CREEP RESISTANT 9-12% Cr STEELS - LONG-TERM TESTING, MICROSTRUCTURE STABILITY AND DEVELOPMENT POTENTIALS

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ABSTRACT

The long-term creep rupture strength of new 9-12%Cr steam pipe steels is now secured with long-term testing up to 100,000 hours, and the steels have been introduced into advanced ultra supercritical steam power plans.

Detailed microstructure studies have improved the understanding of the long-term stability of the 9-12%Cr steels: Precipitate hardening controls the long-term microstructure stability, and solid solution strengthening from Mo and W plays no significant role in the long-term microstructure stability of 9-12%Cr steels. Significant particle strengthening can be obtained by intermetallic Laves phases provided that a large number of particles nucleate during creep exposure. MX carbonitrides rich in V and Nb are extremely stable against coarsening, but they may be dissolved by precipitation of the complex nitride Z-phase (Cr(V,Nb)N). This is mainly a problem in Nb containing steels with Cr contents of 10% and above.

Significant advances in microstructure stability modelling will allow further improvements of the creep properties of this class of alloys.

KEYWORDS

9-12%Cr steels, long-term creep testing, microstructure stability, creep mechanisms, precipitate characterisation, Laves phase, Z-phase, precipitate stability modelling

INTRODUCTION

The creep rupture strength of martensitic 9-12%Cr steels for fossil fired steam power plants has been doubled over the last three decades and this development has been used to construct new advanced fossil fired steam power plants with higher efficiency. Increases in steam parameters from subcritical 180 bar/530-540°C to ultra supercritical values of 300 bar and 600°C have been realised, and this has led to efficiency increases from 30-35% to 42-47%, equivalent to approximately 30% reduction in specific CO_2 emission. With the expected continuous increase in the demand for coal for electricity production, especially in the fast developing Asian countries, this technology progress has clearly the potential to become one of the most significant countermeasures against global CO_2 emissions in the coming decades.

Extensive long-term testing of industrial products is required before a new creep resistant steel can be introduced for pressure vessel design. This means that the lead time for a new alloy from laboratory to power plant approaches 10 years. Further to the long-term testing, detailed studies of microstructure stability of the alloys are made in order to: a) assure the long-term stability of new alloys up to 300,000 hours, which is the expected maximum lifetime of new power plants, and b) give directions for the development of even stronger steels for operation at further advanced steam parameters.

The present paper reviews results from the long-term testing and microstructure stability studies for new 9-12%Cr alloys intended for pressure vessel applications and presents an estimate of development potentials for this group of alloys.

1. ALLOY DEVELOPMENT

The impressive alloy development of the 9-12%Cr steels has been achieved through apparently minor compositional changes to well-established steels like the 9Cr1Mo (P9) and 12CrMoV (X20 CrMoV121). The new alloys include the modified 9Cr1Mo steel P91, the tungsten alloyed 9Cr steel E911 and the tungsten and boron alloyed P92 all intended for thick section boiler components and steam pipes. Similar grades have been developed for cast and forged components in steam turbines. Typical chemical composition, heat treatment and creep rupture strength of the pipe steels are presented in Table 1.

Mass %	12CrMoV	P91	P92	E911
C	0.20	0.09	0.11	0.12
Si	0.26	0.29	0.04	0.13
Mn	0.51	0.35	0.46	0.56
Cr	11.0	8.70	8.96	8.70
Mo	0.90	0.90	0.47	0.97
W	-	-	1.84	0.96
Ni	0.44	0.28	0.06	0.29
V	0.29	0.22	0.20	0.20
Nb	-	0.072	0.069	0.062
N	-	0.044	0.051	0.066
B	-	-	0.001	0.0004
Austenitisation	1050°C	1050°C	1065°C	1060°C
Tempering	750°C	765°C	770°C	770°C
10^5h creep rupture strength at 600°C	59 MPa	≈85 MPa*)	113 MPa	98 MPa

Table 1. Steam pipe steels: Typical chemical composition, heat treatment and mean creep rupture strength according to recent evaluations by ECCC. *) A new evaluation for P91 is currently being made.

- *Steel P91*

Steel P91 was developed in the USA in the middle of the 1970s for nuclear application by Oak Ridge National Laboratories and Combustion Engineering based on the 9Cr-1Mo tube steel P9. By optimization of the alloy composition with small amount of V, Nb and N the creep strength was considerably improved. Later it was realised, that the steel could also be used for conventional steam plant, and it was included in the ASME Code in 1984. The first large scale application of the steel at USC steam conditions followed in the Kawagoe plants in Japan commissioned in 1988 with steam parameters 31 MPa/566°C/566°C.

- *Steel P92*

The Japanese steel NF616 is a tungsten alloyed 9%Cr steel, which was invented by Prof. T. Fujita at Tokyo University in the middle of the 1980´s based on life-long studies of the 9-12%Cr steels. Further development of the steel was made together with Nippon Steel Corporation [1,2]. The basic new alloying concept in the steel is to replace part of the molybdenum with app. 1,8% tungsten and add boron. In international research projects led by EPRI with participation of steel makers, boiler makers and utilities from Japan, USA, UK and Denmark, pipe production, pipe bending, welding procedures and long term creep data for the steel was developed, and this led to ASME Code approval in 1994 under the name P92 [3]. First large scale application of the steel followed in the Danish 400 MW USC power plant Avedoere 2 commissioned in 2001 with steam parameters 30 MPa /580°C/600°C.

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- *Steel E911*

Steel E911 was developed in the European COST 501 Action in 1992 based on the turbine rotor Steel E with similar composition [4]. The basic alloying concept was that the tungsten content should be reduced compared with steel NF616, because this element was believed to be harmful to long term microstructure stability of 9-12%Cr steels. Consequently the composition is based on 9%Cr with 1%Mo and 1%W. The steel was developed by European steel makers and pipe manufacturers and has recently received ASME Code approval under the name P911. First large scale application followed in the German 975 MW lignite fired USC plant Niederaussen commissioned in 2002 with steam parameters 26,5 MPa/565°C/600°C.

2. LONG-TERM TESTING

One of the most demanding tasks for the validation of new high temperature steels for use in pressure vessel design is the development of a comprehensive database of long-term creep test results on industrial products. For approval by the ASME new steels need tests with rupture times of at least 10,000 hours to establish allowable stresses. For approval in Europe rupture times of at least 30,000 hours for five melts of a new steel are needed to make a valid extrapolation to the 100,000 hour mean rupture strength to be used for design. This requirement is the main reason that the lead time from laboratory to application for a new high temperature steel is approximately 10 years as demonstrated by the examples above.

ECCC database statistics

Grade 92 - 2004

Test temperature °C	Number of test results in time interval					
	<10,000h	**10,000h-20,000h**	**20,000h-30,000h**	**30,000h-50,000h**	**50,000h-70,000h**	**>70,000h**
550	92	4(2)	4	(1)	1(2)	
575	9(1)	2(1)	1	2	(2)	(1)
600	196(3)	30(1)	17(1)	16(2)	8(4)	3(1)
625	33	9	2(1)	1(1)	1	
650	211(1)	26	15	15(2)	4	2
675	18					
700	122	5(1)	1			
750	17					

Parantheses denote unbroken tests **Total testing hours:** **7,036,945**

E911-2004

Test temperature °C	Number of test results in time interval					
	<10,000h	**10,000h-20,000h**	**20,000h-30,000h**	**30,000h-50,000h**	**50,000h-70,000h**	**>70,000h**
550	13	1	2(1)		1(2)	
575	25(1)	4(1)	3	2	2	
600	86(1)	21	8	4(2)		(1)
625	49(3)	8	2	1	1	
650	75(8)	14	3(1)	2(1)	(1)	
675	6					
700	2					

Parantheses denote unbroken tests **Total testing hours:** **3,148,115**

P91-2005

Test temperature °C	Number of test results in time interval					
	<10,000h	**10,000h-20,000h**	**20,000h-30,000h**	**30,000h-50,000h**	**50,000h-70,000h**	**>70,000h**
500	71(6)	6(3)	2(6)		1(3)	(1)
525	3(3)		(1)		(1)	(2)
550	262(11)	31(9)	21(7)	12(2)	6(2)	5(1)
575	58(1)	10(1)				(1)
600	524(7)	99(11)	43(7)	29(4)	9(5)	2(8)
625	57(1)	2(1)				
650	358	40(4)	16(5)	6	6	(2)
675	6					
700	40	4				

Parantheses denote unbroken tests **Total testing hours:** **17,852,130**

Table 2. Statistics of ECCC databases for steels P92, E911 and P91.

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In Europe long-term creep tests are carried out and coordinated by the European Creep Collaborative Committee ECCC, where testing efforts are shared between a number of countries, and long-term strength values for European codes and standards are established according to commonly agreed extrapolation methods [5].

For steels P91, E911 and P92 the ECCC have made worldwide data collations resulting in extensive databases, see Table 2. In 2005 new evaluations of the data have been made resulting in ECCC data sheets with validated rupture strength values for the steels up to 100,000 hours. Compared to previous evaluations made by the ECCC 8-10 years ago the new evaluations show a slight reduction of 8-10% in the 100,000 hour strength values for all three steels. This is due to additional data from further suppliers leading to higher scatter and not least to substantial additional long-term data for the steels in the range of more than 30,000 hours.

The ECCC evaluations have been submitted to the ECISS (European Committee for Iron and Steel Standardisation) for inclusion in the relevant steel standards. The comprehensive data from long-term testing has confirmed excellent creep stability and sufficient creep ductility of all three steels up to 600°C.

3. MICROSTRUCTURE STABILITY

The decisions to introduce the new 9Cr steels in plant had to be based on shorter test results leading to less certainty about the long-term stability of the steels. This fact together with conflicting ideas about long-term strengthening mechanisms of especially tungsten and the quest for even better martensitic steels has led to intensive research into the microstructure stability of the 9-12% Cr steels under creep exposure. Further, there is a need to ensure long-term stability up to 300,000 hours, which is a likely lifetime of thermal power plants.

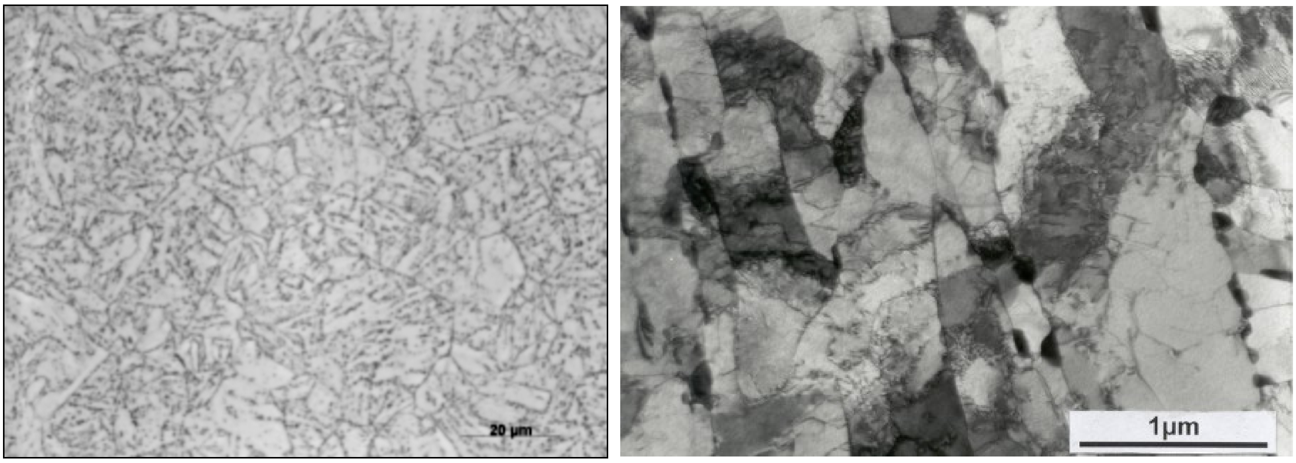

Figure 1. Tempered martensite in steel P92. Light optical micrograph (left). Transmission electron micrograph (right)

The microstructure of the 9-12% Cr steels is tempered martensite formed during a final normalising and tempering heat treatment, figure 1. After normalising at about 1050°C air hardening will lead to a martensitic transformation in sections up to approximately 100 mm thickness due to the chromium content. Tempering in the range 730°C-780°C leads to recovery of ductility by annihilation of dislocations and to the formation of ferrite subgrains. $M_{23}C_6$ Cr-carbide and MX (V,Nb)-nitride particles precipitate in the steels during tempering, on prior austenite grain boundaries, ferrite subgrain boundaries and on dislocations inside subgrains. When the steels are put into operation in power plant at temperatures below the final tempering temperature, further particles may

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precipitate, which are thermodynamically unstable at the tempering temperature, like e.g. the intermetallic $Fe_2(Mo,W)$ Laves phase.

From table 1 it is clear that minor alloy additions of Nb, N produced the first strength increase from 12CrMoV to P91 and the second increase from P91 to P92 was achieved by partly replacing Mo by W and adding B.

The technically relevant stresses and temperatures for creep testing and service exposure of the 9-12% Cr steels are in the range 300 MPa/500°C – 50 MPa/650°C. Relevant loading conditions in this range will lead to rupture times between 100 and 300,000 hours and minimum creep rates between $3 \cdot 10^{-6}$ and $5 \cdot 10^{-12}$ s^{-1}. In the authors opinion there are strong indications that the controlling creep mechanism in the whole of the technically relevant stress/temperature range is dislocation creep. Microstructural sources of creep deformation are then the migration of dislocations and subgrain boundaries [6,7]. This indicates that microstructural explanations for the improved creep strength of the new 9-12%Cr steels should be found in mechanisms which retard the migration of dislocations and subgrain boundaries, and thus delay the accumulation of creep strain with time. Such mechanisms include i) solid solution strengthening by formation of clouds of solute atoms around dislocations, and ii) interactions with precipitate particles.

Solid solution strengthening has often been referred to in discussions of the effect of Mo and W on creep strength of 9-12%Cr steels**.** It has since long been clear that during creep exposure at temperatures around 600°C-650°C most of the Mo and W in the steels will precipitate as intermetallic Laves phase ($(Fe,Cr)_2(Mo,W)$). The dominating opinion was that this would cause creep instability in the steels because the solid solution strengthening effect from Mo and W would be lost, and the precipitation strengthening effect from Laves phase was believed to be insignificant. This opinion seemed to be supported by the breakdown of long-term creep strength of some W alloyed 9-11Cr steels, and by the excellent long-term stability of the Mo alloyed 9CrB turbine material Steel B from the European COST project (Mo causes less Laves phase precipitation than W). However, the excellent long-term stability of the W alloyed 9Cr steel P92 seemed to contradict the opinion.

Recently, it has become clear that solid solution strengthening from W and Mo has no significant effect on long-term microstructure stability of the 9-12%Cr steels. Precipitation hardening by pinning of dislocations and subgrain boundaries should be regarded as the most significant strengthening mechanism in 9-12%Cr steels, and microstructure stability of the 9-12%Cr steels under creep load is equivalent to precipitate stability [8]. This is consistent with two findings: a) the compositional changes, which have improved the creep strength of the 9-12%Cr steels, have also resulted in clear changes in the precipitate populations, and b) breakdowns in creep stability presented below can be explained by unexpected precipitate reactions.

3.1 Precipitate stability

In recent years experimental measurements of particle sizes of individual precipitate types have been obtained with new microstructure characterisation methods like EFTEM and high resolution SEM. Together with new thermodynamic of phase stability and kinetic models of precipitate growth and coarsening these measurements have been used to evaluate the precipitate stability of a number of 9-12% Cr steels.

Figure 2 show evaluations by measurements and modelling of the evolution in mean particle sizes of precipitates in steels P91 and P92 at 600°C [9,10]. In both steels the MX carbonitrides show a similar very high stability against coarsening. The $M_{23}C_6$ carbides show considerable coarsening in steel P91, whereas in steel P92 this carbide is highly stable. This is attributed to the presence of B in steel P92, which seem to improve the carbide coarsening stability. This probably explains the high stability of the mentioned 9CrMoB rotor material Steel B, but the exact mechanism for Boron stabilisation is at present unknown.

Large differences are found between the Laves phase particle sizes in the two steels. After an initial growth phase of app. 10,000 hours the Laves phase particles in steel P92 are more stable against coarsening than the $M_{23}C_6$ carbides in steel P91. In steel P91 the Laves phase particles grow to very large sizes during an extended growth phase, which lasts app. 30,000 hours. The observed differences in particle stability can explain the observed differences in creep rupture strength between the steels. The Laves phases in steel P92 produce a significant precipitate strengthening effect, which together with the observed high stability of the $M_{23}C_6$ carbides provide the high creep strength of this steel.

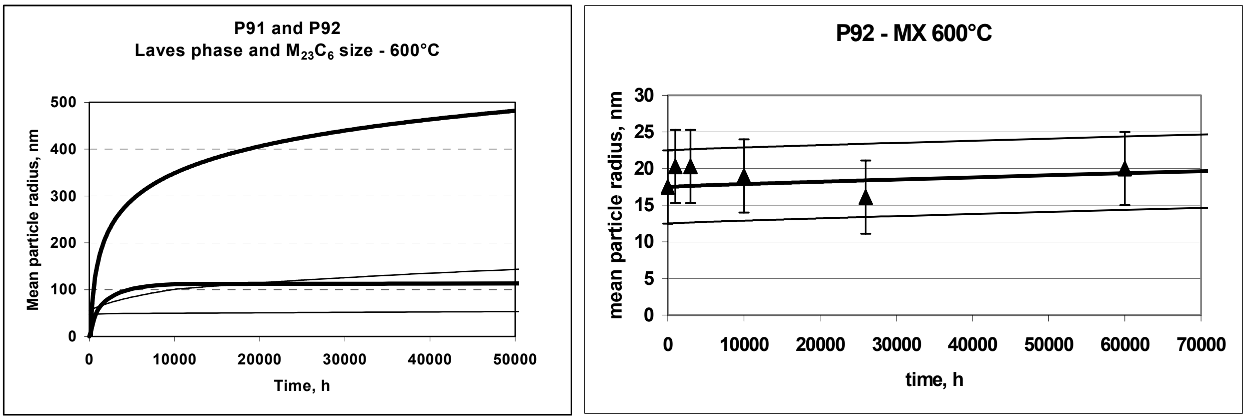
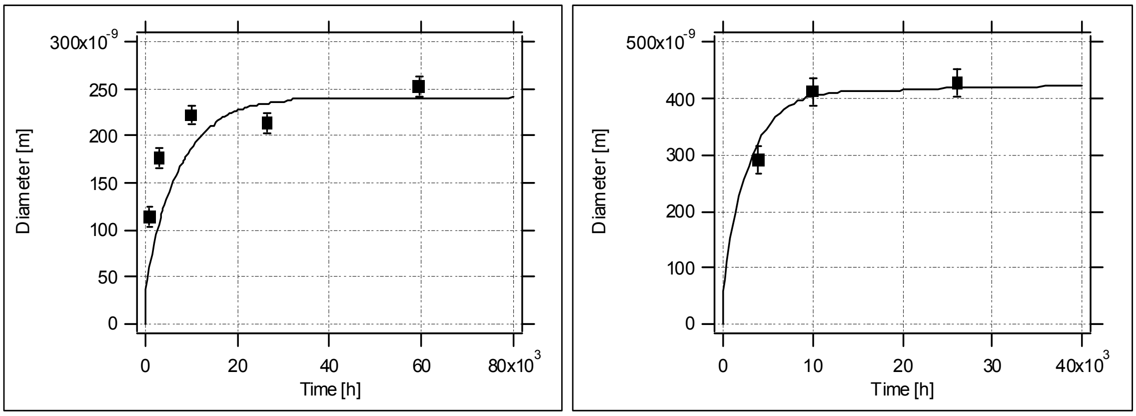


Figure 2. Left: Particle sizes of M23C6 (thin lines) and Laves phase (thick lines) in steels P91 and P92.
Right: Size evolution of MX particles in steel P92. A similar MX evolution is observed in P91.

The observed differences in Laves phase behaviour in steels P91 and P92 is explained by the lower solubility of the Mo Laves phases (650°C) compared with W Laves phases (720°C). When the creep temperature is close to the solution temperature for Laves phase, nucleation of the phase is very difficult, and only few particles nucleate. This results in the extended growth phase and large mean particle size. Similar observations have been made for the 9CrMoB Steel B [11]. This is also seen in the very strong effect of temperature on Laves phase particle size in steel P92, Figure 3 [12]. At 650°C the Laves phase particles are app. 60% larger than at 600°C.



a) b)

Figure 3. Model of growth and coarsening of Laves phase in steel P92. The diameter of Laves phase vs. time. a) 600°C, b) 650°C. Full squares denote measured values.

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It is clear from the discussion above that alloying with either W or B has a strong positive effect on the microstructure stability of 9-12% Cr steels. W produces fine stable Laves phase particles in steel P92 - even finer than the $M_{23}C_6$ carbides in steel P91 - when the creep temperature is sufficiently below the solubility temperature for this phase. The Laves phase particles contribute significantly to particle strengthening. B has a strong stabilising effect on the $M_{23}C_6$ carbides, even though the mechanism for this remains unclear. The MX precipitates appear to be extremely stable against coarsening and could probably be characterised as the backbone of the long-term stability of the new 9-12%Cr steels.

4. MICROSTRUCTURE INSTABILITY

The presented experimental observations produce improved understanding of the excellent stability of the strongest of the new 9-12%Cr pipe steels, i.e. the P92, but the models fail to explain observed microstructure instabilities in a number of steels. These instabilities can however be attributed to precipitate reactions. Imbalances in composition may lead to formation of unwanted phases like AlN in steel P91 at the expense of MX nitrides [13], and unexpected phase transformations like the Z-phase formation may occur with a similar result as described below.

4.1. Z-phase precipitation

In 2001 two tubes of steel T122 (12CrWMoCuVNbN) installed in a test superheater facility at a Danish power plant ruptured prematurely after 30,000 hours exposure at 58 MPa/app. 610°C and after 12,000 hours at 45 MPa/660°C. The rupture times were only app. 10% of the expected minimum lifetimes according to published ASME allowable stresses, Fig. 4. Inspection of the tubes showed no signs of extensive corrosion or oxidation attack, and temperature estimates based on oxide thickness measurements did not indicate any overheating.

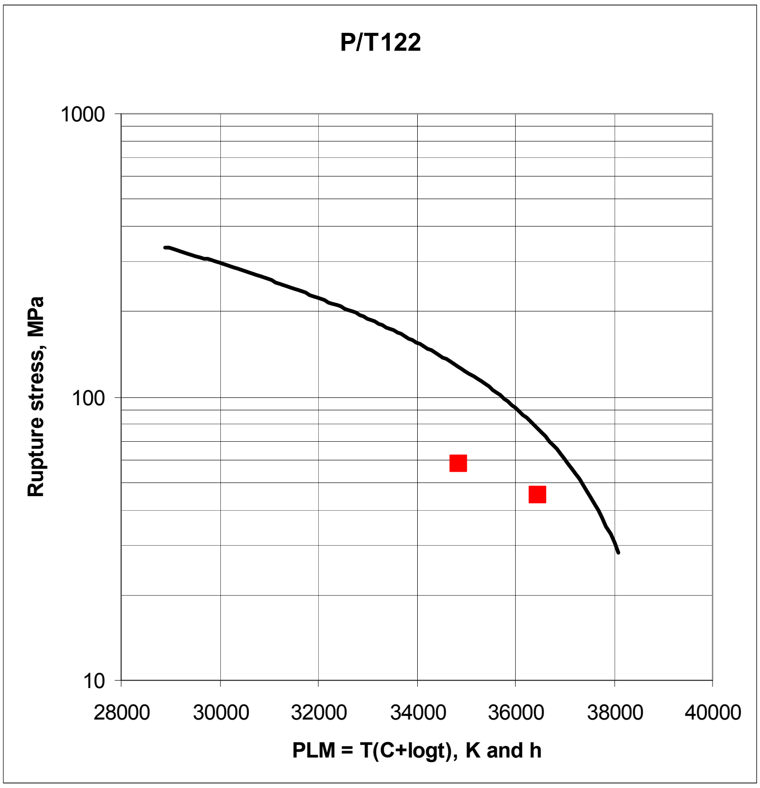

Figure. 4. Mean rupture strength of steel P/T122 and observed lifetimes of T122 superheater test tubes

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Detailed microstructure investigations showed extensive precipitation of the complex nitride Z-phase (Cr(V,Nb)N) in the steels, and this had led to an almost complete dissolution of MX nitrides. The T122 tubes contained 12%Cr. Parallel investigations of a steel P122 pipe with 11%Cr after isothermal exposure 650°C/10,000 hours showed only very little sign of Z-phase formation, and a large population of MX was still present in the steel. It was concluded that the Z-phase transformation was responsible for the breakdown in creep stability of the T122 tubes. Since the only significant difference between the T122 and P122 steels was the Cr content it was further concluded that a high Cr content accelerates Z-phase formation.

Severe creep instabilities have been observed in a number of experimental 10-12%Cr steels, e.g. the 11CrWCo steels TAF 650 and NF12. It was for long believed that this was caused by Laves phase precipitation, but measurements of Laves phase particles in a steel similar to the TAF650 have demonstrated an even more favourable particle size distribution of the Laves phase than in steel P92 [12]. Similarly, a large number of test alloys with 11-12%Cr, and with or without W, intended for operation at 650°C have been manufactured and tested. All of these, which have been tested to long times, show a severe breakdown in creep stability between 5,000 and 30,000 hours at 650°C. Recent investigations have identified Z-phase precipitation as the root cause of this instability in the 10-12%Cr steels. The Z-phase can completely dissolve the MX carbonitrides, which are major contributors to creep strength. The Z-phase precipitate as very large particles, which will not contribute to precipitation hardening, and which may each consume more than 1000 MX particles, figure 5.

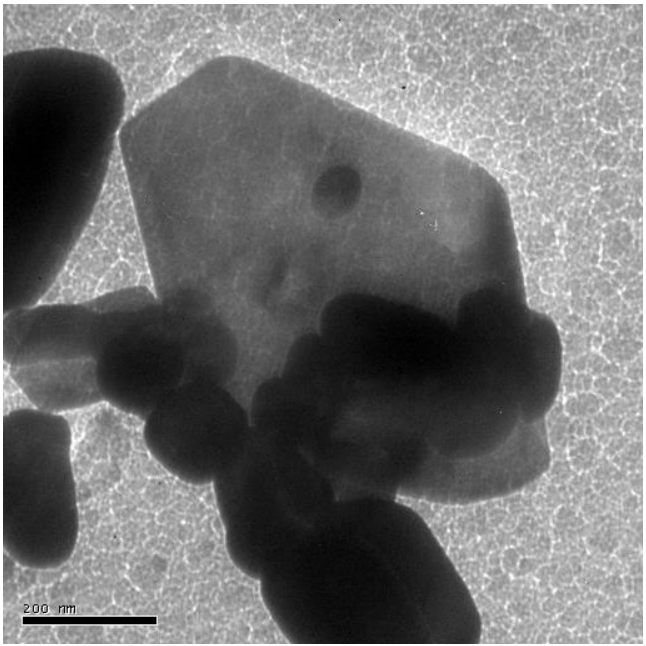

Fig. 5. Z-phase particle in a 11CrWCoVNbNB steel

The Z-phase nitride in the form CrNbN has been known from Nb alloyed austenitic stainless steels since the 1950s, and here it is most often regarded as a beneficial strengthening phase, since it precipitates as very fine particles. In martensitic 9-12%Cr steels alloyed with V and Nb the Z-phase in form Cr(V,Nb)N was first observed in 1986 in the 12%Cr bolting steel X19CrMoVNbN 11 1 [14]. In 1996 detailed investigations of creep specimens of older 12CrMoVNb steels showed that the Z-phase precipitated after 10,000-30,000 hours of creep at 600°C, and that MX (V,Nb)-carbonitrides dissolved. It was concluded that the Z-phase precipitation reaction was responsible for observed creep instabilities in the steels [15]. However, the older 12%CrVNb steels including the X19CrMoVNbN 11 1 have Nb contents, which are app. 5 times higher than the new generation of high strength 9-12%Cr steels, and it was not considered that Z-phase precipitation would occur in the new steels.

Thus, the observed Z-phase precipitation represent a serious problem to the long-term microstructure stability of the new generation of 9-12%Cr steels, in which a major part of the high creep strength is obtained by particle strengthening with MX (V,Nb)-carbonitrides.

Work at TU Denmark has resulted in a first version of a thermodynamic model of the Z-phase [16]. Model predictions indicate that Z-phase is the thermodynamically stable nitride at temperatures below 650°C in *all* of the 9-12%Cr steels, including P91, E911, P92 and even 12CrMoV, which do

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not contain niobium. This means that it is the kinetics of Z-phase precipitation (nucleation and growth), which is the key to understanding of the differences in creep stability of the steels. Estimates of nucleation rates of the Z-phase based on driving force calculations show that Cr is a very significant element to accelerate Z-phase precipitation. Steels with 11-12%Cr can show Z-phase precipitation with complete MX dissolution within a few thousand hours at 650°C.

9%Cr steels like P91 and P92 have only shown beginning Z-phase precipitation after 30,000-40,000 hours at 650°C, with large populations of MX particles still present in the microstructure. At 600°C and lower temperatures Z-phase precipitation in the 9%Cr steels seems to be even slower, and may in fact be insignificant to the long-term stability up to 300,000 hours.

5. DEVELOPMENT POTENTIALS

Current bottlenecks for increases in steam parameters of power plants are the thick section components in boiler, steam line and turbine. The materials in these components are ferritic/martensitic 9-12%Cr steels, which are used for their creep and fatigue properties. Austenitic steels have higher creep strength, but they can not be used in thick section components because of their limited fatigue strength, which will impair the cycling ability of the plant.

As seen the strongest available ferritic/martensitic steels are based on 9%Cr, and in order to increase steam parameters above 610°C/325bar improved steam oxidation resistance is needed together with improved creep strength. The goal for new ferritic/martensitic steels is to reach a creep rupture strength of 100 MPa at 100,000 hours and 650°C.

In the recent decades many attempts have been made to develop ferritic/martensitic steels based on 11-12%Cr, which should have the required steam oxidation resistance and creep strength for 650°C. However, as demonstrated above all of these attempts have failed due do unforeseen microstructure instabilities leading to breakdowns in the long-term creep strength. This demonstrates that the alloy optimisation methods used so far based mainly on empirical trial and error have reached their limit. Further developments should rely on more scientifically based methods in order to reach the goal.

Computer based microstructure stability models based on thermodynamic and kinetic descriptions (like e.g. the Thermocalc and DICTRA) have undergone strong developments recently. It is now possible to model precipitate growth and the stability against coarsening as a function of chemical composition [10,17,18]. Precipitate nucleation rates can also be estimated as mentioned for the Z-phase. Experiences in the field of high temperature steels show high accuracy of the models, and with further development of underlying thermodynamic and diffusion databases they could serve as strong predicive tools for alloy design.

The author believes that it will be possible to double the creep strength of this class of alloys once more based on the novel scientifically based predictive tools. However, due to the problems with Z-phase the alloy development might fail to combine high creep strength based on MX strengthening with high oxdation resistance based on high Cr content into one alloy. In that case it would become necessary to develop surface coatings to obtain the oxidation protection.

Alloy development of this class of steels in Europe is ongoing in the COST 536 ACCEPT action, which began in 2004.

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6. SUMMARY

The long-term creep rupture strength of new steam pipe steels P91, E911 and P92 is now secured with long-term testing up to 100,000 hours. All of these steels have been introduced into advanced ultra supercritical steam power plans.

Detailed microstructure studies have improved the understanding of the long-term stability of the 9-12%Cr steels:

- Precipitate hardening controls the long-term microstructure stability, and solid solution strengthening from Mo and W plays no significant role in the long-term microstructure stability of 9-12%Cr steels.

- Significant particle strengthening can be obtained by intermetallic Laves phases provided that a large number of particles nucleate during creep exposure. Phase stability to high temperature, like for W Laves phases, promotes the nucleation of Laves phase.

- MX carbonitrides rich in V and Nb are extremely stable against coarsening, but they may be dissolved by precipitation of the complex nitride Z-phase (Cr(V,Nb)N). This is mainly a problem in Nb containing steels with Cr contents of 10% and above.

Significant advances in microstructure stability modelling will allow further improvements of the creep properties of this class of alloys.

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