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Assessment of Negligible Creep, Off-Normal Welding and Heat Treatment of Gr91 Steel for Nuclear Reactor Pressure Vessel Application

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GEN IV Nuclear Energy Systems

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ABTRACT

Two different topics of Grade 91 steel are investigated for Gen IV nuclear reactor pressure vessel application. On the first topic, negligible creep of Grade 91 is investigated with the motivation to design the reactor pressure vessel in negligible creep regime and eliminate costly surveillance programs during the reactor operation. Available negligible creep criteria and creep strain laws are reviewed, and new data needs are evaluated. It is concluded that modifications of the existing criteria and laws, together with their associated parameters, are needed before they can be reliably applied to Grade 91 for negligible creep prediction and reactor pressure vessel design. On the second topic, effects of off-normal welding and heat treatment on creep behavior of Grade 91 are studied with the motivation to better define the control over the parameters in welding and heat treatment procedures. The study is focused on off-normal austenitizing temperatures and improper cooling after welding but prior to post-weld heat treatment.

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PART I: ASSESSMENT OF NEGLIGIBLE CREEP

- By Weiju Ren, Oak Ridge National Laboratory

PART II: EFFECTS OF OFF-NORMAL WELDING AND HEAT TREATMENT

- By Thomas Lillo and Terry Totemeier, Idaho National Laboratory

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PREFACE

The present report is composed of studies on two different topics of Grade 91 steel separately conducted for the Gen IV nuclear reactor pressure vessel application. The two topics are included in Part I for "Assessment of Negligible Creep", investigated by the Oak Ridge National Laboratory (ORNL), and Part II for "Effects of Off-Normal Welding and Heat Treatment", investigated by the Idaho National Laboratory (INL).

PART I

ASSESSMENT OF NEGLIGIBLE CREEP

By Weiju Ren

Oak Ridge National Laboratory

1. BACKGROUND FOR NEGLIGIBLE CREEP ASSESSMENT

In considering the design of reactor pressure vessel (RPV) for the Gen IV nuclear reactor systems, the RPV is envisioned of a size ranging from 19 to 24 m in height, 7.06 to 8.2 m in outside diameter, and 100 to 300 mm in wall thickness. The operating temperature is proposed to range from 300 to 495ºC with a transient temperature of 450 ~ 565ºC for the worst case accident; and the design life is targeted for 60 years. The Grade 91 steel, a. k. a. modified 9Cr – 1Mo, is identified as a leading candidate material for the construction [1].

In high temperature services, structural materials often experience thermal creep phenomenon that causes microstructural evolution, property deterioration, and to the worst, significant deformation resulting in functional or structural failure. Severity of such consequences depends on the load and temperature the material is subjected to. To prevent disastrous accidents in important structures serving at high temperatures, surveillance programs are usually employed to monitor creep damage evolution during long-term operations. In the Gen IV nuclear reactor systems, because the RPV size is exceptionally large and its service life extremely long, the surveillance plans required can be technically very complicated, and the implementation of such surveillance programs will be costly and labor intensive.

Significant studies on the creep phenomena over decades indicate that for a material under stress, creep occurs only when its driving force exceeds the threshold value of the material. In the nuclear reactor environment, the driving force can be either thermal energy or radiation energy. Thermal creep occurs when the stressed material is subjected to an elevated temperature that is high enough to provide sufficient thermal energy to motivate thermal creep mechanisms. In the same fashion, irradiation creep occurs only when the stressed material is subjected to a radiation dose that is great enough to provide sufficient radiation energy to motivate irradiation creep mechanisms. If the thermal energy or the radiation energy is restricted to a low level such that insufficient driving force is available to motivate the creep mechanisms, creep deformation of the material can be very limited and negligible.

Based on such considerations, if the RPV of the Gen IV nuclear reactor systems is designed to operate below the temperature and radiation levels that can provide sufficient energies to motivate creep mechanisms, creep deformation in the RPV can become very limited, and ideally, negligible. Since the RPV is operating in the negligible creep regime, expensive surveillance programs can be eliminated.

To explore the possibility of avoiding surveillance programs for monitoring the thermal creep damage of the RPV in Gen IV nuclear reactor operation, a task is established to investigate the thermal negligible creep of the prospective RPV material, the Grade 91 steel. The approach is to review available negligible creep criteria and determine whether they are applicable to the Grade 91 steel, examine existing creep data and the possibility of defining negligible creep based on the data, identify new data needs if any, generate necessary new data to verify and fill the gaps of existing data for negligible creep criterion development. To increase the efficiency and save the cost, collaboration is developed with Framatome-ANP under the Gen IV International Forum

(GIF) structure and through the activities of American Society of Mechanical Engineers (ASME) Codes and Standards Committees.

Regarding the irradiation creep, its occurrence in the RPV depends on the radiation dose received from different types of nuclear reactors. In a thermal reactor, the dose received by the RPV is usually very low and the irradiation creep, if any, is known to be negligible. For a fast reactor, irradiation creep should be evaluated due to the high radiation dose to the RPV. Because the leading Gen IV reactor concept, the Very High Temperature Reactor, is a thermal reactor, the irradiation creep issue will not be covered in the present investigation.

2. MATERIAL

The Grade 91 steel was developed as a creep resistant steel from the standard 9Cr-1Mo steel in an attempt to replace stainless steels for liquid metal reactor (LMR) applications. The development was started in 1975. After 5 years, a modified 9Cr-1Mo steel was shown to achieve creep properties equal to type 304 austenitic stainless steel up to 590°C with adequate weldability. As compared to the standard 9Cr-1Mo chemical composition, the modification included additional requirements for V, Nb, N, Al, and Ni. At the conclusion of the development, the chemical composition as shown in Table 1 was recommended for commercialization. The recommended commercialization composition included an aim value and an allowed range for each element [2]. Depending on product forms, the chemistry of the ASTM and ASME specifications has slight variations from that in Table 1. To ensure the desired microstructures and properties, requirements for heat treatment and welding were also developed through significant experiments. A typical heat treatment would be 1040ºC for 1 hour per inch of thickness, then fan or spray cooling depending on the thickness, followed by tempering at 760ºC for 1 hour per inch of thickness. Procedures and parameters for various heat treatment and welding processes were well developed in fossil energy programs [3].

After its commercialization, Grade T91 was first approved for use in Section I of the ASME Boiler and Pressure Vessel Code in 1984. In February 1993, the ASME Power Piping Code, B31.1, approved Grade 91 for seamless pipe in Specification SA-335 and plate in Specification SA-387. At present, the material is also permitted by ASME Boiler and Pressure Vessel (B&PV) Code Section III Division 1 Subsection NB for Class 1 components operating up to 371°C. For Class 1 components in elevated temperature services, Subsection NH provides additional rules for application above the 371ºC limit.

Table 1: The original commercial chemical composition for modified 9Cr-1Mo steel

	C	**Mn**	**Si**	**P**	**S**	**Cr**	**Ni**	**Mo**	**Cu**	**V**
Minimum	0.08	0.30	0.20	-	-	8	-	0.85	-	0.18
Maximum	0.12	0.50	0.50	0.02	0.01	9	0.2	1.05	0.2	0.25
Target	0.10	0.40	0.20	<0.01	<0.01	8.5	<0.10	0.95	<0.10	0.21
	Nb	**N**	**Al**	**Ti**	**B**	**W**	**Zr**	**O**	**Sb**	
Minimum	0.06	0.03	-	-	-	-	-	-	-	
Maximum	0.10	0.07	0.04	0.01	<0.001	<0.01	<0.01	<0.02	<0.001	
Target	0.08	0.05	<0.02	<0.01	<0.001	<0.01	<0.01	<0.02	<0.001	

3. EVALUATION AND DEVELOPMENT OF NEGLIGIBLE CREEP CRITERIA

To investigate negligible creep of Grade 91 for nuclear reactor RPV application, the negligible creep of the material for ASME B&PV Class 1 components must first be defined. In this effort, criteria for determining negligible creep behavior in the Class 1 components are reviewed and those with potential applicability to Grade 91 are evaluated. In ASME Code Section III Division 1 Subsection NB, metal temperature limits are used as the criteria for not considering creep effects in design and analysis. It should be noted that the metal temperature is used in these criteria to differentiate from environmental temperature around the metal. It is believed that when the materials are serving at temperatures below these threshold limits, insufficient thermal energy is available to motivate creep mechanisms and cause the creep damage. These temperature limits are given in Section II Part D Table 2A as summarized in Table 2.

Table 2: ASME B&PV Code temperature limits for not considering creep effects

Material	**Temperature Limit (°C)**
Carbon steel and low alloy steel	370
Martensitic stainless steel	370
Austenitic stainless steel	425
Nickel-chromium-iron steel	425

It is stipulated in ASME B&PV Code that above those temperature limits, creep effects should be considered, and the design rules with the consideration of creep effects are provided in Section III Division 1 Subsection NH. The rules are developed for evaluating the following failure modes mostly related to the creep damage:

- Ductile rupture from short term loading
- Creep rupture from long-term loading
- Creep-fatigue failure
- Ratcheting
- Buckling and creep buckling.

It should be pointed out that the temperature limits listed in Table 2 are more or less a rule of thumb. They are given just to facilitate engineering judgment when determining the necessity for creep related evaluations in design and analysis. At temperatures above these limits, creep effects may still not be considered under certain circumstances. To cope with these circumstances, some nonmandatory rules for strain, deformation, and fatigue limits at elevated temperatures are provided in Appendix T of Subsection NH. It is considered an acceptable practice for T-1324 Test No. A-3 under T-1300 "Deformation and Strain Limits for Structural Integrity" that a detailed creep-fatigue evaluation is not required if the limits of the test, as shown in Equation (1) and Equation (2), are satisfied. In other words, the creep effects can be deemed negligible. Equation (1) is a time fraction criterion, and Equation (2) is a strain criterion.

The limit of the time fraction criterion in T-1324 Test No. A-1 is presented as follows:

$$\sum_i \frac{t_i}{t_{id}} \le 0.1 \tag{1}$$

where: t_i = total duration of time during the service lifetime that the metal is at temperature T_i. Note that the service lifetime shall never be greater than the sum of all t_i.

t_{id} = maximum allowable time as determined by entering Figs. I-14.6 at temperature T_i and a stress value of 1.5 times the yield stress S_y associated with T_i, denoted as $1.5S_y|T_i$. If $1.5S_y|T_i$ is above the stress values provided in Figs. I-14.6, this test cannot be satisfied. When $1.5S_y|T_i$ is below the lowest stress value provided in Figs. I-14.6, the constant temperature line may be extrapolated to larger t_{id} values using the steepest slope on Figs. I-14.6 for that material. Here, Figs. I-14.6 refer to the series of figures in ASME Code Subsection NH.

For Grade 91, the stress vs. rupture time curves provided in the Code as shown in Fig. 1 should be used to determine t_{id}. To facilitate analysis, the curves are also tabulated in the Code as shown in Table 3.

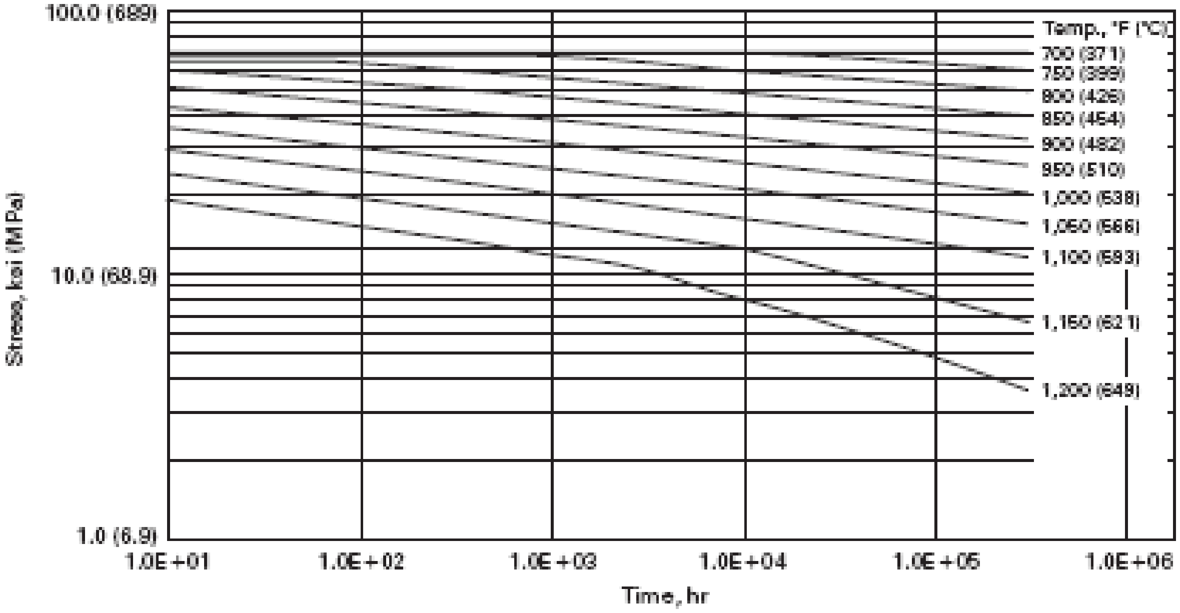
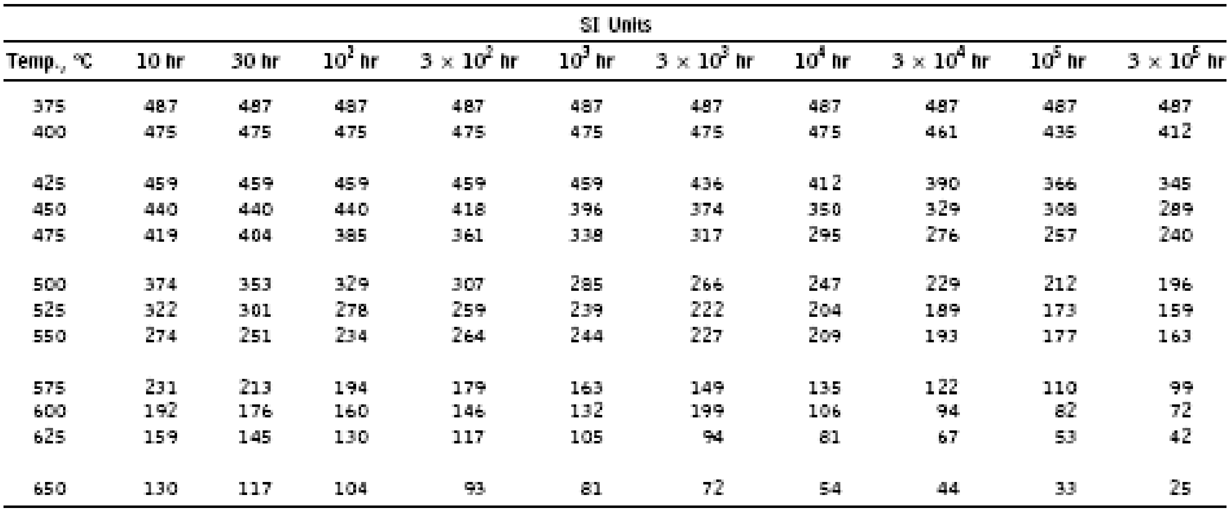


Fig. 1: Expected minimum stress-to-rupture for 9Cr-1Mo-V in ksi (MPa)
(Source: FIG. I-14.6F of Subsection NH Mandatory Appendix I-14)

Table 3: Expected minimum stress-to-rupture values for 9Cr-1Mo-V in MPa



SI Units										
Temp., °C	10 hr	30 hr	10^2 hr	3×10^2 hr	10^3 hr	3×10^3 hr	10^4 hr	3×10^4 hr	10^5 hr	3×10^5 hr
375	487	487	487	487	487	487	487	487	487	487
400	475	475	475	475	475	475	475	461	435	412
425	459	459	459	459	459	436	412	390	366	345
450	440	440	440	418	396	374	350	329	308	289
475	419	404	385	361	338	317	295	276	257	240
500	374	353	329	307	285	266	247	229	212	196
525	322	301	278	259	239	222	204	189	173	159
550	274	251	234	264	244	227	209	193	177	163
575	231	213	194	179	163	149	135	122	110	99
600	192	176	160	146	132	199	106	94	82	72
625	159	145	130	117	105	94	81	67	53	42
650	130	117	104	93	81	72	54	44	33	25

Source: TABLE I-14.6F of Mandatory Appendix I-14

The limit of the strain criterion in T-1324 Test No. A-3 is presented as follows:

$$\sum_i \varepsilon_i \leq 0.2\% \qquad (2)$$

where: ε_i = the creep strain that would be expected from a stress level of $1.25S_y|T_i$ applied for the total duration of time during the service lifetime that the metal is at T_i. When the design lifetime is separated into several time periods, then the service lifetime shall not be greater than the sum of all the time periods. That is:

$$\sum_i t_i|T_i \geq \text{service lifetime} \qquad (3)$$

In the discussion above, the yield stress, S_y, is employed for both Equation (1) and Equation (2) as a base for the reference stresses, $1.5S_y$ and $1.25S_y$. As a matter of fact, the use of S_y as a base for the reference stresses to determine negligible creep is also more or less a rule of thumb for convenient engineering judgment. Depending on materials, other parameters, such as the allowable stress S_m that is closely related to minimum tensile curve, can also be used as a base for the reference stresses. Similarly, the reference strain, 0.2%, employed in Equation (2) can be replaced by other strain values as well, depending on materials and magnitude of conservatism or risk involved in the criterion.

The values of S_y for Grade 91 are summarized in Table 4 for temperatures below and at 525ºC, and in Table 5 for temperatures above 525ºC, together with the calculated reference stresses. All data are also plotted in Fig. 2.

Table 4: S_y and calculated reference stresses for temperatures below and at 525ºC

TºC	S_y, MPa	$1.25S_y\|T_i$, MPa	$1.5S_y\|T_i$, PMa
25	414	517.5	621
65	394	492.5	591
100	384	480	576
125	380	475	570
150	378	472.5	567
175	378	472.5	567
200	377	471.25	565.5
225	377	471.25	565.5
250	377	471.25	565.5
275	377	471.25	565.5
300	377	471.25	565.5
325	375	468.75	562.5
350	371	463.75	556.5
375	366	457.5	549
400	358	447.5	537
425	348	435	522
450	337	421.25	505.5
475	322	402.5	483
500	306	382.5	459
525	288	360	432

Source: ASME B&PV Code Section II Part D Table Y-1

Table 5: S_y and calculated reference stresses for temperatures above 525ºC

TºC	S_y, MPa	$1.25S_y\|T_i$, MPa	$1.5S_y\|T_i$, PMa
550	269	336.25	403.5
575	243	303.75	364.5
600	218	272.5	327
625	193	241.25	289.5
650	165	206.25	247.5

Source: ASME B&PV Code Section III Subsection NH Mandatory Appendix I-14, Table I-14.5

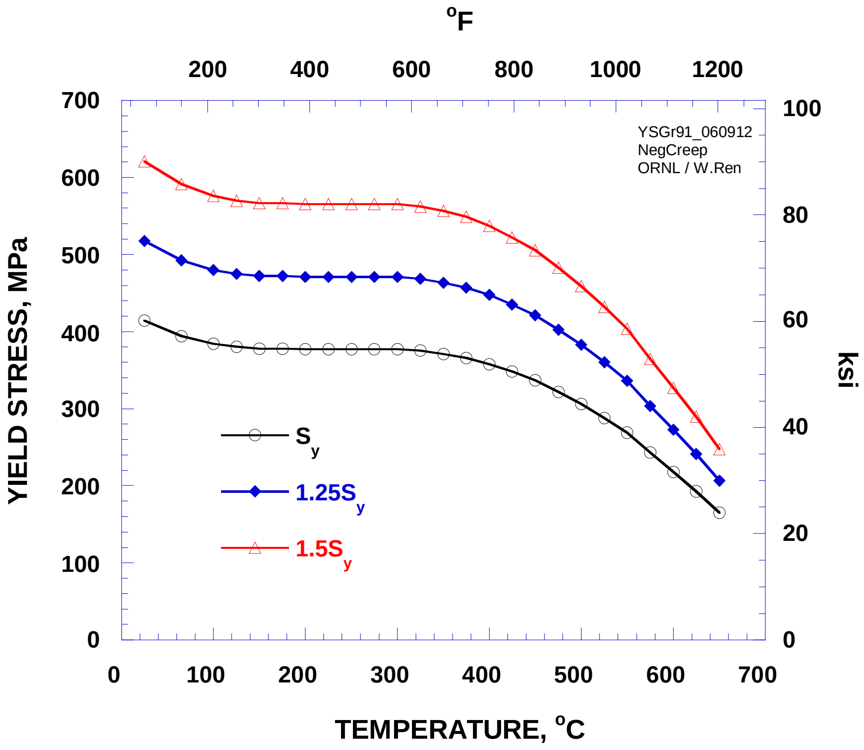


Fig. 2: S_y and the corresponding reference stresses for evaluating negligible creep in Grade 91

To provide a convenient tool for evaluating negligible creep in Grade 91, a negligible creep curve should be developed using Equation (1) or Equation (2). To use Equation (1), $1.5S_y|T_i$ in Table 4 and Table 5 is first employed to determine the value of t_{id} from Fig. 1 or Table 3 at the corresponding temperature T_i. Then the value of t_i, which represents the time durance for negligible creep, can be determined through Equation (1). With a series of $T_i \sim t_i$ values, a negligible creep curve for Grade 91 can be plotted. Creep under any combination of T_i and t_i below the curve can be considered negligible.

In conducting the above operations, it becomes unfortunately obvious that all the values of $1.5S_y|T_i$ listed in Table 5 exceed the corresponding values in Table 3 and Fig. 1 at the given temperatures. For example, at 375ºC, $1.5S_y|T_i$ has the value of 549 MPa according to Table 4. It exceeds the lowest value of 487 MPa for 375ºC in Table 3 and Fig. 1. This suggests that with the given reference stress, $1.5S_y|T_i$, Equation (1) is not applicable to Grade 91 for establishing the negligible creep curve.

To establish a negligible creep curve for Grade 91 using Equation (2), $1.25S_y|T_i$ in Table 4 and Table 5 can be used to determine the value of t_i for the reference strain of 0.2% at T_i from average isochronous stress-strain curves of Grade 91. Some average isochronous stress-strain curves of Grade 91 are provided in FIG. T-1800-E-1 ~ FIG. T-1800-E-11 in Subsection NH. However, these curves include the loading strain. Without the value of the loading strain, it is difficult to derive the needed information. To avoid this inconvenience and to facilitate the calculation, creep strain laws and parameters for Grade 91 introduced in Reference [4] are used as follows:

$$e_c = a_o \cdot t^{1/3} + mcr \cdot t \qquad (4)$$

where: e_c = creep strain (%);
a_o = primary creep strain constant (%/$h^{1/3}$);
mcr = minimum creep rate (%/h)

For the present calculation, the creep strain, e_c, is the reference strain with the value of 0.2%. The a_o and mcr are calculated using the equations below:

$$mcr = C \cdot S^n \exp(V \cdot S) \cdot \exp(-Q/T) \qquad (5)$$

$$a_o = D \cdot S \cdot \exp(V_o \cdot S) \cdot \exp(-Q_o/T) \qquad (6)$$

The parameters in Equations (5) and (6) are given in Table 6.

Table 6: Parameter values for the creep equation

Parameter	**Value**	**Unit**	**Applicable Temperature, ºC**
D	5450000	% / $h^{1/3}$	538 ~ 649
D	847000	% / $h^{1/3}$	427 ~ 482
Q_o	23260	K	538 ~ 649
Q_o	25330	K	427 ~ 538
V_o	0.023	1 / MPa	427 ~ 649
C	2.25×10^{22}	% / h	427 ~ 649
n	5	-	427 ~ 649
V	0.038	1 / MPa	427 ~ 649
Q	77280	K	427 ~ 649

Based on the equations above, a negligible creep curve is developed for Grade 91 as shown in Fig. 3. It should be noted that the lowest temperature covered in Table 6 is 427ºC. Extrapolation is conducted to calculate the portion of the curve below this temperature, as indicated in blue in Fig. 3.

Beside the above equations, parameters, and data that may be used to develop negligible creep curve for Grade 91, other potential equations and parameters also exist, for example, the RCC-MR Code rules used by the French. Assessment indicates that some of the equations and their parameters, such as Equation (1) and its reference stress discussed above, may be applicable for some materials, but not for Grade 91. However, if the equations or their parameters can be properly modified based on sound technical grounds, optimized negligible criteria or curves can still be developed. For example, the reference stress can be chosen from $1.25S_y$, $1.5S_y$, and $2S_m$, while the reference strain chosen from 0.2%, 0.01% etc. Because S_y and S_m are the basic measurements of material strength, and creep strains are the measurements of magnitude of damage, it is an engineering judgment about how much creep damage relative to the strength of the material should be considered negligible when the reference stress and strain are selected. Of course, the judgment must be supported with sufficient test data and sound logical analysis.

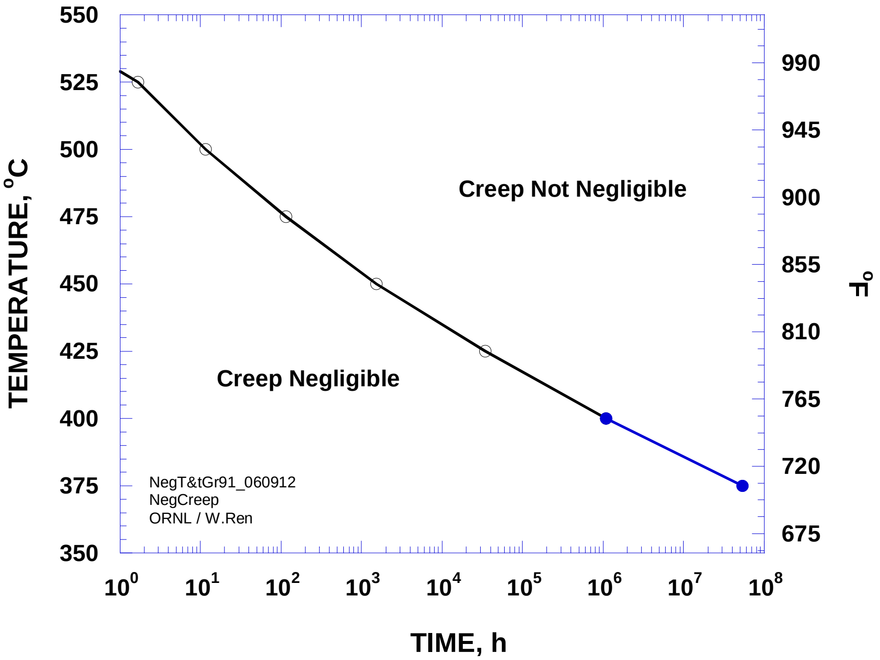


Fig. 3: Negligible creep curve of Grade 91 developed based on Equation (2)

To assess the effect in choosing different equations and reference parameters to define the negligible creep, a negligible creep curve is developed using Equation (1) with S_m as the base for reference stress, $2S_m|T_i$. The S_m is the lowest stress intensity value at a given temperature, T_i, among the time-independent strength quantities which are defined in Section II, Part D. The values of S_m at various T_i, $S_m|T_i$, and the calculated reference stress $2S_m|T_i$ are plotted in Fig. 4. The negligible creep curve is developed and compared in Fig. 5 with that derived from Equation (2). Again, the lowest portion is extrapolated as indicated in brown. Fig. 5 clearly demonstrates that with S_m as the base for the reference stress, the negligible creep curve developed from the time fraction criterion, Equation (1), is different from, but comparable to that developed from the strain criterion, Equation (2).

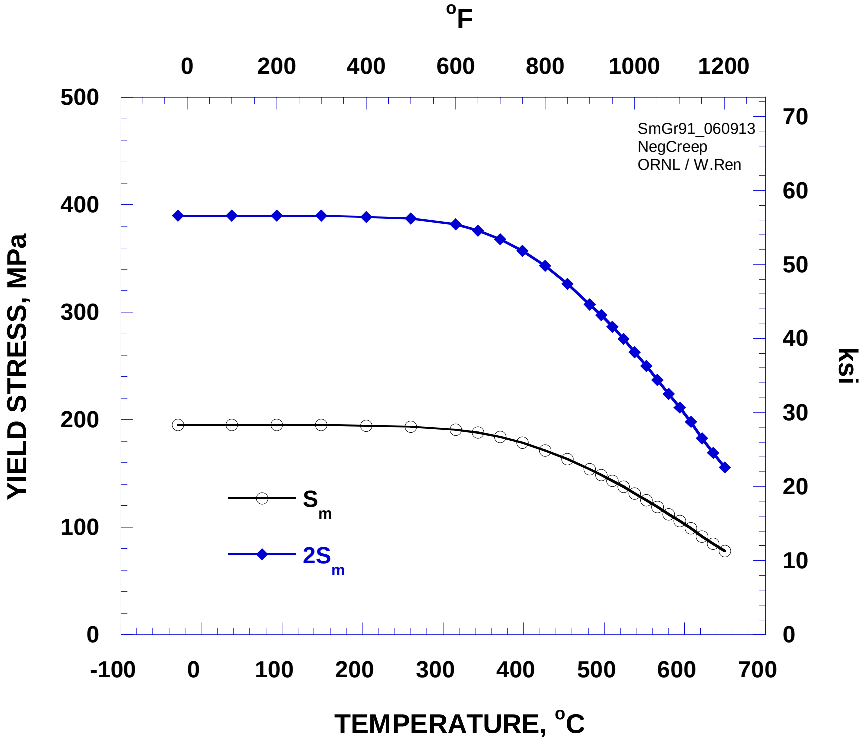


Fig. 4: S_m and the corresponding reference stress for evaluating negligible creep in Grade 91

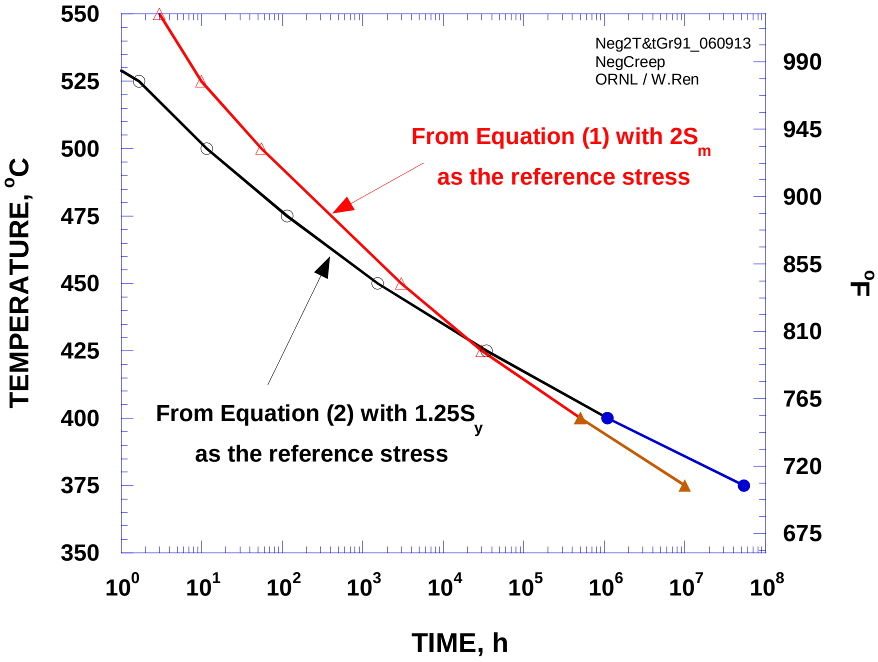


Fig. 5: Negligible creep curve of Grade 91 developed based on Equation (1)

4. ASSESSMENT OF EXISTING CREEP DATA AND DATA NEEDS AT LOW TEMPERATURE AND LONG TIME

It is exhibited in Fig. 3 and Fig. 5 that due to the lack of data in the low temperature and long time regime, extrapolation has to be conducted in developing the negligible creep curves. This observation raises several concerns: First, it is reasonable to question the reliability of the extrapolation. Secondly, if existing creep laws per se are developed based on extrapolation, the applicable region of these creep laws is also questionable. Third, although theoretically a threshold temperature should exist for negligible creep, could this temperature be too low such that the efficiency of the reactor must be compromised?

To address these concerns, existing creep data of Grade 91 are collected for analysis. Creep behavior at low temperature and long time is reviewed. Some creep laws are compared with existing creep data.

The collected creep data are summarized in Table 7. Times marked with D represent the times for test discontinuation. The lack of low temperature data is apparent. Only two tests were conducted at 427ºC. Most of the tests were performed at temperatures above 500ºC, where creep is clearly not negligible. Among the tests listed in Table 7, some original creep curves are collected for analysis. Search is still underway for more original curve data.

To assess the creep behavior at low temperature, the creep curves of the two tests at 427ºC are plotted in Fig. 6. The two tests were conducted at 379 and 414 MPa, corresponding to 83% and 91% of the minimum ultimate tensile strength, respectively. Both tests were conducted on specimens from the same heat (Heat No. 30394). Comparison of the two curves in Fig. 6 indicates that the strain at 414 MPa is more than twice of that at 379 MPa. However, despite of the significant difference in strain, both tests exhibited a trend towards insignificant creep strain accumulation after long testing times. It seems that creep strain hardening occurred in the material after certain amount of creep strain accumulation. It is possible that after the initial creep deformation, the creep mechanisms that only needed low activation energy were exhausted. Higher energy was required to overcome the obstacles to continue creep deformation or further motivate more difficult mechanisms. If this is true, a possibility exists that a short excursion of temperature may help overcome the obstacles and reactivate the initial creep mechanisms to continue creep deformation even after the temperature is returned to the previous level. This possibility should be verified through experiments, and be considered in negligible creep criterion development and the RPV design, if proven true. The experimental verification should also include metallurgical investigation to provide an understanding of the creep cavitation mechanisms in the negligible creep temperature regime. At present, an investigation supported by the ORNL Laboratory Director's R&D (LDRD) Fund is being conducted to study creep cavitation mechanisms in materials including Grade 91 using small-angle neutron scattering and computational modeling [5]. The results will be incorporated into the metallurgical characterization for the present negligible creep investigation.

To compare existing data with available creep laws, the creep times for 0.1% creep strain at 472ºC are derived from the two tests presented in Fig. 6, and plotted with creep strain laws from

References [4] and [6] in Fig. 7. Comparison of the two curves indicates that at the same stress level, the ASME creep strain law predicts much longer time for accumulating 0.1% creep strain at 472ºC. For example, at 500 MPa, the predicted creep time from the ASME curve is approximately 2 orders longer than that from the ORNL curve. This indicates that the ORNL creep strain law is more conservative than the ASME creep strain law. Because both laws were developed by ORNL personnel, judged from the publication time, the ASME creep strain law (published in 1999) should have contained more experimental information, especially the information from longer creep tests, therefore be considered more reliable than the ORNL creep strain law (published in 1987). However, when compared with the two test data points, both curves seem to have provided reasonably good predictions. This observation suggests that either the material is very sensitive to difference in load at 472ºC, or there is serious data scatter from some currently unknown reason. It is obvious that more tests are needed to provide a better understanding of the creep behavior at this temperature regime. And the creep strain laws should be further evaluated for application to Grade 91.

To further compare the creep strain laws and the test data, the ASME and ORNL creep strain curves are plotted with test data at 482ºC in Fig. 8. The ASME curve again predicts creep time approximately 2 orders longer than the ORNL curve does. Compared with the test data points, the ORNL curve obvious agrees much better with the test data at short times than that at long time. This observation more or less supports the speculation that the ORNL creep strain law might have been developed mainly based on short-term test data. It can also be observed from Fig. 8 that the slop demonstrated by the test data points is less steep than that of both curves. This suggests that both creep strain laws may be too pessimistic. However, because there is only one data point at long time, verification is definitely needed by conducting more long-term tests.

Table 7: Summary of the collected existing data on Grade 91

Test No.	Heat No.	Temperature, ºC	Stress, MPa	Time, h
23698	394E	427	379	106523D
21882	394B	427	414	132539D
25424	383B	450	427	2492D
22770	394E	454	379	75645.7
24632	383B	475	350	480.9D
24663	383B	475	350	1916D
24749	394E	482	276	83491D
24772	148A	482	276	83126D
24752	176	482	276	84147D
25936	176	482	414	35.4
4508	182A	482	365	1332
24746	383B	482	276	66470D
21751	5349Y	482	379	151.2
21752	5349Y	482	414	26.8
25317	176	500	448	1638.4
25644	176	500	469	637.2
25663	176	500	496	53.7
23495	383B	500	276	10003D

Test No.	Heat No.	Temperature, ºC	Stress, MPa	Time, h
23566	383B	500	276	2831D
23643	383B	500	240	13532D
23645	383B	500	240	11611D
23738	383B	500	200	5080D
23740	383B	500	200	1028D
23758	383B	500	200	8000D
23789	383B	500	166	5108D
23835	383B	500	370	300
23858	383B	500	200	7900D
23935	383B	500	370	300
23953	383B	500	351	1195
24290	383B	500	310	209D
24438	383B	500	310	670D
24645	383B	500	310	1489D
28183	383B	500	140	5285D
20842	-	538	179	101540
21769	-	538	186	84310
24351	394E	538	207	32460
24820	-	538	166	83348
24847	-	538	166	83050
24899	-	538	166	82707
25371	-	538	83	47516
24357	30176	538	207	32212
24359	30176	538	186	32109

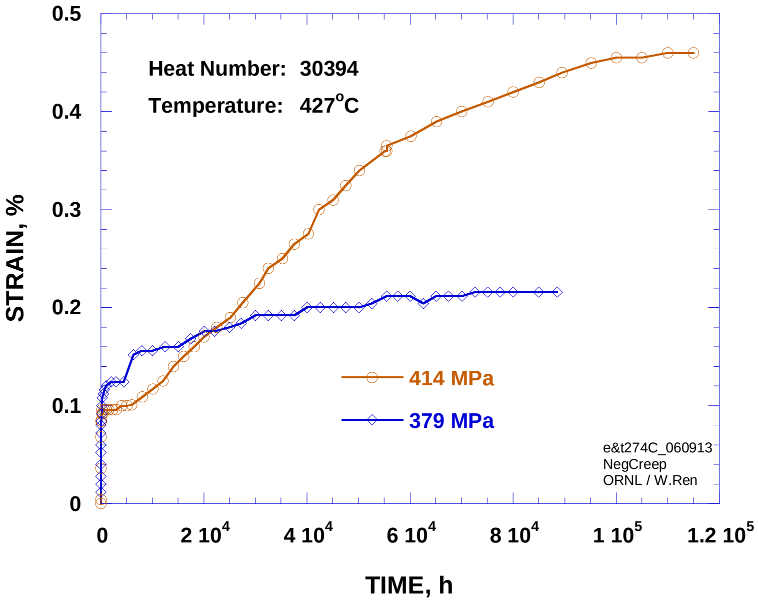


Fig. 6: Creep curves of Grade 91 Heat 30394 at 427ºC

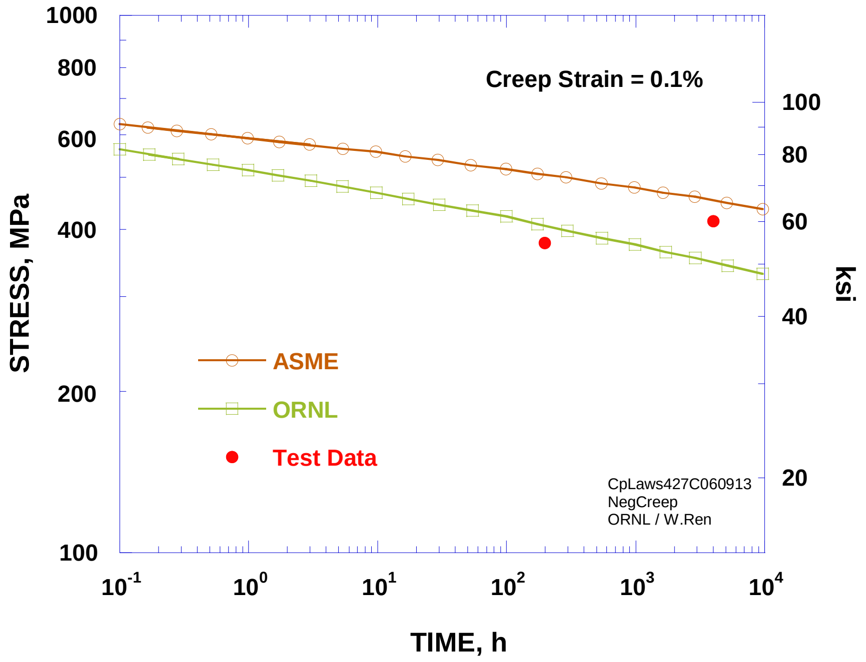


Fig. 7: Comparison of creep strain laws and creep test data at 427ºC of Grade 91

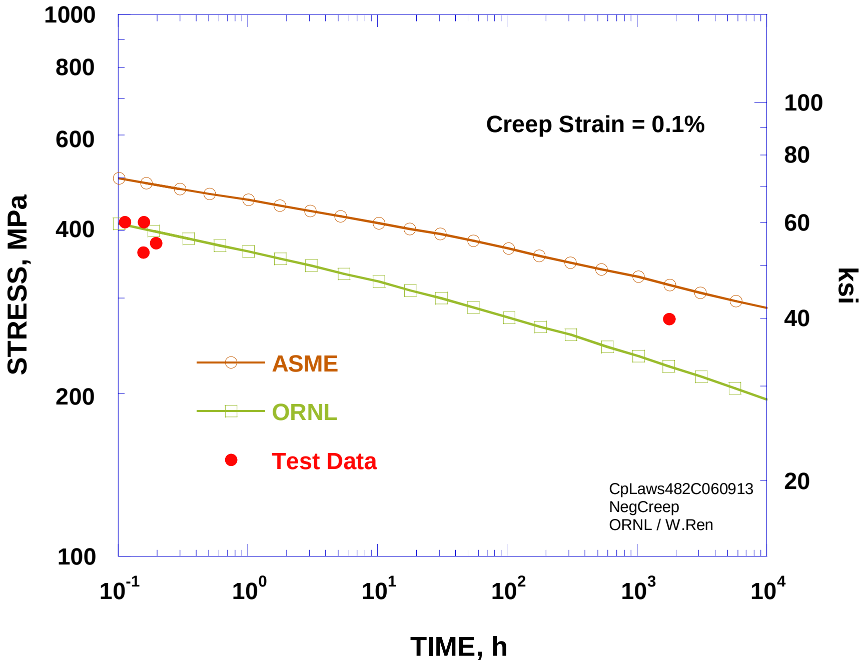


Fig. 8: Comparison of creep strain laws and creep test data at 482ºC of Grade 91

5. PREPARATIONS FOR DATA GENERATION

To generate new data of Grade 91 needed in the low temperature range, a total of 10 creep machines have been under preparation during FY06, as shown in Fig. 9. The large round vessels seen in Fig. 9 are low temperature furnaces left from a previous program on automotive polymer composites. They will be replaced by tube furnaces for testing of Grade 91 in FY07. At present, all the major components have been designed and/or specified. Most of the component acquisitions have been completed. Installation will start as soon as funding becomes available in FY07.

Fig. 9: Creep machines under preparation for negligible creep testing

Because most of the tests will be conducted at low temperatures in the range of 425 ~ 450ºC with low stresses, specimen alignment will become an important factor that may introduce significant error, and therefore must be strictly controlled. Several measures have been taken to minimize possible bending strains resulting from misalignment. U-joints have been installed on both top and bottom of the load train. The specimen is designed with a relatively large gage cross section area so that large load can be applied to facilitate the alignment. Further, the large specimen gage perimeter allows three strain gages to be installed at 120 degrees to each other around the gage section. Procedures will be developed to load the specimen at room temperature up to 10% of the prescribed testing load to facilitate minimizing misalignment through load train adjustment under the surveillance of the three strain gages. The specimen is also designed with a long gage length to facilitate detecting minimal creep strains during the testing. A schematic of the specimen design is shown in Fig. 10.

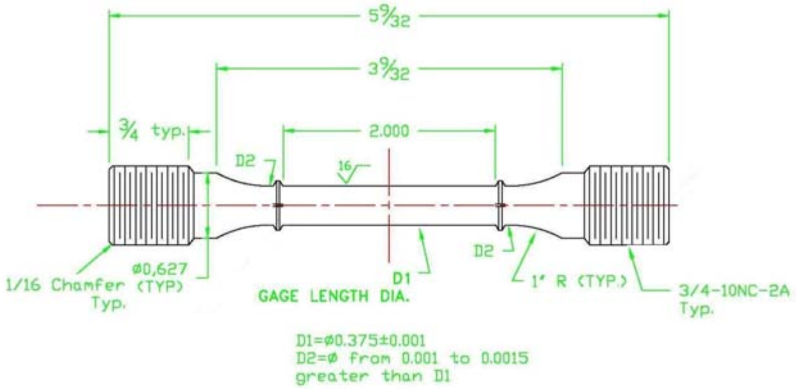


Fig. 10: Specimen design for negligible creep testing

To prepare the material for specimens, a 220 mm thick block of Grade 9, shown in Fig. 11, has been acquired. The material has been identified to be from Heat 30176. The thickness of the material (220 mm) falls into the mid range of the proposed reactor pressure vessel wall thickness (100 to 300 mm), and is therefore considered ideal for investigating negligible creep phenomenon in the intended application.

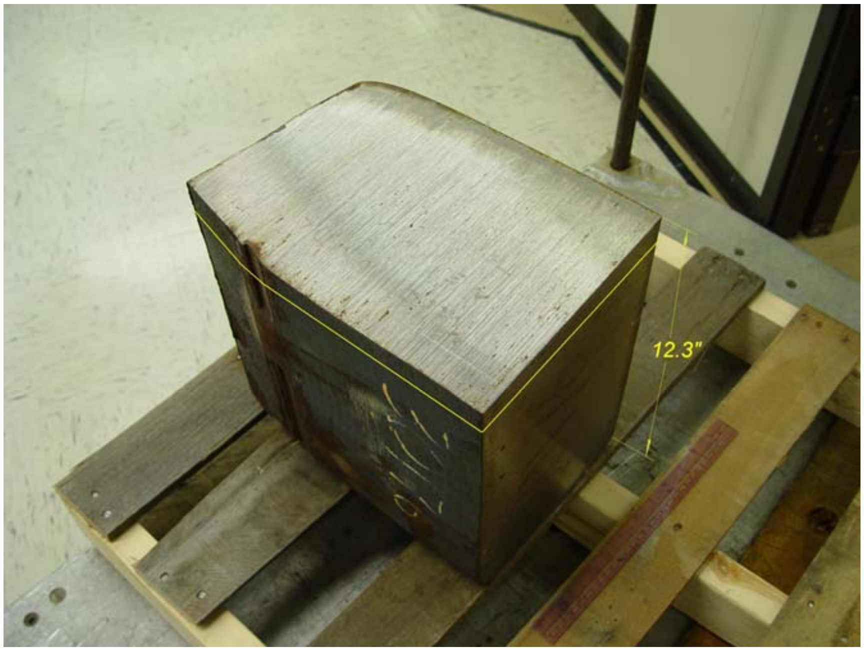


Fig. 11: Grade 91 material acquired for negligible specimen preparation

Because the development of Grade 91 steel was mainly led by the ORNL, a significant amount of information produced in the past can still become available. In FY06, a search for relevant information and data was initiated. The effort reveals that in addition to a 10 years (1975 ~ 1985) summary report on the Grade 91 development program [2] and many other documents, a lot of unpublished information also exists. Further, the material has been aged for 5,000, 10,000, 25,000, 50,000, and 75,000 hours at 482, 538, 593, 649, 704ºC in lab, and more than a decade at several temperatures at Kingston and Tanner's Creek Plants. The information and material will be further collected and used for the present investigation in the future.

As previously mentioned, the present investigation is collaborated with Framatome-ANP under the GIF structure and through the activities of ASME Codes and Standards Committees. Because the testing for negligible creep is a very time-consuming process and the turnover rate of creep testing machines is very low, the collaboration can significantly help reduce research time and cost. At present, some creep machines are also under preparation by the Commissariat à l'Energie Atomique (CEA) for negligible creep testing. It has been agreed that a collaborated test matrix will be developed for data generation. Some preliminary test conditions have already been discussed for the collaboration and a tentative test matrix has been agreed upon by both sides, as shown in Table 8. The simulated post-weld heat treatment (PWHT) conditions in Table 8 are still to be defined. As progress is made in the testing and more information becomes available, the tentative test matrix may be modified to meet new needs and requirements. Possible modification may be an expansion in testing temperatures at the low end.

Table 8: Tentative test matrix for collaboration in negligible creep data generation

Temperature (°C)	**Stress (MPa)**	**Product Conditions**	**Comments**
425	400	As received Simulated PWHT Simulated PWHT + aged	Evaluate the stationary creep rate (which should hopefully be zero). If aged material in as received condition, it might be necessary to foresee tests in three conditions for both stresses: as received, as received + aged, and simulated PWHT.
	375	Simulated PWHT Simulated PWHT + aged	
450	425 (*)	As received Simulated PWHT	(*) Optional tests to failure, to be performed if tests at 400 MPa stopped before failure
	400	As received	Test to failure or stopped at 15,000 h
		Simulated PWHT	Test to failure or stopped at 15,000 h
	375	Simulated PWHT	Target : 1 % creep strain
	350	As received	Target : 0.5 % creep strain
		Simulated PWHT	Target : 0.5 % creep strain
	325	Simulated PWHT	Target : 0.2 or 0.5 % creep strain
475	375 (*)	As received Simulated PWHT	(*) Optional tests to failure, to be performed if tests at 350 MPa stopped before failure
	350	As received	Test to failure or stopped at 15,000 h
		Simulated PWHT	Test to failure or stopped at 15,000 h
	325	Simulated PWHT	Target : 1 or 2 % creep strain
	300	As received	Target : 0.5 or 1 % creep strain
		Simulated PWHT	Target : 0.5 or 1 % creep strain
	275	Simulated PWHT	Target : 0.2 or 0.5 % creep strain

6. SUMMARY AND FUTURE WORK FOR NEGLIGIBLE CREEP ASSESSMENT

The present assessment on existing creep rules and available test data suggests that negligible creep behavior in Grade 91 may be expected in the proximity of 425ºC. However, there is a lack of experimental data in this temperature range for verification. The assessment also indicates that neither negligible creep criteria nor creep strain properties available in nuclear ASME Code are particularly applicable to Grade 91. Further verifications and modifications are needed for the creep strain laws, negligible creep criteria and associated parameters in the temperature domain of 425 ~ 500ºC so that they can become more reliable for the Grade 91 application. Expansion of the investigation into lower temperatures may also be expected depending on the initial results. It is apparent that all the verifications and modifications require generation of new data, especially the data at low temperature regime and long test times. A total of 10 creep machines have been under preparation in FY06 for the data generation, and the preparation is close to completion. Agreement has been reached on a tentative test matrix with Framatome-ANP, and the data generation will be conducted in close collaboration to save time and cost. The material of Grade 91 for the data generation has been acquired in FY06. Heat treatment of the material, machining of specimens, and completion of the final machine preparation will start as soon as funds become available in FY07. Because of the long-term test times required, the testing will be started at the earliest possible time in FY07. Once sufficient data becomes available, the negligible creep curve and creep strain law together with associated parameters particularly for Grade 91 will be developed.

Mechanical metallurgy investigation will also be conducted on the tested creep specimens to provide a mechanistic understanding of the microstructural evolution, creep mechanisms, and investigate the possibility of change of creep mechanisms in the temperature domain of 425 to 500ºC and above. The investigation will be collaborated with an on-going LDRD program studying the creep cavitation mechanisms of materials including Grade 91 using small-angle neutron scattering and computational modeling. The metallurgical investigation will also include effects of post weld heat treatment (PWHT) on negligible creep conditions.

PART II

EFFECTS OF OFF-NORMAL WELDING AND HEAT TREATMENT

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Idaho National Laboratory

7. OBJECTIVE OF OFF-NORMAL WELDING AND HEAT TREATMENT STUDY

Preliminary studies at the INL indicated that certain off-normal heat treatments (such as those that occur near weldments or during fabrication in the field) can result in seriously impaired long-term creep strength without clear indications from room temperature tests and observations. Field fabrication of components, such as high temperature piping and pressure vessels, will likely be required and the level of control over the heat treatment parameters will have to be specified. Research has been performed to study the effects of off-normal heat treatment and off-normal welding procedures on creep behavior to better define the control needed over these parameters.

Work began in March of 2006 and has focused on off-normal austenitizing temperatures and improper cooling after welding, prior to performing the post-weld heat treatment (PWHT). The results and progress during the past six months are summarized below.

8. RESULTS AND PROGRESS

Two large Grade 91 plates, 48” x 48” x 0.75”, were procured from All Metals & Forge (Parsippany, NJ), Heat No. U8552 8A, in the normalized and tempered condition (1040^{o}C, 33 minutes followed by 788^{o}C, 80 minutes). The chemical composition of the plates is given in Table 9.

Table 9: Grade 91 Plate Composition

C	**Mn**	**P**	**S**	**Cu**	**Si**	**Ni**	**Cr**	**Mo**	**V**	**Ti**	**Al**	**Nb**	**N**
0.09	0.46	0.01	0.002	0.13	0.27	0.22	8.40	0.89	0.21	0.002	0.01	0.08	0.041

Samples for the welding and off-normal austenitizing work were cut from these plates.

8.1 Off-Normal Welding Procedures and Associated Creep Behavior of Grade 91 Steel

Field fabrication of components such as pressure vessels will require extensive welding, most likely gas tungsten arc welding (GTAW). High temperature welding processes will require heat treatment after welding to obtain maximum creep properties. Standard welding procedures for this alloy require the weld to fully cool below the martensite finish temperature (M_f) to produce a completely martensitic microstructure which is then tempered. This tempering heat treatment also induces precipitation and the development of the required creep properties. The martensite finish temperature is fairly low for this particular alloy, ~160^{o}C. Normal procedures require the weld to cool to room temperature to ensure the entire microstructure has transformed to martensite. However, in the field, it is possible that the PWHT (760^{o}C for 2 hours) may commence prior to the weld reaching the M_f. In this case, the remaining austenite transforms to ferrite during the PWHT. The presence of ferrite in the final microstructure will degrade the creep resistance. This work was designed to evaluate the effect of ferrite on creep properties. Samples were, therefore, heat treated to develop various volume fractions of ferrite in the final microstructure. The relationship between ferrite volume fraction and creep properties could then be evaluated.

This work looks at the creep behavior and microstructural development of improperly cooled welds, in both the cross-weld and longitudinal configurations – i.e. creep samples that contained either base metal, heat affected zone (HAZ) and weld metal or were composed of entirely weld metal, respectively. Due to the complexity of the microstructure in the cross-weld samples, bulk grade 91 material was heat treated to simulate different areas in the cross-weld samples.

8.1.1 Simulated HAZ and weld metal

The temperature history in the HAZ is a function of distance from the weld and, therefore, the microstructure and the creep properties can also vary as a function of the distance from the weld. In an effort to discern the critical microstructural features giving rise to early creep failure in the HAZ, material was heat treated so as to simulate the thermal history at different locations in the HAZ. Subsequent creep testing and microstructural evaluations will help determine the effects of improper cooling after welding.

Samples that were to simulate the HAZ material were instrumented with thermocouples to continuously monitor the sample temperature during heat treatment. The samples were then inserted into a furnace held at the upper temperature. It was held there for a short period of time (~5 minutes), removed the furnace, allowed to air cool to the lower temperature, e.g. 175-350^{o}C (i.e. improper cooling), and then immediately subjected to the PWHT (760^{o}C, 2 hrs). Two upper temperatures were used – 835^{o}C, which is slightly above A_{c1} (~800^{o}C) and 925^{o}C, which is slightly above A_{c3} (~900^{o}C) – to simulated HAZ material. Samples that were to simulate the weld metal were also included in this study and were subjected to an austenitizing treatment at 1050^{o}C followed by air cooling to the lower temperature and then the PWHT. The matrix of samples heat treated is shown in Table 10.

Table 10: Heat Treatment Matrix of Simulated HAZ and Weld Metal Samples

Sample ID[*]	**Quantity of plates heat treated**	**Upper Temperature and hold time**	**Lower Temperature**	**PWHT Temperature and Time**
SHAZ1 – 200 - 1 SHAZ1 – 200 - 2	2	835^{o}C, 5 minutes	200^{o}C	760^{o}C, 2 hrs
SHAZ1 – 325 - 1 SHAZ1 – 325 - 2	2	835^{o}C, 5 minutes	325^{o}C	760^{o}C, 2 hrs
SHAZ2 – 200 - 1 SHAZ2 – 200 - 2	2	925^{o}C, 5 minutes	200^{o}C	760^{o}C, 2 hrs
SHAZ2 – 325 - 1 SHAZ2 – 325 - 2	2	925^{o}C, 5 minutes	325^{o}C	760^{o}C, 2 hrs
SWM – 175 - 1 SWM – 175 - 2	2	1050^{o}C, 5 minutes	175^{o}C	760^{o}C, 2 hrs
SWM – 250 - 1 SWM – 250 - 2	2	1050^{o}C, 5 minutes	250^{o}C	760^{o}C, 2 hrs
SWM – 350 - 1 SWM – 350 - 2	2	1050^{o}C, 5 minutes	350^{o}C	760^{o}C, 2 hrs

[*] SHAZ – Simulated Heat Affected Zone
SWM – Simulated Weld Metal

Two plates were processed for each heat treatment. Each plate was approximately 2” x 6” x 0.75” from which three creep samples were machined for a total of 42 creep samples. Creep testing is currently under way and is being carried out at 600 and 650°C at various stress levels. The stress levels have been or will be chosen to produce Larson-Miller parameters of >28 where differences in creep behavior have been found to be most pronounced. Microstructural analysis will be carried out on selected samples to document microstructural evolution and relate microstructure to creep behavior.

8.1.2 Creep of improperly cooled welds

Welds with improper cooling were made using GTAW methods. The composition of the filler wire is shown in Table 11 and does not differ significantly from the base metal, Table 9, except the nickel content of the filler wire is approximately three times that of the base metal. The welding system used for making these welds consisted of a standard 450-amp pulsed power supply operating an INL-designed air-cooled GTAW torch, and integrated into a custom-built control system that allowed computer control of torch motion and essential welding parameters via a LabVIEW Real Time operating system, and also incorporated a weld vision system for remote operation and monitoring. Plates were clamped at their edges with the center raised about 0.125 in. before the first pass, which compensated for distortion and yielded nearly flat final weldments. Weld joint design included a 0.094 in. land that allowed the first pass to be made in partial penetration mode; back gouging and welding of the back side was not performed. Pulsed current was used with high and low pulses of 350 A for 0.4 s and 200 A for 0.4 s, at an arc voltage (AVC-controlled) of 11 V, under argon shielding gas. Six passes were made for each weld; surfaces were wire brushed between passes. Travel speed was 1.8 mm/s (4.25 in./min); wire feed rate (0.045 in. diameter ER90S-B9) was 90 in./min (38 mm/s) with no oscillation for Passes 1-5, and an oscillation width of 3 mm for Pass 6. Interpass and final temperatures (before PWHT) were monitored at the center of the weld, beginning immediately after welding, with a surface thermocouple read through a digital meter.

Table 11: Composition of the TIG Filler metal – AWS/ASME Classification: ER90S-B9

C	Mn	P	S	Cu	Si	Ni	Cr	Mo	V	As	Al	Nb	N
0.11	0.53	0.006	0.002	0.04	0.24	0.73	8.95	0.94	0.22	0.007	0.007	0.05	0.044

Fig. 12a shows the joint design, the resulting weld bead and weld cross section. The weld is very uniform and lacks porosity, Fig. 12c. The highly automated nature of the welding equipment allowed multiple welds on multiple plates under virtually the same conditions. After each welding pass, the temperature was measured at the start, middle and end of the weld using a thermocouple in contact with the surface of the weld bead. The maximum temperature recorded after the final weld pass was on the order of 250°C ± 15°C. Therefore, welded plates were allowed to cool to 250 (basically no cooling), 212 and 175°C before being immediately subjected to the PWHT (760°C for 2 hours). The martensite start temperature, M_s, is approximately 360°C while the martensite finish temperature, M_f, is approximately 160°C. Therefore the three different cooling temperatures used in this study should produce a significant range of retained austenite volume fraction which will transform to ferrite during the PWHT. Two welded plates (12” x 12” x 0.75”) at each post welding cooling temperature were prepared. Each plate was

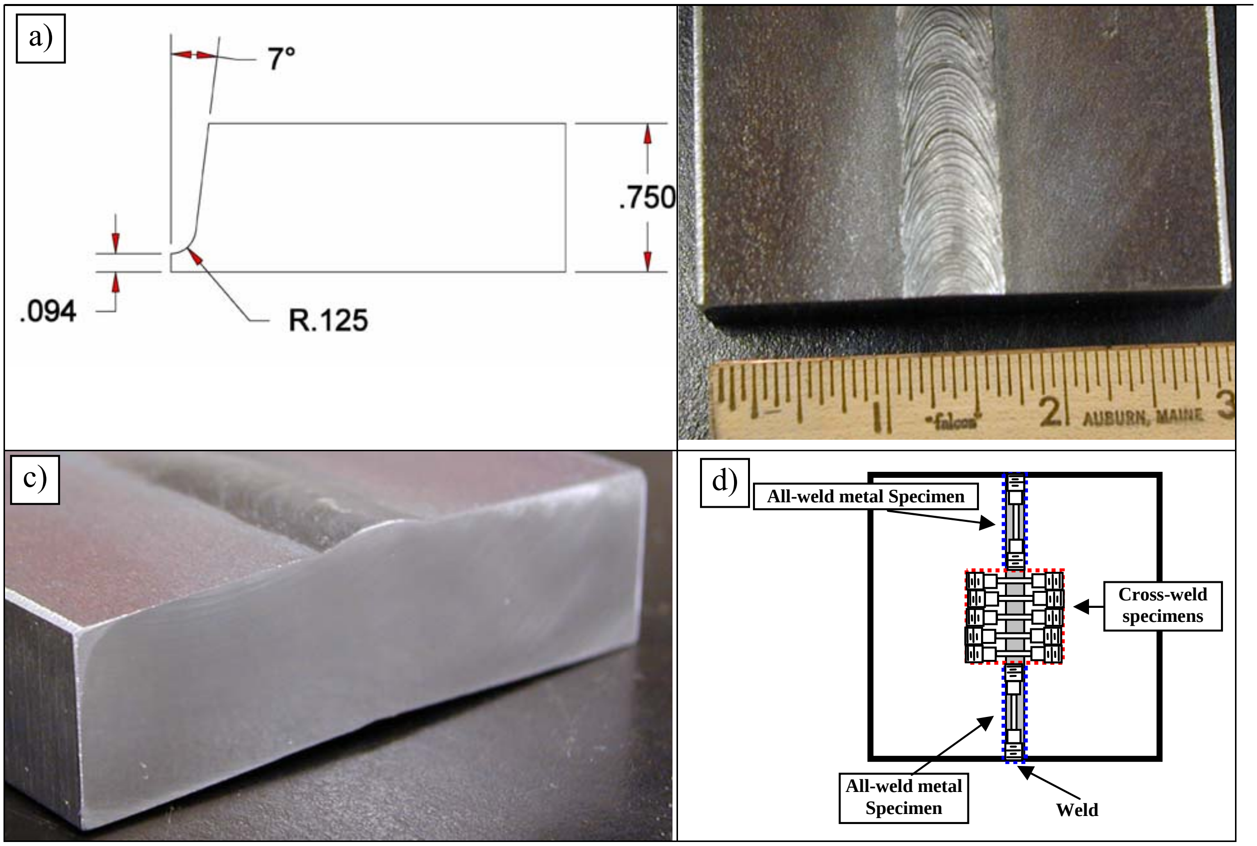

Fig. 12: A schematic of the weld joint design is shown in a) while the actual weld bead and a view of the weld cross section are shown in b) and c), respectively and d) a schematic of creep sample orientation/location in the welded plates.

subsequently sectioned to produce two all-weld-metal creep samples and up to six cross-weld creep samples, Fig. 12d. Therefore, there was a total of four all-weld-metal creep samples and up to twelve cross-weld creep samples for each of the three post weld cooling temperatures, for a total of 48 creep samples, available for creep studies. Creep testing at 650^{o}C and 100 MPa has begun. Additional stress levels at 650^{o}C as well as at 600^{o}C are scheduled for testing. Analysis of the microstructure and documentation of the fracture location of selected samples will be carried out to determine the relationship between microstructure and creep behavior of welded material. Microstructural analyses are also being performed on as-heat treated material to separate creep effects on microstructural development.

8.2 Off Normal Heat Treatment and Associated Creep Properties of Grade 91 Steel

An additional issue associated with field fabrication is the required heat treatment of components that do not conveniently fit into a furnace. This work is evaluating the effect of improper heat treatment on creep behavior to determine the level of control needed during heat treatment of large components in the field.

The work has focused on the incorrect normalization temperature. In previous work, normalization below the recommended normalization temperature of 1050°C resulted in lower creep resistance even though there was no obvious difference in microstructure (at the optical microscopy level) and room temperature tensile properties [7]. This work will further define the critical normalization temperature, below which, creep strength falls to unacceptable levels. In addition to determining the creep strength as a function of the normalization temperature, microstructural evaluations are being performed to ascertain the cause of the decrease in creep strength with decreasing normalization temperature.

8.2.1 Improper normalization and associated creep strength

Billets from a hot-rolled round bar 50 mm in diameter (composition is given in Table 12) were normalized at temperatures ranging from 900°C to 1080°C, air cooled to room temperature and then tempered at 760°C for 2 hours. Seven creep specimens were machined from each billet. Creep parameters were chosen to compliment previous work by Totemeier, et. al., 2006. Creep tests began in May of 2006 and were carried out at 600 and 650°C at stresses ranging from 80 to 170 MPa. The current results are shown in Table 13.

Table 12: Composition of Hot-Rolled Round Bar of Grade 91 Steel

Fe	C	Cr	Mo	V	Cu	Ni	Nb	N	P	S
Bal.	0.09	8.42	0.96	0.21	0.25	0.14	0.073	0.04	0.012	0.004

Table 13: Creep Tests of Improper Normalized Grade 91 Steel

Normalization Temperature, °C	Sample ID	Creep Temperature, °C	Creep Stress, MPa	Minimum Creep Rate, sec^{-1}	Time to Rupture, hrs	Ductility, %	RA, %	LMP
900	TML-9CR-900-4	650	75.47	2.08E-07	72.02	31.1	90.8	29.40
925	TML-9CR-925-1	650	60.6	1.36E-08	795.9	37.7	88.0	30.37
925	TML-9CR-925-2	650	70.4	3.33E-08	271.7	34.4	91.9	29.94
925	TML-9CR-925-3	650	79.3	7.08E-08	179.5	31.4	91.4	29.77
925	TML-9CR-925-4	650	60.19	1.19E-08	725.9	36.7	92.1	30.33
950	TML-9CR-950-4	650	85.7	6.40E-08	173.4	26.3	90.7	29.76
1000	TML-9CR-1000-01	650	80	In progress				
1000	TML-9CR-1000-02	650	90	1.24E-08	524.3	20.9	86.1	30.20
1000	TML-9CR-1000-04b	650	100.3	4.39E-08	160.2	19.7	88.7	29.72
1000	TML-9CR-1000-04	650	100	5.00E-08	228	31.8	91.3	29.87
1000	TML-9CR-1000-05	650	119.7	2.20E-07	62.6	30.4	91.5	29.35
1000	TML-9CR-1000-07	650	144.2	1.33E-06	13.7	36.3	92.3	28.74
1025	TML-9CR-1025-01	650	85	8.61E-09	1008.4	12.9	53.7	30.46
1025	TML-9CR-1025-02	650	89.4	1.33E-08	462.5	15.3	65.8	30.15
1025	TML-9CR-1025-04	650	100	3.19E-08	302.1	25.9	79.5	29.98
1025	TML-9CR-1025-05	650	119.7	1.90E-07	58.4	28.2	90.1	29.32
1025	TML-9CR-1025-07	650	144	1.30E-06	10.9	28.6	90.3	28.65
1050	TML-9CR-1050-1	650	85	5.28E-09	954.8	16.4	80.9	30.44

Normalization Temperature, °C	**Sample ID**	**Creep Temperature, °C**	**Creep Stress, MPa**	**Minimum Creep Rate, sec^{-1}**	**Time to Rupture, hrs**	**Ductility, %**	**RA, %**	**LMP**
1050	TML-9CR-1050-2	650	90	In progress				
1050	TML-9CR-1050-4	650	100	2.22E-08	322.7	17.5	87.7	30.01
1050	TML-9CR-1050-04b	600	130	In progress				
1050	TML-9CR-1050-06	600	170	In progress				
1080	TML-1080-1	650	90	In progress				
1080	TML-1080-2	650	100	In progress				
1080	TML-1080-4	650	129.4	8.30E-08	128.1	18.5	61.1	29.64
1080	TML-1080-5	600	130	In progress				
1080	TML-1080-6	600	140	In progress				
1080	TML-1080-7	600	160	In progress				

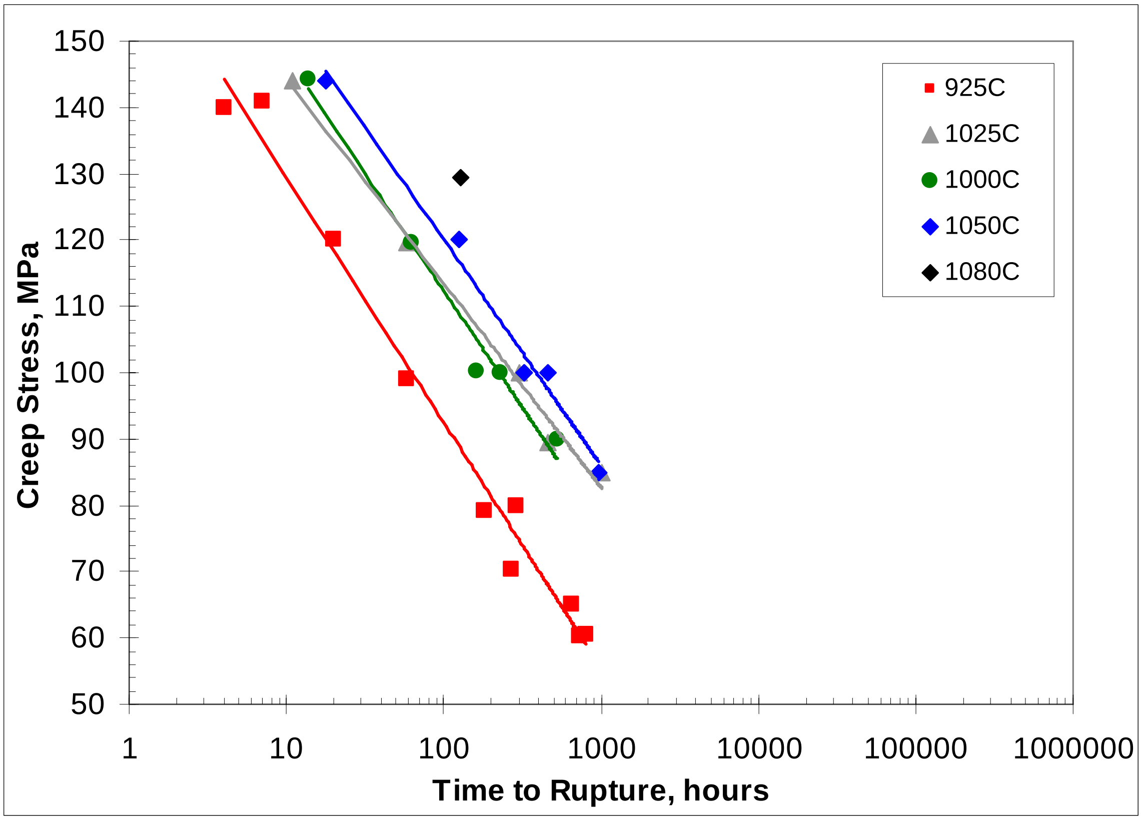

Fig. 13: Plots of creep behavior as a function of the normalizing temperature. After normalization, all samples were tempered at 760°C for 2 hours prior to creep testing.

The testing at 650°C is almost complete with only a few remaining tests in progress. The data in Table 13, along with previous data from Totemeier et. al.,2006, are plotted in Fig. 13. Evident in this figure is the drop in creep strength as the normalization temperature is decreased. It should be noted that the A_{c3} temperature, the temperature at which ferrite is completely transformed to austenite, is approximately 900°C so all creep samples include in Fig. 13 should consist of tempered martensite with no ferrite present since the lowest normalization temperature was 925°C . Interestingly, it appears that the normalization at 1080°C may result in improved creep properties without a large decrease in creep ductility. Completion of the remaining tests should clarify the creep behavior of this material normalized at a temperature higher than standard practice.

Currently we believe the normalization temperature affects the solubility of the various carbides and the extent to which the carbides are dissolved during normalization. Subsequently, carbides are re-precipitated during tempering at 760°C. Low normalization temperatures result in little dissolution of the carbides, growth of existing carbides and reduced precipitation at grain boundaries and subboundaries during subsequent tempering. Growth of existing carbides and reduced re-precipitation during tempering both contribute to reduced creep strength since pinning of grain boundaries, subboundaries and lattice dislocation is also reduced. Work by Hald and Kubon, 1997 [8], on the theoretical thermodynamic stability of the MX-type (mainly V, Nb and N) carbides, which are the most stable and are largely responsible for the long term creep strength in this alloy, indicate that they begin to dissolve around 900°C and are completely dissolved above about 1030°C. Careful microstructural analysis of the precipitate size, number and distribution will provide a clearer picture of the factors that influence the creep strength of this alloy.

8.2.2 Microstructural analysis

In order to determine the root cause for the decrease in creep strength with decreasing normalization temperature, microstructural analysis of materials normalized at different temperatures will be performed. The emphasis of this study is on characterizing the size, number and distribution/location of the carbides. The relatively small size of the carbides requires these studies to be carried out using transmission electron microscopy. TEM samples have been prepared by both extraction replica and electropolishing techniques. Extraction replicas remove the carbides from the sample and allow them to be analyzed without the interference of magnetic matrix. Electropolishing of chemically-thinned, TEM blanks (approximately 0.001" or 25 microns thick) using an ethonal-4% perchloric acid electrolyte at 5°C have yielded very good TEM foils, see Fig. 14. Samples of baseline material, subjected to the typical heat treatment of 1050°C normalization followed by air cooling and tempering at 760°C for 2 hours, and material normalized at 925°C, air cooled and tempered at 760°C for 2 hours have been prepared and are ready for analysis.

Analysis of diffraction patterns from individual precipitates has revealed the presence of mainly chromium/iron carbides ($Cr_{21.34}Fe_{1.66}C_6$ – a $M_{23}C_6$ type carbide) and vanadium nitride (VN) particles. Typically the VN particles, at approximately 30 nm in diameter, are on the order of half the size of the $M_{23}C_6$ type carbides. At this time not enough particles have been analyzed to make a conclusion as to preferential precipitation sites, e.g. grain/sub boundaries or grain interior, for the various particles or the size dependence on normalization temperature. Identification of precipitates using electron diffraction is slow and tedious. It is not feasible to characterize a large number of precipitates using this method. Chemical mapping using the energy dispersive x-ray spectrometer (EDS) will be used in the future to quickly identify a large number of precipitate from various samples. (The EDS detector on the TEM is currently being repaired and should be operational by the end of August, at which time microstructural analysis will resume.)

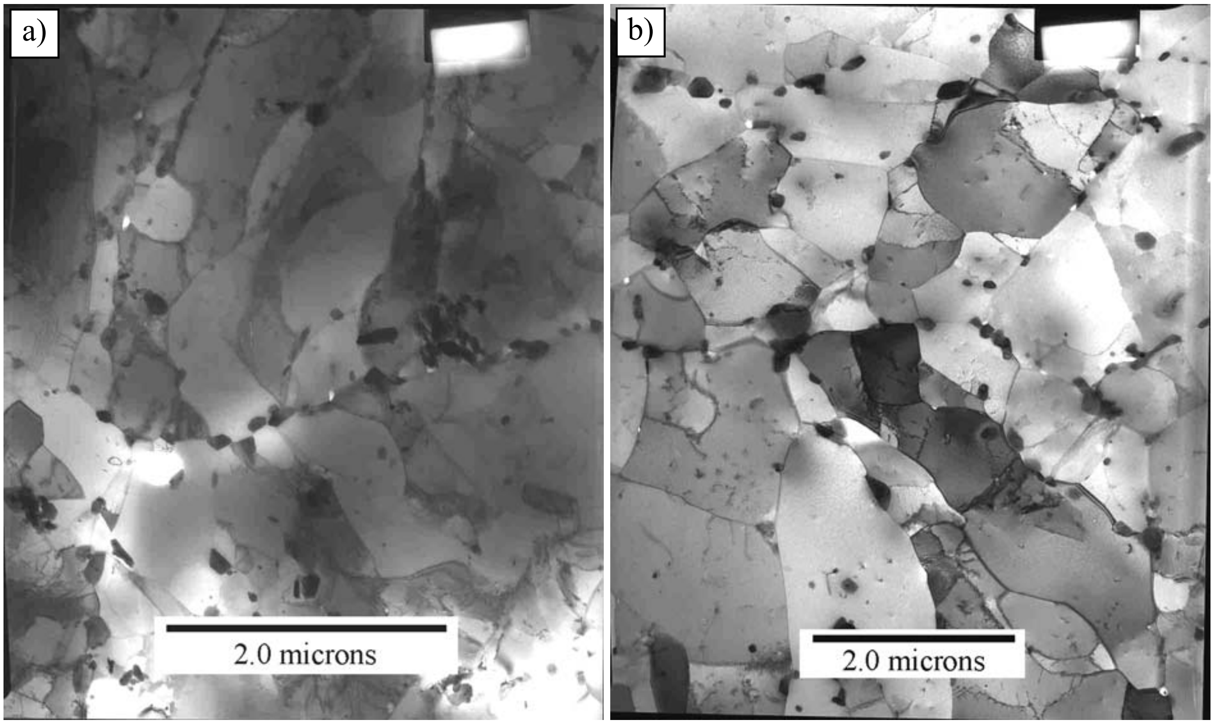

Fig. 14: TEM photomicrographs of Grade 91 creep samples – a) 1050^{o}C normalize and temper and b) 925^{o}C normalize and temper. Creep conditions – a) 650^{o}C, 120 MPa and b) 650^{o}C, 70 MPa. Black particles are carbides.

9. REFERENCES

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