NEW CREEP RUPTURE ASSESSMENT OF GRADE 91

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ABSTRACT

The European collection and assessment of Grade 91 creep rupture data were performed by European Creep Collaborative Committee (ECCC) in 1995 referring to all products - tube, pipe, forging, casting and plate - obtained by industrial and laboratory casts. The database mainly contained data within 50,000 hours. The stress to obtain rupture at 600°C into 100,000 hours was assessed in 94 MPa.
A new collection of creep rupture data was performed by ECCC in 2005: a larger database was generated with several broken tests in the range of 50,000-100,000 hours and some over 100,000 hours. The dataset was assessed with different procedures developed by ECCC and the extrapolations were validated by Post Assessment Tests (PATs). The evaluated creep strength into 100,000 hours at different temperatures reached a high level of confidence and more accurate extrapolations up to 200,000 hours were achieved.

KEYWORDS

High Chromium steel, T/P91, Creep-rupture strength, Creep data assessment, ECCC

INTRODUCTION

In the last two decades, the request of improved efficiency and reduced emissions in power plants has resulted in the design of system utilizing higher and higher steam temperatures and pressures.
This shift towards more severe service conditions required the use of materials with appropriate high-temperature strength, physical properties and metallurgical stability at the operating temperature of the unit to prevent catastrophic failures by several potential long-term mechanisms: leakage due to creep-induced dimensional changes, bursting due to stress rupture, or thermal fatigue due to thermally-induced cyclic stresses.
In the 1970s, Oak Ridge National Laboratory (ORNL) developed a new alloy, commonly referred to as 9Cr-1Mo-V, similar to conventional 9Cr-1Mo grades; modifications included additions of Vanadium, Niobium, Nitrogen and low Carbon content. The chemistry of the 9Cr-1Mo-V steel was especially designed to promote the formation of a microstructure which provides excellent long-term high-temperature strength. The final desired microstructure, which can be achieved by a proper normalizing and tempering heat treatment, consists of tempered martensite, with a fine carbonitride precipitation (MX) inside the matrix, and a extensive carbide precipitation ($M_{23}C_6$) along the grain boundaries [1], [2], [3]. In addition, because of its lower thermal expansion coefficient and higher thermal conductivity, this alloy is much more resistant to thermal fatigue than the austenitic stainless steels.
The 9Cr-1Mo-V steel has been adopted in various forms in ASTM/ASME Specifications: seamless tube T91 (ASTM A213), seamless pipe P91 (ASTM A335), forged pipe FP91, forging F91 and casting C12A. This alloy is also included in European Standardization, EN 10216-2, under the designation X10CrMoVNb9-10. The first large scale application of this steel at USC (UltraSuperCritical) steam conditions was in the Kawagoe plants in Japan, commissioned in 1988 with steam parameter 31MPa/566°C/566°C.

The present paper focuses on long-term creep properties of parent material of 9Cr1Mo0.25V steel (Grade 91). This steel has been at the center of activities in European Creep Collaborative Committee (ECCC), Working Group 3A (WG3A), “Ferritic Steels”, which covers creep data development and analysis for parent materials and welds of all creep resistant steels, ranging from low alloy steels up to 12%Cr steels.
The first European collection and assessment of Grade 91 creep rupture data were performed by ECCC in 1995 referring to all products - tube, pipe, forging, casting and plate - obtained by industrial and laboratory casts. The database mainly contained data within 50,000 hours. The stress to obtain rupture at 600°C into 100,000 hours was assessed in 94 MPa.
A new collection of creep rupture data was performed by ECCC in 2005: a larger database was generated with several broken tests in the range of 50,000-100,000 hours and some over 100,000 hours. The dataset was assessed with different procedures developed by ECCC and the extrapolations were validated by Post Assessment Tests (PATs) [4]. The evaluated creep strength into 100,000 hours at different temperatures reached a high level of confidence and more accurate extrapolations up to 200,000 hours were achieved.

PREVIOUS ASSESSMENT OF GRADE 91

One of the most demanding task for the validation of new high temperature steels for use in pressure vessel design is the development of a wide database of long-term creep test results. The ECCC WG3A, and its predecessors in earlier rounds of ECCC collaboration, assessed the long creep rupture strength of the most advanced 9Cr1MoV steels, Grades 91, E911 and 92, on comprehensive worldwide datasets, including information about material pedigree: chemical composition of the heats, heat treatment details, mechanical properties. Minor compositional changes among those three steels led to major differences of their creep strength, as shown Table 1.

For P92 a first worldwide data collation was completed in 1999 and it resulted in a database containing approximately 4.2 million testing hours. Data assessments in ECCC resulted in a first datasheet published in 1999 with stress to rupture into 100,000 hours at 600°C assessed in 123MPa. In year 2005 a new ECCC database was generated, containing almost 9 million testing hours. A new data assessment was published in 2005: the creep strength was re-assessed in 600°C/113MPa/10^5h and 600°C/101MPa/$2 \cdot 10^5$h [5], [6].

For steel E911 the creep tests performed during COST 501 Program were collated by ECCC in 2000. They amounted to approximately 2.4 million testing hours. Assessments made by ECCC in 2002 have resulted in a provisional datasheet, as the data were not extensive enough to establish validated mean stress rupture strength values beyond 30,000 hours without extended time extrapolation.
The database was updated in 2004 with several other worldwide data (longest available data reached 87,000 hours at 600°C) and more reliable extrapolations were published in ECCC datasheet in 2005: 600°C/98MPa/10^5h [5], [6].

For steel P91 a worldwide data collation was made in 1994, which resulted in a database containing approximately 14 million testing hours. The assessment of the data was published in ECCC datasheet in 1995 with validated mean stress rupture strength up to 100,000 hours at temperatures 500°C up to 670°C [5].

Table 1 – Chemical composition, heat treatment and mean creep rupture strength of advanced Cr steels, Grades 91, E911 and 92

Element	T/P91 (EN 10216-2)	E911 (ASTM 213-335)	T/P92 (ASTM 213-335)
C	0.08 – 0.12	0.09 – 0.13	0.07 – 0.13
Si	0.20 – 0.50	0.10 – 0.50	0.50 max
Mn	0.30 – 0.60	0.30 – 0.60	0.30 – 0.60
P	0.020 max	0.020 max	0.020 max
S	0.010 max	0.010 max	0.010 max
Cr	8.00 – 9.50	8.50 – 10.50	8.50 – 9.50
Mo	0.85 – 1.05	0.90 – 1.10	0.30 – 0.60
Ni	0.40 max	0.40 max	0.40 max
Al tot	0.04 max	0.04 max	0.04 max
Cu	0.030 max	/	/
Nb	0.06 – 0.10	0.06 – 0.10	0.04 – 0.09
Ti	/	/	/
V	0.18 – 0.25	0.18 – 0.25	0.15 – 0.25
N	0.03 – 0.07	0.04 – 0.09	0.03 – 0.07
W	/	0.90 – 1.10	1.5 – 2.0
B	/	0.0003 – 0.006	0.001 – 0.006
Normalising (°C)	1040 – 1090	1040 min	1040 min
Tempering (°C)	730 – 780	730 min	730 min
10^5h creep rupture strength at 600°C	94 MPa (ECCC 1995)	98 MPa (ECCC 2005)	113 MPa (ECCC 2005)

NEW ASSESSMENT OF GRADE 91

More than ten years have passed since last evaluation of Grade 91 creep-strength. Available long term tests have now passed 100,000 hours in the range 575°C-625°C and deviations from previously extrapolated curves in 1995 gave indication that a re-assessment would have been reasonable. For this reason a new ECCC database was generated. The data were collated by Japan, USA and major European Countries (mainly from Germany, France, England and Italy).
Centro Sviluppo Materiali (CSM) and Vallourec Research Center were officially designed as ECCC assessors of new creep-rupture database. The assessors were asked to process the database separately, following ECCC Recommendations and performing the PATs.

CREEP RUPTURE ASSESSMENT BY CSM

A total of 2,195 creep data (including running tests) were selected according to European Standard EN 10216-2, in terms of chemical composition, heat treatment and mechanical properties, for a total of 17,852,130 testing hours. The database mainly consisted of tube and pipe products, with less than 300 data only from bar, plate and forging.
Details of the used database are reported in Table 2. The summary of creep rupture data distribution is shown in Table 3. The testing temperature varies from 427°C up to 730°C, but most of the data are concentrated at 550°C, 600°C and 650°C. Several very long creep tests are included in the database: seven creep data passed 100,000 hours; the longest broken creep test reached 113,431 hours at 600°C and one running test passed 137,928 hours at 600°C.

Table 2 – Details of assessed database in terms of chemical composition, heat treatment, tensile properties and product sizes (according to EN 10216-2:2002 and its permissible deviations of the product analysis from specified limits on cast analysis)

	DETAILS						Specified ranges EN 10216-2:2002	
	Variable	Unit	Mean	Min	Max	Std. Dev.	Min.	Max
Composition	Carbon	wt%	0.099	0.080	0.120	0.008	0.080	0.120
	Silicon	wt%	0.321	0.190	0.490	0.063	0.200	0.500
	Manganese	wt%	0.433	0.310	0.590	0.054	0.300	0.600
	Phosphorous	wt%	0.012	0.001	0.024	0.005	/	0.020
	Sulphur	wt%	0.003	0.001	0.009	0.002	/	0.010
	Chromium	wt%	8.593	8.050	9.300	0.291	8.000	9.500
	Molybdenum	wt%	0.945	0.850	1.060	0.037	0.850	1.050
	Nickel	wt%	0.156	0.010	0.460	0.085	/	0.400
	Vanadium	wt%	0.212	0.150	0.270	0.018	0.180	0.250
	Niobium	wt%	0.076	0.055	0.100	0.010	0.060	0.100
	Nitrogen	wt%	0.046	0.027	0.069	0.010	0.030	0.070
	Aluminium	wt%	0.012	0.001	0.003	0.008	/	0.040
	Copper	wt%	0.064	0.003	0.157	0.032	/	0.300
Heat Treatment	Normalisation temperature	°C		1040	1090		1040	1090
	Tempering temperature	°C		740	780		730	780
Tensile	Rp	MPa	557	450	767	56.8	450	/
	Rm	MPa	712	630	829	48.8	630	830
Products/sizes	Tube/pipe OD	mm		26.7	660			
	Tube/pipe WT	mm		2.67	100			
	Forging, bar, plate	mm						

It is interesting to compare the new database with the previous one from ECCC (Table 4). The differences are consistent at very long time and the new available long-term broken data guarantee higher calculation reliability and extrapolations up to 200,000 hours.

Table 4 – Comparison between ECCC databases, dated 1995 and 2005

	Test Durations						
	h <10.000	h 10.000 to 20.000	h 20.000 to 30.000	h 30.000 to 50.000	h 50.000 to 70.000	h 70.000 to 100.000	h > 100.000
	Number of test points available						
ECCC 1995	1260 (60)	159 (16)	52 (18)	23 (11)	9 (16)	2 (7)	
ECCC 2005	1657 (37)	220 (29)	92 (28)	52 (8)	24 (20)	5 (16)	3 (4)

Two main assessments were performed, the first one according to PD6605 procedure and the second one according to Larson-Miller (LM) method.

The simplified Mendelson-Roberts-Manson (MRM) equation was used to assess the database. This is a complex creep-rupture model according to PD6605 assessment procedure, by the following parametric equation [6], based on a polynomial expansion:

$$\ln(t_r^*) = \left\{ \sum_{k=0}^{n} \beta_k \cdot (\log[\sigma])^k \right\} \cdot (T - T_0)^r + \tau_0 \quad (1)$$

where n is the degree of the polynomial, β_k, τ_0, T_0 and r are constants, t_r^* is the predicted rupture time [hours], T is the absolute temperature [K] and σ is the applied creep stress [MPa].
CSM assessed the database according to the following MRM four degree equation:

$$\ln(t_r^*) = \left[263.8 + 217.1 \cdot \log\sigma - 212.8 \cdot (\log\sigma)^2 + 84.9 \cdot (\log\sigma)^3 - 13.2 \cdot (\log\sigma)^4\right] \cdot (T - 438.7)^{-0.27} - 55.9 \quad (2)$$

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Table 3 - Summary of creep rupture data distribution

Temp. (°C)	h<10000		10000<h<=20000		20000<h<=30000		30000<h<=50000		50000<h<=70000		70000<h<=100000		h>100000		Total broken	Total unbroken	Total B+UB	% of total broken
	B	UB	B	UB	B	UB	B	UB	B	UB	B	UB	B	UB				
427												1		1		2	2	0
454												1				1	1	0.0
482	1									4					1	4	5	0.0
500	71	6	6	3	2	6			1	3		1			80	19	99	3.9
525	3	3				1				1		2			3	7	10	0.1
538	12		3			2		1		5	1	1			16	9	25	0.8
550	262	11	31	9	21	7	12	2	6	2	3	1	2		337	32	369	16.4
575	58	1	10	1										1	68	3	71	3.3
580	1														1		1	0.0
585	1														1		1	0.0
590	1														1		1	0.0
593	39	1	9		3		3	1				1			54	3	57	2.6
600	524	7	99	11	43	7	29	4	9	5	1	6	1	2	706	42	748	34.3
610	1		2												3		3	0.1
615	1		1												2		2	0.1
620	12	2	4						2						18	2	20	0.9
621	10														10		10	0.5
625	57	1	2	1											59	2	61	2.9
630	16	5	2		2										20	5	25	1.0
637	1														1		1	0.0
640	24		5				2								31		31	1.5
645	3														3		3	0.1
649	55		2		3										60		60	2.9
650	358		40	4	16	5	6		6			2			426	11	437	20.7
660	20														20		20	1.0
662	3														3		3	0.1
665	3														3		3	0.1
670	16														16		16	0.8
675	6														6		6	0.3
677	21				1										22		22	1.1
680	16														16		16	0.8
685	2														2		2	0.1
688	1														1		1	0.0
690	6														6		6	0.3
695	2														2		2	0.1
700	40		4												44		44	2.1
704	5				1										6		6	0.3
710	4														4		4	0.2
730	1														1		1	0.0
TOTAL	1657	37	220	29	92	28	52	8	24	20	5	16	3	4	2053	142	2195	100

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To check the good quality of the time and stress extrapolations by MRM approach, following the ECCC guideline, CSM verified that the new Grade 91 extrapolations satisfy all PATs categories.
The calculated creep-rupture isothermals are plotted in Figure 1 for main temperatures, in comparison with experimental data points and ECCC reference curves dated 1995 (PAT 1.1). Data fitting is very good in the range 500°C-600°C and at 700°C, whilst at 650°C the scatter is large, especially at very long time exposure.
To assess the effectiveness of the model in predicting the creep behaviour of the complete dataset, the [predicted time, log t_r^*] versus [observed time, log t_r] diagram was plotted for all tested temperatures (PAT 2.1) (Figure 2). Less than 1.5% of total data falls outside $\log t_r^* = \log t_r \pm 2.5\, S_{[A\text{-}RLT]}$ boundary lines.

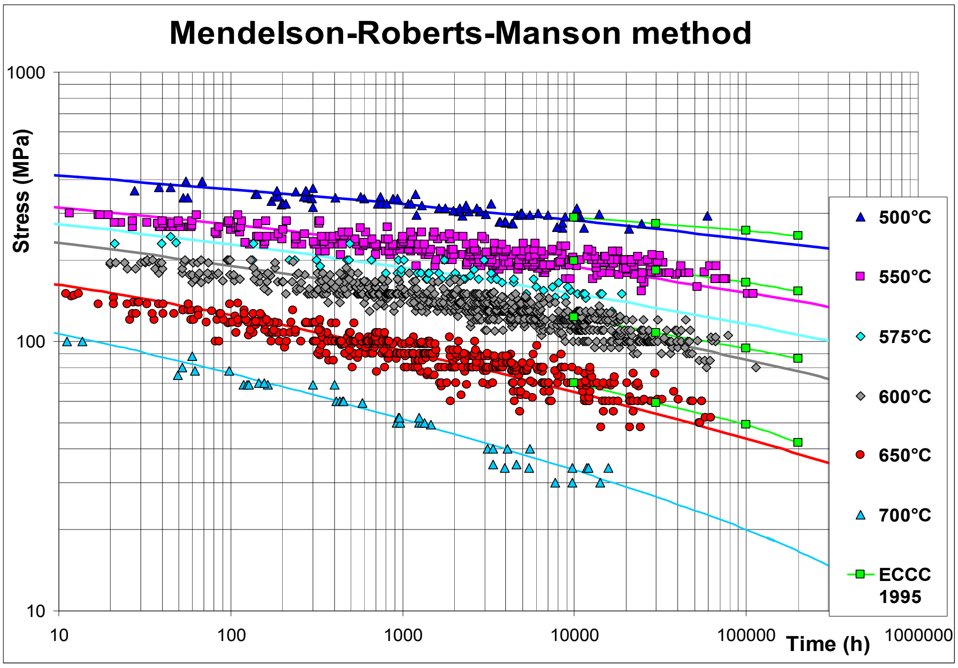

Figure 1 – Isothermal curves by MRM equation (PAT 1.1)

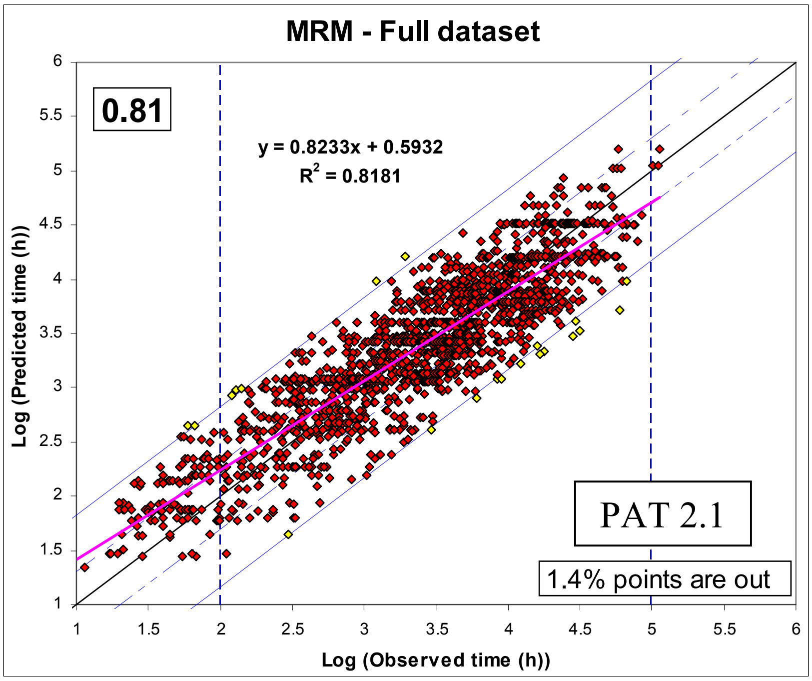

Figure 2 – Predicted time vs observed time plot

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The isothermal creep curve at 600°C and the relative PAT 2.2 plot are showed in Figures 3 and 4. The fitting is very good even at very long time (~100,000 hours) and one point only (0.14%) falls outside $\log t_r^* = \log t_r \pm 2.5\, S_{[A\text{-}RLT]}$ boundary lines in the $\log t_r^*$ vs $\log t_r$ diagram.

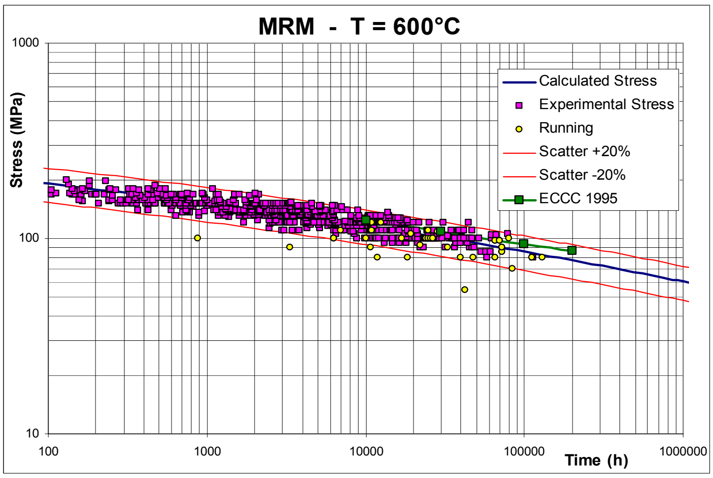

Figure 3 – Isothermal curve at 600°C

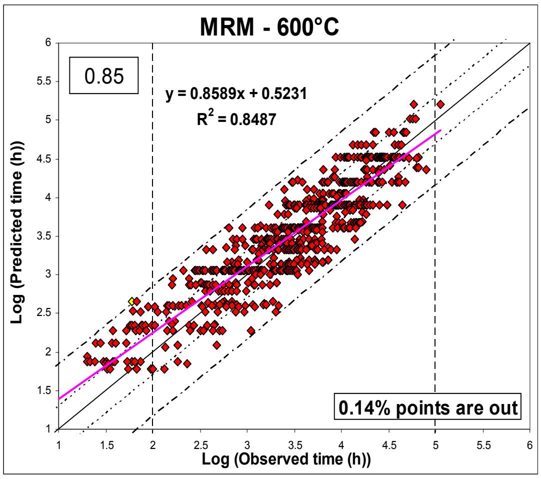

Figure 4 – PAT 2.2 at 600°C

CSM performed a second assessment by Larson-Miller equation. The relationship used to assess the database is the following:

$$\log \sigma_c = a + b \cdot P + c \cdot P^2 + d \cdot P^3 + e \cdot P^4 \qquad (3)$$

where σ_c is the calculated stress [MPa], a, b, c, d and e are constants and P is the Larson-Miller parameter, defined as:

$$P = T \cdot (C_{LM} + \log(t_r))/1000 \qquad (4)$$

where C_{LM} is a constant optimised by regression, t_r is the time to rupture [hours] and T is the temperature [K]. The calculated creep-rupture isothermals are plotted in Figure 5 for main temperatures, in comparison with experimental data points and ECCC reference curves dated 1995. A dangerous stress overestimation is obtained by LM method at all temperatures, especially at 600°C. The requirement of physical realism of the extrapolations is not completely fulfilled.

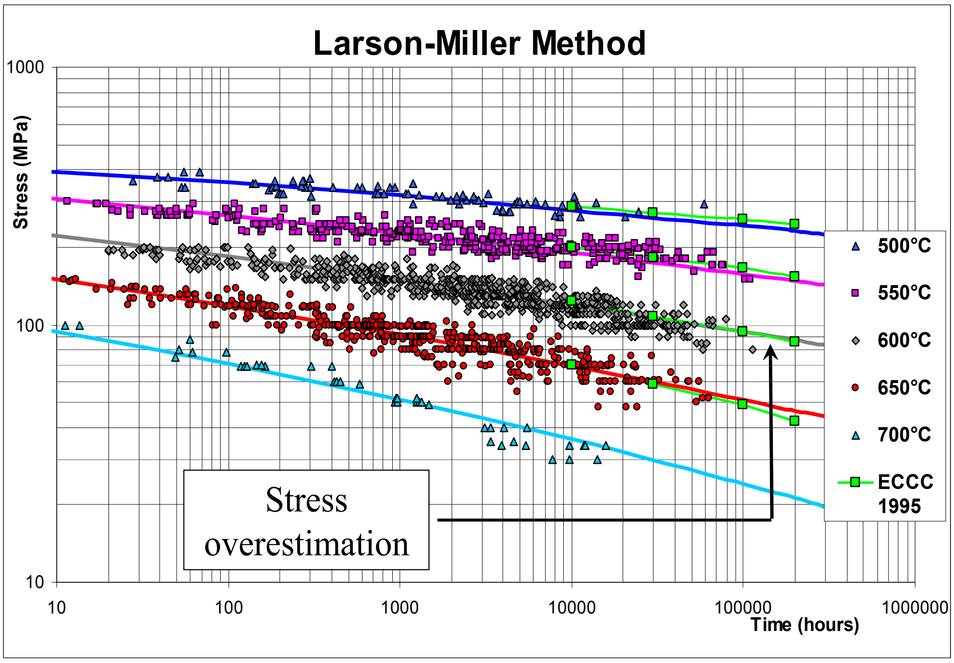

Figure 5 - Isothermal curves by LM equation

CREEP RUPTURE ASSESSMENT BY VALLOUREC

Vallourec Research Center assessed the same ECCC database by Minimum Commitment (MC) equation [7]:

$$\log t_r^* = a_0 + a_1 \cdot \log\sigma + a_2 \cdot \sigma + a_3 \cdot \sigma^2 + a_4 \cdot T + \frac{a_5}{T} \qquad (5)$$

where t_r^* is the predicted time [hours], σ is the stress [MPa], T is the temperature [K], and a_0, a_1, a_2, a_3, a_4 and a_5 are constants. The constants determined for Grade 91 database by MC equation are:

$$a_0= -0.73;\ a_1= -5.15;\ a_2= -0.0059;\ a_3= -1.68\cdot10^{-5};\ a_4= -0.0089;\ a_5= 21058.5. \qquad (6)$$

The visual check of the credibility of predicted model shows a good correlation between the individual data points and the calculated isothermal lines, as illustrated in Figure 6.

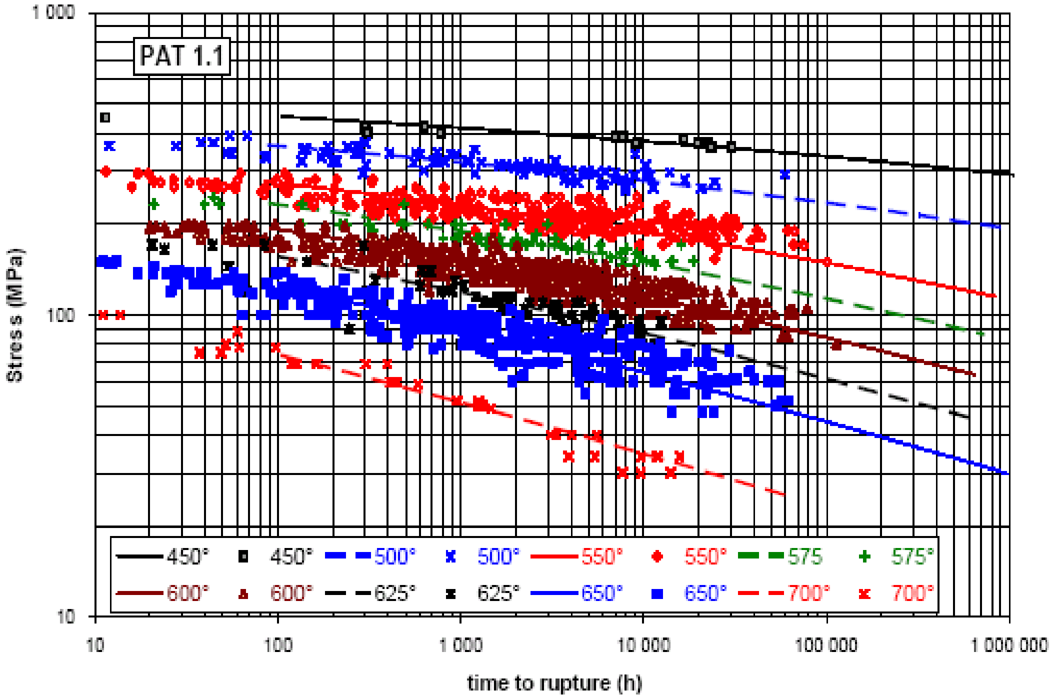

Figure 6 - Isothermal curves by MC equation

DISCUSSION AND RESULTS

The results of three different assessments by MRM, LM and MC equations, are summarized in Table 5 for the main testing temperatures. In the range of 550°C-600°C the large amount of data allowed extrapolations up to 200,000 hours. The values within parentheses indicate the deviation percent of assessed values with respect to ECCC 1995 extrapolations.
A graphical comparison of the experimental data with the assessed isothermal curves is given in Figures 7 and 8, at 550°C and 600°C respectively. Differences between MRM and MC equation are very small, while the LM method is the less conservative and seems to be less able to describe a strong curvature between the high and low stress creep regime. The correction to ECCC assessment is consistent and a general reduction of creep-rupture strength was achieved.

Table 5 – Results of new creep-rupture assessments (*values with extended time extrapolation)

Assessment Method	Temperature °C	Creep rupture strength (MPa) for times			
		10,000 h	30,000 h	100,000 h	200,000 h
ECCC 1995	550	199	183	166	154*
MRM equation		189 (-5.0%)	171 (-6.5%)	152 (-8.4%)	141 (-8.4%)
Larson-Miller		190 (-4.5%)	174 (-4.9%)	157 (-5.4%)	147 (-4.5%)
MC equation		189 (-5.0%)	169 (-7.6%)	149 (-10.2%)	137 (-11.0%)
ECCC 1995	600	123	108	94	86*
MRM equation		117 (-4.8%)	101 (-6.5%)	86 (-8.5%)	77 (-10.4%)
Larson-Miller		120 (-2.4%)	107 (-0.9%)	94 (0%)	86 (0%)
MC equation		116 (-5.7%)	100 (-7.4%)	84 (-10.5%)	76 (-11.6%)
ECCC 1995	650	70	59	49*	42*
MRM equation		65 (-7.1%)	54 (-8.4%)	44 (-10.2%)	38* (-9.5%)
Larson-Miller		69 (-1.4%)	60 (+1.7%)	51 (+4.1%)	46* (+9.5%)
MC equation		65 (-7.1%)	54 (-8.4%)	44 (-10.2%)	39* (-7.1%)

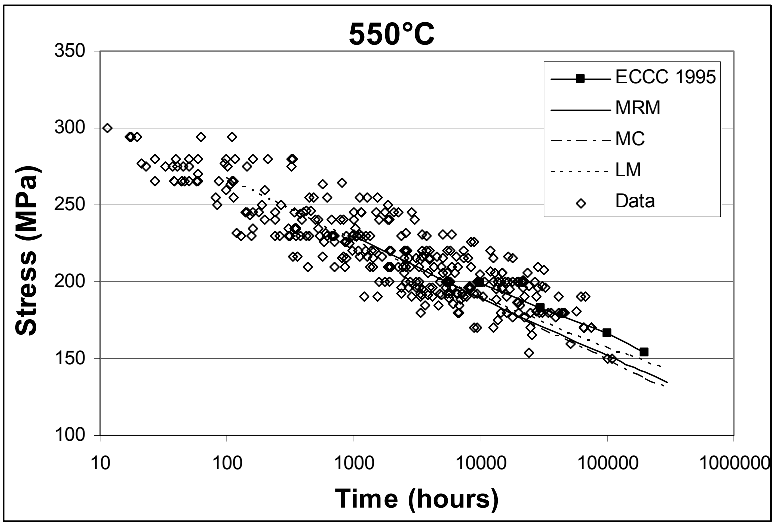

Figure 7 – Comparison of Grade 91 test data with the assessed mean curves at 550°C

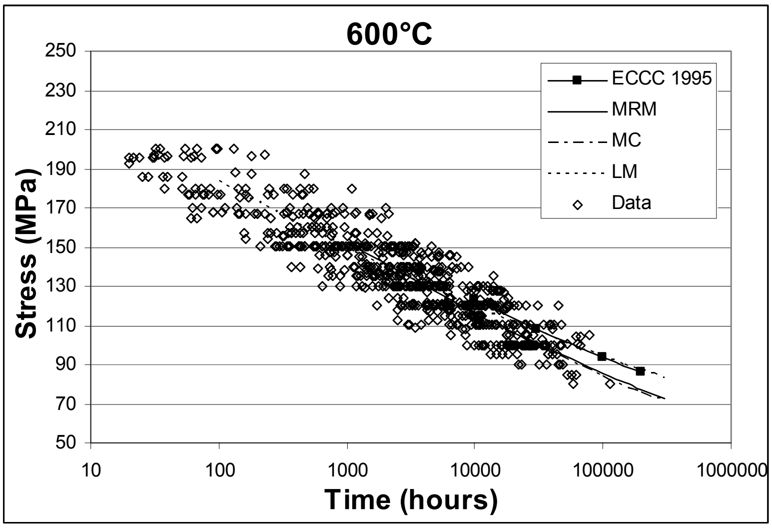

Figure 8 – Comparison of Grade 91 test data with the assessed mean curves at 600°C

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At this point, a new comparison among the creep-rupture strength of the three most advanced ferritic steels, Grades 91, E911 and 92, can be approached, see Table 6 and Figure 9 [8], [9].
A final official ECCC creep-rupture assessment of Grade 91 must be approved yet, but in the comparison with Grades E911 and 92, the average creep-rupture values between MRM and MC methods were used.

Table 6 – Comparison of evaluated creep rupture strength values of Grades 91, E911 and 92

Temperature °C	100,000 h creep rupture strength		
	Grade 91 average MRM-MC values	Grade E911 (ECCC 2005)	Grade 92 (ECCC 2005)
550	150	173	187
575	116	134	146
600	85	98	113
625	62	71	77
650	44	/	56

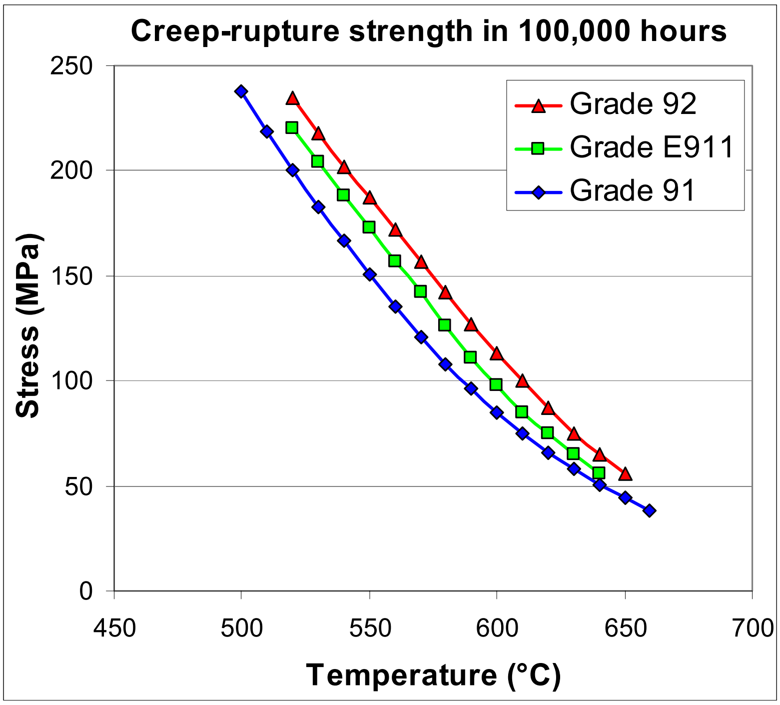

Figure 9 – Comparison of creep strength within 100,000 hours of Grades 91, E911 and 92. Average values between MRM-MC methods were adopted for Grade 91

CONCLUSIONS

The agreement between the assessments of new ECCC Grade 91 database by MRM and MC equations was very good, while by adopting a LM equation a dangerous overestimation of creep-rupture strength at all temperatures, especially at 600°C, was obtained.
Very long time extrapolations, up to 200,000 hours at 600°C, were achieved according with ECCC Recommendations.
A general reduction of creep resistance of Grade 91 with respect to ECCC official datasheet, dated 1995, was assessed.

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