

## Z-PHASE IN 9-12%Cr STEELS

H. Danielsen  
IPL-MPT, TU Denmark

J. Hald  
Elsam/Energy E2/ IPL-MPT, TU Denmark

### Abstract

The complex nitride Z-phase, Cr(V,Nb)N, has recently been identified as a major cause for premature breakdown in creep strength of a number of new 9-12%Cr martensitic steels, especially the high Cr variants. A thermodynamic model of the Z-phase has been created based on the Thermo-Calc software. The model predicts the Z-phase to be stable in all of the new 9-12%Cr martensitic steels, and this has generally been confirmed by experimental observations. Z-phase precipitation seems then to be a kinetic problem, and driving force calculations, using Thermo-Calc with the developed model, have been used to predict steel compositions, which could delay Z-phase precipitation. The model also predicted the existence of a new niobium free Z-phase variant, which has since been discovered in a niobium free 12CrMoV steel.

### Introduction

Great success has been achieved over the last 25 years with the developments of new modified martensitic 9-10%Cr steels like the P91, E911 and P92 for steam boilers and steam pipes and of other martensitic grades for steam turbine rotors, blades, valves and casings. The developed steels have creep rupture strength up to twice as high as the previously available martensitic grades, and this improvement has formed a basis for increased efficiencies in steam power plants in Europe and Japan by introduction of steam parameters up to 4351 psi(300 bar) and 1112°F(600°C).

Based on the success of the new 9-10%Cr steels developments of new alloys intended for operation up to 1202°F(650°C) steam temperature was initiated a decade ago. Due to accelerated steam oxidation at the increased temperature the new grades contained Cr at levels of 11-12%. Most of the new alloys showed promising improvements of creep rupture strength during 5,000 to 10,000 hours of testing at 1202°F(650°C), but at longer testing times up to 30,000 hours all of the new alloys have experienced dramatic breakdowns in microstructure stability, figure 1. Very recently the precipitation of the complex nitride Z-phase (Cr(V,Nb)N) has been identified as the root cause of the observed microstructure instabilities in the new steels.

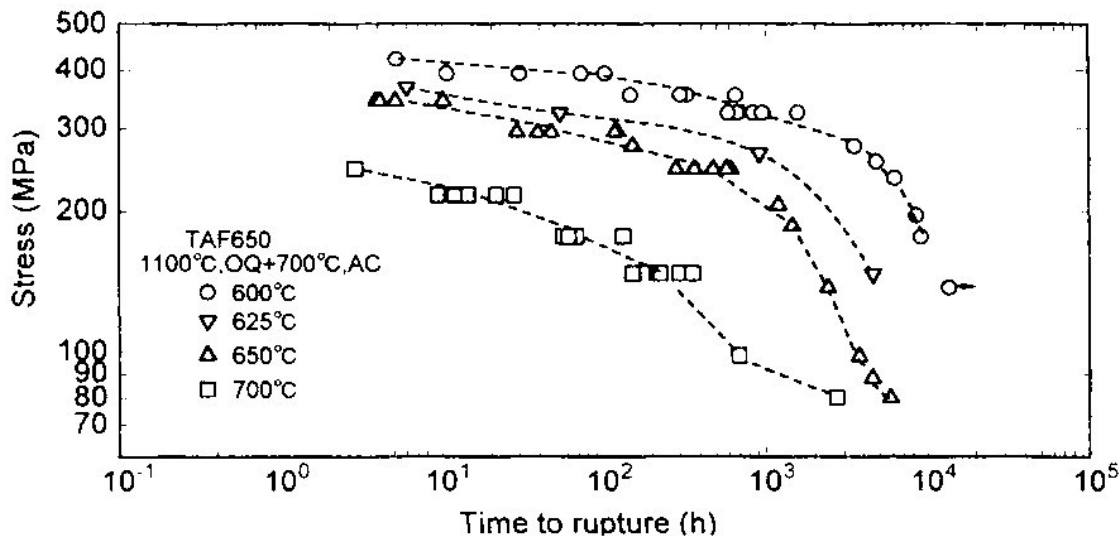


Figure 1. High hopes were pinned on steels like e.g. the TAF650 (11Cr3W3CoVNbN) but as creep tests reached 10,000h their strength had dropped dramatically (1)

Precipitate particles are seen as the most important microstructure elements contributing to creep strength and microstructure stability of the 9-12%Cr steels. The MX carbonitrides, which mainly consist of Nb(C,N) or VN, have been identified as highly important to the creep stability. The Z-phase consists of the same elements as the MX, and its precipitation is then accompanied by dissolution of the beneficial MX particles. The Z-phase precipitates as large particles, which do not contribute to particle strengthening, and thus the creep strength of the steel is considerably lowered as seen in figure 1.

In the present article observations of Z-phase precipitation is reviewed, and experimental and theoretical work to develop a thermodynamic model of the Z-phase is described. Such a model allows predictions of the specific conditions, which cause Z-phase precipitation.

### Observations of the Z-phase

The Z-phase was discovered as early as 1950 by Binder (2) in Nb alloyed austenitic steels, where it has since been frequently observed and credited with beneficial strengthening effects. The Z-phase precipitated very quickly as small finely distributed rod-like particles, and it was often the first particle to appear. Many attempts were made to determine the crystal structure of the Z-phase (3, 4), but it was not until 1972 that Jack & Jack identified it as Cr<sub>2</sub>Nb<sub>2</sub>N<sub>2</sub> (5), figure 2. In addition to the main elements, some minor concentrations of Fe, Mo and traces of other elements were also present.

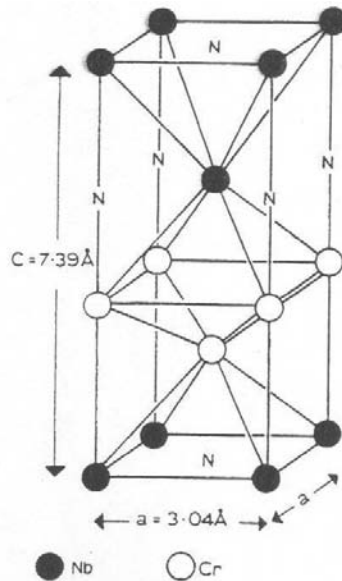


Figure 2. The crystal structure of a pure Nb Z-phase,  $\text{Cr}_2\text{Nb}_2\text{N}_2$  (5)

In 1984 Andren and co-workers (6) discovered a particle with a Z-phase composition, but half of the Nb had been replaced with V. It was not positively identified as Z-phase though, but as a metastable precursor to the Z-phase. A year later Schnabel and co-workers (7) discovered a vanadium containing Z-phase in the martensitic 11%Cr steel, X19 (CrMoVNbN 11 1), which shows a very dramatic drop in creep strength similar to some of the more modern steels, figure 3. However, there was no connection drawn between the appearance of the Z-phase and this drop in strength. This had to wait a decade, until Strang and Vodarek (8) introduced the term modified Z-phase in 1996. The modified Z-phase is a normal Z-phase, which contains vanadium, the unit cell formula being  $\text{Cr}_2(\text{V},\text{Nb})_2\text{N}_2$ . The crystal structure remains the same, but modified Z-phase has a slightly smaller unit cell,  $a = 2.86\text{Å}$  and  $c = 7.39\text{Å}$ . The new Z-phase did not behave like the old one at all. It only precipitated after long exposure times, and then only as very few and coarse particles.

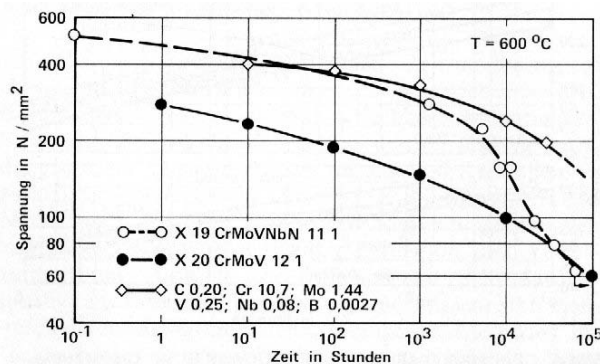


Figure 3. A comparison of creep strength of three 10-12%Cr steels at 1112°F(600°C); Z-phase was observed in X19 (CrMoVNbN 11 1) by Schnabel and co-workers (7)

In 1996 Bursik (9) and in 2001 Kimura (10) observed the Z-phase in the modified 9%Cr P91, after long term creep testing at 1112°F(600°C) and 1202°F(650°C). In P91 the Z-phase precipitation had not led to a complete dissolution of the MX particles and no severe breakdown of the creep strength had occurred.

As mentioned Z-phase has recently been observed precipitating in a number of 11-12%Cr steels similar to the TAF 650 (11, 12), which had experienced severe breakdowns in creep strength after 10,000h at 1202°F(650°C), figure 1. In these steels the Z-phase was much more numerous than in steel P91 and only few MX particles were observed.

In order to supplement the Z-phase observations from literature we conducted investigations of a number of steels, which had been exposed to temperatures close to 1202°F(650°C) for several thousand hours. Details of our investigations can be found in (13). Investigations included lower Cr steels (P91, P92, E911, P122), and higher Cr steels (FN5, NF12, T122). All of the high Cr steels had shown severe breakdown of creep strength during either laboratory testing or plant exposure, whereas none of the low Cr steels had shown such behaviour. Z-phase was observed in all of the investigated steels, but the precipitation reaction seems to be more sluggish in the low Cr steels.

## Literature survey

Thermodynamic calculations with computer programs like the Thermo-Calc have been successfully applied to evaluate phase stability of precipitates and matrix phases of the 9-12%Cr steels as a function of temperature and chemical composition. Such calculations have led to improved understanding of the influence of precipitate phases (carbides, intermetallic phases and nitrides) on long-term microstructure stability of creep resistant 9-12% Cr steels. A major problem with the Z-phase precipitation was that no thermodynamic model of the Z-phase existed, and thus thermodynamic calculations were unable to predict the stability range of the Z-phase.

In order to make a thermodynamic model of the Z-phase, an extensive literature survey was conducted. The intention was to find solid results, which could be used in the model, i.e. the composition of the Z-phase and its solution temperature, together with the overall composition of the steel. Other factors such as exposure time, size of the Z-phase particles and information about the other present precipitates were of secondary importance, since the model only describes equilibrium. Such information does not contribute directly to the model, but helps the general understanding of the Z-phase.

The most important sources found on Z-phase are listed in tables 1 and 2. The sources on steels without vanadium are generally of an older date, but they give nice all-round information, except for lower temperatures. For the vanadium containing steels, the total lack of results concerning the solution temperature is a large setback for the creation of a thermodynamic model. Finding this temperature was seen as a prerequisite for a reliable model so experiments were necessary.

Table 1: Sources for non-Vanadium containing steels

Author(s)	Steel type	Publ. Year	Solution temp.	Composition
Gerlach et al (14)	$\gamma$	1968	Yes	Yes
Vodarek (15)	$\gamma$	1990	Yes	Yes
Hughes (3)	$\gamma$	1967	Yes	Yes
Uno et al (16)	$\gamma$	1991	Yes	No
Erneman et al (17)	$\gamma$	2003	No	Yes
Kurosawa et al (18)	$\alpha$	1980	Yes	No
Baumel (19)	$\alpha$	1965	Yes	No

Table 2: Sources for Vanadium containing steels

Author(s)	Steel type	Publ. Year	Solution temp.	Composition
Andren et al (6)	$\gamma$	1985	No	Yes
Strang et al (20)	$\alpha'$	2002	No	Yes
Ishii et al (21)	$\alpha'$	2002	No	Yes
Svoboda et al (11)	$\alpha'$	2002	No	Yes
Kimura et al (10)	$\alpha'$	2001	No	Yes
Bursik et al (9)	$\alpha'$	1996	No	Yes
Zies (12)	$\alpha'$	2003	No	Yes
Suzuki et al(22)	$\alpha'$	2003	No	Yes

As the model began to take shape it also predicted the existence of a pure vanadium Z-phase, CrVN, in steels which did not contain niobium. This result could not be confirmed by literature. Ettmayer (4) had made experiments with the goal of creating Z-phases in ternary systems. He was successful in the case of CrNbN and CrTaN Z-phases, but he failed to create a CrVN Z-phase. The investigations of the ternary CrVN system showed no Z-phase at temperatures of 1472°F(800°C) and above.

## Experiments

The experimental work was based on TEM observations of extraction replicas. Precipitate compositions were analysed with EDS, which formed the basis of the identification of the particles. Carbon and nitrogen could not be measured in this way, so no measurements of these elements were obtained.

### **X20 CrMoV 12 1**

As mentioned the Thermo-Calc model had predicted Z-phase to be stable in 9-12%Cr steels which contained vanadium but no niobium, but this could not be confirmed by the literature search. This Z-phase would have the chemical formula CrVN and to our knowledge had never been observed before. Several samples of the 12CrMoV steel X20 (CrMoV 12 1), which does not contain Nb, were investigated. In one of these samples, exposed at 1112°F(600°C) for

Table 3: Composition of some of the steels used for experiments in this work (wt%)

Element	X20	P91	P92	T122	NF12	FN5
C	0.19	0.10	0.106	0.09	0.085	0.11
N	0.025	0.053	0.051	0.0609	0.045	0.027
Si	0.23	0.36	0.04	0.25	0.25	0.06
Mn	0.49	0.37	0.46	0.60	0.44	0.55
Cr	11.5	8.30	8.96	12.20	11.60	11.20
Mo	0.91	0.95	0.47	0.35	0.14	0.26
W	-	-	1.84	1.97	2.68	2.63
Ni	0.66	0.15	0.06	0.18	0.17	0.40
Nb	-	0.07	0.069	0.06	0.08	0.065
V	0.31	0.21	0.20	0.25	0.20	0.22
Cu	0.054	-	-	0.43	0.01	-
Al	0.016	0.15	0.007	0.004	-	-
B	-	-	0.001	0.0021	0.0026	0.0010
Co	0.021	-	-	-	2.48	2.66
Heat treatment	1050°C/1h 750°C/2h	1050°C/1h 780°C/1h	1050°C/2h 770°C/2h	N+T	1100°C/0.2h 760°C/1h	1100°C/4h 570°C/8h 715°C/24h

150,000h, Z-phase was found. EELS measurements confirmed presence of N in amounts indicating Z-phase composition. As there has been no previous reporting of a Nb free Z-phase to our knowledge, investigations with electron diffraction are ongoing to verify if the observed particles are indeed Z-phase.

### ***Solution temperature experiments***

Heat treatment experiments were made to determine the solution temperature of Z-phase in vanadium containing steels. Steels which had already developed Z-phase were used, so a short heat treatment at higher temperatures could provide a quick way of determining the solution temperature.

The materials used where the T122 exposed at 1220°F(660°C) for 12,000h and NF12 exposed at 1202°F(650°C) for 17,000h. Later investigations of the Nb free Z-phase were carried out on the X20 sample exposed for 150,000h/1112°F(600°C). The Z-phase was present in all of these exposed materials. The experiments gave a solution temperature of the Z-phase between 1472°F(800°C) and 1525°F(830°C) in the samples. The solution temperature of Z-phase in vanadium free steels is around 2192°F(1200°C), so there is a large stability difference.

In samples, where the Z-phase had been dissolved, it had been replaced by (or transformed into) large particles consisting mainly of V and Nb. The composition of the metallic elements was (at%): 66% V, 20% Nb, 13% Cr. In the case of X20 Nb was replaced by V. It is assumed that these are MX particles, but no real effort has been made to identify them. In the temperature

interval between 1472°F(800°C) and 1525°F(830°C) these particles seemed to coexist with the Z-phase, but this could be because of insufficient time to dissolve the Z-phase.

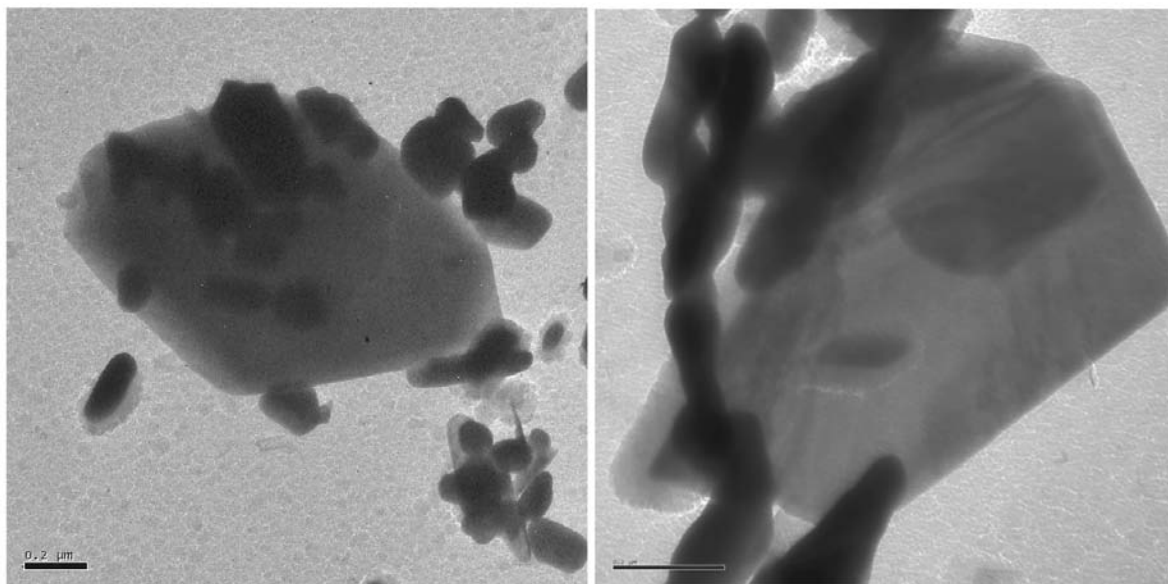


Figure 4. Left, a Z-phase found in an NF12 sample exposed at 1202°F(650°C) for 17,000h. Right, a CrVN Z-phase found in an X20 sample exposed at 1112°F(600°C) for 150,000h. Both scalebars are 0.2μm

### The thermodynamic Z-phase model

A thermodynamic model of the Z-phase was created using the Thermo-Calc software. This is based on the regular solution model developed by Hillert and Staffanson (23) and extended by Ågren and Sundman (24). Thermo-Calc calculates the Gibbs energy for every phase in a multi-component system, and can then tell which phases will be stable at certain temperatures and in which quantity and composition they will appear. Thermo-Calc can however only calculate equilibrium conditions.

In order to calculate the Gibbs energy for the Z-phase, the energy for every possible combination of compositions has to be calculated. As Z-phase contains minor amounts of Fe and Mo, the full formula being (Cr,Fe)(Nb,V,Mo)N, there are many combinations of pure compounds: CrVN, CrMoN, FeVN etc. The calculations are essentially done by adding the standard element references enthalpies for each element and a polynomial, in our case  $K_1 + K_2T$ .  $K_1$  is the total energy of formation of the compound from the elements at zero Kelvin and  $K_2$  is a simplified temperature dependence. More information about the model can be found in (13). For example the calculations for CrNbN would be:

$$G_{CrNbN} = {}^{SER}G_{Cr} + {}^{SER}G_{Nb} + {}^{SER}G_{N} + K_1 + K_2T \quad (1)$$

THERMO-CALC (2004.08.16:14.53) : ZET

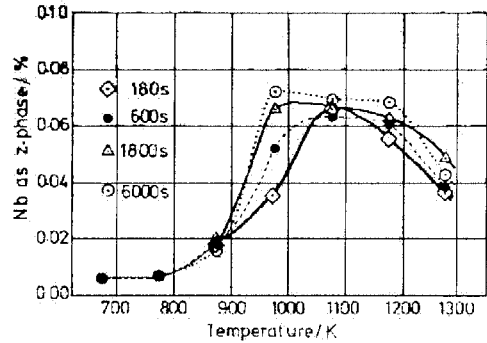
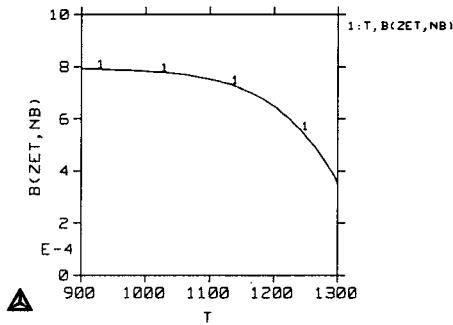


Figure 5. Quantity of Nb precipitated as Z-phase calculated by Thermo-Calc compared to the observed quantity of Nb precipitated as Z-phase (same scales are used). The absence of Z-phase at lower temperatures in the experimental data is caused by kinetic effects (18)

The model was made by fitting the model constant to all available data from the literature survey and experiments. A total number of 22 constants were fitted. The approach was first to fit the CrNbN system (original Z-phase), because it was simple and well described. Then the model was expanded by adding Fe and Mo, and finally V which integrated the modified Z-phase into the model. Figure 5 gives an example of how data has been used to fit the model.

### Results from the model

THERMO-CALC (2004.01.22:13.28) : ZET

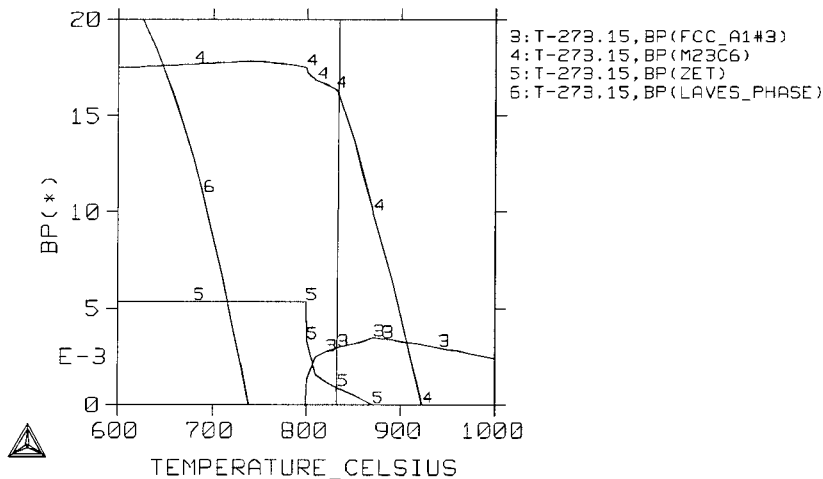


Figure 6. Thermo-Calc calculations showing the phase quantity as a function of temperature of a T122 steel. Z-phase (curve 5) is replaced by MX (curve 3) at approximately 1472°F(800°C)



Overall, the model seems to give good predictions of the Z-phase compositions found in the literature and our experimental data. The calculations done on the solution temperatures all coincide within 122°F(50°C) of the measured values in own experiments and in the literature survey.

There are some difficulties for the modified (vanadium containing) Z-phase compositions as many of the investigated vanadium containing steels are probably not in equilibrium. In many cases the experimentally determined niobium contents in the Z-phase are higher than the model predictions. This could be explained by the fact that the Z-phase initially prefers to precipitate close to grain boundaries and niobium containing carbo-nitrides, thus absorbing relatively more niobium in its initial stages. Figure 7 shows how the niobium rich MX particles dissolve first in steel P91.

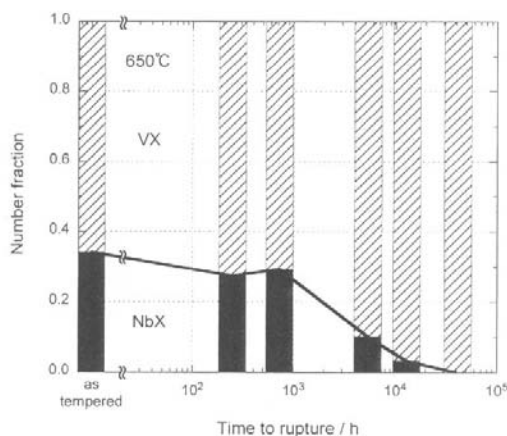


Figure 7. Changes in number fraction of primary MX (NbX) and secondary MX (VX) with increasing creep exposure time at 1202°F(650°C) in p91 (22)

The Thermo-Calc model predicts the Z-phase to be stable in practically all 9-12%Cr steels containing V and N, even in those without niobium contents. The experiments so far have all confirmed these calculations, including the presence of Z-phase in steels without Nb. It is then important to recognise Z-phase precipitation as a kinetic problem, it probably cannot be stopped, but only slowed down.

Just because the Z-phase seems to be the most stable nitride in 9-12%Cr steels, it does not make these steels obsolete. Since Z-phase only precipitates very slowly, the steels need not be affected by it within their lifetime, which is around 200,000-300,000h. Indeed the X20 steel seems more than capable to survive at these exposure times (it has been used in power plants for more than 30 years). In the P91 and P92 steels Z-phase has primarily been observed at 1202°F(650°C), and the steels show no strong indications of a complete breakdown in creep strength up to approximately 45,000 hours at this temperature. Discovery of one Z-phase particle in a steel does not necessarily spell disaster for it, a few Z-phases cannot render a steel useless. Observation of numerous Z-phases accompanied by widespread MX dissolution is an entirely different matter.

## Driving force calculations

Thermo-Calc can only calculate systems at equilibrium, but it can still calculate the driving force for the Z-phase precipitation reaction, which is directly dependent on the chemical composition. The driving force is calculated by comparing the change in Gibbs energy for a steel with Z-phase to the same steel without Z-phase. This effectively means that the driving force is the stability difference between Z-phase and MX, the second most stable nitride. Steels with smaller driving forces are likely to precipitate Z-phase slower than steels with higher driving forces.

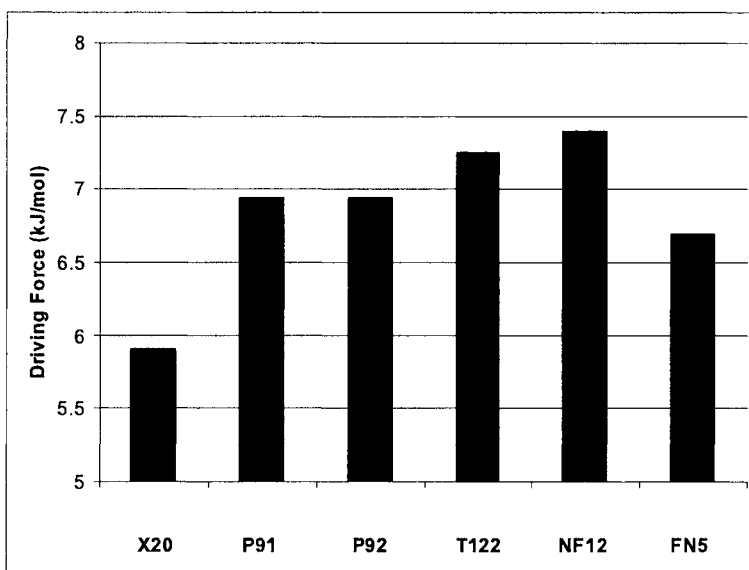
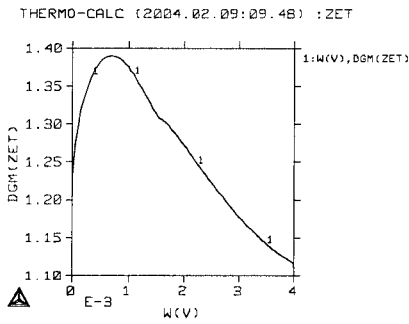
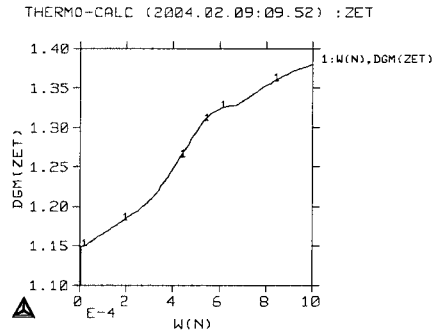


Figure 8. The driving forces of Z-phase for several different steels at 1202°F(650°C). Even the small differences seen here may have large consequences on the precipitation speed

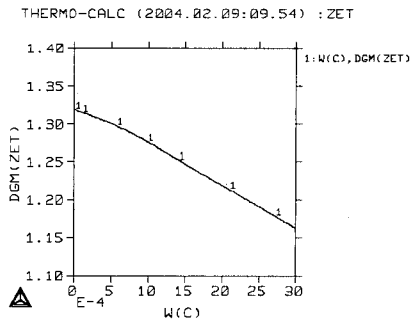
The results of the driving force calculations give the approximate order of steels, which most readily precipitate Z-phase. The T122 and NF12, which showed relatively quick Z-phase precipitation, have higher driving forces than steels like P92 and X20, where Z-phase precipitation is more sluggish. One glaring exception seems to be the FN5 steel where the calculated driving force is lower than for P92. However, the investigations showed Z-phase to be much more active in FN5 than in P92. This could possibly be explained by the tempering process, which is quite different for the two steels, table 3. The driving force will only be one piece in a larger puzzle, which gives a complete picture of the precipitation speed of the Z-phase. Other factors, including microstructure and heat treatments, may be responsible for the kinetics of Z-phase precipitation.



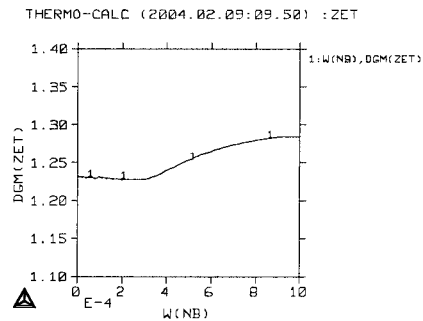
Driving force from 0 to 0.4 wt% V



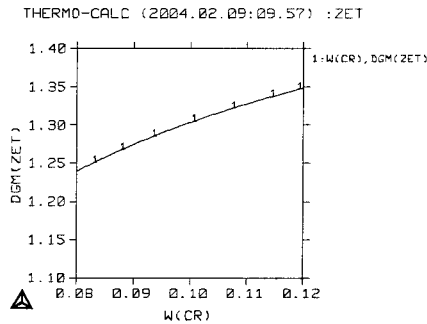
Driving force from 0 to 0.1 wt% N



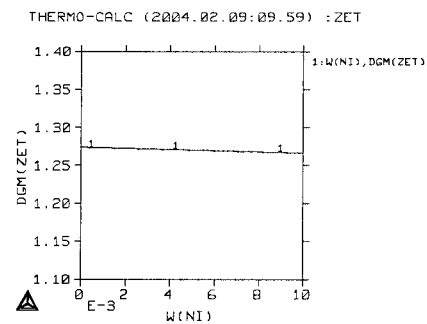
Driving force from 0 to 0.3 wt% C



Driving force from 0 to 0.1 wt% Nb



Driving force from 8 to 12 wt% Cr



Driving force from 0 to 1 wt% Ni

Figure 9. Driving force calculated as a function of concentration of certain elements at 1202°F(650°C). Driving force unit: J/mol divided by RT. Composition of P92 used (wt%):  
Cr 8.96 C 0.106 N 0.051 Ni 0.06 V 0.20 Nb 0.069

Driving force calculations with systematic variation of single elements give us some indications as to which elements affect the Z-phase precipitation speed. Figure 9 shows some observations using the Thermo-Calc model.

The Z-phase observations indicate that variations in the Cr content is the most significant difference between stable steel (like P91 or P92) and unstable steels (like T122 or NF12). However, the calculated change in driving force for Cr variations seems to be small compared to the effect of varying other element contents.

The greatest variations in the driving force are seen when changing the V or N contents, two of the major elements in the Z-phase. The three nitrides, MX, M<sub>2</sub>X and Z-phase, seem to be competing for nitrogen, and they all contain vanadium. The Z-phase is the most stable nitride, and will be present in basically all steel compositions. At relatively high N/V ratios, the M<sub>2</sub>X phase will form as it contains a lot of readily available Cr and only a little V, which will be in short supply. When equal amounts of V and N are available the Z-phase will be most stable. At low N/V ratios the MX particles will become more stable as they can form carbo-nitrides, V(C,N), and will thus use the N most “economically”. This feature is most pronounced in steels with a high C content. In Nb containing steels, a high carbon content (or low nitrogen content) will stabilise NbC, leaving the Z-phase with only V, and thus lowering its stability.

The model predicts V(C,N) forming in low N/high C steels, but this should probably be checked out. In this work no indications have been seen of this, but very few sources mention the C or N content in the particles. Equipment capable of measuring these contents has not been readily available in the course of this work, so no measurements have been made on this.

## Conclusion

The complex nitride Z-phase, Cr(V,Nb)N, has recently been identified as a major cause for the premature breakdown in creep strength of a number of new 9-12%Cr martensitic steels. A review of reported observation of Z-phase precipitation together with own investigations indicate that the Z-phase problem is most severe for the high Cr variants of the 9-12%Cr steels.

A thermodynamic model of the Z-phase has been created using the Thermo-calc software. The model was made on the basis of data from literature and own experiments. The model seems to correspond very well with the great majority of data.

The model predicts Z-phase to be the most stable nitride in all 9-12%Cr steels containing N and V. After it was predicted by the new model, a Nb free CrVN Z-phase was discovered in a steel, which does not contain niobium. To our knowledge this is the first observation of a CrVN Z-phase. Electron diffraction and other measurements are currently being performed in order to verify its structure.

Driving force calculations with the developed Thermo-calc model give the general trends of the effect of different elements on the speed of Z-phase precipitation. The lowest driving forces for

Z-phase precipitation would be obtained by having low N, Cr, Nb contents and high V, C contents. In its present form the model seems to underestimate the effect of Cr on the driving force.

Even though our predictions show Z-phase to be the most stable nitride in all 9-12%Cr steels, it does not make them obsolete. Z-phase has to appear in large quantities in order to affect the creep strength. In low Cr steels, like P91 or P92, the Z-phase precipitation seems to be very slow at application temperatures, 1112°F(600°C), and may in fact be insignificant to the long-term stability up to 300,000 hours

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