Project No. 15-8548

Assessment of Aging Degradation Mechanisms of Alloy 709 for Sodium Fast Reactors

Reactor Concepts Research Development and Demonstration (RCRD&D)

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Project 15-8548: Assessment of Aging Degradation Mechanisms of Alloy 709 for Sodium Fast Reactors
FINAL REPORT

1. Introduction

This project was initiated in October 2015 to investigate the creep and creep-fatigue behavior of Alloy 709 for structural components in Gen IV sodium cooled fast spectrum reactors. The goal of this project is to develop an understanding of the mechanisms responsible for the behavior of austenitic stainless steel alloy 709 under accelerated testing conditions to confidently predict long term creep and creep-fatigue behavior at the times (500,000 hours) and temperatures (550°C) of interest for fast reactor structural applications. Alloy 709 has exhibited significantly better creep resistance than Alloy 316H, which is currently specified in design of fast reactor structural components. However, the alloy has not been code qualified for elevated temperature nuclear design. The elevated temperature microstructure stability and creep and creep-fatigue behavior need to be better understood so service life can be accurately predicted. Alloy 709 was developed for fossil boiler applications, which operate at a higher temperature than the nuclear components. Thus, there is a strong need to characterize its creep and creep-fatigue behavior at these lower temperatures and corresponding longer service lifetimes. The overall objective of this project is to develop sufficient understanding of the mechanisms responsible for behavior of Alloy 709 under accelerated testing conditions to confidently predict long term creep and creep-fatigue behavior at the times (500,000 hours) and temperatures (550°C) of interest for fast reactor structural applications. The following report summarizes the findings of the creep-fatigue and creep behavior with respect to the research questions identified during the course of the project. Additionally, the training and professional development that occurred through the project and the products resulting from the project are summarized.

2. Creep Fatigue Behavior

The objective of this research was to develop an understanding of the deformation and damage mechanisms responsible for creep-fatigue failure of Alloy 709 across a range of accelerated testing conditions. A mechanistic understanding of LCF and creep-fatigue behavior provides a basis for reliable extrapolation of short-term test data to long-term service conditions relevant to nuclear applications. Specifically, the effects of microstructural evolution during accelerated laboratory testing were investigated with respect to creep-fatigue behavior and performance, and compared to the effects of long-term microstructural evolution during service. The conclusions of this study are addressed here with respect to the specific research questions posed.

How do the cyclic and time-dependent deformation mechanisms at 550 °C, compared to 650 °C, affect the cyclic stress-strain response, internal damage formation and propagation, and reduction in creep-fatigue life compared to pure LCF?

This question was primarily addressed through tests on the solution annealed condition (1100 °C anneal and water quench). Cyclic plasticity and time-dependent creep deformation have different effects on the formation and propagation of damage that lead to creep-fatigue failure. Specifically, creep deformation is responsible for grain boundary cavitation and void formation. At both temperatures the accumulation of creep strain correlates with creep damage (*i.e.* grain boundary voids). On the other hand, cyclic plastic deformation reduces the strain energy available for propagation of grain boundary creep damage.

The amount of time-dependent creep strain accumulated during creep-fatigue at 550 °C is a small fraction of that accumulated at 650 °C, due to less stress relaxation during the tensile hold. Despite a greater resistance to creep deformation at 550 °C, the creep-fatigue life is reduced significantly more at 550 °C compared to 650 °C, relative to LCF. Therefore, the significant difference in creep-fatigue behavior and life at 550 and 650 °C is attributed to differences in cyclic deformation mechanisms when testing from a solution annealed condition. Specifically, at 550 °C, slip is primarily planar for the duration of the test due to a strong inhibition of dislocation cross-slip, which prevents recovery and leads to high stresses during cycling. The lack of dynamic recovery results in propagation of grain boundary damage and failure by intergranular fracture. At 650 °C, the deformation character changes during creep-fatigue testing from planar to wavy, which is associated with cyclic softening and subgrain formation. Recovery during creep-fatigue testing results in lower stresses, blunting of internal damage, and mixed mode fracture propagation (transgranular and intergranular). Although the accumulated creep damage is greater in creep-fatigue at 650 °C, compared to 550 °C, a greater amount of plastic deformation during cyclic loading retards the propagation of the grain boundary damage. The shortest creep-fatigue lives occur under conditions of minimal cyclic plasticity, while the best creep-fatigue performance is realized with a moderate amount of cyclic deformation and a minimal amount of creep deformation.

How does initial microstructure and microstructural evolution (specifically precipitation) of Alloy 709 affect LCF and creep-fatigue behavior in accelerated laboratory testing compared to the behavior expected during long-term service exposure?

The microstructural instability of solution annealed Alloy 709 results in an evolution of precipitate and solid solution strengthening during short-term LCF and creep-fatigue testing at elevated temperatures. Intragranular precipitation of carbides and nitrides ($M_{23}C_6$ and Nb(C,N)) increases the strength initially, followed by a decrease as precipitates coarsen. Additionally, dynamic precipitation leads to a reduction in solid solution strengthening as solute concentration decreases. The microstructural evolution, and thus the evolution of strengthening mechanisms, is a strong function of test temperature. The higher strength of Alloy 709 at the lower testing temperatures in this study is a result of finer precipitates and higher solute content compared to higher temperatures.

Solute atoms responsible for dynamic strain aging (DSA) promote planar deformation and strain-hardening through an increase in friction stress on partial dislocations, which inhibits cross-slip and recovery. Dynamic precipitation decreases the solute content, lowers the friction stress, and leads to a transition in slip character from planar to wavy, in turn promoting recovery. Dynamic recovery correlates with lower strain-hardening rates and lower dislocation densities. Therefore, a reduction of the solute content also leads to a lower magnitude of Taylor hardening. The reduction in strength and the transition of slip character as a result of dynamic aging results in more cyclic plasticity and lower stress at peak strain.

Static aging of Alloy 709 under conditions which promote significant carbide and nitride precipitation prior to creep-fatigue testing results in a substantial increase in the creep-fatigue life at 550 and 650 °C, compared to the solution annealed condition. A reduction of the total interstitial concentration (C + N) from aging promotes cross-slip and dynamic recovery at lower stresses than in the solution annealed condition. Aging prior to testing also results in a reduction in precipitate strengthening, due to the coarse nature of the $M_{23}C_6$ carbides that form during static aging compared to those that precipitate dynamically. Although fine Z-phase also forms during static aging, the small volume fraction does not result in significant strengthening. The enhanced cyclic plasticity and relatively low peak tensile stresses after static aging result in transgranular fracture in creep-fatigue under the same test conditions where failure occurs by intergranular fracture in the solution annealed condition.

The significant improvement in creep-fatigue life at 550 °C with the pre-aged microstructure is due to more cyclic plasticity with approximately the same amount of creep strain per cycle compared to the solution annealed condition. Although the cyclic plasticity is also greater at 650 °C in the aged condition, a smaller increase in creep-fatigue life over the solution annealed condition is due to the relatively high creep strain per cycle compared to the test at 550 °C. These results support the hypothesis that creep-fatigue performance of Alloy 709 is optimized by enhancing the cyclic plasticity and minimizing the creep strain.

Creep resistance is reduced somewhat in Alloy 709 as a result of the aging treatment used in this study. For the creep-fatigue conditions studied, the creep strain per cycle is approximately the same in both the aged and the solution annealed microstructures, although the stresses are significantly lower in the aged condition. A lower creep resistance in the aged condition has important implications for constant stress creep conditions.

The results of this study indicate that the creep-fatigue performance during accelerated testing from a solution annealed condition is not representative of the performance during long-term service. Although precipitation during static aging of Alloy 709 at the expected service temperature of 550 °C is relatively slow compared to the higher temperatures studied, significant precipitation is expected in a short time compared to the expected service life. Therefore, the deformation behavior in the aged microstructure at the expected service temperature of 550 °C, which results in slow propagation of intergranular creep damage, is more indicative of the behavior during long-term nuclear service.

3. Creep Behavior

The objective of this component of the project was to evaluate creep behavior across a range of accelerated test conditions to determine the ability to extrapolate minimum strain rate predictions from high stress, shorter time data. It is established that austenitic steels often exhibit a transition in creep behavior from the high stress to low stress regimes, where high stress data have a higher creep rate exponent than the low stress data. These different regimes are also present in Alloy 709. The following research questions were designed to address this behavior.

How does Alloy 709's creep rate exponent and activation energy respond to creep conditions at moderate temperatures, varying stress, and varying thermal history?

Solution annealed specimens of Alloy 709 were subjected to constant load creep testing at 650 °C with an applied stress ranging from 110 MPa to 300 MPa. The alloy exhibits a minimum creep rate exponent of 6.4 during creep testing at applied stresses above 180 MPa and a minimum creep rate exponent of 3.95 during creep testing at applied stresses below 180 MPa. Alloy 709 has a minimum creep rate exponent of 3.13 while creep testing at 550 °C with an applied stress ranging from 180 MPa to 300 MPa.

The activation energy of creep measured between the two temperatures varied with stress. The activation energy is calculated to be 190 kJ/mol at 180 MPa applied stress. In contrast, the activation energy for creep is about 292 kJ/mol at 300 MPa. The activation energy of self-diffusion of similar alloys to Alloy 709 is closer to the activation energy of creep calculated at 300 MPa than the activation energy for creep calculated at 180 MPa. Several solute species in 316 are reported to have a lower activation energy of solute interdiffusion than self diffusion in austenitic stainless steels and more closely match the calculated activation energy of creep at 180 MPa than the activation energy calculated at 300 MPa.

Specimens of Alloy 709 were aged at 650 °C for 2,500 hrs and at 750 °C for 150 hrs. The two aging conditions produce two different microstructures. Both conditions precipitate out $M_{23}C_6$ on grain boundaries and as needles with grains. 650 °C produces a fine distribution of MX phase on dislocations. The precipitates are about 10 to 20 nm in diameter. At 750 °C, the alloy produces a fine distribution of z-phase on dislocations with size of about 10 to 20 nm. Creep testing Alloy 709 specimens aged at 650 °C and 750 °C increased the minimum creep rate during constant load creep testing at 650 °C at stresses ranging from 145 MPa to 300 MPa. Aging at 750 °C increased the minimum creep rates more than aging at 650 °C. In contrast, aging lowered the minimum creep rate during creep testing at 650 °C and an applied stress of 110 MPa. For creep testing at 650 °C, aging Alloy 709 produced a minimum creep rate exponent larger than that produced by constant load creep testing of solution annealed material. Alloy 709 has a minimum creep rate exponent of 8.1 when aged at 650 °C and 7.9 when aged at 750 °C. These data indicate that, for the aging times chosen, aging temperature has little effect on minimum creep rate exponent for Alloy 709.

Why is a transition in creep rate exponent as a function of stress observed in Alloy 709 at intermediate temperatures?

It is interpreted that the creep rate exponent changes from the high stress to low stress regimes due to a change in creep deformation mechanism from climb-controlled creep at higher stresses to glide/solute drag controlled creep at lower stresses. Two models were applied to Alloy 709 to predict minimum creep rate. One model assumes climb of dislocations past particles is the controlling process, and the model fits minimum creep rate data believed to be under climb controlled creep. Another model assumes the glide of dislocations is the controlling process, and the model fits minimum creep rate data believed to be under glide controlled creep.

Alloy 709 exhibits two different types of constant load creep curves. It is interpreted that one type of constant load creep curves is associated with climb controlled creep and the other is associated with glide controlled creep. During constant load creep testing at 650 °C and with an applied stress above 180 MPa, the alloy exhibits creep curves associated with climb controlled creep. The curves have pronounced primary creep regions. The minimum creep rate occurs as an infliction point prior to the material transitioning to tertiary creep. During constant load creep testing at 650 °C and with an applied load less than 180 MPa, the alloy exhibits constant load creep curves associated with glide controlled creep. The tests have a very brief period of conventional primary creep before the material reaches the minimum creep rate. Most tests then experience a period of inverted primary creep as the creep rate increases and establishes a constant creep rate at a slightly higher value. A creep test at 110 MPa and a temperature of 650 °C shows sigmoidal creep. Sigmoidal creep is a period of inverted primary creep followed by a period of conventional primary creep, and this behavior is also associated with glide controlled creep. Creep tests conducted at 250 MPa and a temperature of 550 °C produced a creep curve associated with glide controlled creep. The curve shows a similar brief period of primary creep followed by a period of inverted primary creep like those occurring at low stresses at 650 °C.

4. Training and Professional Development

The following personnel participated in the project from the Colorado School of Mines and Idaho National Laboratory:

Kip Findley (PI, Mines), Michael Kaufman (co-PI, Mines), Alan Carter (Ph.D. student, Mines), Ty Porter (Ph.D. student, Mines), Jie Song (post-doctoral researcher, Mines), Olivia DeNonno (undergraduate researcher, Mines)

(Richard Wright (PI, INL), Michael McMurtry (INL)

The project enabled training of all of the personnel in critical materials issues related to future nuclear reactor design. The project team involved the industrial sponsors of the Advanced Steel Processing and Products Research Center and the Center for Non-Ferrous Structural Alloys to obtain industrial mentorship and perspective on relevant steel production and metallurgy. During the course of the project, the graduate students had the opportunity to perform experiments at Idaho National Laboratory and thus had exposure to both academic and national laboratory research environments. Both the graduate students and the post-doctoral research had opportunities to mentor other students during the project. Both graduate students also had multiple opportunities to develop written and oral communication skills through reports, papers, and presentations at meetings and conferences.

5. Products Resulting from the Work

DOE Reports

DOE 1st Year Progress Report
DOE 2nd Year Progress Report
DOE 3rd Year Progress Report
DOE FINAL REPORT
DOE Deliverable: Computational Simulation to Determine Accelerated Aging Conditions
DOE Deliverable: Characterization of Baseline and Aged Alloy 709 Microstructures
DOE Deliverable: Assessment of Mechanical Property Results

Publications

A. Carter, T. Porter, K.O. Findley, M.K. Kaufman, "Time Temperature Precipitation Diagram and Qualitative Validation for Alloy 709," Proceedings of the ANS Winter Meeting, 2017.

T. Porter, K.O. Findley, R. Wright, M. McMurtrey, "Assessment of Creep-Fatigue Behavior of Alloy 709," Proceedings of the ANS Winter Meeting, 2017.

T.D. Porter, K.O. Findley, M.J. Kaufman, R.N. Wright, "Assessment of creep-fatigue behavior, deformation mechanisms, and microstructural evolution of alloy 709 under accelerated conditions." International Journal of Fatigue 124 (2019): 205-216.

Presentations

M.J. Kaufman, K.O. Findley, R.N. Wright, A. Carter, T. Porter, J. Song, "DOE-NEUP Project: Assessment of Aging Degradation Mechanisms of Alloy 709 for Sodium Fast Reactors," presentation at Alloy 709 Kick-off meeting, N.C. State University, December 2015.

A. Carter, T. Porter, K. Findley, M. Kaufman, R. Wright, "Assessment of Aging Degradation Mechanisms of Alloy 709," poster presented at DOE ART Advanced Materials Review, Germantown, MD, June 2016.

T.D. Porter, K.O. Findley, J. Song and M.J. Kaufman, "Assessment of Aging Degradation Mechanisms of Alloy 709 for Sodium Fast Reactors," semi-annual presentation to the sponsors of the Advanced Steel Processing and Products Research Center, Colorado School of Mines, September 20, 2016.

A. Carter, K.O. Findley, and M.J. Kaufman, "Assessment of Aging Degradation Mechanisms of Alloy 709 for Sodium Fast Reactors," semi-annual presentation to the sponsors of the Advanced Steel Processing and Products Research Center, Colorado School of Mines, September 20, 2016.

K.O. Findley, M.J. Kaufman, R.N. Wright, A. Carter, T. Porter, J. Song, “DOE-NEUP Project: Assessment of Aging Degradation Mechanisms of Alloy 709 for Sodium Fast Reactors,” presentation at DOE NEUP Alloy 709 Teleconference Review, August 18, 2016.

T.D. Porter, A. Carter, K.O. Findley, J. Song and M.J. Kaufman, "Assessment of Aging Degradation Mechanisms of Alloy 709 for Sodium Fast Reactors," semi-annual presentation to the sponsors of the Advanced Steel Processing and Products Research Center, Colorado School of Mines, March 21, 2017.

R.N. Wright, "Assessment of Aging Degradation Mechanisms of Alloy 709 for Sodium Fast Reactors," Presentation at the NEUP ART Materials Program Review, June 6-7, 2017.
A.T. Carter, K.O. Findley, and M.J. Kaufman, “Aging Effects on Time Dependent Deformation and Damage Mechanisms of Alloy 709,” Presentation at ASPPRC Semi-Annual Meetings, Fall 2017.

T.D. Porter, K.O. Findley, and M.J. Kaufman, “Assessment of Creep-Fatigue Behavior and Damage Mechanisms of Alloy 709 Under Accelerated Conditions,” Presentation at ASPPRC Semi-Annual Meetings, Fall 2017.

A.T. Carter, K.O. Findley, and M.J. Kaufman, “Aging Effects on Time Dependent Deformation and Damage Mechanisms of Alloy 709,” Presentation at CANSFA Semi-Annual Meetings, Fall 2017.

T.D. Porter, K.O. Findley, and M.J. Kaufman, “Assessment of Creep-Fatigue Behavior and Damage Mechanisms of Alloy 709 Under Accelerated Conditions,” Presentation at CANSFA Semi-Annual Meetings, Fall 2017.

A. Carter, J. Song, K. Findley, M. Kaufman, "Assessment of Aging Degradation Mechanisms of Alloy 709 for Sodium Fast Reactors Reactors," presented at the Materials for Nuclear Energy Applications Symposium at MS&T 2017, Pittsburgh, PA, October 8-12, 2017.

T. Porter, J. Song, K. Findley, M. Kaufman, “Assessment of Creep-fatigue Behavior and Damage Mechanisms of Alloy 709 Under Accelerated Conditions,” presented at the Materials for Nuclear Energy Applications Symposium at MS&T 2017, Pittsburgh, PA, October 8-12, 2017.

T. Porter, K.O. Findley, R. Wright, M. McMurtrey, "Assessment of Creep-Fatigue Behavior of Alloy 709," presented at the ANS Winter Meeting, October 29-November 2, 2017.

A. Carter, T. Porter, K.O. Findley, M.K. Kaufman, "Time Temperature Precipitation Diagram and Qualitative Validation for Alloy 709," presented at the ANS Winter Meeting, October 29-November 2, 2017.

A.T. Carter, K.O. Findley, and M.J. Kaufman, “Aging Effects on Time Dependent Deformation and Damage Mechanisms of Alloy 709,” Presentation at ASPPRC Semi-Annual Meetings, Spring 2018.

T.D. Porter, K.O. Findley, and M.J. Kaufman, “Assessment of Creep-Fatigue Behavior and Damage Mechanisms of Alloy 709 Under Accelerated Conditions,” Presentation at ASPPRC Semi-Annual Meetings, Spring 2018.

Ty Porter, Alan Carter, Kip Findley, Michael Kaufman, Michael McMurtrey, Richard Wright, "Assessment of Aging Degradation Mechanisms of Alloy 709 in Creep and Creep-Fatigue," presentation at DOE Advanced Reactor Technology Advanced Materials Program Review, Germantown, MD, 2018.

T.D. Porter, K.O. Findley, and M.J. Kaufman, “Assessment of Creep-Fatigue Behavior and Damage Mechanisms of Alloy 709 Under Accelerated Conditions,” Presentation at ASPPRC Semi-Annual Meetings, Fall 2018.

T.D. Porter, K.O. Findley, and M.J. Kaufman, “Assessment of Creep-Fatigue Behavior and Damage Mechanisms of Alloy 709 Under Accelerated Conditions,” Presentation at CANFSA Semi-Annual Meetings, Fall 2018.

T.D. Porter, K.O. Findley, M.J. Kaufman, “Assessment of Creep-Fatigue Behavior and Damage Mechanisms of Alloy 709 Under Accelerated Conditions,” Presentation at the semi-annual meetings of the Advanced Steel Processing and Products Research Center, Spring 2019.

T.D. Porter, "The Effects of Microstructural Evolution on Deformation and Damage Mechanisms During Creep-Fatigue Testing of Alloy 709," Ph.D. Thesis Defense Presentation, Colorado School of Mines, June 2019.

A.T. Carter, K.O. Findley, and M.J. Kaufman, “Assessment of Alloy 709 Accelerated Creep Properties for Use in Sodium Cooled Fast Reactors,” Presentation at ASPPRC Semi-Annual Meetings, Fall 2019.

Other Reports

A. Carter, K.O. Findley, M.J. Kaufman, "Assessment of Aging Degradation Mechanisms of Alloy 709 for Sodium Fast Reactors," Advanced Steel Processing and Products Research Center semi-annual report, Spring 2016.

T.D. Porter, K.O. Findley, M.J. Kaufman, “Assessment of Aging Degradation Mechanisms of Alloy 709 for Sodium Fast Reactors,” Advanced Steel Processing and Products Research Center semi-annual report, Spring 2016.

T.D. Porter, K.O. Findley, J. Song and M.J. Kaufman, "Assessment of Aging Degradation Mechanisms of Alloy 709 for Sodium Fast Reactors," semi-annual report to the sponsors of the Advanced Steel Processing and Products Research Center, Colorado School of Mines, Fall 2016.

A. Carter, K.O. Findley, and M.J. Kaufman, "Assessment of Aging Degradation Mechanisms of Alloy 709 for Sodium Fast Reactors," semi-annual report to the sponsors of the Advanced Steel Processing and Products Research Center, Colorado School of Mines, Fall 2016.

T.D. Porter, K.O. Findley, J. Song, and M.J. Kaufman, "Assessment of Creep-Fatigue Behavior and Damage Mechanisms of Alloy 709 under Accelerated Conditions," semi-annual report to the sponsors of the Advanced Steel Processing and Products Research Center, Colorado School of Mines, Spring 2017.

A. Carter, K.O. Findley, and M.J. Kaufman, "Assessment of Aging Degradation Mechanisms of Alloy 709 for Sodium Fast Reactors," semi-annual report to the sponsors of the Advanced Steel Processing and Products Research Center, Colorado School of Mines, Spring 2017.

A.T. Carter, K.O. Findley, and M.J. Kaufman, “Aging Effects on Time Dependent Deformation and Damage Mechanisms of Alloy 709,” ASPPRC Semi-Annual Report, Fall 2017.

T.D. Porter, K.O. Findley, and M.J. Kaufman, “Assessment of Creep-Fatigue Behavior and Damage Mechanisms of Alloy 709 Under Accelerated Conditions,” ASPPRC Semi-Annual Report, Fall 2017.

A.T. Carter, K.O. Findley, and M.J. Kaufman, “Aging Effects on Time Dependent Deformation and Damage Mechanisms of Alloy 709,” ASPPRC Semi-Annual Report, Spring 2018.

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A.T. Carter, K.O. Findley, and M.J. Kaufman, “Assessment of Alloy 709 Accelerated Creep Properties for Use in Sodium Cooled Fast Reactors,” ASPPRC Semi-Annual Report, Fall 2018.

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A. Carter, K.O. Findley, M.J. Kaufman, “Assessment of Alloy 709 Accelerated Creep Properties for use in Sodium Cooled Fast Reactors,” Semi-annual report to the sponsors of the Advanced Steel Processing and Products Research Center, Spring 2019.

T.D. Porter, K.O. Findley, M.J. Kaufman, “Assessment of Creep-Fatigue Behavior and Damage Mechanisms of Alloy 709 Under Accelerated Conditions,” Semi-annual report to the sponsors of the Advanced Steel Processing and Products Research Center, Spring 2019.

T.D. Porter, "The Effects of Microstructural Evolution on Deformation and Damage Mechanisms During Creep-Fatigue Testing of Alloy 709," Ph.D. Thesis, Colorado School of Mines, June 2019.

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