

HARMONIZING OF CREEP-FATIGUE TEST METHODS THROUGH DEVELOPMENT OF ASTM STANDARDS

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ABSTRACT

Creep-fatigue crack formation (endurance) and crack growth rate data are necessary inputs for assessing the structural integrity and for estimating the design life of high temperature components in power generation and aircraft engine industries. Ensuring consistency in the reported test data, as well as an understanding of the inherent scatter and its source in the data, are both necessary for assuring quality and limitations of the analyses that rely on the data. In 2008, the American Society for Testing and Materials (ASTM) under the umbrella of its subcommittees E08.05 on Cyclic Deformation and Crack Formation and E08.06 on Crack Growth, and the sponsorship of Electric Power Research Institute (EPRI) through its international experts' working group on creep-fatigue embarked on the task of developing separate standard test methods for creep-fatigue crack formation and creep-fatigue crack growth. The first standard entitled, "E-2714-09: Standard Test Method for Creep-fatigue Testing" was developed in 2009 and was followed up with a round-robin consisting of 13 laboratories around the world for testing the newly developed standard. This paper discusses the results of this round-robin concluded in 2012 using the widely used P91 steel that led to the formulation of the Precision and Bias statement contained in the version of the ASTM standard E2714 that was successfully balloted in the year 2013.

BACKGROUND

Time-dependent inelastic deformation resulting from stresses at elevated temperatures is known as *creep* [1,2]. Creep is often the life-limiting design consideration in high temperature components. Engineering applications also often involve components that experience cyclic loads during service. The term creep-fatigue (C-F) refers to conditions that involve cyclic deformation and damage in conjunction with time-dependent deformation and damage due to creep.

The practice of conducting laboratory experiments to characterize a material's creep-fatigue response is common in the scientific community for the past years. Because of the large number of experimental variables involved during laboratory C-F testing that can introduce variability in the data, it is beneficial to code the best practices in a standard test method. To respond to this need, the ASTM (American Society for Testing and Materials) Task Group on Creep-fatigue Crack Formation (E08.05.08) with global support enabled by EPRI (Electric Power Research Institute) developed a new standard E2714 in 2009 [3]. Specifically, this standard deals with and is limited to the formation of macroscopic cracks in a specimen that was initially un-cracked, to a size detectable by a technique stated in the standard.

A voluntary round-robin (RR) program began in 2009 with the primary objective of conducting C-F tests to characterize the number of cycles required for crack formation of an elevated temperature material. This RR also included pure fatigue and creep tests that were expected to be conducted as per existing ASTM standards E606 [4] and E139 [5], respectively for completeness with regard to characterizing the test material. With the test results obtained from this RR, a measure of precision and bias and specifically inter- and intra-laboratory variability within the results was derived and successfully balloted through the ASTM process. This paper describes the results of this round-robin. Even though previous RR studies have been conducted under low-cycle fatigue (LCF) loading conditions at elevated temperatures [6,7,8], none have been conducted under conditions with hold time.

TEST MATERIAL

The test material chosen for this RR is the modified 9% chromium (Cr)-1% molybdenum (Mo) steel that is designated by the ASTM as grade P91 steel wherein the prefix P denotes piping application. All the P91 specimens used for the current RR were obtained from a retired pipe donated by EPRI, Charlotte, USA. The pipe material was re-normalized to ensure consistency with the original tempered martensitic/ferritic microstructure of these steels. The physical dimensions of the pipe section were as follows: outer diameter: 482 mm, wall thickness: 47.5 mm and a length of approximately 1 m. The pipe was cut along its length to obtain approximately 3 equal sections. The 3 cut segments were respectively labeled as sections 1, 2 and 3 and only the cut Section 2 was used for the current RR. All RR participants were given their choice of either receiving machined specimens or specimen blanks that they would use for machining specimens. A comprehensive collection of all the specimen drawings and

machining layouts, along with other test matrix details, used for the current RR is provided in a recent EPRI report and publication [9]. It was stipulated that all of the test specimen geometries must comply with the guidelines recommended by the test standard E2714-09. The chemical composition of P91 steel used in the RR testing in weight% is given in Table 1.

Table 1: Chemical composition of test material (in weight %).

C	Si	Mn	P	S	Ni	Cr	Mo	As	V	Nb	Al	Cu	N	Sb, Sn	Fe
0.11	0.31	0.45	0.011	0.009	0.19	8.22	0.94	0.005	0.21	0.07	0.006	0.16	0.039	0.001	Bal.

Tensile tests and creep deformation and rupture tests on P91 steel were conducted at 625⁰C to fully characterize the material. The creep deformation and rupture tests were conducted as per the recommended guidelines prescribed by the ASTM standard E139 [5]. The test durations varied from as low as few days to as high as a few months. The test parameters were finalized by using a model based on the Larson-Miller parameter (LMP). These results clearly showed that the creep characteristics of the RR material compare favorably with data from the literature.

ROUND-ROBIN TEST RESULTS

The RR participants were requested to send their completed test results to the University of Arkansas (UA) for further data analysis. UA along with EMPA in Switzerland were responsible for the post-test inspection and optical microscopy analysis of the tested specimens. All the test results from all the participants are shown in Fig. 1.

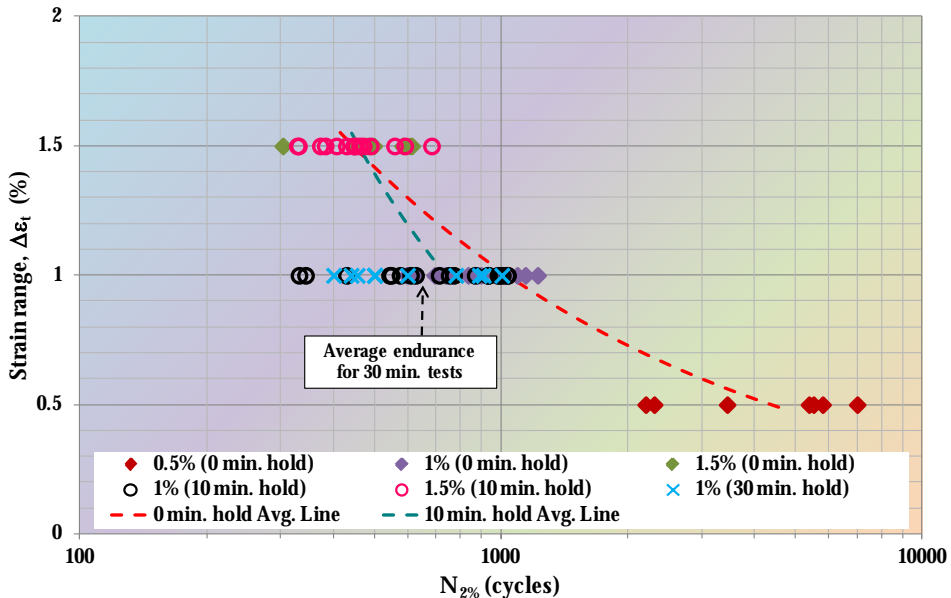


Figure1: Creep-fatigue endurance plot containing all the round-robin test data for P91 steel at 625°C.

The RR participants were requested to determine 2% load drop as the end-of-life criterion for C-F crack formation and record their interpretation of C-F endurance for each completed test using the method described in the ASTM standard. The stress-strain response and stress-relaxation response during hold times were also recorded and reported as part of the participant reports. The RR participants were randomly coded and each was assigned an arbitrary identification number from 1 to 15. A total of 32 tests were completed by 10 RR participants under three strain ranges of 0.5%, 1% and 1.5% without hold time at 625°C while following the test procedure prescribed in the C-F test standard. In addition, 46 tests were completed with a hold time of 10 and 30 minutes under strain ranges of 1% and 1.5% by 11 participants.

Plots of maximum and minimum stress (σ_{max} and σ_{min} , respectively) vs. cycles obtained under cyclic testing without hold time consistently showed that P91 steel undergoes significant cyclic softening. The magnitude of peak stress decreases as loading cycles increase during the initial cycles followed by a linear softening stage for considerably longer duration before softening accelerates again, marking the onset of crack formation [3]. The endurance results presented for discussion in this section are based on 2% percent deviation in the maximum stress from the extended linear softening phase, as reported by the RR participants.

All test results for C-F crack formation under the condition of no hold time are provided in Fig.2. The standard deviations in the mean values are also presented if there had been more than one test conducted at any given strain amplitude by any of the RR participants. The number of cycles for C-F crack formation decreases with increasing strain amplitude. In addition to this obvious

result, several significant observations can be made from the results. (1) At the lowest strain range, the resistance heating method seems to result in lower lives as compared to radiation heating but significantly higher compared to the one test performed with induction heating. At the other strain ranges, no biases in lives were detected that could be associated with the heating methods. (2) Specimens from one of the locations in the pipe appear to yield the highest lives at all the 3 strain ranges. (3) It appears that considerable scatter exists in the endurance data for a given strain range but on the basis of the information provided by the participants, there is no basis for eliminating any data from being considered. Further, the scatter observed was comparable or less than those reported in the earlier round-robins [6,7,8] (4) As part of analysis, we developed a method for analytically determining the 2% stress drop point. The differences between the average trends and the 95% confidence interval from this method versus those reported by the participants are both shown in the plot. The difference between the two approaches for estimating lives is marginal. For more details, refer to reference [9]. The metallography results will be considered later in the paper.

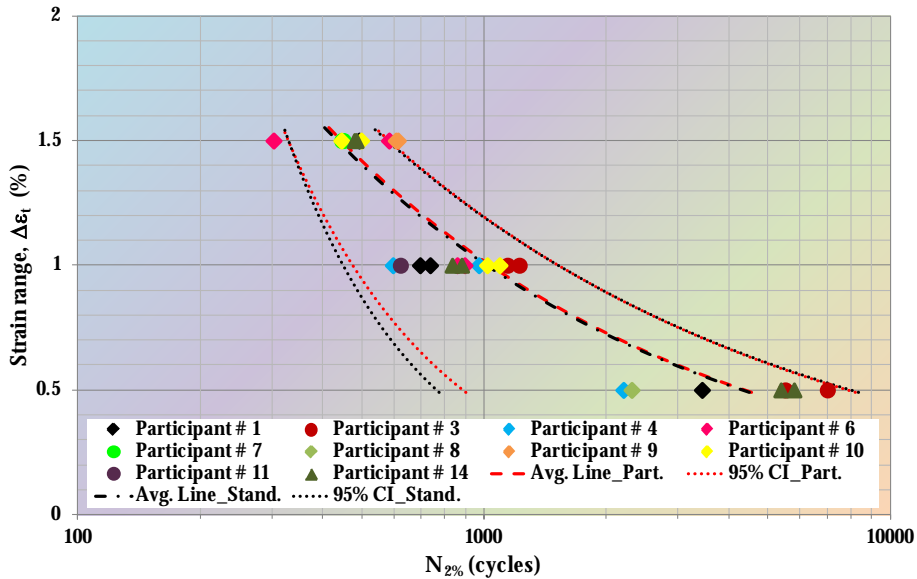


Figure 2: Creep-fatigue endurance plot for P91 steel at 625°C containing the round-robin data from tests conducted at different strain range with no hold time. The 95% confidence interval is provided considering C-F endurance data as per both the Participants' assessment (CI_Part.) and Analytical method (CI_Stand.).

Next we focus on the results obtained from cyclic tests conducted with hold time – 10 and 30 minutes – at the maximum (tensile) peak strain during loading, Figures 3 and 4. It is expected that under such loading conditions, the C-F endurance is still dominated by fatigue with creep deformation contributing somewhat marginally to the accumulated damage. It was observed that P91

steel qualitatively shows similar cyclic softening behavior in the presence of hold time as in tests without hold time. Also, the C-F lives were on the average shorter under the hold time conditions. Increasing the hold time for the same strain range was found to further decrease the C-F life. Although these observations were not noted in all of the RR tests, the majority of the test results followed this trend. In the tests conducted by RR participants 4, 6, 9, 10 and 11, the introduction of hold time led to approximately equal or higher C-F lives compared to tests with no hold time. This indicates that perhaps the effects of creep are small on the life of the specimens with the scatter in the data masking the effects due to hold time under the adopted conditions.

Considerable scatter in the mean C-F endurance for a given strain range arises by including the test results corresponding to some of the data. These tests indicate the possibility of being potential outliers and the reasons, if any, will be discussed later based on post-test metallographic inspection. These results correspond to tests conducted by participants 4, 9, and 11 who also reported significantly different C-F endurances for the zero hold time tests. This raises the possibility of issues with the test set-up and/or practices for participants 4 and 9 that consistently persisted during the RR testing. This deserves further examination using metallography and post-test analyses.

Besides the problems encountered with data contributed by participants 4 and 9, there were additional issues that are identified among the reported results that merit further discussion. For 1% strain range tests with 10 minute hold time, the participants reported excellent repeatability but the reproducibility among the various participants was not as good. Participant 11 reported considerably reduced C-F endurance in both their tests. Participant 6 had significant repeatability scatter in the tests conducted at 1% strain range with either 10 or 30 minutes hold. Participant 4 reported increasing C-F endurances in both their tests with the introduction of hold time, as compared with LCF tests conducted in their laboratory. Participant 8 had a standard deviation of ~ 40% as a percentage of the mean C-F endurance in the 2 tests conducted at 1% strain range, as compared with that of < 1% for 2 tests conducted at 1.5% strain range (both with hold time) in the same facility. The results from Participant 8 reported considerably reduced C-F endurances at 1.5% strain range with 10 minute hold. Post-test inspection and metallographic results will provide more definitive insight into these trends.

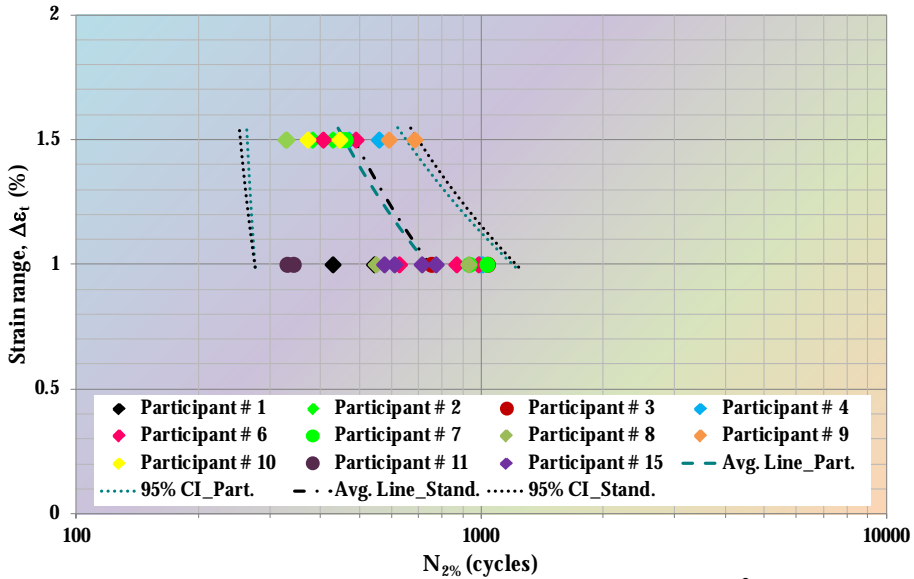


Figure 3: Creep-fatigue endurance plot for P91 steel at 625°C containing the round-robin data from tests conducted at different strain ranges and 10 minutes hold time. The 95% confidence interval is provided considering C-F endurance data as per both the Participants' assessment (CI_Part.) and Analytical method (CI_Stand.).

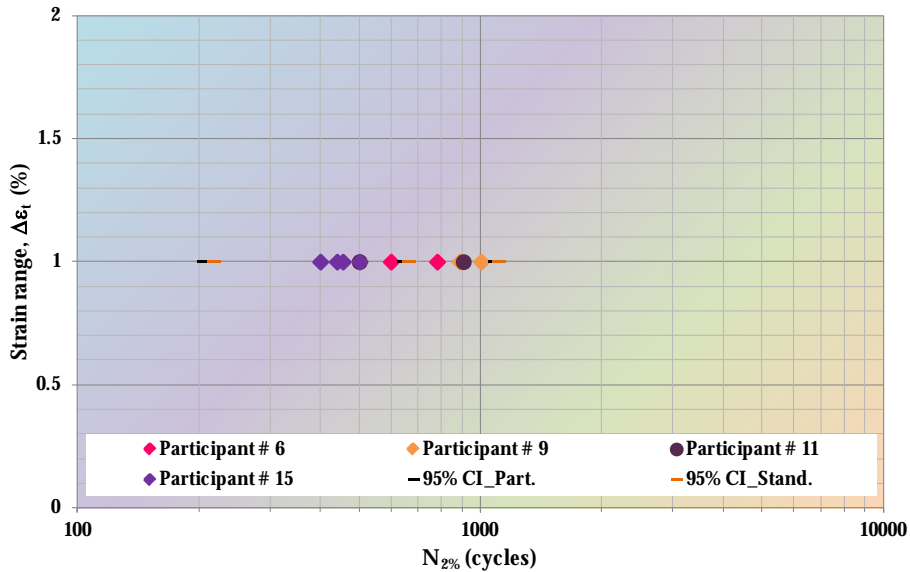


Figure 4: Creep-fatigue endurance plot for P91 steel at 625°C containing the round-robin data from tests conducted at different strain ranges and 30 minutes hold time. The 95% confidence interval is provided considering C-F endurance data as per both the Participants' assessment (CI_Part.) and Analytical method (CI_Stand.).

POST-TEST INSPECTION

Before the tested specimens were mechanically sectioned for metallography and further optical microscopic examination, high resolution digital photographs of the as-tested specimens were obtained for the records and are reported elsewhere in detail [9] with only a few select examples here such as in Fig.5. Visual inspection of these specimens was carried out and prominent characteristics of the specimen morphology and the presence of any geometric instability (or bulging) were carefully noted. The main features from these visual and digital observations are summarized as follows:

- 1) Pure LCF (no hold time) test specimens show multiple crack initiation sites characteristic of transgranular failure and no significant bulging.
- 2) All C-F (hold time) test specimens showed limited crack branching, but bulging becomes more pronounced with increasing hold time.
- 3) Bulging was much more noticeable for tests conducted with induction heating as compared to tests conducted using resistance furnaces under the same test condition. This is indicative of localized hot spots and perhaps non-uniform temperatures in the specimen during induction and radiation heating.
- 4) Oxidation and specimen dimensions may also play a vital role in the formation of bulges; Specimens tested by Participant 11 (that have comparatively smaller diameters) do not show any indication of bulging even on 30 minute hold time tests.
- 5) Specimen location does not seem to play any role on the type and morphology of C-F crack(s) for any of the tests.
- 6) In several tests conducted by Participant 6, the fatal cracks originate at the ceramic bead applied to avoid extensometer slippage. The thermal expansion mismatch between the metal and the ceramic may have influenced the test results in these cases.
- 7) One of the participants (Participant 2) used both induction and resistance heating systems and reported variations in cycles to failure; however, no visible differences existed in the external appearance of the tested specimens.

The test specimens were then mounted onto transparent specimen holders followed by two rounds of grinding and polishing to reveal the microstructure. These specimens were then examined primarily using an optical microscope and scanning electron microscopic analyses followed, if warranted.

Rather than at inclusions and/or other internal microstructural features, cracks predominantly formed on specimen surfaces and propagated normal to the loading direction with secondary branching of cracks in specimens tested under C-F loading with a hold time. The surfaces of almost all the primary cracks that propagated and opened due to plastic/creep deformation were covered with thick oxide layers although the thickness of these layers seemed to vary significantly for specimens tested under similar conditions but by different participants. These observations resemble those of Fournier *et al.* [10].

Creep damage, normally associated with grain and lath boundary cavitation, is not visible in any of the “chemically unetched” metallographic specimens. Thus,

these specimens were etched using chemical etchants that are known to preferentially etch these boundaries. Since 1% strain range is the only test condition under which three different hold times (0, 10 and 30 minutes) were employed, it is the most suitable for such examination. The specimens were taken from three different RR participants all using resistance heating method system. It was clearly seen that creep damage was present in the specimens tested with 30 minute hold time but not in specimens tested with 0 minute hold time. There is evidence of some cavitation damage in the 10 minute hold time tests. These examinations indicate that creep might be actively involved in enhancing deformation micromechanisms with increasing hold times, but does not seem to contribute significantly to damage for hold times less than 30 minutes.

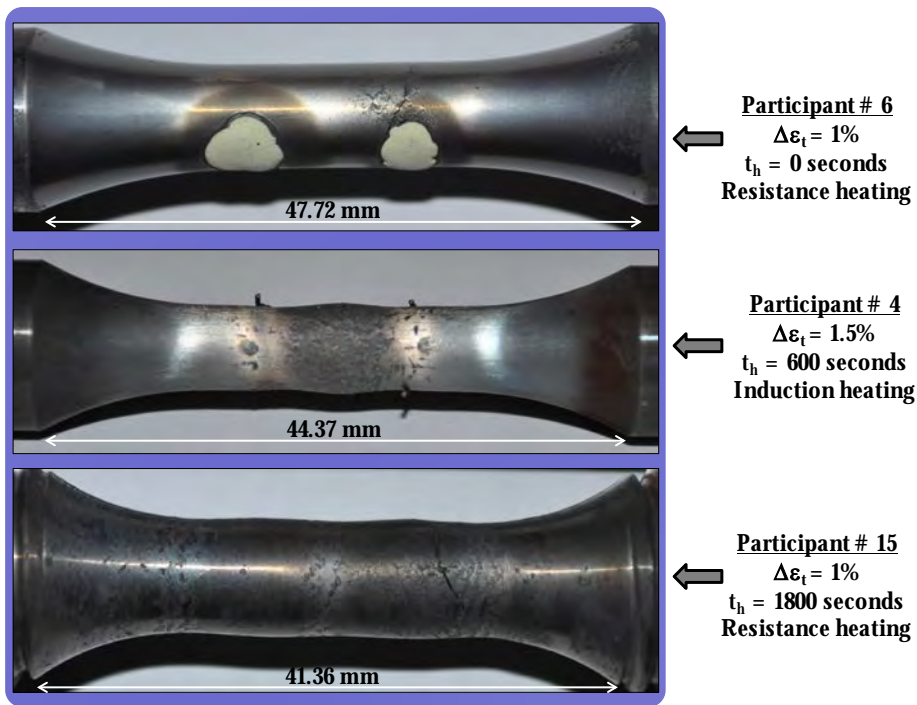


Figure 5: High resolution digital photographs of different test specimens as received from the round-robin participants.

Figure 6 shows the micrographs of two specimens tested at 1% strain range and 30 minutes hold at peak tensile strain, one from Participant 15 who used resistance heating and reported a life of 400 cycles and the other from Participant 2 who used induction heating and reported a life greater than 1039 cycles. The damage characteristics between the two specimens were quite different in that the specimen with the lower life (resistance furnace heated) showed lot more creep and oxidation damage than the one with longer life (induction heated). The significant difference in C-F endurance between the two specimens and the accompanying damage states indicates that differences in

heating method can contribute to systematic biases in the C-F life, with resistance heating associated with comparatively lower C-F lifetimes. This may be more the case for ferritic materials and something to keep in mind during C-F testing. Also, additional studies to investigate the differences due to heating methods must be undertaken.

DISCUSSION

To formulate the precision and bias statements needed for supporting the C-F test standard, statistical analyses of the reported RR data were performed. These analyses provide a measure of the inter- and intra-laboratory variability also termed as the reproducibility and the repeatability, respectively. Statistical analyses performed are as per the guidelines prescribed in the ASTM standard E691-09 [11].

Repeatability and reproducibility are statistical measures indicative of the intra- and inter-laboratory variability, respectively in the test results obtained among the different laboratories while following the test procedure prescribed in the C-F test standard E2714-09. Formally, *repeatability limit* (r) means that two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the “ r ” value for that material; “ r ” is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment in the same laboratory. *Reproducibility limit* (R) means two test results shall be judged not equivalent if they differ by more than the “ R ” value for that material; “ R ” is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipments in different laboratories.

The potential sources of uncertainty in the test results are expected from hardware related issues such as the accuracies of strain and force transducers, temperature measurement and control, type of heating systems employed and machine alignment, along with inherent microstructural variability within the nominally homogeneous test material. Additional factors such as load-cell calibration and/or bending misalignment might also contribute to further uncertainty. All these issues are highlighted in the test standard and the recommended test practices are detailed to minimize these effects. The RR data from this study provides an ideal opportunity to quantify the uncertainties that are expected due to these factors. The influence of specimen size on the C-F endurance has not been systematically investigated even for sizes that fall within the recommended test specimen configurations. Hence, a small number of tests were conducted to investigate if there is any systematic bias of specimen size on C-F lives of specimens.

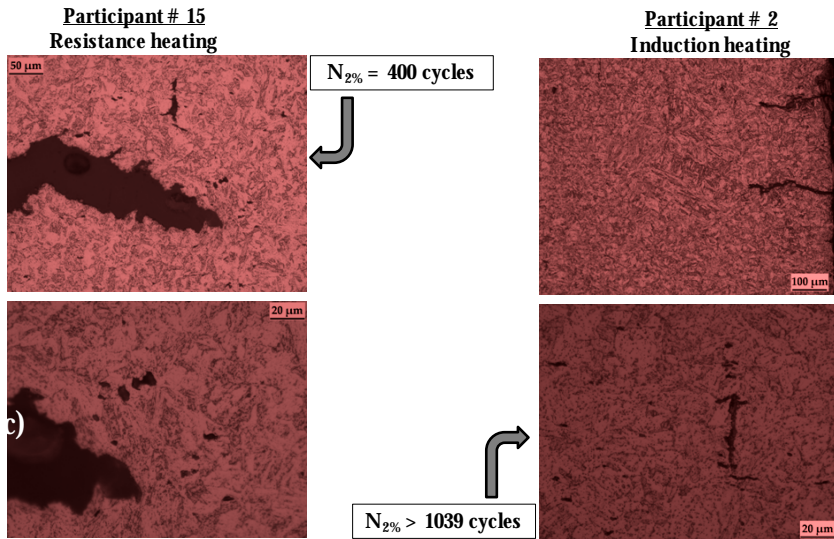


Figure 6: Optical microscopic images showing the effect of type of test-furnace heating during the C-F deformation of P91 steel at 1% strain range and 30 minutes hold at peak tensile strain. More creep cavitation is seen in specimens tested with resistance heating.

Table 2 shows the intra- and inter-laboratory variability (or equivalently, repeatability and reproducibility, respectively) estimated using statistical calculations with the C-F endurance data. The end-of-life as reported by the RR participants following the current guidelines in E2714-09 is tabulated under *Participants' Assessment* and the end-of-life based on an analytical five step procedure developed as part of this study is tabulated separately and labeled as the *Analytical Method*. Although the mean C-F endurance and its standard deviation did not significantly vary between the two assessment methods, the repeatability in tests involving higher hold times, was better when the analytical procedure was utilized. The reproducibility, on the other hand, appears to be substantially unchanged. This indicates that utilizing the analytical procedure for determining 2% force reduction life in C-F testing is beneficial but the benefits are marginal. Thus, if the analytical procedure is included in a future version of the standard, it should not be made mandatory but rather be treated as a technical note.

The data obtained from few test specimens such as those specifically from participants 6, 10 and 11 resulted in unusually reduced C-F lives and thus significantly increased the scatter among the results. The tested specimens showed that in one case the crack emanated from the region where the extensometer was attached and in the other two cases there was clear evidence of bending in the specimen. These and other test specimens that contributed to increased scatter were analyzed thoroughly via visual examination and optical metallography. Some of these test results were deemed to not qualify as valid data to meet the current test standard. Figure 7 shows the C-F endurance plots containing the RR data that exclude these test

results from those shown earlier in Figure 2. It can be readily seen that the overall scatter reduced considerably.

Table 2: A measure of mean creep-fatigue endurance is defined as the C-F life corresponding to 2% load drop and the standard deviation in the data. The intra- and inter-laboratory variability is also computed and presented as per both assessment procedures.

<u>Test Condition</u>	Participants' Assessment		Analytical Method	
	Mean N _{2%} ± Std. Dev. (cycles)	Repeatability/ Reproducibility Std. Dev. (cycles)	Mean N _{2%} ± Std. Dev. (cycles)	Repeatability/ Reproducibility Std. Dev. (cycles)
± 0.25%	5920 ± 474	547 / 612	5938 ± 499	523 / 621
± 0.5%	911 ± 174	47 / 177	880 ± 180	47 / 183
± 0.75%	491 ± 68	40 / 74	489 ± 71	37 / 75
± 0.5% (10 min. hold)	741 ± 235	51 / 238	753 ± 244	51 / 246
± 0.75% (10 min. hold)	457 ± 97	18 / 98	479 ± 102	27 / 103
± 0.5% (30 min. hold)	697 ± 203	82 / 211	672 ± 207	44 / 210

To quantify the reduction in scatter before and after excluding few invalid test results, the variability factor – ratio of maximum to minimum C-F life obtained from the 95% CI bands – was calculated for the different test conditions. For LCF tests, the variability factor reduced from 10, 3 and 2 to 3, 3 and 2 for the strain ranges of 0.5%, 1% and 1.5%, respectively. For 10 minute hold time tests, the variability factor reduced from 4 and 3 to 3 and 3 for the strain ranges of 1% and 1.5%, respectively. The variability factor remained at 5 for the 30 minute hold time tests at 1% strain range, as none of the test results from this condition could be excluded from the data set.

PRECISION AND BIAS STATEMENTS

These statements were developed by the Inter-laboratory studies (ILS) group in ASTM from the analysis provided from the round-robin data [11].

The Repeatability, “r” and reproducibility “R” limits are listed in Tables 3 to 5 below. Any judgment in accordance with the above for “r” and “R” would normally have an approximate 95% probability of being correct, however the precision statistics obtained must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The limited number of laboratories reporting replicate results under certain conditions guarantees that there will be times when differences greater than predicted by these results will arise, sometimes with considerably greater or smaller frequency than the 95% probability limit would imply. Consider the repeatability limit and the reproducibility limit as general guides, and the associated probability of 95% as only a rough indicator of what can be expected.

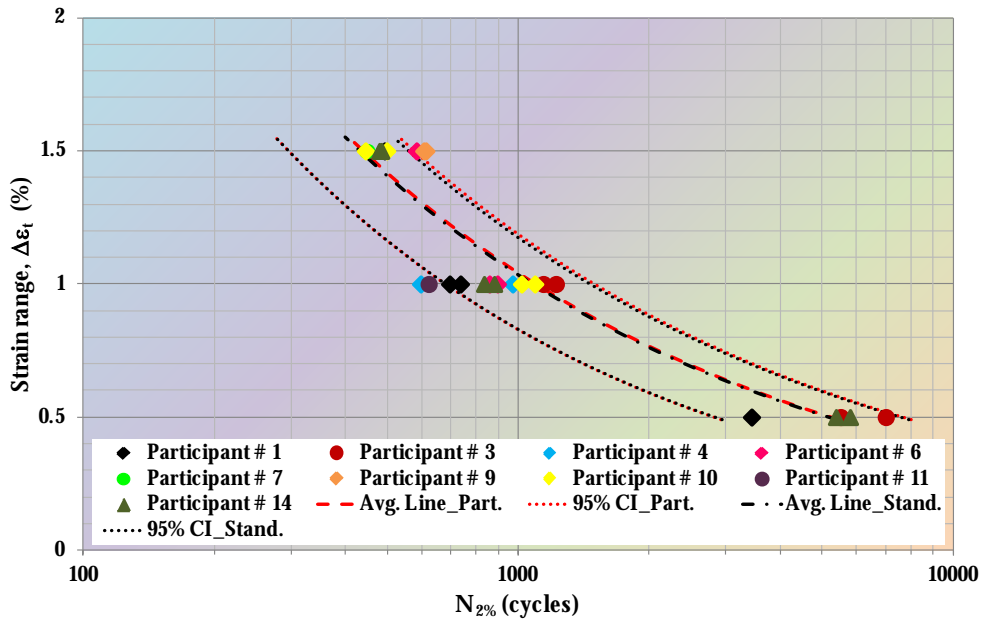


Figure 7: Creep-fatigue endurance plot containing the round-robin data excluding invalid test results from tests conducted at different strain ranges and no hold time. The 95% confidence interval is provided considering C-F endurance data as per both the Participants’ assessment (CI_Part.) and Analytical method (CI_Stand.).

Table 3- Statistics from all tests conducted with no hold time

Strain Amplitude/No. of Labs/ No. of Tests	Average \bar{X}	Repeatability Standard Deviation, S_r	Reproducibility Standard Deviation, S_R	Repeatability Limit, r	Reproducibility Limit, R
$\pm 0.25/5/7$	3957.5	488.9	1902.8	1368.9	5328
$\pm 0.50/7/13$	869.8	105.8	207	296.3	579.5
$\pm 0.75/6/11$	491.5	82.3	84.0	230.5	235.3

Table 4- Statistics from all tests conducted at $\pm 0.5\%$ with hold times of 10 and 30 minutes

Hold Time/No. of Labs/ No. of Tests	Average \bar{X}	Repeatability Standard Deviation, S_r	Reproducibility Standard Deviation, S_R	Repeatability Limit, r	Reproducibility Limit, R
10/8/19	740.8	143.7	266.3	402.3	745.8
30/4/10	696.3	163.8	247.3	458.5	692.4

Table 5- Statistics from all tests conducted at $\pm 0.75\%$ with hold time of 10 minutes

Hold Time/No. of Labs/ No. of Tests	Average \bar{X}	Repeatability Standard Deviation, S_r	Reproducibility Standard Deviation, S_R	Repeatability Limit, r	Reproducibility Limit, R
10/7/14	456.7	47.1	102.9	132.0	288.2

SUMMARY AND CONCLUSIONS

- A round-robin (RR) was conducted under the sponsorship of ASTM Task Group E08.05.08 on Creep-fatigue Crack Formation to assess the variability in creep-fatigue endurance results. Multiple laboratories conducted creep-fatigue tests using P91 steel at a temperature of 625°C as per guidelines outlined in the ASTM standard test method E2714-09.
- To assess inter-laboratory variability (or reproducibility) in the results, tests were conducted under three strain amplitudes of $\pm 0.25\%$, $\pm 0.50\%$, and $\pm 0.75\%$ and at hold times ranging from 0 to 30 minutes. Each laboratory tested duplicate specimens under identical conditions and in some cases, more than 2 tests were conducted under identical conditions to assess repeatability.
- Metallographic assessment of the tested specimens was performed to determine the extent of fatigue and creep damage under each test condition:
 - The damage mode in tests conducted under the conditions of no hold time and with 10 minutes of hold time was *predominantly* transgranular fatigue with some presence of oxidation spikes
 - The damage mode in tests conducted under the condition of 30 minutes hold time consisted of an *interaction* of transgranular fatigue with dominant oxide spikes and creep cavitation
- The overall variability in C-F endurances as measured by the 95% confidence interval varied with the strain range and hold times as follows (*after excluding invalid test results and rounding off to the nearest integer*):
 - For tests with no hold times, the variability factor (ratio of maximum to minimum life) for the 95% confidence interval bands was **3**, **3** and **2** for strain amplitudes of $\pm 0.25\%$, $\pm 0.5\%$ and $\pm 0.75\%$, respectively
 - For tests conducted with a hold time of 10 minutes, the variability factor for the 95% confidence interval bands was **3** and **2** for strain amplitudes of $\pm 0.5\%$ and $\pm 0.75\%$, respectively
 - For tests conducted with a hold time of 30 minutes, the variability factor for the 95% confidence interval bands was **5** for a strain amplitude of $\pm 0.5\%$

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