples exhibited increased density of cracks and creep cavities at higher temperature along with Cr₂₃C₆ precipitates and high dislocation density.

³ This chapter is to be published.
6.1. Introduction

Generation IV (GEN IV) nuclear reactors are an important source of base load power in the middle–long term (2030–2050) and there are many designs of these nuclear power plants but only some of them will be deployed [1]. GEN IV nuclear reactors are designed to be safer, more reliable, relatively more efficient and have longer lifetimes (60-80 years) than current nuclear reactors that are reaching their life expectancy. The only possible mid-term available fast reactor is Sodium-Cooled Fast Reactor (SFR) with an acceleration in research and development and reduction in economic competitiveness [1]. SFR operates at temperatures as high as 550 °C and uses sodium as coolant and moderator due to its low specific heat, high melting point, high thermodynamic efficiency and heavy atoms compared to water [2, 3]. As reported, SFR is the most researched type of fast reactors and its fast spectrum is able to convert fertile material to fissile, thus increasing the efficiency of usage of the nuclear fuel [1]. The structural material of SFR should have excellent mechanical properties that can withstand harsh environments such as high temperatures (550-750 °C), high corrosive environments and high radiation doses.

Austenitic stainless steels are used in many engineering applications including conventional and nuclear power plants because they are corrosion-resistant, have high-temperature strength, and economic feasibility of the alloys due to their high nickel and chromium contents. They are primarily composed of a Fe-Cr-Ni alloys; chromium content is generally over 12 wt% to form a passive chromium oxide film providing absence of staining, rusting and corroding in various aqueous or corrosive chemical environments, and nickel content to improve the corrosion resistance as well as the high-temperature strength of the material. During the operation of SFRs, structural materials experience cyclic thermal stresses during start-ups and shut-downs while time-dependent effects like creep occur due to stress relaxation during on-load periods. The combination
of these lead to creep-fatigue damage including intergranular creep cavitation damage (creep) as well as transgranular crack initiation/propagation (fatigue) thereby decreasing the crack initiation endurance. Preliminary data on Fe-25Ni-20Cr austenitic stainless steel (known as Alloy 709) suggests that it is an excellent candidate as structural material for SFR with good corrosion-resistance, high strength, sodium compatibility, thermal stability and good creep properties [4]. While the service temperature for structural components of SFRs is 550 °C, other components get exposed to higher service temperatures leading to shorter lifetimes. It is thus important to investigate and understand the creep-fatigue interaction of advanced austenitic stainless steels such as Alloy 709 at higher temperatures (~650 °C) in order to generate enough data in a reasonable time and then extrapolate to the service conditions expected in SFRs. Creep-fatigue tests are performed following ASTM standard E2714-13 that provides a clear indication of the influence of cyclic loading on creep deformation characteristics and the influence of creep deformation on cyclic loading response [5].

In this chapter, the effect of temperature on creep-fatigue interaction of Alloy 709 is investigated by preforming strain-controlled creep-fatigue tests with tensile hold times of 0, 60, 600, 1,800 and 3,600 seconds at strain range of 1% at 650 °C and 750 °C [6] in air following ASTM standard E2714-13 [5]. During ramp up and ramp down to the peak strain amplitude in tension and compression, the strain rate was maintained at $2 \times 10^{-3} \text{ s}^{-1}$ (0.1 Hz).

6.2. Experimental Setup

6.2.1. Material

The experimental material is a Fe-25Ni-20Cr advanced austenitic stainless steel (Alloy 709) received in the form of a rectangular plate that had undergone hot-rolling followed by solution-annealing heat-treatment at 1100 °C for 75 minutes then followed by subsequent water quenching.
The chemical composition of the alloy is given in Table 6.1. Creep-fatigue tests were performed using test specimens machined from the as-received plate with 3 mm diameter gage section and 12 mm gage length along rolling direction as shown in Figure 6.1(a).

Table 6.1. Chemical composition (wt%) of Alloy 709.

<table>
<thead>
<tr>
<th>Element</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>N</th>
<th>Ti</th>
<th>Nb</th>
<th>B</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wt%</td>
<td>0.07</td>
<td>0.91</td>
<td>0.44</td>
<td>&lt;0.014</td>
<td>&lt;0.000</td>
<td>19.93</td>
<td>24.98</td>
<td>1.51</td>
<td>0.148</td>
<td>0.04</td>
<td>0.26</td>
<td>0.0045</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

6.2.2. Method

Creep-fatigue tests were performed in an electro-dynamic creep-fatigue machine equipped with a resistive heating furnace, in which the temperature is measured and controlled via K-type thermocouples connected to a readout unit and maintained within ±2 °C as shown in Figure 6.1(b). The elongation was measured and controlled using Linear Variable Differential Transducer (LVDT) attached to a high-temperature extensometer fixture connected to the grip sections of the sample. Microstructural characterization was performed using Optical Microscopy (HIROX microscope), scanning electron microscopy (SEM) (FEI Quanta 3D FEG and SEM Verios) and transmission electron microscopy (TEM) (JEOL 2000FX and FEI Talos F200X) at Idaho National Laboratory (INL). Sample preparation for each microstructure examination was performed according to the preparation procedures mentioned in chapter 3, section 3.3.

Strain-controlled creep-fatigue tests were conducted at 1% strain range with tensile hold times of 0, 60, 600, 1,800 and 3,600 seconds at 650 °C and 750 °C [6] and at a strain rate of 2×10⁻³ s⁻¹ (0.1 Hz).
6.3. Results and Discussion

6.3.1. Effect of DSA on Creep-Fatigue Behavior

The number of cycles to failure vs. hold time at 1% strain range and temperatures of 650 °C and 750 °C is shown in Figure 6.2. In general, at each hold time investigated at 1% strain range, the number of cycles to failure at 650 °C is higher than at 750 °C meaning that the creep-fatigue life decreases at higher temperatures. Furthermore, the creep-fatigue life of Alloy 709 is found to decrease with increasing hold time at 750 °C [6] while at 650 °C, the material exhibits fluctuations in which the number of cycles to failure is higher at 60 seconds hold time than at 0 seconds, and it is lower at 600 seconds hold time than at longer hold times. These fluctuations are attributed to the effect of Dynamic Strain Aging (DSA) during the hold time period.
DSA occurs due to the interaction between moving dislocations and solute atoms during deformation, in which stress-strain curves during tensile deformation exhibit serrations known as *Portevin-Le Chatelier Effect (PLE)* of type $A+C$ at 650 °C and a strain rate of $2 \times 10^{-3}$ s$^{-1}$ thereby affect the mechanical properties of the material such as fatigue life [7-9]. During hold time in the Alloy 709, it is suggested that piled-up dislocations at the carbides may not be moving and carbon diffuses to the dislocations to form Cottrell atmospheres. The formation of the Cottrell atmosphere around dislocations near the carbides leading to an increased number of pile-ups in which fatigue cracks may initiate. Therefore, when applying hold time in strain-controlled fatigue tests, the dislocation density in each pile-up will decrease and thus improve the fatigue life of the material [10]. This kind of behavior has also been observed in other steels such as AISI 4140 at strain ranges from 1% to 3.2% at room temperature and up to 200 °C with 30 to 60 seconds hold time [10]. Furthermore, DSA has been observed to influence the strain-controlled fatigue and creep-fatigue in nickel-base solid solution alloys such as Alloy 617 and Alloy 230 at 800 °C [11]. However, the influence of DSA on creep-fatigue properties is still not well known and requires more investigations [11-13].
Figure 6.2. Number of cycles to failure vs. hold time at 1% strain range and temperatures of 650 °C and 750 °C [6].

6.3.2. Effect of Temperature on Time to Fracture, $t_f$

With increasing tensile hold time in the Alloy 709 under creep-fatigue tests at 650 °C, the time for the material fracture increases (Figure 6.3). Also, at each hold time, the time to failure is higher at lower temperatures (~650 °C) except at 600 seconds hold time due to the effect of DSA as mentioned previously.

Furthermore, time variations of the maximum tensile stresses at investigated hold times (Figure 6.3(a)) indicate small initial increases followed by distinct decreases at longer hold times. This behavior has been also observed in Alloy 709 at higher temperatures (~750 °C) as mentioned in Alsmadi, et al. [6].
Figure 6.3. Maximum stress with respect to time at 650 °C (a) and effect of hold time, \( t_h \), on the time to failure, \( t_f \), at 650 °C and 750 °C (b) (all at 1% strain range and different hold times).

### 6.3.3. Stress-Strain Behavior

The second-cycle stress-strain hysteresis loops at 1% strain range at 650 °C are plotted at different hold times in Figure 6.4, while half-cycle stress-strain hysteresis loops at same strain range and temperature are plotted in Figure 6.5. As shown, the width of the second-cycle and half-cycle hysteresis loops increases with hold time since introducing tensile hold time during loading cycle results in stress relaxation and therefore, increases the inelastic strain which suggests the occurrence of creep damage [14-17]. Furthermore, at all hold times, the width of the half-cycle hysteresis loops is less than the width of the second-cycle loops which indicates the degradation of creep-fatigue life of the material.
Figure 6.4. Second-cycle hysteresis loops for the creep-fatigue tests at 1% strain range and 650 °C at hold times of 0, 1,800 and 3,600 seconds (a), 60 seconds (b) and 600 seconds (c).
Figure 6.5. Half-cycle hysteresis loops for the creep-fatigue tests at 1% strain range and 650 °C at hold times of 0, 1,800 and 3,600 seconds (a), 60 seconds (b) and 600 seconds (c).

### 6.3.4. Crack Initiation Properties

The point of macro-crack initiation and failure is determined following the approach applied by Alsmadi, et al. [6] and Totemeier, et. al., [18], where macro-crack initiation is defined as the point at which the stress ratio deviates from linearity while failure is defined by a 20% drop in stress ratio from the point of deviation in the plot of the absolute value of the ratio of maximum tensile stress to compressive stress as a function of cycle number. Figure 6.6 shows the effect of
hold time on the number of cycles to crack initiation and failure, in which they fluctuate with increasing hold time. Also, the difference between number of cycles to crack initiation and failure does not follow a specific trend due to the effect of DSA on the material during hold time as mentioned previously.

Figure 6.6. The effect of hold time on number of cycles to crack initiation and failure under creep-fatigue tests of Alloy 709 at 1% strain range and 650 °C.

6.3.5. Effect of Temperature on Degree of Work Hardening

During cyclic deformation, the material undergoes cyclic hardening or softening until the saturation stage is reached [6]. The degree of work hardening can be determined using the stress amplitude, $\Delta \sigma / 2$, measured at half-life cycle and first cycle as given below [19, 20]:

\[
\text{Degree of Hardening} = \frac{(\Delta \sigma / 2)_{\text{Half-Cycle}} - (\Delta \sigma / 2)_{\text{First-Cycle}}}{(\Delta \sigma / 2)_{\text{First-Cycle}}} \quad (6.1)
\]
The effect of temperature on degree of work hardening of the Alloy 709 at 1% strain range and different hold times is depicted in Figure 6.7, in which work hardening increases with decreasing temperature (~650 °C) due to the generation and interactions of dislocations whereas with increasing temperature to 750 °C, dislocations start to annihilate and their decreased density results in a decreased degree of work hardening.

![Figure 6.7](image)

Figure 6.7. The effect of temperature on degree of work hardening under creep-fatigue tests of the Alloy 709 at 1% strain range and different hold times.

6.3.6. Creep-Fatigue Interaction Diagram of the Alloy 709

Creep-fatigue interaction diagram is constructed according to linear damage summation (LDS) as applied by ASME [21], where creep and fatigue damage fractions are determined independently as following [6]:

\[
\Delta \varepsilon = 1.0\
\dot{\varepsilon} = 2 \times 10^{-3} \text{ s}^{-1}
\]
\[
\sum_j \left[ \frac{n_j}{N_{d_j}} \right] + \sum_k \left[ \frac{\Delta t_k}{T_{d_k}} \right] \leq D
\] (6.2)

where \( n \) and \( N_d \) are the number of cycles of type \( j \) and the allowable number of cycles of type \( j \), respectively, and \( \Delta t \) and \( T_d \) are the actual time at stress level \( k \) and the allowable time at the same stress level, respectively, and \( D \) is the allowable combined damage fraction which is usually assumed to be equal to one [6, 14, 15, 21]. Figure 6.8 shows the creep-fatigue interaction diagram for Alloy 709 at different strain ranges, temperatures and hold times along with the ideal failure criterion according to LDS. As shown, almost all data points lie below or above the ideal curve with some damage values lying very close to or on the \( D=1 \) curve such as 0.8% strain range at 1,800 seconds hold time and 750 °C [6] and 1% strain range at same hold time and 650 °C. On the other hand, at 1,800 seconds hold time and 1% strain range, creep damage fraction is higher at 650 °C than at 750 °C while it is the opposite for fatigue damage fraction. Furthermore, at 650 °C, data points lie closer to the \( D=1 \) curve with increasing hold time.

As mentioned previously, more data points are needed to help establish the creep-fatigue bilinear damage envelope of Alloy 709 at different strain ranges, temperatures and hold times [6].
Figure 6.8. Creep-fatigue interaction diagram of Alloy 709 at different strain ranges, temperatures and hold times; the green dashed line shows the ideal failure criterion according to linear damage summation ($D=1$).

6.3.7. Microstructural Damage

Figure 6.9 shows the free surfaces of Alloy 709 taken at the transverse cross section perpendicular to the stress axis under creep-fatigue tests at 1% strain range and 650 °C at different hold times characterized using optical microscopy. As shown, with increasing hold time, dominant mode of transgranular cracks (Figure 6.9(a)) on the surface changes to a mixed mode of cracks where both transgranular fatigue cracking and intergranular creep cavitation are noted near the edge of the free surfaces of the Alloy (Figure 6.9(b,c)) [22]. Furthermore, at lower temperatures (~650 °C) and long hold times at 1% strain range (Figure 6.9), cracks appear in a branched and almost closed shapes propagating in the specimen where oxygen is shown to penetrate along these
cracks as revealed by a thin oxide layer at the crack tip whereas at higher temperature (~750 °C) and same strain range (Figure 4.11) [6], cracks appear in a widely opened shapes and are almost straight filled with oxide indicating faster propagation since oxidation is a time-dependent process in which the penetration depth of oxidation will be higher with increasing hold time [23].

Figure 6.10 shows the SEM fractographs of the fracture surface of Alloy 709 after creep-fatigue tests at 1\% strain range and 650 °C at different hold times. As shown, with increasing hold time, the number of cracks increases to a mixed mode of intergranular cracks (indicated by yellow arrows) and interior grain boundary cracking (indicated by blue arrows) along with creep cavities developed on the grain boundary (indicated by green circles). Also, striations start to disappear with increasing hold time indicating that the dominant damage mode is comprised of both creep and fatigue damage unlike the case when no hold time is applied where the density of striations is higher due to dominant fatigue damage. At 600 seconds hold time (Figure 6.10(c)), the fracture surface is featured with rough dimples due to the hard inclusions in the material that lead to the formation and interlinkage of voids. Furthermore, the density of cracks and creep cavities is higher at 750 °C (Figure 4.14 and Figure 4.15) [6] than at 650 °C (Figure 6.10) at all hold times. Crack nucleation and propagation of Alloy 709 are discussed in detail in Alsmadi, et al. [6].
Figure 6.9. Fractographs of the free surface of Alloy 709 after creep-fatigue tests at 1\% strain range and 650 °C at hold times of 0 seconds (a), 1,800 seconds (b) and 3,600 seconds (c) (Stress axis perpendicular to the page).
Figure 6.10. Fractographs of the fracture surface of the Alloy 709 after creep-fatigue tests at 1% strain range and 650 °C at hold times of 0 seconds (a), 60 seconds (b), 600 seconds (c), 1,800 seconds (d) and 3,600 seconds (e).
To study the dislocation structure and precipitation behavior of Alloy 709 under creep-fatigue tests at different temperatures, Figure 6.11 shows bright-field STEM image of the Alloy 709 under creep-fatigue tests at 1% strain range and 1,800 seconds hold time at 650 °C, whereas Figure 6.12 shows bright-field STEM image of the Alloy 709 under creep-fatigue tests at same strain range and hold time at 750 °C at a zone axis [110] along with EDS maps. As shown at 650 °C (Figure 6.11), precipitates with different shapes such as spherical, rectangular and some nodular are randomly distributed in the matrix as well as on subgrain boundaries, while at 750 °C (Figure 6.12), precipitates with mostly nodular shape are highly and also randomly distributed in the matrix and on subgrain boundaries. EDS analyses at 750 °C (Figure 6.12(b)) indicate that these precipitates are Nb/P-rich with S/Mn-rich and carbon dioxide (CO₂) but mostly Cr-rich (Cr₂₃C₆) which have detrimental effects on corrosion resistance, toughness and ductility [11, 24]. The frequency of the intergranular Cr₂₃C₆ precipitates becomes higher with increasing aging time in the material [25]. Figure 6.11(b) and Figure 6.12(b) show the process of precipitate fragmentation and diffusion to the interface, in which the Cr diffusion from the incoherent matrix to the interface contributed to the formation of these precipitates and the high interfacial energy of the incoherent interface between the intergranular Cr₂₃C₆ precipitates and the matrix was the driving force for the growth of intergranular Cr₂₃C₆. Therefore, the loss of ductility near the intergranular precipitates weakens the grain boundaries and accelerates the micro-voids formation [25]. As shown in Figures 6.11(b) and 6.12(b), the elongated shape of Cr₂₃C₆ precipitates on the grain boundaries suggests that the growing speed along the grain boundaries is higher than that towards the austenite matrix [25]. The very small particles shown in Figure 6.11(a) and Figure 6.12(a) are mainly Cr-rich. Furthermore, higher density of dislocation networks of tangled dislocations with free dislocation segments is observed at 650 °C (Figure 6.11(a)), whereas less dislocation density is observed at
750 °C. As mentioned previously, the intrinsic dislocations provide nucleation sites for the intragranular precipitates when the material undergoes aging [25, 26].

Overall, the microstructural damage will influence the mechanical properties thereby contributing to the degradation of creep-fatigue life of the material at different strain ranges, temperatures and hold times.

Figure 6.11. Bright-field STEM image of the Alloy 709 under creep-fatigue tests at 1% strain range and 1,800 seconds hold time at 650 °C at zone axis [110] showing dislocations (a) and precipitates (b).
Figure 6.1. Bright-field STEM image of the Alloy 709 under creep-fatigue tests at 1% strain range and 1,800 seconds hold time at 750 °C at zone axis [110] showing dislocations and precipitates (a) along with EDS maps (b).
6.4. Conclusions

Creep-fatigue interaction is expected to occur in many reactor components in Sodium-Cooled Fast Reactors (SFRs), in which their structural material should have excellent mechanical properties to withstand the harsh environments in the reactor. Fe-25Ni-20Cr (wt%), an advanced austenitic stainless steel (known as Alloy 709) is a potential structural material for SFR due to its good mechanical properties. The effect of temperature on creep-fatigue interaction of the Alloy 709 is investigated by conducting strain-controlled creep-fatigue tests with tensile hold times of 0, 60, 600, 1,800 and 3,600 seconds at strain range of 1% at temperatures of 650 °C and 750 °C [6] and 2x10^{-3} \text{s}^{-1} (0.1 \text{ Hz}) strain rate. The concluding remarks are:

- Creep-fatigue life of Alloy 709 is found to decrease with increasing hold time at 750 °C while at 650 °C, the material exhibits fluctuations due to the effect of Dynamic Strain Aging (DSA) during the hold time period.
- In general, with increasing tensile hold time at 650 °C, the fracture time increases.
- At all hold times at 1% strain range and 650 °C, the width of the half-cycle hysteresis loops is less than that of the second-cycle loops indicating degradation of creep-fatigue life of the material.
- Work hardening is found to increase at 650 °C due to the generation and interaction of dislocations whereas at 750 °C, dislocations start to annihilate thereby resulting in decreased degree of work hardening.
- Creep-fatigue interaction diagram of the Alloy 709 is presented at different strain ranges, temperatures and hold times according to linear damage summation (LDS).
• Free surfaces of the deformed samples of Alloy 709 at 650 °C exhibited a mixed mode of cracks with increasing hold time and a branched and almost closed shapes propagating in the specimen.

• The fractographs of the deformed samples show an increased density of cracks and creep cavities with increasing hold time at 650 °C, while this density is higher at 750 °C.

• STEM images of Alloy 709 subjected to creep-fatigue tests at 650 °C and 1% strain range show high randomly-distributed Cr$_{23}$C$_6$ precipitates along with higher dislocation density than at 750 °C.
6.5. References


7. CONCLUSIONS AND SUGGESTED FUTURE WORK

7.1. Conclusions

To understand high temperature creep-fatigue interaction of the Alloy 709; a candidate structural material for next-generation Sodium-Cooled Fast Reactors (SFRs), strain-controlled low-cycle fatigue (LCF) and creep-fatigue tests were performed at different hold times, strain ranges in air at $2 \times 10^{-3}$ s$^{-1}$ strain rate (0.1 Hz) at 650 °C and 750 °C following ASTM standard E2714–13.

Strain-controlled LCF tests were performed at strain ranges from 0.3% to 2.5% with fully reversible cycle of triangular waveform at 750 °C in air and $2 \times 10^{-3}$ s$^{-1}$ strain rate (0.1 Hz). It is found that the number of cycles to failure decreases with increasing strain range and the predicted fatigue life of Alloy 709 shows a better correlation with the characteristic slopes predictive method rather than the universal slopes method.

To investigate the effect of hold time on high-temperature creep-fatigue behavior of Alloy 709, strain-controlled fatigue tests were performed with hold times of 60, 600, 1,800 and 3,600 seconds introduced at the maximum tensile strain of 1% strain range at 750 °C and $2 \times 10^{-3}$ s$^{-1}$ strain rate (0.1 Hz). The following conclusions are made:

- During creep-fatigue tests, the width of the hysteresis loops is found to increase with hold time suggesting the rise of inelastic strain with hold time indicating the occurrence of creep damage.
- The creep-fatigue life of the alloy decreases with increasing hold time until it saturates at longer hold times.
- With increasing hold time, the number of cycles to macro-crack initiation and failure decrease; indicating faster crack initiation and higher growth rates.
• The optical microscopy revealed highly branched and almost closed crack growth in the specimen during low hold time (600 seconds) while almost straight and widely opened cracks filled with oxide were observed during longer hold time (3,600 seconds).

• The fractographs of the fracture surface of Alloy 709 after low-cycle fatigue (LCF) test at 1% strain range using the SEM, showed crack nucleation sites, striations due to crack propagation and dimple features representing the region of overload fracture. On the other hand, the fracture surfaces of Alloy 709 after creep-fatigue tests showed much lower density of striations with increasing hold time indicating that the dominant damage mode is related to both creep and fatigue damages. In addition, the density of creep cavities and cracks is shown to increase with hold time, where typical intergranular and transgranular cracks are observed.

• Creep-fatigue life is evaluated by a linear summation of fractions of cyclic and creep damages according to ASME code. However, more studies are needed to confirm the creep-fatigue damage envelope for this alloy at different strain ranges and hold times.

The effect of strain range on high-temperature creep-fatigue interaction of Alloy 709 is investigated by conducting strain-controlled creep-fatigue tests with tensile hold times of 0, 600, 1,800 and 3,600 seconds at strain ranges varying from 0.6% to 1.2% at 750 °C and 2x10⁻³ s⁻¹ (0.1 Hz) strain rate. The concluding remarks are:

• In general, the number of cycles to failure decreases with increasing strain range at a given hold time.

• The half-cycle peak stress amplitude, \( \Delta \sigma / 2 \), increases when strain range is increased at different hold times indicating a higher degree of cyclic hardening.
• The plastic strain range, $\Delta \varepsilon_p$, and the number of cycles to failure at a given hold time follow Coffin-Manson relation.

• At all strain ranges, the cyclic stress response exhibits a region in which there is a plateau in stress extending to the failure point defined by 90% drop in peak tensile stress.

• For high-cycle fatigue (HCF) at low strain ranges, crack initiation occurs near the end of life, while for low-cycle fatigue (LCF) at high strain ranges, crack initiation occurs early and the fatigue life is dominated by crack propagation.

• In stress relaxation during hold time, serrations are formed due to increase in the stress corresponding to dislocations locking by the solute atoms and decrease in the stress due to the release of these dislocations from the obstacles via thermal activation.

• Creep-fatigue interaction diagram of Alloy 709 at different strain ranges and hold times is constructed according to linear damage summation (LDS). Although LDS is used because it is applicable over a wide variety of conditions with the availability of application data, it still has some limitations as discussed earlier.

• The fractographs of the deformed samples show increased number of cracks and decreased number of creep cavitation with increasing strain range.

• TEM images show that the microstructure is rich of $M_{23}C_6$ precipitates which have detrimental effects on corrosion resistance, toughness and ductility, and high dislocation density at different strain ranges. The intrinsic dislocations provide nucleation sites for the intragranular precipitates when the material undergoes aging.

• Under creep–fatigue interaction, subgrains are formed with well-organized boundaries consisting of arrays or networks of dislocations while in continuous cycling, the material exhibits cellular structure with dislocations tangled in a disorganized manner.
Finally, the effect of temperature on creep-fatigue behavior of Alloy 709 is investigated by conducting strain-controlled creep-fatigue tests with tensile hold times of 0, 60, 600, 1,800 and 3,600 seconds at 1% strain range at 650 °C and $2 \times 10^{-3}$ s$^{-1}$ (0.1 Hz) strain rate. The following conclusions are made:

- Creep-fatigue life of Alloy 709 is found to decrease with increasing hold time at 750 °C while at 650 °C, the material exhibits fluctuations due to Dynamic Strain Aging (DSA) during the hold time period.
- In general, with increasing tensile hold time at 650 °C, the fracture time increases.
- At all hold times at 1% strain range and 650 °C, the width of the half-cycle hysteresis loops is less than that of the second-cycle loops which indicates the degradation of creep-fatigue life of the material.
- Work hardening is found to increase at 650 °C due to the generation and interactions of dislocations whereas at 750 °C, dislocations start to annihilate resulting in decreased degree of work hardening.
- Creep-fatigue interaction diagram of the Alloy 709 is presented at different strain ranges, temperatures and hold times according to linear damage summation (LDS).
- With increasing hold time, free surfaces of the deformed samples of Alloy 709 at 650 °C exhibited a mixed mode of cracks, and branched closed shaped propagating in the specimen.
- The fractographs of the deformed samples exhibited increased density of cracks and creep cavities with increasing hold time at 650 °C, while this density is higher at 750 °C.
• STEM images of Alloy 709 subjected to creep-fatigue tests at 650 °C and 1% strain range show randomly distributed Cr$_{23}$C$_6$ precipitates along with higher dislocation density than that at 750 °C.

7.2. Suggested Future Work

Creep-fatigue interaction is a type of plastic deformation that includes a lot of factors which can be investigated and studied to understand and predict the behavior of structural materials in SFRs. Some of these factors have been already investigated in this work under strain-controlled mode such as tensile hold time, strain range and temperature while there are other factors that can be addressed in the future to examine their variations on creep-fatigue life of the Alloy 709. These factors can be:

• *Neutron Irradiation*

One of the most important factors to study is the effect of neutron irradiation on creep-fatigue behavior of the Alloy 709 under typical operating conditions expected in SFRs. It is important in the future to prepare creep-fatigue samples of Alloy 709 for performing neutron irradiation at high temperatures. Following irradiation, strain-controlled creep-fatigue tests should be performed at same temperature at desired strain range, tensile hold times and strain rate. Furthermore, microstructural evaluation of the fractured irradiated samples should be performed using SEM, TEM, x-Ray diffraction (XRD) and atom probe tomography (APT) to investigate the damage mechanisms responsible for deformation and failure at different loading conditions.
• **Environmental Effect**

The environmental effect on creep-fatigue damage of Alloy 709 can be studied by conducting creep-fatigue tests in different environments like air, vacuum or corrosive environments expected in SFRs.

• **Stress-Controlled Creep-Fatigue Testing**

In stress-controlled creep-fatigue tests, the stress is held constant during hold periods while strain range varies, and all the factors investigated under strain-controlled mode can be investigated also under stress-controlled mode. It is suggested that the main advantage of stress-mode tests compared to strain-mode tests lays in the fact that they enable a higher viscoplastic strain to be applied during hold time period for the same test duration. Also, for a similar amount of viscoplastic strain applied during hold time, a stress-mode test can be more deleterious than a strain-mode test and for the same hold time period, stress-mode tests exhibit higher creep-fatigue life reduction than in strain-mode [1] due mainly to the absence of stress relaxation during hold times. Furthermore, in stress-controlled creep-fatigue tests, creep damage accumulated during hold time can be calculated using strain-fraction method, i.e., *Ductility Exhaustion* and then incorporated in design codes and assessment procedures at high temperatures. This can be represented graphically by the creep-fatigue interaction diagram.

• **Interrupted Creep-Fatigue Testing**

An analysis of the evolving failure modes during creep-fatigue tests can be investigated by interrupting the test during cycling for ex-situ microstructural investigation. In fatigue, test interruptions usually occur within the plateau or saturated stress region of the cyclic deformation
curve while in creep–fatigue tests, the interruptions are usually in the middle of cyclic softening after the maximum peak stress is reached [2]. Therefore, it is important to study the microstructural evolution of Alloy 709 during creep-fatigue tests in order to understand and predict the final failure mechanism of the alloy.

- **Sequential Creep-Fatigue Testing**

  An alternative to simultaneous creep-fatigue testing method is when the material is subjected to a combined test cycle; where the first part is load-controlled creep test and the second part is strain-controlled fatigue test, or vice versa. One of the purposes of performing sequential creep-fatigue tests is the assessment of the effect of prior creep deformation on fatigue properties and the effect of prior fatigue on creep properties [3]. Creep and fatigue damage fractions can be calculated the same way in simultaneous creep-fatigue tests which was presented in this work. However, they require complicated material-property database although they give more realistic values [3].

- **Lifetime Prediction methods**

  There are more than 100 creep-fatigue lifetime prediction methods that have been proposed to assess and validate the prediction capabilities and explain the failure mechanisms under creep-fatigue loading conditions [4]. A common method is linear damage summation (LDS) method which was employed in this work on Alloy 709. Other methods like frequency separation method, strain range partitioning method, damage function method based on hysteresis energy, damage rate model, cavitation model and extended strain-life equation can be investigated in the future to calculate creep and fatigue damage fractions of Alloy 709 subjected to creep-fatigue tests [5].
• **Effect of DSA on Creep-Fatigue Life**

As discussed in Chapter 6, Dynamic Strain Aging (DSA) affects creep-fatigue life in which it occurs during the hold time period due to the interaction between moving dislocations and solute atoms during deformation, and thus can improve the fatigue life of the material under strain-controlled testing mode [6]. This kind of behavior has been studied in other steels such as AISI 4140 [6] and also nickel-base solid solution alloys such as Alloy 617 and Alloy 230 [7]. However, the influence of DSA on creep-fatigue properties of Alloy 709 is still not well known and requires more investigations in the future under different loading conditions and testing modes.

• **Effect of Mean Stress**

During creep-fatigue tests, mean stress usually equals zero. However, changing the mean stresses can affect number of cycles to failure where fatigue life decreases with increasing mean stress.

• **Effect of Oxidation**

The effect of oxidation on creep-fatigue endurance is an important case of study since oxidation is a time-dependent process and accumulates mostly during hold time which in turn, allows to study the effect of creep damage accumulated during hold periods.

• **Test Specimen Geometry**

Creep-fatigue specimen geometry varies between hour-glass specimens, notched specimens, parallel-gage specimens, ridged specimens and tubular specimens [8-10]. Each of these different
geometries is selected for certain experimental conditions to minimize the difficulties associated with buckling, thermal gradients and experimental setup.

- **Unequal Strain Rate (Tension and/or Compression)**

  Unequal strain rate has a great effect on the final failure mechanism and thus creep-fatigue endurance as discussed in Chapter 2.

- **Grain Size and Creep Ductility**

  The effect of grain size is more significant under creep-fatigue test conditions that result in intergranular failure, while creep ductility strongly affects the extent of creep-fatigue interaction as discussed in Chapter 2.
7.3. References


ACKNOWLEDGMENTS

The author would like to acknowledge the financial support from the Nuclear Energy University Programs (NEUP/ Project #15-8582) of the Department of Energy, Office of Nuclear Energy for performing this research, and Dr. Sam Sham of Argonne National Laboratory for supplying the experimental material, and Dr. Nilesh Kumar, Dr. Abdullah Alomari and Dr. Boopathy Kombaiah for support and discussions. The SEM work was performed in part at the Analytical Instrumentation Facility (AIF) at North Carolina State University, which is supported by the State of North Carolina and the National Science Foundation (award # ECCS-1542015). The AIF is a member of the North Carolina Research Triangle Nanotechnology Network (RTNN), a site in the National Nanotechnology Coordinated Infrastructure (NNCI). The TEM work was partially supported by the U.S. Department of Energy, Office of Nuclear Energy under DOE Idaho Operations Office Contract DE-AC07-051D14517 as part of a Nuclear Science User Facilities project. The author gratefully acknowledges the partial financial support by the U.S. National Science Foundation (grant # CMMI 1727237), and the use of optical microscopy at the Industrial and Systems Engineering at North Carolina State University, Raleigh, USA.
Appendix A

List of Publications and Presentations

**Peer Reviewed Journal Articles and Conference Proceedings:**


Posters and Presentations:


Appendix B

Transactions, SMiRT-25
Charlotte, NC, USA, August 4-9, 2019
Division IX

SIMULATION AND EXPERIEMENTS OF MULTI-LAYER DRY CASK SHIELDING MATERIALS

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ABSTRACT

The demand for radiation shielding studies has grown with the increasing utilization of radioactive materials. A variety of locations such as nuclear reactors, medical centers, forensic laboratories, nuclear research centers, and nuclear waste storage facilities are potential exposure sites of not only gamma rays but also neutron and/or charged particles. This research investigates the concept of a dry cask with multi-layer shielding and evaluation for high burnup spent fuel storage by using MacroShield as a deterministic code. The materials selected as a central theme for dosimetric assessment, are Stainless Steel 304 and 316; and carbon steel. Previously investigated work on glass oxides and concrete forms suggested the utilization of specific forms of glasses and concretes for the multi-layered cask. Conducted gamma ray attenuation experiments compares favorably to MicroShield modeling. Coated samples have shown minimal to nil corrosion rates.

INTRODUCTION

Spent nuclear fuel is stored primarily within dry casks, which involves a stainless steel canister surrounded by concrete overpack. A schematic of a traditional dry cask is shown in Figure 1. The termination of the Yucca Mountain nuclear waste repository has changed the long-term storage plan of spent nuclear fuel. Dry casks storage will continue to be stored at nuclear utilities sites located outside of nuclear power plants. As this storage method involves the dry casks being permanently located in their storage units for up to 300 years, the integrity of the dry cask storage materials must maintain their integrity over this lifetime.

![Figure 1. Schematic of the dry cask storage method in both horizontal and vertical orientations retrieved from NRC illustration.](image-url)
MATERIALS & METHODS

Dry casks are typically made of both 304 and 316 stainless steels. In order to improve the lifetime of the dry cask storage method, a multilayer coating will be added to the surface of the stainless steel canisters as shown in Figure 2. Stainless steel 1" diameter puck substrates are coated with single layers using Physical Vapor Deposition (PVD) in order to study the individual layer performance prior to multilayer analysis.

The multilayer coating involves an external layer of MoS₂ which serves as a solid lubricant as shown by Singh et al. (2015) to improve the handling and transportation of dry casks in order to prevent the formation of micro cracks. The middle layer is formed using as a tri-oxide layer known as zirconolite, which serves to improve the corrosion resistance of the steel substrate as corrosion is one of the primary mechanisms of material failure as seen by TiO₂ thin films by Shan et al. (2008). The inner layer of TiN acts as a diffusional barrier, preventing interaction between the multilayer coatings and the underlying stainless steel as observed by Kwon et al. (2006). The longevity of the dry cask storage method requires the integrity of the steel waste package wall and the concrete overpack to be maintained for 300 years. The gamma attenuation and corrosion performance of these materials are investigated.

![Diagram showing multilayer coating components](image)

Figure 2. Shielding barriers surrounding the nuclear waste package.

**Gamma Attenuation**

The attenuation of gamma rays is tested both experimentally and modelled in MicroShield using single layer coated stainless steel samples and concrete overpack of various compositions. The gamma attenuation experimental setup is shown in Figure 3 below. Sources being used include, Cs-137, Ba-133, and three Co-60.

![Gamma attenuation experimental setup](image)

Figure 3. Gamma attenuation experimental setup.
The gamma ray attenuation is performed by measuring the fractional radiation intensity, labelled as \( I \) below, compared to the radiation source intensity, labelled as \( I_0 \) below. The thickness of each individual sample, labelled as \( x \) below, is measured and input into the exponential law in order to obtain the attenuation coefficient:

\[
I = I_0 e^{-\mu x}
\]  

(1)

Computational analysis is performed using MicroShield version 9.05 package and Serpent in order to calculate the attenuation coefficient and half value layer (HVL) depending on material density and composition. Dry cask configurations of multi-layered concentric cask designs containing a special inner glass-oxide layer are used as shown in Figure 4. The computational results are compared to experimental results for single layer coated steel substrates.

Figure 4. Computational configuration of modelled dry cask using various sources and shielding layers.

**Corrosion Testing**

Coated single layer steel substrates are placed within a salt-brine circulator using a pH of 5.8 for 40 days to analyse the corrosion effects to the material over this time frame. The salt-brine circulator is shown in Figure 5. A steel substrate sample is placed within the circulator for long periods of time in order to determine the long-term material degradation due to corrosion. Sample mass measurements are taken daily over the course of the experiment.

Figure 5. Salt-brine circulator system for corrosion testing.
RESULTS & DISCUSSION

The linear attenuation of coated steel substrates and concrete samples of various compositions is determined both experimentally and computationally. The experimental setup on the coated steel substrates use Cs-137, Ba-133, and three Co-60 sources while the concrete testing additionally adds three low-energy Cd-109 sources.

Experiments and Modelling on Coated Materials

The effect of adding coatings to stainless steel substrates is shown in Figure 6 & 7 where samples coated with different single and double layered coatings decrease the linear attenuation coefficient between 302 keV to 356 keV.

![Graph of Canister Coated Stainless Steel Attenuation Using High-Energy Photon Sources](image)

Figure 6. Measured counts of coated stainless steel substrates showing high-energy peaks.

![Graph of Linear Attenuation Coefficient in Canister Coated Samples Using High-Energy Photon Sources](image)

Figure 7. Determination of linear attenuation coefficients of various coatings on stainless steel substrates.

Computational modelling using MicroShield is performed to calculate the linear attenuation coefficient and half value layer of ZrO2 and TiO2 to compare the experimental results to computational modelling results. A strong correlation between experimental results and computational models is shown.
in Figures 8 & 9. The calculated half value layers are close except in the case of a double layer on 316 SS where the HVL is slightly increased as shown in Figure 10.

**Figure 8.** Comparison of experimentally and computationally calculated linear attenuation coefficients of ZrO₂.

**Figure 9.** Comparison of experimentally and computationally calculated linear attenuation coefficients of multi-layered ZrO₂ and TiO₂.

**Figure 10.** Determination of Half Value Layers of coated substrates on 316 SS.
Experiments and Modelling on Concrete

Similar experimental analysis is performed for concrete samples of various compositions and compared to simulated conditions using MicroShield software. Different mixtures of concrete are used, labelled Mix A and Mix B. Both mixtures have a composition of 50% special concrete, 25% boron, and 25% granite. The special concrete portion of Mixture A refers to a composition of 13.98% cement, 7.63% water, 10% lead, and the remaining filled with either granite, sand, or a half and half mixture of sand and granite.

The effect of concrete as a strong shielding material is shown in Figure 11. Samples of concrete mixtures with various compositions show slightly higher linear attenuation coefficients between 302 keV to 662 keV as seen in Figure 12.

![Graph showing overpack concrete attenuation using high-energy photon sources.](image)

**Figure 11.** Measured counts of concrete samples of various mixtures showing high-energy peaks.

![Graph showing linear attenuation coefficient in overpack concrete using low-energy and high-energy photon sources.](image)

**Figure 12.** Determination of linear attenuation coefficients of two different mixtures of concrete overpack material.

Computational modelling using MicroShield is performed to calculate the linear attenuation coefficient and half value layer of concrete samples of Mixture A with sand and granite to compare the
experimental results to modelling results. A strong correlation between experimental results, literature values from Lamash and Baratta (2001), and computational models is shown in Figures 13 & 14 at high-energy values. The calculated half value layers are very close, thus not dependent on the choice of aggregate as shown in Figure 15.

Figure 13. Comparison of experimentally and computationally calculated linear attenuation coefficients of Mixture A concrete with sand added using high-energy sources.

Figure 14. Comparison of experimentally and computationally calculated linear attenuation coefficients of Mixture A concrete with granite added using high-energy sources.
Figure 15. Determination of Half Value Layers of concrete Mixture A with various aggregates using high-energy sources.

The effect of concrete as a shielding barrier against low-energy sources is shown in Figure 16. Differences between the experimentally determined linear attenuation coefficient and the computationally determined linear attenuation coefficient are distinct at low-energies as seen in Figures 17 & 18. A poor correlation between the experimental and computational data occurs at energies below 88 keV. This is most likely due to the insufficient compositional data input into the MicroShield software. The calculated half value layers are very close, thus not dependent on the choice of aggregate as shown in Figure 19 at low-energies.

Figure 16. Measured counts of concrete samples of various mixtures showing low-energy peaks.
Figure 17. Comparison of experimentally and computationally calculated linear attenuation coefficients of Mixture A concrete with sand added using low-energy sources.

Figure 18. Comparison of experimentally and computationally calculated linear attenuation coefficients of Mixture A concrete with granite added using low-energy sources.

Figure 19. Determination of Half-Value Layers of concrete Mixture A with various aggregates using low-energy sources.
Corrosion Testing

Single layer coated samples of MoS₂, ZrO₂, and TiN over a 40 day circulatory cycle show no significant mass loss from corrosion. The maximum change in mass over the 40 day period is calculated to be a 0.65% change. Stainless steel materials are known for their strong corrosion resistance, thus it is expected that over a short time period, no corrosion degradation is observed.

CONCLUSION

Thin film coatings provide sufficient corrosion resistance to stainless steels used in nuclear waste storage applications. The material coatings show slight variations in the linear attenuation coefficient at higher energies, thus the chosen material as the coating is important. The experimentally determined linear attenuation coefficient values for coated stainless steel substrates show good correlation to computationally generated values. Experimentally determined linear attenuation coefficients of concrete mixtures under high-energy sources show agreement with computationally determined values. The half value layer does not depend on the type of filler aggregate used. Under low-energy sources, the experimental and computational linear attenuation coefficients show differing values likely due to the limitation in modelling the composition of the concrete mixtures within the modelling software. Once again, the half value layer does not depend on the type of aggregate added at low energies. Both thin films and various mixtures of concretes prove to be effective shielding barriers, thus various thin film coatings and concrete mixtures should be used for long-term storage of spent nuclear fuel.

REFERENCES